

FIBRE-REINFORCED MATERIALS  
FABRICATED BY POWDER EXTRUSION

by

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ABSTRACT

The powder extrusion process as a consolidation technique for pre-alloyed aluminium and aluminium alloy powders has been thoroughly investigated. Analyses have been proffered to predict extrusion load, strain rate and "steady state" temperature during the extrusion process. Classical hot working theory has been applied to powder compacts during extrusion and to the resulting extrudes relating flow stress, strain rate and temperature. The controlling effect of the extrusion parameters on the product structure and mechanical properties has been clearly demonstrated. Al-Mg-Si powder extrudes have been subjected to various heat treatments and the resulting mechanical properties investigated.

Glass fibre reinforced rods have been produced, by this powder extrusion route, from mechanically mixed quantities of aluminium alloy powder and glass powder. The extrusion process has served three purposes:-

- (1) Fibre formation (in situ in the deformation zone and die neck)
- (2) Fibre alignment
- (3) Matrix consolidation

The structure and mechanical properties of the resulting reinforced extrudes have been observed in some detail. A knowledge of the glass content, extrusion temperature and ram speed required to give optimum properties has been obtained.

In all, the results of several hundred extrusions, the corresponding mechanical tests and microscopical examinations are reported in this thesis.

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## CHAPTER 1

## INTRODUCTION

1.1.1 Powder Metallurgy

In the early fifties considerable interest was shown in S.A.P. products manufactured by A.I.A.G. These possessed high strength at elevated temperatures due to the high oxide content, (6-15%), but low ductility due to the brittle nature of the oxide. Various alloying additions were used in an attempt to improve the ductility but little success was achieved. Atomised Al alloy powders have a lower oxide content (0.2-5%) depending on the process used. However, due to the high cooling rate from the liquid state, atomized powders possess a very fine, homogeneous, (though non-equilibrium) structure which improves the ductility considerably. Alloys which are very difficult to manufacture by normal casting procedures, due to poor solid solubility of a particular phase in the bulk metal e.g. Al-Fe, can be produced by atomization. The resulting powder may be consolidated by a number of mechanical working processes including forging, rolling, and extrusion. To ensure complete consolidation in the extrusion process a minimum extrusion ratio (5:1 to 10:1) must be exceeded. However, evidence <sup>19</sup> exists that lower extrusion loads and faster extrusion speeds (compared to cast billet extrusion) are possible. These advantages compensate for the restricted minimum extrusion ratio.

1.1.2 Precipitation Hardening

Many of the Aluminium alloys are very responsive to heat treatments. The Al-Cu and Al-Mg-Si (which behaves as an Al-Mg<sub>2</sub>Si binary alloy) systems are classic



examples of precipitation hardening alloys. The usual form of the heat treatment consists of the three stages:-

- 1) heat soaking
- 2) quenching
- 3) ageing

Such a treatment is fairly costly when applied to a fabricated component and in the extrusion industry a compromise treatment is used. This much more convenient process involves:-

- 1) heating the billet to the soaking temperature
- 2) press quenching the extrude
- 3) ageing the extrude

Part of this project involved finding out if extruded Al-Mg-Si alloy powders would prove responsive to a suitable heat treatment resulting in an increase in the strength of the product.

#### 1.2.1 Fibre Reinforcement

Approximately ten years ago a series of papers concerning fibre reinforcement was published when certain firms, including Rolls-Royce, were interested in carbon-fibre products. The possibility of including strong, high elastic modulus fibres in a ductile metal matrix was and indeed still is very interesting. The main advantage of such a system is that the rigidity as well as the strength of fairly light metals, such as aluminium, may be significantly improved. However, the large drawback of the high production cost of the composite has greatly hindered general development. Such composites have been manufactured on a limited scale for special purposes, especially in the aerospace industry, where production costs are often outweighed by performance requirements.

### 1.3.1 Composite Production by Powder Route - Present Work

The method of composite production employed in this investigation overcomes many of the usual difficulties and enables the strengthening mechanisms of dispersion strengthening, precipitation hardening and fibre reinforcement to be used on a major architectural alloy, HE9. The method is simple and fast so that production costs are kept to a minimum.

## CHAPTER 2

## LITERATURE REVIEW

2.1.1 Introduction

To achieve optimum composite properties a thorough knowledge of the factors which dictate these properties is required. The method of production and the type of system employed in the composite are obviously important. A knowledge of the press capacity required, estimated from various load prediction models, is also advantageous. Process conditions, defined by temperature and strain rate, together with material properties determine the product structure which in turn determines its properties. Methods of estimating these conditions obtaining during extrusion must therefore be examined in considerable detail. Possible strengthening mechanisms for improving the composite properties, e.g. heat treatment, are also of considerable importance.

2.2.1 Cold compaction of metal powders

Metal powders are frequently cold compacted to facilitate handling and pre-heating operations prior to the application of consolidation processes such as hot forging and/or extrusion. The powder compaction process has been extensively studied. These studies have usually involved the formulation of a relationship between the applied pressure and the resulting compact density. For most metal powders it is generally agreed that the compaction process consists of three distinct stages<sup>1</sup> (Fig. 1). Kunin and Yurchenko<sup>2</sup> suggested that the following mechanisms operate during the various stages:-

Stage 1 Packing of the undeformed particles into a smaller volume.

Stage 2 Elastic and plastic deformation of the particles. During deformation the particles slide over each other. In metals having oxide surface layers e.g. aluminium, this sliding movement causes rupturing of the oxide layers. This leads to metal-metal interparticle point contact which results in cold welding. With increasing pressure these areas of contact increase in size. Easterling<sup>3</sup> showed that the presence of these oxide layers greatly reduced compact strength as well as lowering the response of the compacts to induction heating prior to further hot working. It will be shown in the present investigation that mains frequency induction heaters are incapable of preheating aluminium alloy powder billets compacted to > 90% theoretical density. Strömgen<sup>9</sup> measured the strength of aluminium powder compacts pressed to 100% theoretical density. He suggested that the low strength was due to the oxide particles causing stress concentrations at interparticle boundaries.

Stage 3 Bulk compression i.e. cold working of the compact as a whole. It should be noted that the processes mentioned in stages 1 and 2 sometimes occur concurrently.

One of the most popular relationships between pressure and compact density was first suggested by Shapiro and Kolthoff<sup>4</sup> and by Konopicky<sup>5</sup> :

$$\ln \left( \frac{1}{1-D} \right) = K'P + \text{constant} \quad (1)$$

D = relative density

P = compaction pressure

K' = a compaction constant.

Equation (1) is also plotted in Fig. 1. Good agreement between theory and experimental results exists over the linear region of the experimental curve (Stage 2). The overestimation of the density given by equation (1) at low pressures is due to the equation not taking into consideration particle packing prior

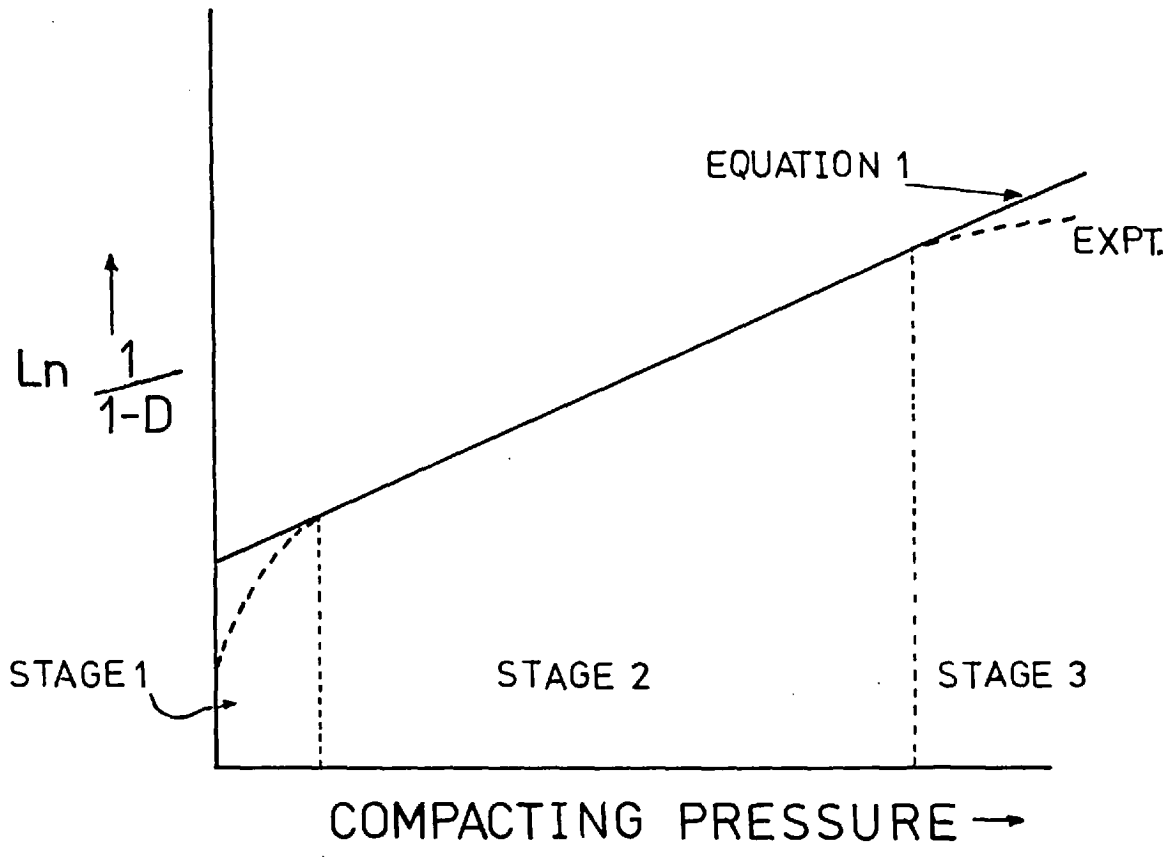
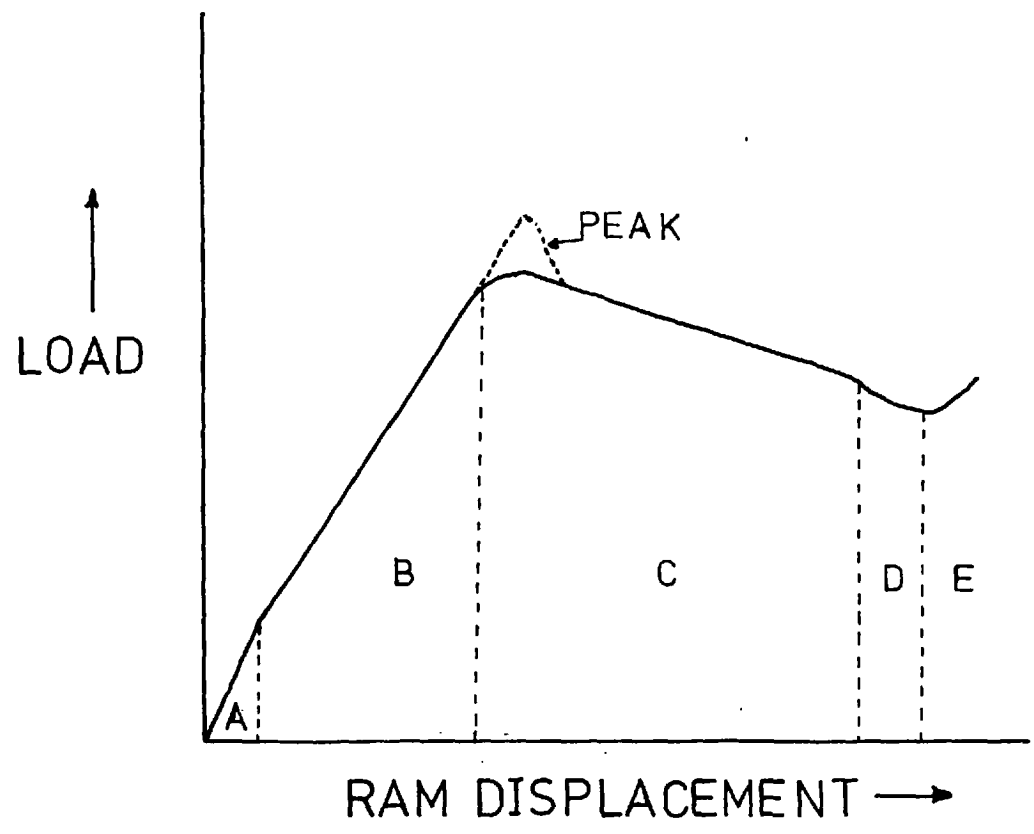


FIG. 1 Relationship between compacting pressure and relative density

FIG. 2 Extrusion pressure as a function of ram displacement (Idealised)



to deformation. Hewitt<sup>6</sup> attempted to incorporate the effects of strain hardening during compaction. His experiments with iron powder showed that the compaction behaviour during stage 2 could be predicted very accurately. However he found that the deviation from linearity during stage 3 could not be attributed to strain hardening. Hewitt suggested that stage 3 involved the closure of pores and defects and therefore could not be explained by a macroscopic model.

Heckel<sup>7</sup> agreed with the operating mechanisms suggested by Kunin and Yurchenko<sup>2</sup> and proposed a theoretically derived pressure-density relationship similar to equation (1). He considered powder compaction to be analogous to a first order chemical reaction. The pores were assumed to be the reactant so that :-

$$\frac{\text{change in density}}{\text{change in pressure}} \propto \text{pore fraction}$$

Kawakita<sup>8</sup> recently proposed another empirical relationship based on volume and pressure measurements. He derived the equation :-

$$C^* = \frac{V_0 - V}{V_0} = \frac{a b p}{1 + bP} \quad (2)$$

$C^*$  = degree of volume reduction

$V_0$  = initial apparent volume

$V$  = actual volume under pressure

$a, b$  = constants

None of the relationships discussed in this section explain the behaviour of compacts under high pressure, although Hewitt attempted to explain this behaviour in terms of work hardening. All the relationships deal with the effect of compaction

pressure on theoretical density. A more useful relationship would relate the compaction pressure to compact strength as past experience has shown that 100% theoretical density does not always correspond to 100% theoretical strength (ie. the strength of a cast and wrought material).

### 2.3.1. Extrusion Process

The process of extrusion seems, initially, to be a simple method of fabricating components. Basically it involves a metal billet being pushed through a hole or orifice of a particular shape and size to give a desired product. Most metals are extruded at elevated temperatures for two main reasons:

(1) To prevent work hardening increasing the required load and thus the energy used.

(2) An extrude with a "cold work" structure (ie. a high dislocation density) is very brittle. Fig. 2<sup>10</sup> depicts an idealised extrusion curve. During the linear region, (A), the billet is upset to fill the container. The billet is then compressed until metal begins to flow through the die, (B). After this breakthrough a "steady state" is established during which extrusion continues, (C). The load actually decreases slightly during this pseudo "steady state" region because the friction forces between billet and container wall decrease as the billet length decreases. Eventually a piping<sup>11</sup> defect tends to occur, (D). This happens when the rear-centre of the billet "caves" in and material from the sides of the billet flows laterally to occupy the newly created void. This often leads to a central core of undesirable, oxidised material running through the latter stages of the extrude. Finally the billet becomes so short that flow is inhibited, (E). The pressure increases

rapidly as the material tries to flow almost perpendicularly to the direction of extrusion.

A number of variations in Fig.(2) are commonly observed. Steel and nimonic<sup>12</sup> alloys exhibit a "steady state" region in which the load increases with ram displacement. This is due to the billet cooling during the extrusion process, (typical temperatures 1100-1200°C). As the temperature decreases the flow stress increases due to reduced dynamic softening. Very often, as in the case of Aluminium alloys,<sup>13</sup> a peak in the load-displacement curve is present, (dotted line, Fig.(2)). This peak is probably due to a thermally activated process involving a build-up of dislocations followed by their annihilation.

In the extrusion of light metal alloys graphite is sometimes put on the die face and die-land to act as a kind of "lubricant". This is not full-film lubrication. The extrusion of steel and nimonic alloys involves the use<sup>14</sup> of fibre glass as a lubricant. This prevents the billet from being welded to the container (thus protecting the container liner) and also lowers the working load. Interest in the cold extrusion of steel using a zinc phostate-sodium<sup>16</sup> stearate lubricant has grown recently.

A process which greatly reduces the effects of friction is indirect extrusion.<sup>15</sup> In this process the ram is used to seal the container end. The ram and container are then moved simultaneously at the same speed and in the same direction. This technique prevents any relative motion between the container walls and the billet. This action pushes the billet through the die. Thus the only frictional forces which must be overcome are those due to the billet/



die face contact and the extrude/die land contact. On an industrial scale, these friction effects are considerable and by reducing them large energy savings become possible.

The process of extrusion does have several disadvantages. One of the biggest is that variations in strain, strain rate and temperature throughout the deforming metal produce an extrude of varying structure. Thus the mechanical properties of the extrude vary with position. The press capacity is dictated by the peak load (Fig. 2). Therefore the press capacity is only fully utilized for a fraction of the duration of each extrusion.

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Recent developments in the extrusion field include the semi-continuous hydrostatic process, suggested by Alexander and Lengyel<sup>17</sup>, and the continuous ("conform") extrusion process<sup>18</sup> of Green. In the past the billet length has always been limited to minimize the amount of power wasted in overcoming billet-container wall friction. Both of these new processes actually make use of this friction force to "drag" the feedstock to the die. The "conform" process basically involves<sup>124</sup> the feeding of a rod into a peripheral square groove in a rotating wheel, the groove side being slightly smaller than the feedstock. A stationary shoe placed around part of the wheel periphery completes the fourth side of an "extrusion container". The friction between rod and groove continuously drags the feedstock into this "extrusion container". An abutment held by the shoe intrudes fully into the groove to act as a container end. The rotating wheel feeds the rod to this point and the force generated at the abutment upsets the feedstock to fill the container. The increasing frictional force generated between the

groove and the upset material generates a pressure which is sufficient to cause extrusion through a hole in the abutment. This process appears to have an important part to play in the future developments of the extrusion industry.

### 2.3.2. Extrusion of Powders

The most widely used method for fabricating a component from metal powder is hot forging. On an industrial scale the extrusion of powders is in its infancy. In experimental powder investigations several methods have been used and these are listed below :-

- (i) loose powder extruded.
- (ii) powder cold compacted, then extruded.
- (iii) powder cold compacted, sintered then extruded.
- (iv) loose powder placed in can, evacuated, sealed, then extruded.
- (v) powder isotatically compacted, then extruded.
- (vi) powder isostatically compacted, canned, evacuated, sealed, then extruded.

The only industrial powder extrusion processes in operation at present involve nimonic, stainless steel and tool steel powders. Method (iv) is usually employed.<sup>20</sup> The evacuation of the can prevents oxidation of the powder. The can, usually made of mild steel, acts as a lubricant although fibre-glass is normally also used. The main disadvantage of this method is that after extrusion the can material must either be machined from the surface or else removed by soaking in a pickling bath. Recent experiments

have shown that method (V) may be successfully used in the extrusion of sponge iron powder<sup>21</sup> and high speed steels.<sup>22</sup> Method (ii) is usually used by investigators working<sup>23</sup> with low melting point alloys. This facilitates the easy handling and preheating of the powder prior to extrusion.

Sheppard and Chare<sup>23</sup> showed that much lower loads were required for the extrusion of aluminium alloy powder compacts than for cast billets of the same composition. They suggested that this load reduction was due to the powder compact not achieving complete consolidation until late in the deformation cycle. Sheppard also showed that the strain-rate sensitivity of a powder compact was much less than that of the corresponding cast billet. These two, important findings are thought to hold for other high stacking fault energy metals in which dynamic recrystallisation does not occur. This strain-rate sensitivity effect indicates that extrusion speeds could be increased if powder compacts were used instead of cast billets. Another advantage of the powder route is that oxide particles, resulting from the oxide layer disintegration, cause an increase in strength due to dispersion strengthening effects. As the oxide particles are very stable this strengthening effect is present even at elevated temperatures. The inherently fine structure of the atomized powders also prevents macrosegregation, a danger when using cast ingots.

However one important limitation to powder extrusion is that a minimum extrusion ratio must be exceeded to ensure complete densification. Gurney et al<sup>24</sup> found this ratio to be approximately 6:1 for 7075 aluminium alloy powder.

#### 2.4.1. Prediction of Load Slip-Line Field Theory.

At present several methods are used in attempts to relate the extrusion pressure to the flow stress of the material. The two most widely used are based on :

1. Slip-line field theory.
2. Load bounding (upper bound) theory.

The slip-line field technique enables fairly complex plastic flow equations to be solved accurately by establishing the maximum shear line field. Solutions for the extrusion process have been developed for a variety of friction conditions ranging from perfectly rough billet/die contact surfaces to perfectly smooth ones. Various die shapes have also been considered<sup>26,27,28</sup> (square, wedged-shaped and concave). These solutions have usually consisted of graphs of extrusion pressure versus extrusion ratio. This theory is mathematically correct but its use is rather tedious. Johnson and Kudo<sup>25</sup> suggested the following method for employing slip-line field theory:-

- (1) Construct the slip-line field which satisfies given boundary conditions in terms of stresses, starting from certain boundary surfaces of the material and assumed rigid plastic slip-lines.
- (2) Obtain the velocity distribution by means of the slip-line field and from some of the given velocity boundary conditions. Then find if the remaining boundary conditions are fulfilled.

- (3) Check for a positive rate of plastic work everywhere in the deformation zone.
- (4) Check that the yield criterion in the assumed non-deforming region is obeyed. If the boundary conditions are not satisfied the field must be redrawn.

Over a limited range of extrusion ratios Johnson showed that the theoretical solutions could be approximated by:-

$$\frac{P}{2K} = A + B \ln R$$

A and B are friction dependent constants. The shear yield stress, K, is a constant for an ideal, rigid, perfectly plastic material. In real materials, especially powder compacts, K varies throughout the deformation zone. To allow for work hardening Dodeja and Johnson<sup>29</sup> suggested the use of a mean shear flow stress, found from plane strain compression tests. More recently Farmer and Oxley<sup>30</sup> produced a modified solution for work hardening materials. However this modification permitted maximum reductions of only 50% to be considered.

The two biggest disadvantages<sup>25</sup> of the slip-line field technique are :-

- (1) It may only be rigorously applied when conditions of plane strain are present.
- (2) Strictly it only applies to rigid, perfectly plastic materials.

It is therefore almost impossible to use in the case of powder extrusion as this is not a plane strain process and metal powder is not a rigid perfectly plastic material.

Due to the tedious nature of the application of this technique and the limitations involved, Johnson developed the upper bound method of analysis.

#### 2.4.2. Plane Strain Upper Bound Solution

The upper bound solution, developed by Johnson<sup>31</sup>, produces loads which are in excess of the true values and in excess of those predicted by the slip-line field technique. By increasing the complexity of the upper bound solution (i.e. the number of velocity discontinuities in the field) the solution is brought nearer and nearer to that given by the slip-line field method. (It should be noted that as the upper bound solution gives an overestimate on the load, it must be minimized to give the solution). In view of the approximations of plane strain, mean yield stress and frictional boundary conditions, common to both techniques, the great saving in effort afforded by the upper bound technique makes the less accurate answer acceptable in the majority of cases.

This technique is based on strain rather than stress equilibrium conditions. Elements of material are assumed to offer maximum resistance to deformation i.e. they deform in such a way as to require the maximum amount of work to produce a given strain. Matter is assumed to be incompressible and only velocity discontinuities and boundary conditions

are considered. The material is assumed to deform as rigid plastic blocks.

The most difficult part of the procedure is the determination of the original field. Kudo and Johnson<sup>25</sup> suggested the following method:-

(1) Assume a velocity field for the body of material which is restricted only by the need to conform to the velocity boundary conditions and the law of constant density.

(2) Calculate the rate of work due to the plastic deformation and due to the slip of the material over the tool surfaces, using the assumed velocity field.

(3) Hence find the extrusion load from this rate of working.

Step (1) may be simplified by basing the field on a known slip-line field or on a knowledge of the flow pattern gained experimentally. Sheppard and Raybould<sup>32</sup> showed that a velocity field could be established by considering the sub-grain size at different positions in the deformation zone of a partial extrusion. Using a minimized plane strain upper bound solution Johnson obtained solutions for extrusion through wedge-shaped and square dies for conditions of zero and sticking friction.

Although simpler to use than slip-line field theory, plane strain upper bound solutions suffer from serious drawbacks:-

- (1) Strictly, they may be applied to plane strain conditions only. ( Extrusion is an axisymmetric process).
- (2) They must be minimized by the application of mathematical techniques.
- (3) Intermediate friction conditions prove very difficult to consider.
- (4) The load predicted is always an overestimate.

#### 2.4.3. Axisymmetric Upper Bound Solution

As stated in the previous section extrusion is rarely, if ever, carried out under plane strain conditions. Therefore an axisymmetric upper bound solution is required. Thomsen<sup>33</sup> made the following experimental observations :-

- (1) Material flow is similar for geometrically similar shapes in axisymmetric and plane strain extrusion.
- (2) The velocity during axisymmetric extrusion is the square of the velocity during plane strain extrusion.

Using this information Sheppard and Raybould<sup>34</sup> derived a simple axisymmetric upper bound solution by first finding a minimized plane strain solution. The areas of the discontinuity surfaces were then obtained by revolving the plane strain field around the centre axis and the magnitude of the velocity discontinuities by squaring those given by the plane strain hodograph.



The first rigorous axisymmetric solution based on a kinematically admissible axisymmetric velocity field was suggested by Kobayashi<sup>35</sup> and by Halling and Mitchell<sup>36</sup> for extrusion through conical dies. The pattern chosen by Halling and Mitchell involved a double triangle arrangement of velocity discontinuities. Basically, they allowed the material inside the triangles to accelerate along the flow lines, producing circumferential straining, and modified the plane strain hodograph accordingly. Suggestions were also made for the incorporation of work hardening and friction effects. The optimization process was carried out on a computer. Adie and Alexander<sup>37</sup> extended this work by producing three triangle arrangements of velocity discontinuities for greater accuracy. Kinematically admissible velocity fields were drawn for rod-can, can-can and tube-can combined extrusions.

This investigation will show that the axisymmetric upper bound technique may be applied to powder extrusion if allowances are made for the effects of strain rate and strain on the powder compact flow stress.

Recent advances in computer technology have enabled steady state plastic deformation processes to be treated by numerical techniques. Zienkiewicz<sup>121</sup> applied a finite element method to extrusion and produced results which agree well with slip-line field solutions. He improved the accuracy of the solution by increasing the number of elements used in the mesh and noted that any frictional conditions could be accommodated.

With the availability of modern computers which possess large storage capacities the finite element method of analysing metal working processes such as extrusion, will probably become predominant.

### 2.5.1. Strain Rate Determination

The strain rate during extrusion is a very difficult parameter to define. It is obviously a function of extrusion ratio and ram speed. However, the strain rate varies throughout the deformation zone. Sheppard and Raybould<sup>34</sup> applied the upper bound analysis to the problem of strain rate determination. The strain rate was calculated along a series of streamlines by means of the following method :

$$\epsilon = \frac{u}{v}$$

$\epsilon$  = strain

$v$  = velocity of material perpendicular to discontinuity

$u$  = magnitude of discontinuity

The time to extrude a unit volume of material of unit area past the discontinuity =  $\frac{1}{v}$

Therefore the strain rate,

$$\dot{\epsilon} = \left( \frac{u}{v} \right) / \left( \frac{1}{v} \right) = u$$

An average strain rate along each stream line is found by considering the number of velocity discontinuities intersected. This method does give an indication of the strain variation across the section.

However it possesses two main disadvantages:

- (1) The deformation is assumed to occur instantaneously at a number of discontinuities.
- (2) A mean overall strain rate has to be calculated from a knowledge of the strain rate along each stream line.

Feltham <sup>38</sup> proposed a mean strain rate calculated by assuming that the deformation occurred within a cone of  $45^\circ$  semi-angle. He also assumed the total strain =  $\ln(\text{Extrusion ratio})$ . By dividing the volume of material extruded per unit time into the volume of the cone he obtained a mean strain rate,

$$\bar{\epsilon} = \frac{6V \ln R}{D_B} \quad (12)$$

$D_B$  = Billet diameter.

$V$  = Ram speed.

<sup>39</sup>

Alexander suggested that

$$\epsilon = a + b \ln R$$

$$\text{giving : } \bar{\epsilon} = \frac{6V (a + b \ln R)}{D_B} \quad (4)$$

(  $a, b$  = constants in the load equation).

However, the deformation zone is rarely a cone of semi-angle  $45^\circ$ . Wilcox and Whitton <sup>40</sup> pointed out that equation (4) should contain a

term to allow for a variation in semi-angle.  
 Chandra and Jonas<sup>(41 - 43)</sup> produced the following  
 equation :-

$$\dot{\epsilon}_x = \frac{4V_D B^2 \tan \omega}{D^3}$$

D = Deformation zone diameter at any point  
 in zone.

$\omega$  = Deformation zone semi-angle.

Unfortunately, all these expressions assume  
 a conical deformation zone whereas, in practice,  
 the deformation zone / dead metal zone interface  
 is usually curved. However they do have the  
 advantage of simplicity.

#### 2.6.1. Temperature Variation During Extrusion

It is generally agreed that during the  
 extrusion process 90 - 95 % of the work done  
 is converted to heat energy, the remaining  
 5 - 10 % being stored in the new structure of  
 the extrude. The temperature change in the material  
 during extrusion depends on many parameters  
 including :-

- (1) The temperatures of the billet and tools.
- (2) The conductivities of the billet and  
 tools.
- (3) The heat capacity of the billet and  
 tools.
- (4) The ram speed.

- (5) The extrusion ratio.
- (6) The flow stress of the billet material.

Clearly temperature prediction during such a process is very difficult.

Tanner and Johnson<sup>(44)</sup> used a plane strain upper bound analysis and assumed the following:-

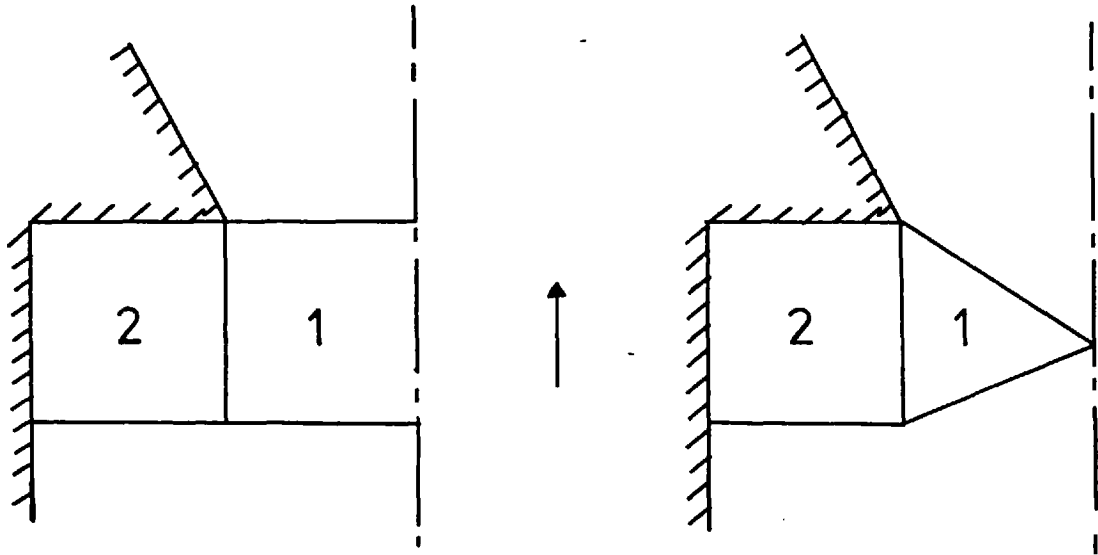
- (1) Plane strain extrusion
- (2) Temperature of billet = Temperature of tools
- (3) No conduction.

This model considered the temperature of the billet material to remain constant until a velocity discontinuity was encountered. The resulting shear produced a strain. This strain was then expressed in terms of a quantity of work done. Hence a temperature rise, proportional to this work, was obtained. By considering the number of discontinuities encountered by each stream line the final temperature distribution in the die-throat was obtained.

Johnson and Kudo<sup>45</sup> extended this work for axisymmetric extrusion through square and conical dies. The three fields used by Johnson and Kudo are shown in Fig. 3. Terms were introduced to allow for the work done inside regions 1 and 2 in addition to that done at the discontinuities.

Sheppard and Raybould<sup>34</sup> also employed an upper-bound analysis. The validity of this theory was confirmed by performing several extrusions with thermocouples inserted in the dead metal zones. The measured temperatures agreed reasonably well with those predicted by the analysis. Sheppard and Raybould also developed an integral profile model (see theory section) which predicted

## SQUARE DIES

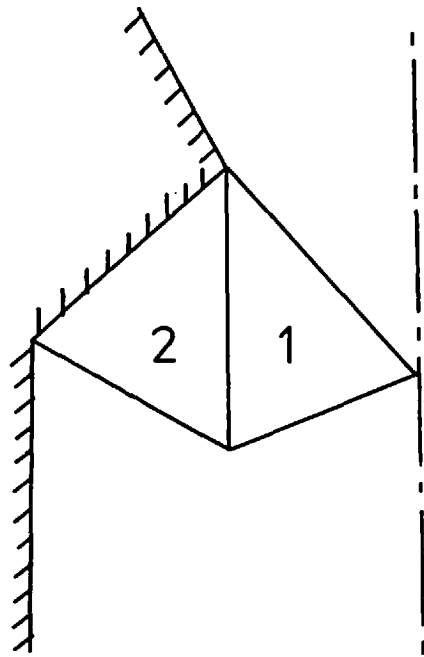


HOMOGENEOUS-HOMOGENEOUS

HOMOGENEOUS-TRIANGULAR

FIG. 3 Velocity fields for different types of extrusion

## CONICAL DIE



TRIANGULAR-TRIANGULAR

values close to those measured experimentally.

The method of Singer and Al-Samarrai<sup>46</sup> involved correcting the mean adiabatic temperature rise to give the mean temperature rise for the existing conditions. They used an empirical load formula and allowed for heat conduction to the die and container. All of the deformation was assumed to occur in the plane of the die. The results obtained were reasonably close to emergent extrude temperatures measured with a two pronged thermocouple.

Akeret<sup>47</sup> used a numerical analysis. He divided the billet into a series of blocks (Fig (4) ) and treated extrusion as a " stepwise " process. Heat conduction from the heat source block to the billet and to the container was also taken into account. Once again the results agree reasonably well with experimentally measured values.

The analyses discussed in this section all make one of two assumptions :

- (1) Deformation occurs in one localized zone.
- or (2) Deformation occurs in steps at velocity discontinuities.

Neither of these assumptions is entirely valid ( especially not the former ). Great difficulty is experienced in dealing with initial temperature differences between billet and tools. This is significant as in industry the billet and tools are rarely at the same temperature. Once again, with the aid of modern computers, finite element numerical analysis will probably prove to yield the best results.

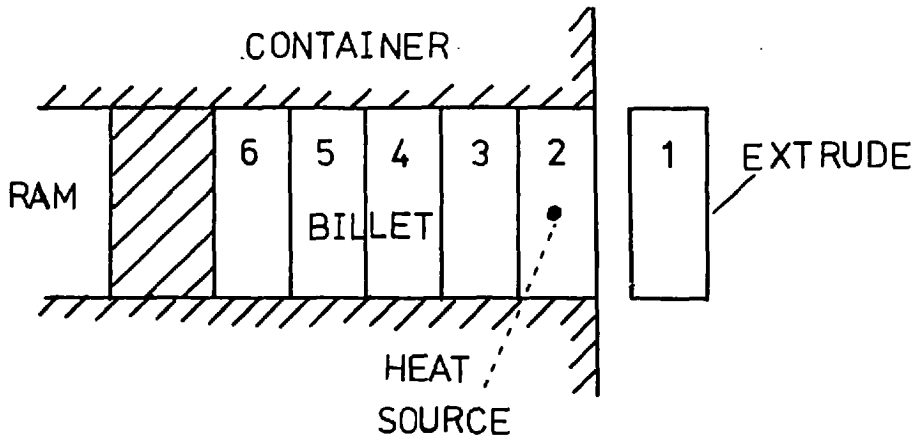
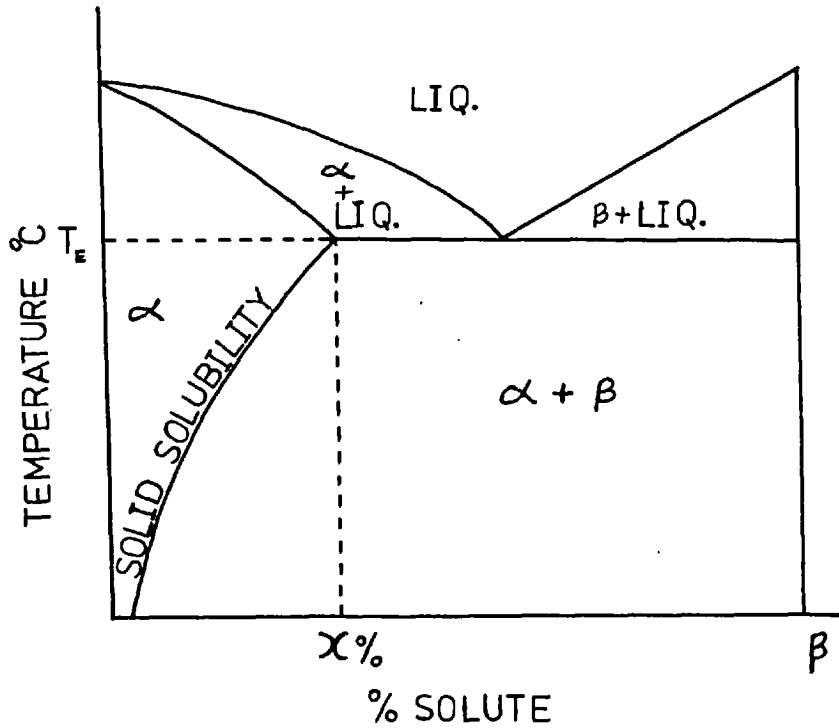


FIG. 4 Temperature rise model involving stepwise movement of the billet

FIG. 5 Phase diagram for ideal precipitation hardening system





On an industrial scale, Reynolds<sup>48</sup> measured the emerging extrude temperature with a lead sulphide - cell radiation pyrometer. This proved to be a quick and convenient method. However although the results were within  $\pm 4^{\circ}$  C of those obtained using a contact thermocouple for pure aluminium extrusion at  $450 - 500^{\circ}$  C, the error increased to  $\pm 9^{\circ}$  C at  $380 - 450^{\circ}$  C. Unfortunately results for aluminium - magnesium silicide alloys produced much larger errors. Thus the accuracy of this method seems to depend on the emissivity of the material. It is therefore unsatisfactory.

#### 2.7.1. Extrude Structure

The structure produced by extruding aluminium, aluminium alloys or most high stacking fault energy metals at elevated temperatures may be wholly recrystallised, partly recrystallised or unrecrystallised depending on the extrusion conditions, especially the temperature. The tendency is for the recrystallised percentage to increase with extrusion temperature and with the amount of deformation. A variation in structure across the section usually exists. Frequently recrystallisation only occurs near the surface due to the greater deformation occurring there. Sometimes an unrecrystallised structure may result from an extrusion carried out at a temperature above the nominal recrystallisation temperature. Such a structure usually resembles a " fibrous " cold worked structure under the optical microscope. However a substructure consisting of subgrains of low misorientation produced by dynamic recovery is revealed by the electron microscope.<sup>48</sup>

Jonas<sup>49</sup> demonstrated that annealing a dynamically recovered aluminium structure failed to cause any recrystallisation and merely increased the subgrain size slightly. This indicated that dynamic recovery during hot working was more effective than static recovery produced by conventional annealing of a cold worked structure. He discovered that the subgrain size and the perfection of the wall increased with extrusion temperature.

This tendency for high stacking fault energy metals to recover instead of recrystallise, is due to their lack of extended dislocations and can be explained in terms of dislocation theory. During deformation glide and forest dislocations intersect forming tangles and increasing the dislocation density. However at typical hot extrusion temperatures and under the action of the deforming stress and stress fields of other dislocations the ability of the dislocations to climb and cross slip is enhanced<sup>50</sup>. The process enables<sup>51</sup>:-

- (1) Dislocations to escape from the specimen surface.
- (2) Dislocations of opposite sign to annihilate each other.
- (3) Dislocations to arrange themselves in sub-boundaries. Thus cells form and become subgrains as dislocations migrate from within the cells to the walls. The subgrain size tends to increase by subgrain coalescence in order to lower the free energy even further.

Recrystallised structures may be classified in three categories depending on the stage at which recrystallisation occurs<sup>51</sup>. These are:-

- (1) After deformation, during cooling to room temperature i.e. static recrystallisation
- (2) During deformation by recovery processes (in situ recrystallisation)
- (3) During deformation i.e. dynamic recrystallisation.

The difference between the processes in 2 and 3 is that in the former the crystallographic orientation of each element

of volume is basically unchanged whereas in the latter the orientation is completely changed. This change is due to large angle boundaries sweeping through the material.

The recrystallisation process involves the nucleation and growth of new grains in a deformed structure. These new grains are relatively free from strain and dislocations. The nucleation process probably involves the growth of subgrains both in size and misorientation by subgrain coalescence.

The type of recrystallisation occurring during aluminium and aluminium alloy extrusion remains uncertain. Castle<sup>52</sup> found an "elongated recrystallised" structure in some Al-Cu-Mg-Mn extrudes which indicated that dynamic recrystallisation had occurred. However, he correctly stated that this was not conclusive proof of dynamic recrystallisation. He questioned the effectiveness of the extrude quench and suggested that the structure was really produced by static recrystallisation with a preferred orientation in the extrusion direction. Li<sup>53</sup> and Hu<sup>54</sup> proposed in situ recrystallisation as the mechanism which operated during aluminium extrusion whereas Jonas<sup>55,49</sup> argued in favour of dynamic recrystallisation. Again these investigators failed to provide conclusive proof of the validity of their theories.

Thus we may conclude that in the extrusion of cast aluminium and its alloys the extrude structure may be wholly or partly recovered or recrystallised depending on the extrusion conditions and the alloy composition. The type of recrystallisation which occurs is uncertain. This lack of knowledge is serious in that a recrystallised structure is undesirable, being weak in comparison to a "fibrous" cold worked structure. For this reason many alloys contain recrystallisation inhibitors to prevent recrystallisation occurring.

eg. Zr is added to Al-Zn-Cu-Mg alloys.

Extrudes fabricated by Sheppard and Chare<sup>23</sup> from aluminium alloy powders exhibited no recrystallisation. He suggested that this was caused by oxide particles obstructing grain boundary migration. Nobili<sup>56, 57</sup> studied the behaviour of aluminium / alumina ( S A P ) alloys and reached a similar conclusion. Plumtree and Deep<sup>58</sup> recently extruded cast aluminium billets of different purity. By means of a Kikuchi line analysis they showed that an impurity content of 0.3 % was enough to prevent recrystallisation under certain extrusion conditions.

It therefore seems that recrystallisation is not a problem in the extrusion of aluminium powders. This is another advantage the powder route possesses over the traditional cast billet extrusion process.

#### 2.8.1 Deformation Mode

In the previous section the processes occurring during hot working, namely recovery and recrystallisation, were discussed. Various theories have been put forward to explain the mechanisms by which these processes occur. Most of these theories have been proposed for creep deformation. Three basic models have been suggested to explain the operating mechanism during dynamic recovery. The three models are based on, respectively :-

- (1) The motion of jogged screw dislocations.
- (2) The climb of edge dislocations.

- (3) The existence of a network of dislocations inside the subgrains.

All three models assume that the activation enthalpy is the same as that for self-diffusion. The differences arise in the terms comprising the structure factor and activation volume.

Barret and Nix<sup>59</sup> proposed the first model for steady state creep. A jog is part of a screw dislocation which does not lie in the slip plane. Under the influence of a shear stress the screw dislocation glides and the jog is "pulled" with it leaving a trail of vacancies. The stress required to move the dislocation depends on the vacancy concentration and its effect on the jog. This model unfortunately does not permit the effect of alloying to be considered. Jonas<sup>49</sup> showed, qualitatively, the applicability of this theory to high strain rate deformation.

Weertman<sup>60</sup>, <sup>61</sup> proposed the second model, based on the climb of edge dislocations. Dislocation sources are assumed to be distributed throughout the material. Under the effects of the applied shear stress they emit loops. These loops expand until they are blocked by the stress fields of loops on parallel planes. The climb of piled up dislocations out of the slip plane then becomes the rate controlling process. Once again, this model was proposed for steady state creep. It does not allow for alloying additions to be considered. Objections to this model have been raised<sup>66</sup> because the predicted substructure is not usually found in specimens deformed in creep tests.

The third model, based on a three dimensional dislocation network within the grains, was suggested by McLean<sup>62</sup>. Moving dislocations intersect this network and, under the influence of the applied shear stress, the unpinned parts of the dislocations bow out decreasing the size of the network. Jogs migrate along the dislocations causing edge dislocations to climb. As a result, the network grows. The rate of recovery depends on the diffusion coefficient. McLean<sup>63</sup> considered the effects of alloying additions and concluded that the dispersed phase particles anchored the network.

Unfortunately, to date, the validity of all three models remains in some doubt. As all were proposed for creep deformation their applicability to high strain rate hot working processes must also be doubtful.

Stüwe and Ortner<sup>64</sup> proposed a model for the mechanism by which dynamic recrystallisation occurs. They based the model on the nucleation of recrystallised grains followed by deformation within the grains. However, experimental verification of this model has not yet been produced.

### 2.9.1 The Z Parameter

Garofalo<sup>66</sup> investigated creep deformation and proposed the following stress-strain rate relationships:-

$$\dot{\epsilon} = A_2 \sigma^{n_2} \quad \text{at low stresses}$$

$$\dot{\epsilon} = A_3 \exp(\beta \sigma) \quad \text{at high stresses}$$

$A_2$  and  $A_3$  are temperature dependent constants

$\beta$  and  $n_2$  are temperature independent constants

Zener and Holloman<sup>65</sup> also investigated creep deformation and found the activation energy for plastic deformation to be the same as that for self-diffusion. They introduced a temperature compensating factor into the strain rate term in an attempt to combine stress-strain rate information at different temperatures. The temperature compensated strain rate,

$$Z = \dot{\epsilon} \exp \frac{\Delta H}{RT}$$

$\Delta H$  = activation energy.

Sellars and Mc G. Tegart<sup>67</sup> combined the work of Garofalo with that of Zener and Holloman and produced an empirical relationship for torsion:-

$$\dot{\epsilon} = A_1 (\sinh \alpha \sigma)^n \exp\left(-\frac{\Delta H}{RT}\right) \quad (6)$$

i.e.  $Z = A_1 (\sinh \alpha \sigma)^n$

where  $A_1$ ,  $\alpha$ ,  $n$  are temperature independent constants.

Jonas<sup>68</sup> showed that this equation could be applied to the hot working of aluminium over a range of strain rates and temperatures. Chare<sup>19</sup> showed this equation to be valid for the extrusion of aluminium alloy powders.

### 2.10.1 Precipitation Hardening

The phase diagram in Fig. (5) shows the essential requirement for precipitation hardening in an alloy system. The decrease in solid solubility with temperature is the aforementioned requirement. (N.B. This is a necessary condition but it may not be sufficient.) The usual heat treatment consists of three stages:-

- 1) Heat soaking
- 2) Quenching
- 3) Ageing

For the maximum response the alloy must contain just less than  $x\%$  solute. (See Fig. 5). Examples of systems which respond are Al-Cu and Al-Mg<sub>2</sub>Si.<sup>69</sup>

Stage 1 in the treatment involves heating the alloy to a temperature in the single phase,  $\alpha$ , region. Maintaining this temperature enables the second phase,  $\beta$ , to dissolve. The temperature should not exceed  $T_E$  (Fig. 5) in order to prevent incipient melting occurring if a cored, non-homogeneous structure is present. Usually the  $\beta$  phase is brittle and optimum properties are obtained with 5-10% by volume of precipitate. Ideally the time of solution is that required to produce a homogeneous  $\alpha$  structure. In practice such a structure may be unattainable if large grains are present as this automatically means large diffusion distances.

In stage 2 the specimen is quenched, usually in water. This prevents precipitation and maintains a high vacancy concentration. Quenching in water does not produce cracking because the  $\alpha$  phase is usually very ductile. The importance of the quenched-in vacancies must be stressed<sup>70</sup> and it has been estimated that they increase the diffusion rate by a factor of  $10^6$  times. The importance<sup>76</sup> of vacancies as heterogeneous nucleation sites is also demonstrated by precipitate free zones which sometimes occur adjacent to grain boundaries because the vacancies in these zones have been absorbed by the grain boundaries.

Stage 3 involves an ageing treatment. The specimen is heated for several hours at a temperature just below the solid solubility curve in Fig. 5 (usually  $0.4-0.5T_m$ ). Precipitate particles form and start to grow. The optimum particle size is  $10^2-10^3 \text{ \AA}$ . The effect of the ageing temperature on mechanical properties is schematically represented in Fig (6). High temperatures cause rapid rates of precipitation and precipitate



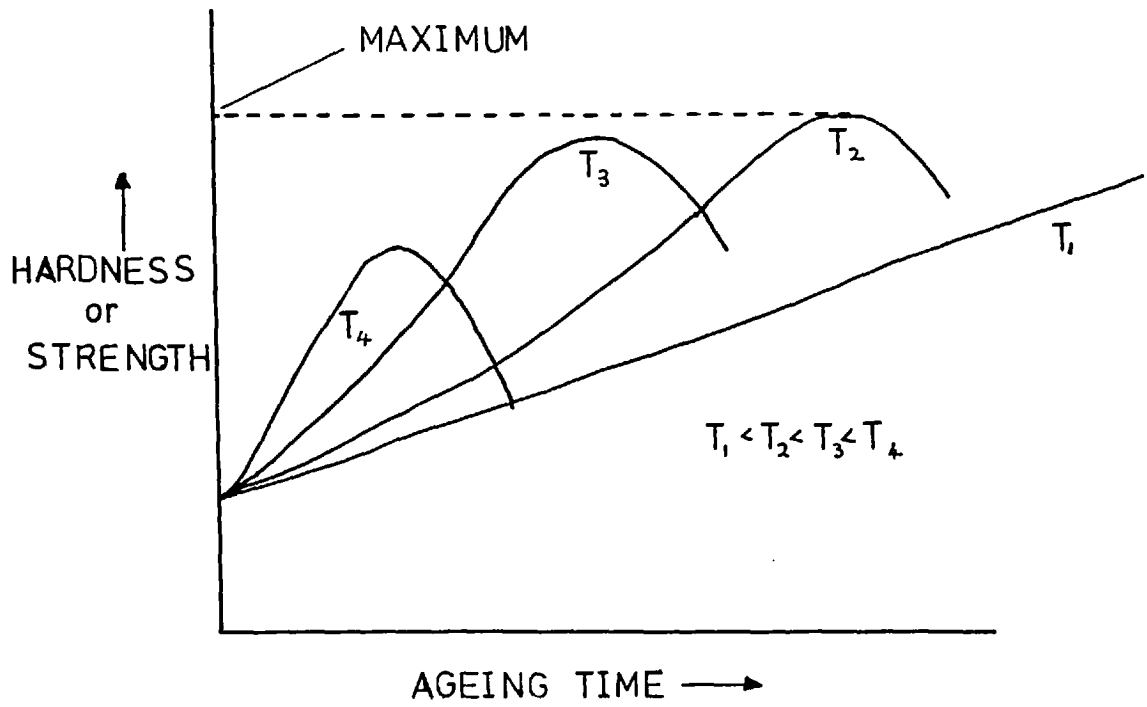
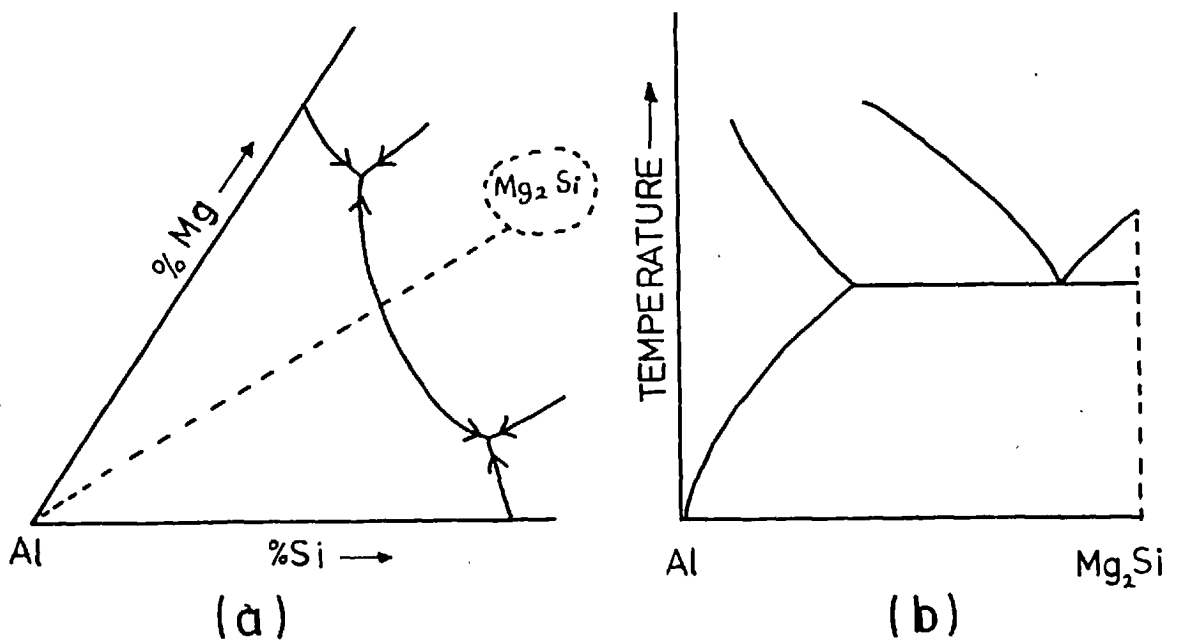


FIG. 6 Effect of temperature on age hardening curve

FIG. 7 (a) Al-Mg-Si phase diagram  
(b) Al-Mg<sub>2</sub>Si pseudo "binary" phase diagram



particle growth. Lower temperatures produce a greater quantity of precipitate and finer particles due to the greater supersaturation. If the specimen is "overaged" the particles grow larger than the optimum size, the interparticle spacing increases and the mechanical properties deteriorate. It should be noted that if an age hardened alloy is employed at elevated temperatures overaging may occur during service causing a rapid deterioration in properties.

The precipitation process was first investigated in 1938 by Guinier and Preston. They used x-ray techniques on Al-Cu alloys and discovered that solute concentration occurred in the matrix prior to precipitation. These concentrations were named G.P. zones after their discoverers. The Al-Cu alloys were found to exhibit G.P. zones in the form of discs 1 or 2 atoms thick and up to 100A in diameter. The discs were found to grow preferentially along {100} planes. These discs caused coherency straining due to the difference in atomic diameter between aluminium and copper. In 1942 the formation of a fully coherent ( $\theta''$ ) metastable precipitate, after the G.P. zones, was discovered. Further research showed that the next step in the precipitation process involved the formation of a partially coherent, metastable precipitate ( $\theta'$ ) followed by the equilibrium, non-coherent precipitate ( $\theta$ ). The optimum mechanical properties were found to correspond to the presence of one of the intermediate precipitates.

The Al-Mg-Si system is equivalent to a binary system for compositions lying close to the Al-Mg<sub>2</sub>Si section.(Fig 7a and b.) Guinier and Lambot<sup>71</sup> investigated this system and showed that G.P. zones occurred in the form of  $\langle 100 \rangle$  needles. Thomas<sup>72</sup> also investigated this system by means of thin foil electron microscopy and discovered the full precipitation sequence to be:-

needles                      → rods      → Plates (Mg<sub>2</sub>Si)  
 (G.P. zones)                ( $\theta'$ )                (equilibrium  $\theta$ )

Jacob<sup>77</sup> agreed with this precipitation sequence but, in addition, suggested that the rods (hexagonal unit cell:  $a = 0.705\text{nm}$ ,  $c = 0.405\text{nm}$ ) were coherent until they grew to about  $1\mu\text{m}$  in length whence they began to lose coherency along their major axis. Pashley<sup>73</sup> showed that some alloys in the Al-Mg<sub>2</sub>Si system underwent room temperature ageing while awaiting (for several days) the "official" ageing treatment.

On a laboratory scale a three stage heat treatment process is, of course, acceptable but on an industrial scale it is expensive. In the large scale extrusion of cast Al-Mg-Si alloys the heat treatment schedule consists<sup>74</sup> of:-

- (1) Preheating the billet to a temperature just below the solid <sup>solubility line</sup> ~~line~~ so that the temperature rise during extrusion dissolves the Mg<sub>2</sub>Si.
- (2) Quenching the extrude on emergence from the die.
- (3) Ageing the extrude.

This is a much more economical process than subjecting the extrude to a traditional three stage heat treatment.

In conclusion, the precipitation hardening process in aluminium alloys has been thoroughly investigated and is fairly well understood.

### 2.11.1 Dispersion strengthening

Since sintered aluminium products, possessing high strength at elevated temperatures, were first produced e.g. by A.I.A.G. in 1948, the interest in dispersion strengthening has grown tremendously. In 1952 Lyle<sup>78</sup>

demonstrated the high tensile strength, at elevated temperatures, of extruded aluminium powder. He discovered that the strength increased with oxide content over the range examined (0-16 wt.% oxide). Towner<sup>79</sup> found that Al-Fe and Al-Cr ( up to 8%) powder extrudes also possessed excellent high temperature mechanical properties. He reasoned that this was due to three main factors:-

- (1) The very fine oxide dispersion caused by the initial particle size.
- (2) The insolubility of the oxide in the matrix, even at high temperatures.
- (3) The retardation of recrystallisation at elevated temperatures by the oxide dispersion.

Several models have been proposed to explain the dispersion strengthening mechanism. The dispersed particles may be classified according to their strength as below:-

- (1) undeformable particles
- (2) deformable particles

Orowan<sup>80</sup> considered undeformable particles and proposed that the yield strength was governed by the stress required to "bow out" dislocations between particles (Fig. 8). The dislocations eventually "short circuit" around the particles leaving behind Orowan loops. He presented the following equation for the flow stress:-

$$\tau = \tau_s + \frac{2T'}{b\lambda} \quad (7)$$

where  $T'$  = line tension in the dislocation  
 $\tau_s$  = yield strength of matrix

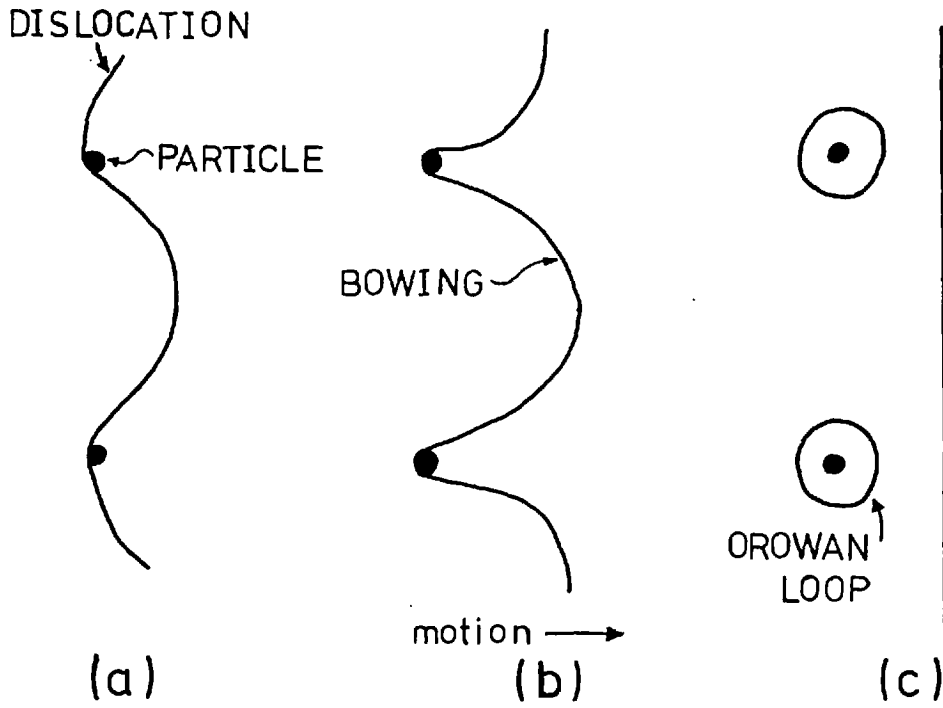


FIG. 8 Effect of second phase particles on dislocation motion

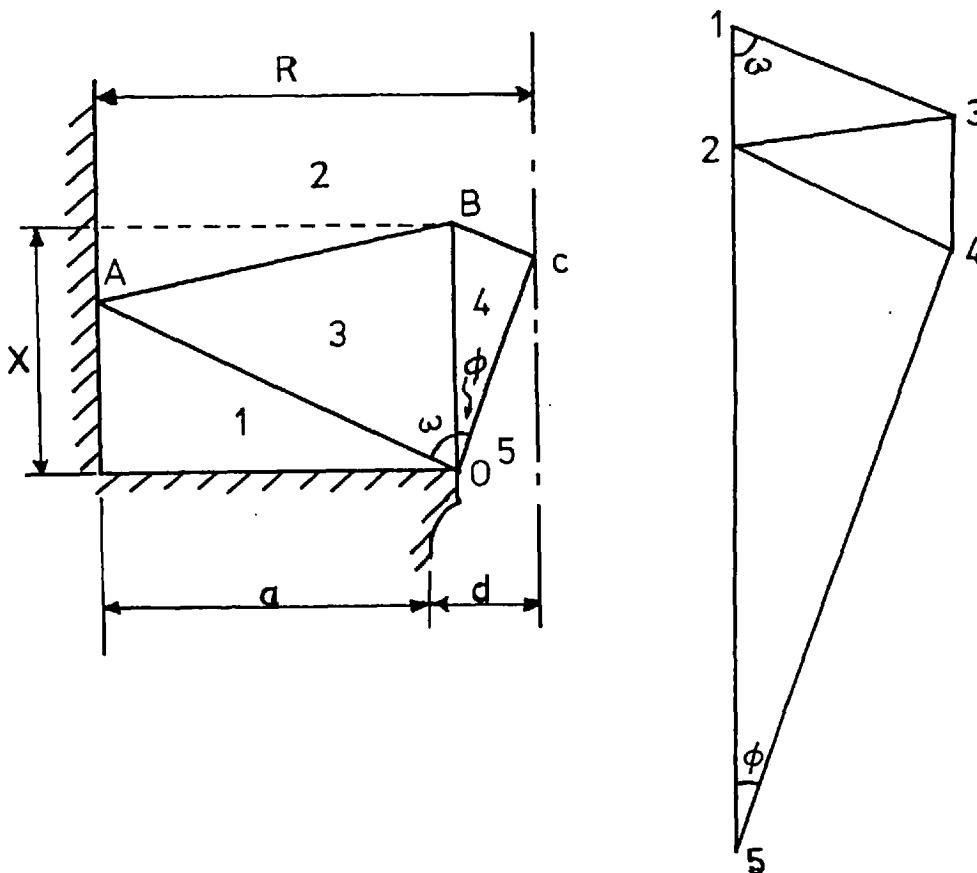


FIG. 9 Plane strain upper bound double triangle velocity field and hodograph

$b$  = Burgers vector of matrix

$\lambda$  = interparticle spacing

Kelly and Nicholson<sup>81</sup> produced the following equation for the line tension (when the dislocation forms a semi-circular loop):-

$$T' = \frac{Gb^2}{4} \theta^* \ln \frac{\lambda}{2b} \quad (8)$$

where  $\theta^*$  = averaging term (for screw and edge dislocations)

$G$  = shear modulus

Ansell and Lenel<sup>82</sup> studied deformable particles and proposed that yielding occurred when the shear stress due to dislocation pile ups at the particles was sufficient to plastically deform or fracture the particles. They suggested the following equation:-

$$\tau = \tau_s + \text{const.} \lambda^{-\frac{1}{2}} \quad (9)$$

Conclusive proof of particle shearing on yielding has yet to be found.

Fisher et al<sup>83</sup> found that dispersed particles enhanced the effect of normal work hardening. An important shortcoming of the two models discussed is that both assume non-coherent, spherical, uniformly distributed particles, an ideal situation.

Of direct relevance to the present investigation is the work carried out by Hansen<sup>84</sup> on the strengthening of aluminium by  $\text{Al}_2\text{O}_3$  particles. His fabrication procedure involved the extrusion of aluminium powder. He found that  $\text{Al}_2\text{O}_3$  particles behaved as a three-dimensional network and he proposed a flow stress equation similar to that of Ansell.

### 2.12.1 Extrusion Defects

#### Surface finish

Johnson and Mamalis<sup>11</sup> classified extrusion defects under three headings:-

- (1) cracking defects
- (2) contraction defects
- (3) skin-inclusion defects

Peripheral cracking is an example of the first type. Caused by hot or cold shortness accompanied by secondary tensile stresses generated in the region of the die, it leads to the so-called "fir-tree" defect. Central bursts or chevrons are also occasionally encountered. Piping at the back end of the billet is an example of a contraction defect. During the final stages of extrusion, when the billet length is very small, the centre sometimes "caves in". This produces a central core of material containing voids at the rear of the extrude. Consequently, in industry, the final length of extrude is discarded. A common example of a skin-inclusion defect involves foreign matter or lubricant on the side of the billet, being dragged between the flowing metal and the dead metal zone. This produces blisters on the extrude. The familiar 'extrusion defect' is a combined contraction-skin-inclusion defect. In the final stages of extrusion, after the billet centre has caved in, oxidised material from the surfaces of the billet flows laterally to occupy this vacant space. This produces a final length of extrude with a core of oxide and inclusions.

In the particular case of aluminium extrusion another problem is caused by the tendency of the aluminium to stick to the die land surface. This accumulation can lead to heavy scoring of the extrude surface. Williams<sup>75</sup> reported that this could be prevented by two methods:-

- (1) Starting and stopping the press which, unfortunately, leaves stop marks on the extrude.
- (2) Using a die with a conical lead-in.

It is difficult to imagine how the first method prevents scoring. One point worth bearing in mind is that although the surface finish of the extrude may appear satisfactory on emerging from the die it may not do so following subsequent heat treatment or anodizing.

An adequate surface finish is even more difficult to obtain in powder extrusion. Chare<sup>19</sup> found that a pad of cast material, placed in front of the aluminium powder billet, produced a fairly smooth extruded surface. El Wakil and El Sabbagh<sup>122</sup> extruded Al powder billets using graphite spray, fat free lubricant with limited success. Very little research has been carried out on verification of the theories put forward to explain extrusion defects. In industry the achievement of a good surface finish is an art rather than a science. Much work remains to be done on the correlation of extrusion conditions with extrude surface finish.

### 2.13.1 Fibre reinforcement of Metals

In a recent review<sup>86</sup> Morley discussed the present state of development of the science and technology of fibre reinforced metals. He suggested that metal matrix composites should be classified according to the type of fibre employed. The four main types of fibre are listed below:-

- (1) whisker crystals e.g.  $Al_2O_3$  or SiC
- (2) continuous ceramic or glass fibres  
e.g.  $SiO_2$
- (3) metal wire e.g. steel or W
- (4) fibres produced 'in situ' in lamellar eutectics e.g. chromium fibres in Cu-Cr



These four types of fibre give rise to the four types of composite discussed in the following sections.

### 2.13.2 Whisker Crystal Composites

Whiskers are defined as unbranched, needle-like, single crystals produced by the filamentary growth of a solid. Examples of typical whisker crystals employed in fibre reinforcement are  $\text{Al}_2\text{O}_3$ , Si, C, and SiN. When pure and undamaged, whiskers of such materials have high values of tensile strength, melting point and elastic modulus together with high strength to weight ratios<sup>87</sup>. Unfortunately the slightest damage greatly reduces their strength from the theoretical value of 0.1 elastic modulus, for a single crystal.

Interest in whiskers as reinforcing fibres grew when it was demonstrated that their use enabled metals to bear considerable loads at elevated temperatures (i.e. near the melting point of the metals). This was shown<sup>88</sup> using  $\text{Al}_2\text{O}_3$  reinforced silver. The percentage theoretical strength\* attained for a 16%  $\text{Al}_2\text{O}_3$  composite, varied from 90% at room temperature to 54% at 870°C. Whisker reinforced silver was also found to be much stronger than dispersion strengthened silver over a temperature range of 20-900°C. However, when the  $\text{Al}_2\text{O}_3$  whisker content was raised to 30% the elongation of the composite dropped to only 1.5% of the whisker fracture strain. The  $\text{Al}_2\text{O}_3$ -Ni system was also investigated for possible uses in the aerospace industry. It was found that 60-70% of the theoretical strength was attainable.

Whiskers were first produced using a batch process. In the case of  $\text{Al}_2\text{O}_3$  an oxidizing atmosphere was passed

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\* The theoretical strength calculated from the rule of mixtures equation:

$$\sigma_C = \sigma_m v_m + \sigma_w v_w$$

$\sigma_C$  = composite tensile strength

$\sigma_m, \sigma_w$  = tensile strengths of matrix and whiskers

$v_m, v_w$  = volume fraction of matrix and whiskers

over a boat of molten aluminium held in a furnace at 1500°C. The resulting whiskers were collected on the side of the boat. Unfortunately slight variations in flow rate, temperature etc. caused a considerable variation in whisker quality from batch to batch. A continuous process using a molybdenum-base collecting surface moving relative to a vapour source produced better results.

Several methods have been used to produce the composites. The first method involved the three stages mentioned below:

- (1) chemical cleaning of the whiskers.
- (2) the formation of bundles of whiskers.
- (3) vacuum infiltration of molten metal into the bundles.

Another method used a powder metallurgical route. The whiskers were mixed with a metal powder e.g. Ni and a wetting agent, dispersed in water and filtered. The filtrate was then mixed with a binder and more water and extruded to align the whiskers. Finally the extrude was dried, sintered and cold rolled.

Further development of whisker composites looks unlikely for several reasons. The production of whiskers and their composites is slow and expensive. The whiskers are very easily damaged which greatly reduces their strength. Even during the growth of the whiskers surface steps sometimes occur causing considerable weakening. Adequate wetting of the whiskers by the matrix is necessary to ensure good bonding. Interface degradation by matrix-whisker interaction<sup>89</sup> often reduces the composite strength e.g. delayed failure<sup>90</sup> in Al<sub>2</sub>O<sub>3</sub> reinforced composites above 630°C. Some doubt even remains about the effect of whisker size on strength. Larger whiskers are easier to handle but they tend to have poorer surfaces and consequently are weaker. Further laboratory investigation of this last effect would be useful.

### 2.13.3 Continuous Ceramic or Glass Fibre Composites

Most of the research carried out on glass fibre reinforcement has concerned the various silicate based glasses in various light metal matrices, the most common combination being "E" glass in aluminium. (Silicate glasses are viscous over a large temperature range, a necessity for successful hot drawing). An excellent review by Kelly and Davies<sup>87</sup> gives details of the production techniques and the results obtained by various investigators. The best method involved conventionally drawn glass fibres being coated with aluminium. Zinc or cadmium was added to improve the coating process. The composite was then formed by hot pressing or vacuum infiltrating bundles of coated fibres. The fracture surfaces of specimens showed no indication of fibre delamination, evidence of good bond strength. Composites containing 20-50% glass were considerably stronger, at temperatures above 300°C, than conventional aluminium alloys. However, the fibre strength, calculated from the measured composite strength, was much less than the theoretical values. This was probably caused by glass-metal chemical interaction.

The usefulness of silicate glasses as reinforcing materials is limited by two main factors.

- (i) The elastic modulus of these glasses is similar to that of aluminium. This dictates that no reinforcement is possible until the metal yields.
- (ii) At temperatures in excess of 300°C the strength of silicate glasses drops markedly.

Silica fibres, produced by a hot drawing process, have been widely used in investigations of ceramic fibre reinforcement. A review by Cratchley<sup>91</sup> discusses the production techniques and the results of several workers.

One successful method of production involved the passing of the fibres through a molten bead of the matrix metal, usually aluminium. This protected the fibre from handling damage and also served to form the matrix. The coated fibres were then hot pressed, at 400–550°C, to form the composite. The applied pressure was found to be very important in that it was required to produce bonds between adjacent fibre coatings while not causing damage to the fibres. An exothermic reaction between silica and aluminium at 643°C<sup>86</sup> was found to weaken the fibres considerably. However, below about 400°C no such weakening effect occurred. Cratchley and Baker<sup>85,92,93,94</sup> studied the mechanical properties of silica-aluminium composites in detail. They discovered that composites containing 50% silica were much stronger than RR58 alloy or a 10% S.A.P. alloy between room temperature and 400°C. The strengths of all three decreased dramatically above 300°C. (This would seem to contradict the findings, mentioned above, concerning the exothermic reaction between silica and aluminium at temperatures below 400°C). Cratchley and Baker found that the mode of failure depended on the fabrication pressure. Specimens fabricated at low pressures exhibited fibre "full out" on fracture whereas those produced at high pressures did not. The fatigue properties were found to be fairly good although the fibre ends did generate some cracks in the matrix. These cracks were unable to pass directly into undamaged fibres. They travelled parallel to the fibres, causing delamination, until reaching cracks in the fibres. This delamination process at the interfaces also produced high notch ductility values for these composites.

Two other materials are worth considering as possible fibre reinforcing materials. Carbon fibres have a high modulus of elasticity and are very strong, even at temperatures of the order of 2000°C. Once again aluminium is a good matrix material as carbon reacts unfavourably with titanium and cobalt at modest temperatures and with

Nickel<sup>96</sup> at temperatures of the order of 900°C. Baker<sup>97</sup> produced composites by hot vacuum pressing aligned fibre-aluminium sheets previously produced by electrodeposition and chemical vapour deposition techniques. Some carbon fibre-aluminium wires were also produced by molten infiltration. Jackson<sup>98</sup> et al found the strength of composites produced by the former method to be lower than expected although the composites were fairly stable at temperatures up to 600°C. The wires produced by molten infiltration were found to have a uniaxial tensile strength close to the theoretical value. The big drawback to the widespread use of carbon fibres in metal is the slow and costly method of fibre production from polymer textile precursor fibres<sup>95</sup>.

High strength boron fibres, produced by chemical vapour deposition onto a heated (1200°C) tungsten wire substrate from boron trichloride in the presence of hydrogen, have also been examined<sup>86</sup>. No really suitable matrix metal has been found due to the high reactivity of boron with most metals such as aluminium; the fibre surfaces becoming oxidised. To avoid this the fibres require coating with silicon carbide. Thus the production process becomes expensive and tedious.

#### 2.13.4 Metal Wire Composites

Metal wires are generally produced by drawing or extrusion. Generally, they are ductile and therefore do not experience weakening effects due to crack propagation caused by surface damage during handling operations. As most metal wires exhibit some plasticity at room temperature they usually fail by partial necking. At higher temperatures they lose strength

fairly rapidly due to enhanced dislocation movement and, in some cases, suffer additional strength losses due to surface alloying.

One of the first systems to be examined was tungsten wire reinforced copper. This served as a good model system because it was found that although the copper wet the tungsten, ensuring a good matrix-fibre bond, no reaction occurred. McDaniels<sup>99</sup> et al prepared composites by infiltrating bundles of tungsten wires (within ceramic tubes) with molten copper at 1200°C. They found that the rule of mixing predicted the composite tensile strength fairly well for both continuous and discontinuities wires. They calculated a critical aspect ratio of 16:1. Observation of the fracture surfaces of tensile specimens showed that fibre "pull out" did not occur, emphasising the strength of the copper-tungsten bond. Metallographic examination showed that continuous fibres fractured at weak points during loading and then behaved as discontinuous fibres. On further loading random breaks continued and finally localization of breaks in a particular region resulted in failure. Cratchley<sup>100</sup> and Baker<sup>101</sup> investigated aluminium reinforced with steel wires. They prepared composites by placing steel wires in a mould with aluminium powder and then pressing at an elevated temperature to achieve consolidation. Care was taken to ensure that the temperature was not sufficiently high to cause excessive reaction between the two metals. The tensile strength of the composite was found to be linearly dependent on the volume fraction of fibres. Cratchley<sup>100</sup> also showed that misalignment of fibres increased with decreasing aspect ratio producing a decrease in strength. Other examples

of systems investigated<sup>91</sup> include:

- 1) Titanium reinforced by molybdenum wires.
- 2) Nickel reinforced by tungsten wires.
- 3) Cobalt reinforced by tungsten wires.

In these three systems composites were produced by mixing pieces of wire with the matrix metal powders, cold pressing and/or sintering and then extruding and rolling to align the fibres and fabricate the final product.

Metal wire reinforced composites seem quite promising. The fabrication process is fairly simple and quick and the wires are much more robust than any glass or ceramic fibres. The only drawback is that the elastic modulus of such composites is much less than that attainable in some glass/ceramic-metal systems.

#### 2.13.5 Lamellar Eutectics

The advantages of composites produced by "in situ" growth of fibres are considerable. The problem of producing a uniform distribution of aligned fibres no longer exists. Fibres do not suffer damage during handling operations. Concern about matrix-fibre chemical interaction weakening the fibres is unnecessary and, finally, wetting problems governing the bond strength are avoided. The necessary condition for an eutectic alloy to be lamellar in nature is that the eutectic phases must have low entropies of melting. Unidirectional controlled solidification of a suitable alloy is used<sup>102</sup> to fabricate such composites. Several methods may be employed to achieve the controlled, unidirectional growth of a solid phase into a liquid phase, with a plane interface. The mould, containing the molten metal, may be withdrawn from a furnace held at a steady temperature or the mould may be left to cool slowly in a furnace with a temperature gradient. The most commonly used method involves the

the downward withdrawal of the molten alloy from a vertical furnace held at a steady temperature. The rate of solidification upwards depends on the rate of withdrawal and on the heat flow conditions. The inter-fibre distance,  $\lambda$ , is related to the freezing rate,  $R$ , by an equation of the form:-

$$\lambda = \text{const. } R^{-\frac{1}{2}} \quad (10)$$

The systems on which most work has been carried out are<sup>103</sup>:-

- 1) Chromium fibres grown from the copper-chromium eutectic (2 wt.% fibres)
- 2)  $\text{Al}_3\text{Ni}$  fibres grown from the  $\text{Al}-\text{Al}_3\text{Ni}$  eutectic (10 wt.%).

To increase the volume fraction of fibres more complex<sup>103</sup> systems must be used. A minimum surface energy criterion predicts lamellae formation instead of fibre formation for volume fractions in excess of  $\sim 32$  wt.%. Thompson and Lemkey<sup>103</sup> suggested that the relative advantages and disadvantages did not leave much to choose from between lamellae and fibres. They showed that the interfibre spacing had an important effect on composite tensile strength and proposed a relationship of the form:-

$$\sigma = \sigma^* + \text{const. } \lambda^{-\frac{1}{2}} \quad (11)$$

$\sigma^*$  is a frictional stress.

Some directionally solidified, complex eutectic superalloys have been produced with unidirectional properties superior to those of conventional nickel and cobalt superalloys. A eutectic consisting of  $\text{Ni}_3\text{Al}$  fibres in a  $\text{Ni}_3\text{-Ta}$  matrix was found<sup>86</sup> to be stronger, above  $1010^\circ\text{C}$ ,



than two cast nickel-based superalloys (MAR-M2005D and TRW-NAS AVIA). A lamellar  $\text{Ni}_3\text{Al-Ni}_3\text{Nb}$  composite exhibited greater strength than either of these conventional alloys, its superiority increasing with temperature.

In common with most other systems, the fracture toughness of these composites increased with interfibre spacing. Good fatigue properties were exhibited with endurance limits of  $\sim 60\%$  of the tensile strengths.

Potentially, the largest scope for these eutectic composites is in the gas turbine industry. However, in this field resistance to oxidation and corrosion is important and protective, diffusion coatings are required to provide adequate protection. Advanced air-cooling technology which allows conventional alloys to be employed at ever higher operating temperatures probably also prevents a breakthrough in the use of "in situ" composites.

## CHAPTER 3

## THEORY

3.1.1 Introduction

In the following chapter the various analyses used in the present work are presented in detail. The equations predicting extrusion load, flow stress, temperature and strain rate are derived together with the various relationships describing the behaviour of fibre reinforced composites under load. A general hot working theory is also presented. This enables flow stress to be correlated with both strain rate and temperature in a single equation which holds for widely varying conditions of temperature and strain rate.

3.2.1 Plane Strain Upper Bound Solution - Prediction of P/K

The formulae most widely used to predict extrusion loads are those developed by Johnson<sup>(25)</sup> and are of the form:-

$$\frac{P}{\sigma} = a + b \ln R \quad (12)$$

It has been shown that this form of equation may also be applied to powder extrusion but the magnitude of the flow stress,  $\sigma$ , is difficult to assess and the constants  $a$  and  $b$  are dissimilar to those observed for as-cast billet extrusion. Extrusion pressure is assumed to depend only on reduction ratio and not on flow pattern or die shape.

An alternative approach based on the flow of metal through the die is possible using load bounding or slip-line field techniques which are only strictly

applicable when plane strain conditions exist. Of these two, the upper-bound approach is more flexible because there is not the requirement of stress equilibrium. The major consideration involves strain increments and the conditions they have to fulfil in a fully plastic body<sup>125</sup>. The solution must commence with a proposed deformation field obtained experimentally by the examination of partial extrusions, or by consideration of a slip-line field solution. The problem then becomes one of finding a rigid triangle hodograph which satisfies both the velocity boundary conditions and the condition for incompressibility of the material. Thus the load produced by an upper bound solution is always higher than the experimental value and hence the load expression must be minimized by some mathematical technique.

Figure 9 shows a simple deformation field defined by the angles  $\omega$  and  $\phi$  and by the deformation zone depth  $x$ . The hodograph which is obliged to satisfy the deformation conditions is also shown. Let us assume that the ram velocity is unity. The material to the rear of ABC has been compacted to 100% density but has undergone no shear deformation and therefore has effectively zero shear strength. On encountering the velocity discontinuity AB which, from the hodograph, is of magnitude  $\overline{23}$ , the powder material undergoes a substantial shear deformation and is forced to flow parallel to AO with velocity  $\overline{13}$ . Thus all metal in triangle ABO is assumed to attain this velocity and direction. This shear deformation is theoretically assumed to occur instantaneously over zero distance which clearly is impossible in practice. The material next encounters discontinuity BO, of magnitude  $\overline{34}$ , giving it a velocity in direction  $\overline{14}$  and finally velocity discontinuity OC ensures that metal leaves the die such that  $\overline{15}$  is equal in magnitude to the plane-strain extrusion ratio.

The rate of energy dissipation at each discontinuity is given by the product of the area of the discontinuity, the magnitude of the discontinuity and the shear yield strength of the metal. Thus for linear discontinuities we may write:-

$$\frac{dw}{dt} = \sum k \cdot v \cdot s \quad (13)$$

The plane strain solution may then be written (considering unit depth):-

$$\text{pressure} \times \text{area} \times \text{velocity} = \sum k \cdot v \cdot s$$

$$p \times (a + d) \cdot (1) \times 1 = \sum k \cdot v \cdot s$$

$$p(a + d) = [ k_{AB} \overline{AB} \overline{23} + k_{BC} \overline{BC} \overline{24} + k_{BO} \overline{BO} \overline{34} + k_{OC} \overline{OC} \overline{45} + k_{AO} \overline{AO} \overline{13} ] \quad (14)$$

The lengths and velocities may be defined in terms of  $\omega$ ,  $x$  and  $\phi$ :-

$$AO = \frac{a}{\sin \omega}, \quad \overline{13} = \frac{a}{x \sin \omega}$$

$$OC = \frac{d}{\sin \phi}, \quad \overline{45} = \frac{a}{x \sin \phi}$$

$$BO = x, \quad \overline{34} = [1 + \frac{a}{d} - \frac{a}{x} \cot \omega - \frac{a}{x} \cot \phi]$$

$$AB = \left[ \frac{a^2}{\sin^2 \omega} + x^2 - 2ax \cot \omega \right]^{\frac{1}{2}},$$

$$\overline{23} = \frac{1}{x} \left[ \frac{a^2}{\sin^2 \omega} + x^2 - 2ax \cot \omega \right]^{\frac{1}{2}}$$

$$BC = \left[ \frac{d^2}{\sin^2 \phi} + x^2 - 2dx \cot \phi \right]^{\frac{1}{2}},$$

$$\overline{24} = \frac{a}{xd} \left[ \frac{d^2}{\sin^2 \phi} + x^2 - 2dx \cot \phi \right]^{\frac{1}{2}}$$

Differentiation of equation (14) is complicated, for powder materials, by the inclusion of shear stress values varying at each discontinuity. It may be assumed, however, that the values of  $\omega$ ,  $\phi$  and  $x$  giving minimum pressure for constant shear stress conditions will also yield the minimum pressure for powder compact extrusion. Thus equation (14) may be written:

$$\text{LOAD} = AK \left[ 2x + \frac{2ax}{d} + \frac{2a^2}{x \sin^2 \omega} + \frac{2ad}{x \sin^2 \phi} - 3a \cot \omega - 3a \cot \phi \right] \quad (15)$$

Differentiation with respect to  $x$  and equating to zero gives:-

$$x^2 = \frac{a^2}{(1 + a/d) \sin^2 \omega} + \frac{ad}{(1 + a/d) \sin^2 \phi}$$

Differentiation with respect to  $\omega$  yields:-

$$\cot \omega = 3x/4a$$

and with respect to  $\phi$  yields:-

$$\cot \phi = 3x/4d$$

As  $\omega$  and  $\phi$  are not independent of  $x$  the function must be minimised by iteration. The program given in appendix 1 does this. By assuming a single, mean, shear flow stress in equation (14) values of  $P/K$  can thus be obtained by means of this program.

### 3.2.2 Use of Plane Strain Upper Bound Solutions for Axisymmetric Extrusion

Analyses such as that presented in the previous section may be applied to axisymmetric extrusion (as an approximation) because Thomsen<sup>33</sup> has shown that:

a) the material flow is similar for identical geometrical configurations

and

b) the magnitude of the velocity during axisymmetric extrusion is approximately the square of the velocity during plane strain extrusion.

There is much experimental evidence to support this second conclusion and it quite clearly satisfies the velocity boundary conditions.

Therefore, for axisymmetric extrusion the surface area of the discontinuities must be considered, and the magnitude of the velocities must be squared. Hence equation (14) may be rewritten as:-

$$p \times \pi(a+d)^2 \times 1 = \left[ k_{AB} A_{AB} (\overline{23})^2 + k_{BC} A_{BC} (\overline{24})^2 + k_{BO} A_{BO} (\overline{34})^2 + k_{OC} A_{OC} (\overline{45})^2 + k_{AO} A_{AO} (\overline{13})^2 \right] \quad (16)$$

The velocities  $\overline{13}$ ,  $\overline{23}$  etc. are identical to the plane-strain analysis and the surface areas by simple geometry become:-

$$A_{OC} = \frac{\pi d^2}{\sin \phi}, \quad A_{AO} = \frac{\pi a(a+2d)}{\sin \omega}, \quad B_O = 2\pi x d,$$

$$A_{AB} = \pi(a+2d) \left[ \frac{a^2}{\sin^2 \omega} + x^2 - 2dx \cot \omega \right]^{\frac{1}{2}},$$

$$A_{BC} = \pi d \left[ \frac{d^2}{\sin^2 \phi} + x^2 - 2dx \cot \phi \right]^{\frac{1}{2}}$$

Since metal flow patterns for plane-strain and axisymmetric extrusions of similar geometry i.e. cross-section are similar, the minimized values of the geometric variables will also be identical. Hence the values of  $x$ ,  $\phi$  and  $\omega$  obtained for a plane strain extrusion ratio of  $R$  may be

used in conjunction with an axisymmetric extrusion ratio of  $R^2$ .

### 3.2.3 Prediction of Load Using Plane Strain Upper Bound Solution

Equations (14) and (16) may be used to predict the extrusion pressure if values for the shear flow stress at each discontinuity are supplied. The results of tensile tests, performed during this investigation indicated a stress/strain relationship for magnesium silicide powder compacts of the form:-

$$\sigma = \sigma_0 \dot{\epsilon}^m \quad (\text{see 5.9.3})$$

where  $\sigma_0$  is a strain rate sensitive constant. A combination of tensile and torsion tests suggested a relationship between <sup>STRESS</sup> $\sigma_0$  and strain rate given below:-

$$\sigma_0 = A \dot{\epsilon}^n \quad (\text{see 5.5.1})$$

Using the Von Mises yield criterion, the shear yield stress at a discontinuity may be written:-

$$K_{ij} = \frac{A}{\sqrt{3}} \dot{\epsilon}^n \dot{\epsilon}^m \quad (17)$$

As the strain rate at a discontinuity is  $\infty$ , an average  $\dot{\epsilon}$  may be found from Feltham's equation (see 3.5.1.). Thus to calculate the shear flow stress at each discontinuity the strain at each discontinuity must be estimated. The relative shear deformation at each 'jump' is given by the magnitude of the velocity 'jump' divided by the velocity perpendicular to that 'jump'. The material is also elongated in the direction of its deflected flow and in the three dimensional case there will be deformations of small magnitude perpendicular to this. Figure (10) depicts the relevant parameters. For example the shear

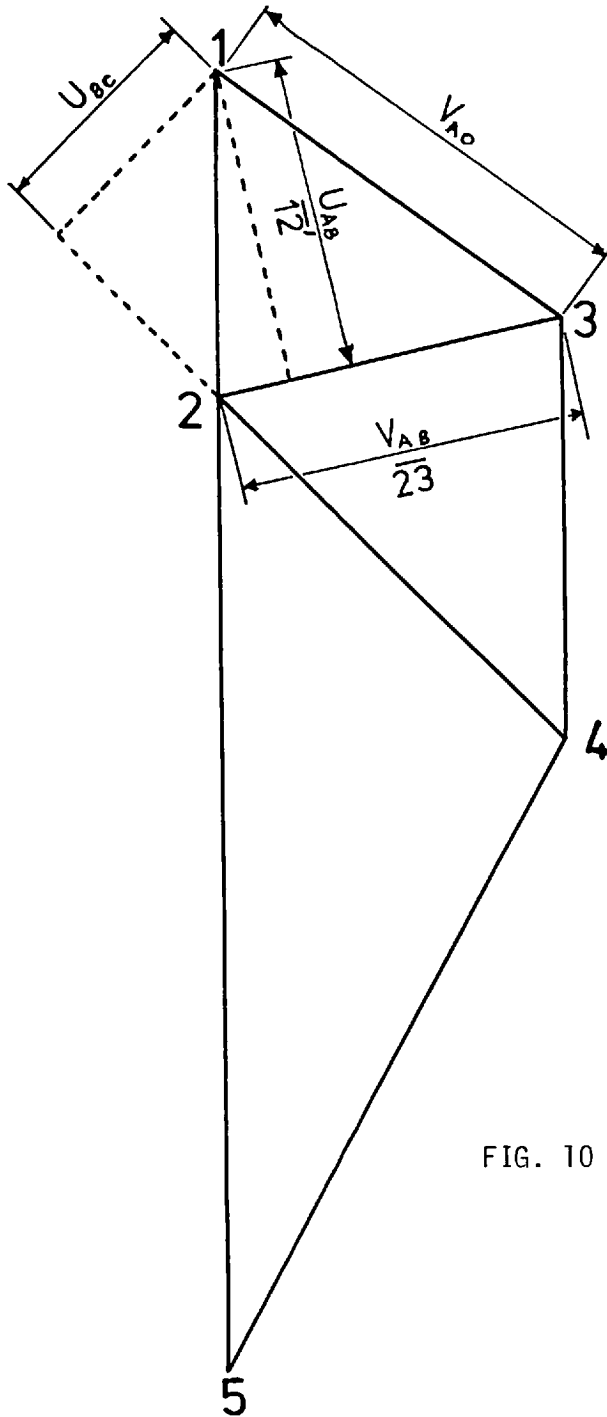


FIG. 10 Extrusion hodograph showing incident velocity components



deformation at AB is of magnitude:

$$\ln (v_{AB}/U_{AB})^2 = \ln (\overline{23}/\overline{12'})^2 = \gamma_{AB}.$$

The deformation in the flow direction =  $\ln (\overline{13})^2 = \epsilon_{AB}$ .

The strains in directions perpendicular to these are an order of magnitude less so that the total equivalent strain may be written:-

$$\overline{\epsilon}_{AB} = \sqrt{\frac{2}{3}} \left( |\epsilon_{AB}|^2 + \frac{1}{2} |\gamma_{AB}|^2 \right)^{\frac{1}{2}} \text{ etc.}$$

It should be noted that the use of the velocity squared relationship is also necessary to determine the shear yield stress to be used in the plane strain analogue; otherwise the total strain will not correlate with that actually obtaining in the extrusion process. In fact as shear deformations do not occur instantaneously i.e. a gradual deformation actually occurs, we must assume that the material properties at each discontinuity are related to those on either side of the discontinuity. This value may best be estimated by taking the mean equivalent strain such that the deformation at each discontinuity is given by:-

$$\frac{1}{2} \overline{\epsilon}_{AB} \quad \text{at} \quad AB$$

$$\frac{1}{2} \overline{\epsilon}_{BC} \quad \text{at} \quad BC$$

$$\overline{\epsilon}_{AB} \quad \text{at} \quad AO$$

$$\overline{\epsilon}_{AB} + \frac{1}{2} \overline{\epsilon}_{BO} \quad \text{at} \quad BO$$

$$\overline{\epsilon}_{AB} + \overline{\epsilon}_{BO} + \frac{1}{2} \overline{\epsilon}_{OC} \quad \text{at} \quad OC$$

These strains can then be substituted into equation (17) and an estimate of the shear flow stress found at each discontinuity. Hence the extrusion load may be predicted.

### 3.3.1 Axisymmetric Upper-Bound Solution

A kinematically admissible velocity field for axisymmetric extrusion, satisfying incompressibility, continuity and velocity boundary conditions was produced graphically by Halling and Mitchell<sup>36</sup>. They assumed a mode of deformation and determined the velocity components needed to produce the required hodograph by considering stream-line geometry. The solution they produced was for axisymmetric extrusion through conical dies. This can be applied to unlubricated extrusion through square dies, as in this case the dead metal zone/deformation zone boundary, effectively forms a conical lead in. A simple deformation field and hodograph for a single triangle arrangement of velocity discontinuities is shown in Figure 11. Assuming that all metal flow within region 2 is parallel to AB i.e. the dead metal zone boundary, then points on radial lines through  $O_2$  have the same velocity vector. The rate of energy dissipation may be expressed:-

$$\dot{E} = \sum_{i,j=1}^n \left( \iiint_V \bar{\sigma} \bar{\epsilon}_i dV + \iint_S \tau_{ij} V_{ij} dS \right) \quad (18)$$

(n = number of regions into which the problem is divided)

This expression requires minimizing to give the best solution. The various terms in (18) must now be calculated. The rate of energy input on the left hand side can be expressed:-

$$\dot{E} = P \times V \times A_B \quad (19)$$

where P = extrusion pressure, V = ram velocity and  $A_B$  = cross-sectional area of ram

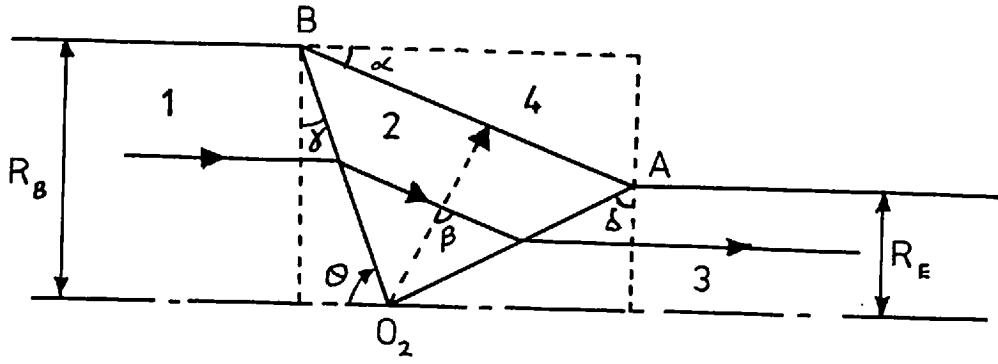
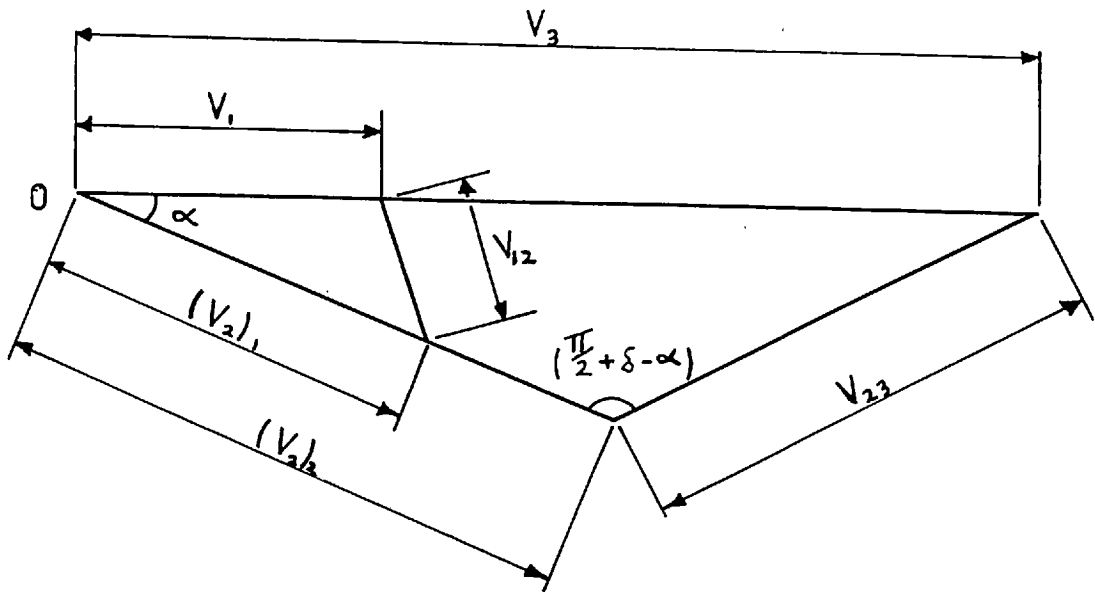


FIG. 11 Axisymmetric upper bound velocity field and hodograph



The first term on the right hand side represents the rate of energy dissipation within region 2. Alexander<sup>37</sup> demonstrated that the effective strain rate may be written:-

$$\bar{\dot{\epsilon}}_1 = \sqrt{\left[ \frac{2}{3} (\dot{\epsilon}_{\theta\theta}^2 + \dot{\epsilon}_{\phi\phi}^2 + \frac{1}{2} \dot{\epsilon}_{r\theta}^2) \right]} \quad (20)$$

where

$$\left. \begin{aligned} \dot{\epsilon}_{\theta\theta} &= \frac{1}{r} \left[ v \cos \beta \left( 1 + \frac{\partial \beta}{\partial \theta} \right) + \sin \beta \frac{\partial v}{\partial \theta} \right] \\ \dot{\epsilon}_{\phi\phi} &= \frac{v}{r} \left[ \cos \beta + \cot \theta \sin \beta \right] \\ \dot{\epsilon}_{r\theta} &= \frac{1}{r} \left[ -v \sin \beta \left( 1 + \frac{\partial \beta}{\partial \theta} \right) + \cos \beta \frac{\partial v}{\partial \theta} \right] \end{aligned} \right\} \quad (21)$$

where  $\beta = \theta - \alpha$ ,  $v =$  velocity in region 2

W.B.  $\partial v / \partial r = \partial \beta / \partial r = 0$

In this case, the distance from  $O_2$  to any point on AB may be expressed in terms of  $\theta$  thus:-

$$r = \frac{R_B \cos(\alpha + \gamma)}{\cos \gamma \sin(\theta - \alpha)} \quad (22)$$

Using a mass balance  $v$  is given by:-

$$v = \frac{(v_2)_1 R_B}{r \sin \theta} \quad (23)$$

Using (20) and (21) we may write:-

$$\bar{\dot{\epsilon}} = \sqrt{\left( \frac{2}{3} \right)} \frac{1}{r} \epsilon_i(\theta) \quad (24)$$

where  $\epsilon_i(\theta)$  is a function of  $\theta$  only. The first term on the right hand side of (18) thus becomes:-

$$\dot{E}_2 = \pi \bar{\sigma}_2 \sqrt{\left( \frac{2}{3} \right)} \int_{\theta_1}^{\theta_2} \left[ r^2 \right]_{r_1(\theta)}^{r_2(\theta)} \epsilon_i(\theta) \sin \theta d\theta \quad (25)$$

The limits  $r_1(\theta)$ ,  $r_2(\theta)$ ,  $\theta_1$  and  $\theta_2$  define the boundaries of region 2, the deformation zone.

Equation (25) may be evaluated by use of Simpson's rule. In the present work the range was divided into twelve steps.

The second term on the right hand side of (18) must now be evaluated. The material deforming in region 2 is assumed to obey the Von Mises yield criterion. Therefore at all the discontinuities within the material  $K_{ij} = \bar{\sigma}_{\text{yield}}/\sqrt{3}$ . This also applies along boundary AB as the dead metal zone, region 4, also consists of the extruding material. All the velocity discontinuities are either constants or functions of  $r$ . Therefore

$$\dot{E}_{ij} = \int \int_s \tau_{ij} v_{ij} ds \quad (26)$$

can be evaluated exactly.

The velocity discontinuities are given below:-

$$\begin{aligned} (v_2)_1 &= \frac{v_1 \cos \gamma}{\cos(\gamma + \alpha)} \\ (v_2)_2 &= \frac{v_1 R_B^2 \cos \delta}{R_E^2 \cos(\alpha - \delta)} \\ \overline{v_{12}} &= \frac{v_1 \sin \alpha}{\cos(\gamma + \alpha)} \\ \overline{v_{23}} &= \frac{v_1 R_B^2 \sin \alpha}{R_E^2 \cos(\alpha - \delta)} \\ \overline{v_{24}} &= \frac{v_1 \cos \gamma R_B}{r \sin \theta \cos(\gamma + \alpha)} \end{aligned} \quad (27)$$

Substituting (27) into (26) we obtain:-

$$\begin{aligned} \dot{E}_{12} &= \frac{\pi R_B^2 \sigma_{12} v_1 \sin \alpha}{\sqrt{3} \cos \gamma \cos(\gamma + \alpha)} \\ \dot{E}_{23} &= \frac{\pi R_B^2 \sigma_{23} v_1 \sin \alpha}{\sqrt{3} \cos \delta \cos(\alpha - \delta)} \\ \dot{E}_{24} &= \frac{2\pi \sigma_{24}}{\sqrt{3}} \int_{\theta_1}^{\theta_2} \frac{v_1 \cos \gamma R_B r d\theta}{\cos(\gamma + \alpha) \cos \alpha} \end{aligned} \quad (28)$$

If we assume that the values of  $\alpha$  and  $\delta$  giving the minimum value of  $P/K$  for a rigid perfectly plastic material also give the minimum for a powder material we can write:-

$$\frac{\sigma_{24}}{\sqrt{3}} = \frac{\sigma_{23}}{\sqrt{3}} = \frac{\sigma_{12}}{\sqrt{3}} = \frac{\sigma_2}{\sqrt{3}} = K \quad (29)$$

Equation (18) can now be rewritten with  $P/K$  on the left hand side.

A computer program has been written to perform the numerical calculations and to minimize the  $P/K$  value on a CDC 6400 computer. The variables which are optimized are the angles  $\alpha$  and  $\delta$ . The only restrictions imposed are that  $\alpha$  and  $\delta$  cannot  $> 90^\circ$ . The optimization procedure used, a standard library routine, is an adaption of the quasi-Newton method of Davidson<sup>126</sup> and eliminates the requirement for derivations. The program is listed in appendix 2.

### 3.4.1 Prediction of Load Using Axisymmetric Upper Bound Solution

In the previous section it was shown that, for a rigid perfectly plastic material, continuous functions describing the deformations along a streamline could be written and the integral (18) evaluated exactly. However, for a material having a wide variation in rheology throughout the deforming zone the integral must be evaluated numerically. To do this the deformation zone must be divided into elements. In this investigation the region was divided into trapezoidal toroidal elements as shown in Figure 12. The strain and strain rate at each grid point must be found.

Elements at a discontinuity boundary such as  $[r_n, \theta]$  in Figure 12 are the most general case and evaluation of the energy dissipated at this element must include the internal energy dissipation, the work done at the velocity discontinuity  $\overline{12}$  and the work done at  $\overline{24}$ , due to sticking friction.

The plastic energy dissipation due to the velocity discontinuity  $O_2 B$  in element  $M$  may be written:-

$$\begin{aligned} \dot{E}_{12} &= k_{12} (\delta S)_{12} v_{\overline{12}} \\ &= 2\pi k_{12} (\delta r)_{n\xi} (r)_{n\xi} v_{\overline{12}} \end{aligned} \quad (29a)$$

In this expression  $k_{12}$  is a function of both strain and strain rate (as discussed in 3.2.3) and theoretically  $\dot{\epsilon} = \infty$  at a discontinuity. Hence the strain rate must be assumed to be that obtaining in the first element after the boundary in region 2.  $k_{12}$  may then be ascertained by calculating the strain at the discontinuity as shown below.





Energy is also dissipated at the dead metal zone boundary due to sticking friction. Hence:-

$$\begin{aligned} \dot{E}_{24} &= K_{r\theta} (\delta S)_{24} V_{24} \\ &= 4\pi r_{\theta} K_{r\theta} V_{24} R_B \frac{\sin \frac{d\theta}{2}}{\sin \beta \sin(\beta - \frac{d\theta}{2})} \end{aligned} \quad (29b)$$

A similar method may again be used to determine  $K_{r\theta}$ ; calculating  $\dot{\bar{\epsilon}}_{r\theta}$  at the boundary element  $(r, \theta)$  and evaluating as shown below.

The internal energy dissipation due to circumferential straining is written:-

$$\begin{aligned} \dot{E}_3 &= \sigma_{r\theta} \bar{\epsilon}_{r\theta} (dV)_{r\theta} \\ &= \frac{\sigma_{r\theta} \bar{\epsilon}_{r\theta} 4\pi r_{\theta}^2 (\delta r)_{\theta} \sin \frac{d\theta}{2}}{\sin(\beta - \frac{d\theta}{2})} \end{aligned} \quad (29c)$$

Therefore the total energy input is given by:

$$\sum (\dot{E}_{12} + \dot{E}_{24} + \dot{E}_3) \quad (29d)$$

(The  $\dot{E}_{12}$  and  $\dot{E}_{24}$  terms applying to the boundary regions only). The external energy input is given by:-

$$\dot{E} = p \times A_B \times V$$

and hence the forming pressure  $p$  may be determined.

Calculation of the flow stress

The flow stress is both strain and strain rate sensitive and hence the strain in each element must be considered. The mean equivalent strain at a velocity discontinuity may be calculated by finding the amount of plastic work done per unit volume at the discontinuity. Thus, for the element considered, the total plastic work done per unit time is given by

$$\dot{E}_{12} = 2\pi K_{12} (\delta r)_{n\xi} (r)_{n\xi} V_{12} \quad (29a)$$

The volume flow past this discontinuity is:

$$dV = 2\pi (\delta r)_{n\xi} (r)_{n\xi} \sin\xi V_1 \quad (29e)$$

$$(W_p)_{r,\xi} = \frac{K_{12} V_{12}}{V_1 \sin\xi} \quad (29f)$$

The plastic work done per unit volume may be written (see Fig. 13)

$$\int_{\varepsilon_1}^{\varepsilon_2} \bar{\sigma} d\varepsilon = \bar{\sigma}_m (\varepsilon_2 - \varepsilon_1) \quad (29g)$$

Hence across the increment of velocity discontinuity under consideration

$$\bar{\varepsilon}_2 - \bar{\varepsilon}_1 = \frac{V_{12}}{\sqrt{3} V_1 \sin\xi} \quad (29h)$$

and from (17) in (3.2.3)

$$K_{12} = \frac{A \bar{\varepsilon}^n}{\sqrt{3} (m+1)} \left[ \frac{\bar{\varepsilon}_2^{m+1} - \bar{\varepsilon}_1^{m+1}}{\bar{\varepsilon}_2 - \bar{\varepsilon}_1} \right] \quad (29i)$$

The material is then further strained as it traverses the element. The strain imparted may be calculated because

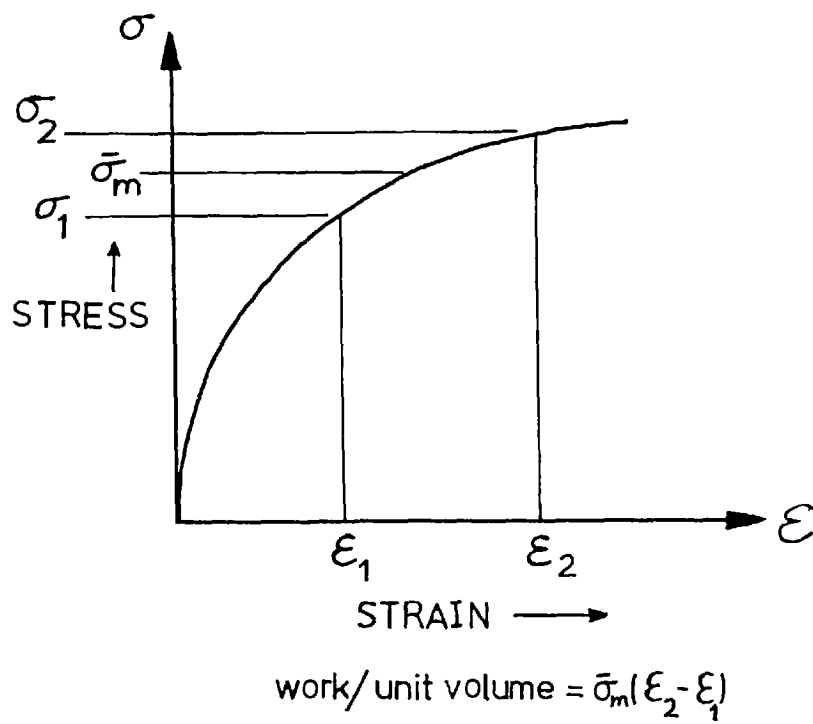


FIG. 13 Definition of mean flow stress

$$d\bar{\epsilon} = \bar{\epsilon} dt = \bar{\epsilon}_3 - \bar{\epsilon}_2 \quad (29j)$$

where  $\bar{\epsilon}$  is calculated using equation (20) in (3.3.1) and

$$dt = \frac{2 r_{n\theta} \sin(\frac{d\theta}{2})}{V_n \theta \sin(\beta - \frac{d\theta}{2})} \quad (29k)$$

thus,

$$\sigma_{r,\theta} = \frac{A \bar{\epsilon} n}{m+1} \left[ \frac{\bar{\epsilon}_3^{m+1} - \bar{\epsilon}_2^{m+1}}{\bar{\epsilon}_3 - \bar{\epsilon}_2} \right] \quad (29l)$$

The program in appendix (3) performs the numerical calculations summing the energies and strains and calculating the pressure. The variables optimized are angles  $\alpha$  and  $\delta$  and the flow stress equation constants  $A$ ,  $m$  and  $n$ . The optimization procedure adopted was written to solve the constrained problem to minimize a general objective function of many independent variables subject to bounds on the variables. A sequential penalty function technique developed by Lootsma<sup>127</sup> is utilised. A routine to calculate the value of the function, in this case the extrusion pressure, is included. The method involves iteration and therefore requires an initial estimate of the least possible value of extrusion pressure. The optimization routine solves the constrained problem by transforming the problem into a sequence of unconstrained problems whose solutions tend to the solution of the original problem.

#### 3.4.2 Calculation of mean flow stress during extrusion

The flow stress may be calculated using the upper bound solutions mentioned in the previous sections. The minimum pressure at the end of extrusion divided by the load constant  $c$  (which =  $p/\sigma_{yield}$ ) gives the mean flow

stress. In the present work the actual  $c$  values used were produced by the simple axisymmetric field discussed in section 3.3.1 and listed in appendix (2). The use of the minimum pressure in the calculation eliminates the need to consider frictional effects between the billet surface and the container wall.

### 3.5.1 Strain rate

It is quite obvious that material flowing along different streamlines through the deformation zone is subjected to different strain rates. Therefore it is very difficult to define a mean strain rate for a particular extrusion ratio and ram speed. Feltham<sup>38</sup> originally proposed the following definition for mean strain rate:

$$\bar{c} = \frac{\text{total strain}}{\text{time for strain to occur}} \quad (30)$$

The time in which the strain occurs may be calculated by assuming that the deformation occurs effectively in a cone of semi-angle  $\omega$  (Fig. 9) and base diameter  $D_B$ . The volume of this cone is given by:-

$$V = \frac{\pi (D_B^3 - D_E^3)}{24 \tan \omega}$$

Therefore the time

$$t = \frac{V}{\text{volume extrusion rate}}$$

$$\text{Now the volume extrusion rate} = \frac{\pi D_B^2 v}{4}$$

$$\therefore t = \frac{D_B^3 - D_E^3}{6D_B^2 \tan \omega v}$$

Thus the time mentioned in equation (30) is obtained.

Now the work done by the ram in moving a distance L

$$= p \times \pi \frac{D_B^2}{4} \times L.$$

∴ The work done per unit volume = p

The work per unit volume may also be expressed:-  
stress x strain.

Now  $p = \sigma_{\text{yield}} c$  (where  $c = \frac{D}{\sigma}$ , from theory).

∴ c may be equated to the mean strain

∴ equation (30) becomes:-

$$\dot{\epsilon} = \frac{6 c \tan \omega D_B^2 v}{(D_B^3 - D_E^3)} \quad (31)$$

Equation (31) permits the calculation of the strain rate during extrusion.

The axisymmetric upper bound solution, given in 3.4.1., calculates the strain rate at the centroid of each element in the deformation zone grid. Thus an average strain rate during extrusion may also be obtained this way. The values produced by this latter method are compared with those obtained from Feltham's equation (31) in the discussion in Chapter 5.

### 3.6.1 General hot working equation

A relationship between strain rate, flow stress and temperature during the hot deformation (i.e. creep testing) of metals was first proposed by Garofalo<sup>66</sup>. It was later applied to conventional hot working processes (i.e. extrusion) by Sellars and McG. Tegart<sup>67</sup>. The relationship proposed involves a sinh term and the governing equation is given below:-

$$\dot{\epsilon} = A_1 \left[ \sinh (\alpha \sigma) \right]^n \exp \left( -\frac{\Delta H}{RT} \right) \quad (32)$$

Here  $A_1$ ,  $\alpha$  and  $n$  are temperature and stress independent constants and  $\Delta H$  is the activation energy which is also assumed to be independent of temperature and stress. To make use of equation (32) and find  $\Delta H, \alpha$  must first be determined.

### 3.6.2 Determination of constants in hot working equation

At high stress levels the relationship between strain rate and stress may be written:-

$$\dot{\epsilon} = A_3 \exp(\beta \sigma) \quad (33)$$

where  $\beta$  is a temperature independent function. Also at high stresses ( $\alpha \sigma > 1.2$ ) and constant temperatures equation (32) can be rewritten:

$$\dot{\epsilon} = A_1 \sinh(\alpha \sigma)^n \quad (34)$$

On expanding the sinh term this approximates to:-

$$\dot{\epsilon} = \frac{A_1}{2^n} \exp(n \alpha \sigma) \quad (35)$$

At low stress levels:-

$$\dot{\epsilon} = A_2 \sigma^{n_2} \quad \text{i.e. } (\alpha \sigma < 0.8) \quad (36)$$

where  $n_2$  is also assumed temperature independent.

Comparing (33) and (35) gives:-

$$\beta = n \alpha \quad (37)$$

Now  $n \approx n_2$

Therefore  $\alpha = \frac{\beta}{n_2}$ .  $\beta$  is found by plotting  $\ln \dot{\epsilon} v \sigma$   
 $n_2$  is found by plotting  $\ln \dot{\epsilon} v \ln \sigma$

hence  $\alpha$  is evaluated.

### 3.6.3 The activation energy

To find the activation energy we must reconsider equation (32). Taking natural logarithms we obtain:-

$$\ln \dot{\epsilon} = n \ln \left[ A_1 \sinh(\alpha\sigma) \right] - \left( \frac{\Delta H}{RT} \right) \quad (38)$$

Now at constant flow stress:-

$$- R \frac{\partial \ln \dot{\epsilon}}{\partial \frac{1}{T}} = \Delta H \quad (39)$$

Equation (39) may be expanded<sup>107</sup> to give:-

$$- R \left( \frac{\partial \ln \dot{\epsilon}}{\partial \ln \sinh(\alpha\sigma)} \right)_T \left( \frac{\partial \ln \sinh(\alpha\sigma)}{\partial \frac{1}{T}} \right)_{\dot{\epsilon}} = - \Delta H \quad (40)$$

Hence from plots of  $\ln \dot{\epsilon}$  vs  $\ln \sinh(\alpha\sigma)$  and  $\ln \sinh(\alpha\sigma)$  vs.  $\ln \frac{1}{T}$  the activation energy may be obtained.

### 3.7.1 Introduction to fibre-reinforcement theory

There are several conventional methods which may be used to strengthen metals. The metal can be work hardened to increase both the dislocation density and the strength. This method is useless if the metal is to be employed at elevated temperatures as recovery and/or recrystallisation will occur causing softening and loss of strength.

Other obstacles to dislocation motion such as solute atom atmospheres and second phase particles may be used to improve the strength. Such strengthening mechanisms can be quite effective, even at elevated temperatures.

Yet another way of increasing the strength of a metal is to introduce fibres of high ultimate tensile strength and, preferably, of high elastic modulus. Basically three



types of fibre are available (neglecting special eutectic alloys):-

- (1) Glassy type fibres which have a high U.T.S but unfortunately an elastic modulus similar to that of most light metals. Such fibres do not deform plastically. This is the type used in the present series of investigations.
- (2) Ceramic materials such as alumina, graphite, boron, silicon nitride and silicon carbide all of which can be prepared as long, thin fibres with diameters of about  $10^{-4}$  to  $10^{-3}$  cms. These materials have smooth surfaces free from deep notches and steps. They are light, have high melting points and high elastic moduli.
- (3) Metallic wires such as steel or nickel wires. These have higher values of U.T.S. and elastic modulus than most matrix metals such as aluminium.

Materials in groups (1) and (2) are inherently strong with those in group (2) having high Pierls-Nabarro forces presenting great difficulties to the motion of dislocations. However, although these materials have high "shear" strength they are brittle and have low resistance to crack initiation and propagation. Thus the theoretical high strength of materials in groups (1) and (2) can only be made use of if they are produced relatively defect free. Their surfaces must also be protected against damage which might lead to crack initiation, propagation and "premature" failure. This protection can be provided by embedding these fibres in a ductile metal matrix of relatively low flow stress. Plastic flow of the matrix occurs early in the loading cycle and the load is transferred to the fibres. The product is called a fibre reinforced composite. This matrix must serve two other important purposes:-

- (1) It must prevent direct fibre-fibre contact. If the fibres contact one another a crack in one fibre may spread catastrophically to another fibre and so on. However, the ductile matrix can terminate crack propagation by "blunting" the crack tip thus giving the overall composite quite a high fracture toughness.
- (2) It must prevent the application of the load from causing any damage to the fibres which might lead to crack initiation.

### 3.7.2 Stress/strain behaviour of continuous fibre composites

In the following sections on fibre reinforcement two assumptions are made concerning composite structure and behaviour. They are:-

- (1) The fibres are uniaxially aligned.
- (2) The matrix-fibre bond is sufficiently strong, to ensure efficient load transference.

During loading, the Al-B<sub>2</sub>O<sub>3</sub> system under investigation exhibits three distinct modes of deformation in succession:-

- (1) Both fibres and matrix deform elastically
- (2) The fibres deform elastically and the matrix plastically. The transition from (1) and (2) occurs at strains slightly greater than the yield strain of the bulk, unreinforced matrix metal.
- (3) Fracture of the fibres followed by composite failure. It should be noted that composite failure occurs immediately upon fibre fracture only when the fibre volume fraction exceeds a minimum value. This minimum volume fraction will be discussed later in section 3.7.3.

The three modes of composite deformation are discussed below.

(a) Elastically deforming matrix and fibres

The elastic moduli of aluminium and  $B_2O_3$  glass are similar. Thus when a load is applied to the composite transference occurs, due to the interfacial bond, and the stresses and strains in the metal and fibres are equal. As the load increases the strain in the matrix and fibres continues to increase linearly until the yield strain of the matrix is reached (Fig. 14).

If the elastic modulus of the fibre is greater than that of the matrix i.e. Fig. 15, it has been shown experimentally that:-

$$E_{\text{composite}} = E_{\text{fibre}} V_{\text{fibre}} + E_{\text{matrix}} V_{\text{matrix}} \quad (41)$$

Hill<sup>130</sup> has shown that (41) is the theoretical lower bound to the modulus when the Poisson's ratios of the components are equal. Thus  $E_{\text{composite}}$  will never be less than the value given by (41) no matter what the poisson's ratios of the components are.

(b) Elastically deforming fibres - plastically deforming matrix

During this stage in the deformation the strain in the matrix is greater than the strain in the fibre for a given stress and so a shear force exists at the interface and contributes to load transference.

The stress/strain behaviour of the composite may be described as quasi-elastic. If at any stage the load is removed the specimen returns almost to its original length. At first this recovery is fully elastic but the final stage of recovery sees the matrix being deformed plastically in compression. When the load has been completely removed the specimen retains a small

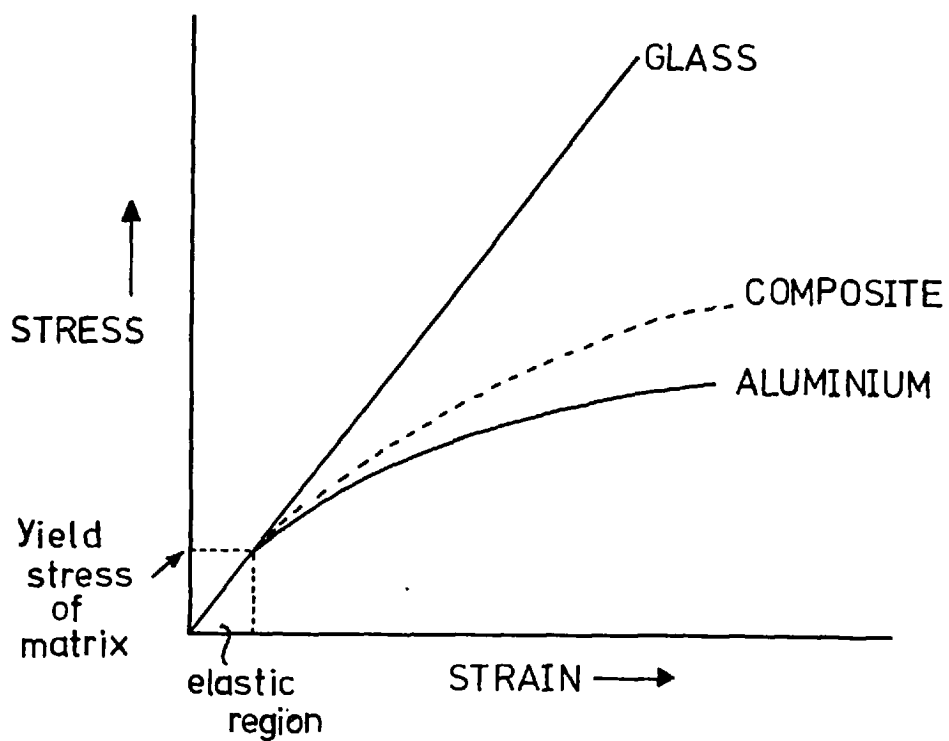
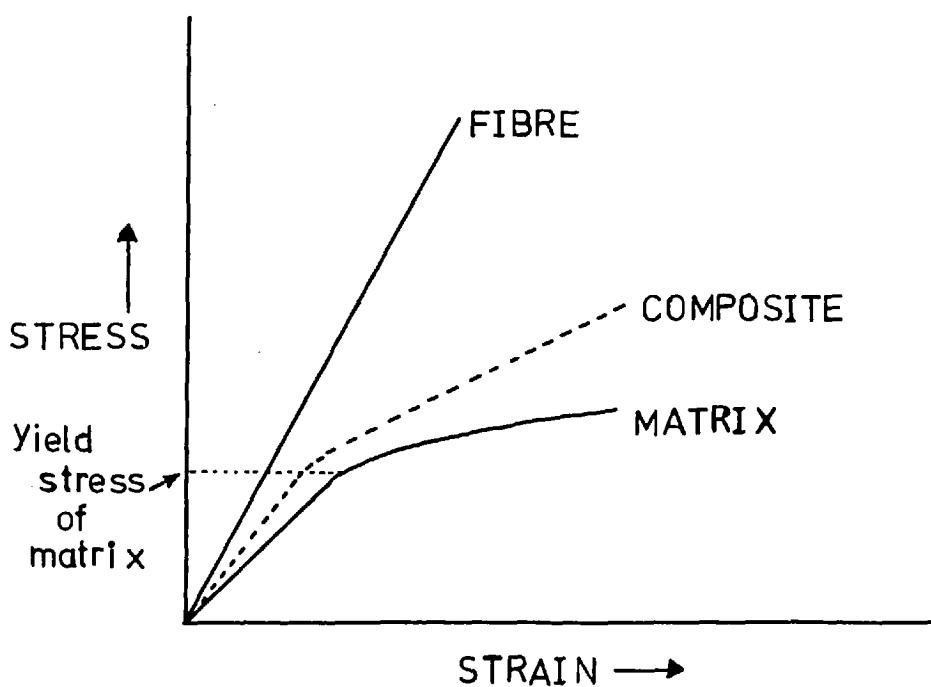


FIG. 14 Stress-strain behaviour of Al-glass composite

FIG. 15 Stress strain behaviour of composite in which  $E_F > E_M$



permanent set (Fig. 16) with the fibres in tension and the matrix under compression. Fig. (16) also shows the result of repeating<sup>85</sup> the stress cycle. On loading again the initial elastic region is extended because the matrix was initially in ~~compression~~ and hence, on completing the cycle, the permanent set is removed.

This is the most important part of the stress/strain curve when the load bearing capacity of the fibres is being utilised. In this region:-

$$E_{\text{composite}} = E_{\text{fibre}} V_{\text{fibre}} + \left( \frac{d\sigma_m}{d\epsilon_m} \right) \epsilon V_m \quad (42)$$

where  $d\sigma_m/d\epsilon_m$  = slope of stress/strain curve of the matrix at a strain,  $\epsilon$ , to which the composite has been subjected. Effectively as:

$$\frac{d\sigma_m}{d\epsilon} \lesssim \frac{E_m}{100}, \quad E_C = E_F V_F \quad (43)$$

(c) Fracture of the composite occurs when the stress in the fibres reaches their U.T.S.

### 3.7.3 Strength of continuous fibre composites

The general expression for the strength of the composite may be written:-

$$\sigma_C = \sigma_F V_F + \sigma'_m (1 - V_F) \quad (44)$$

when  $V_F$  exceeds a value denoted by  $V_{\text{min}}$ . Failure of all the fibres results in immediate failure of the composite only if  $\sigma_C$ , given by (44), is greater than  $\sigma_u (1 - V_F)$ . ( $\sigma_u$  = U.T.S. of matrix). This defines

$$V_{\text{min}} = \frac{\sigma_u - \sigma'_m}{\sigma_F + \sigma_u - \sigma'_m} \quad (45)$$

Another important limiting value must be noted and considered. No. fibre reinforcement at all occurs unless

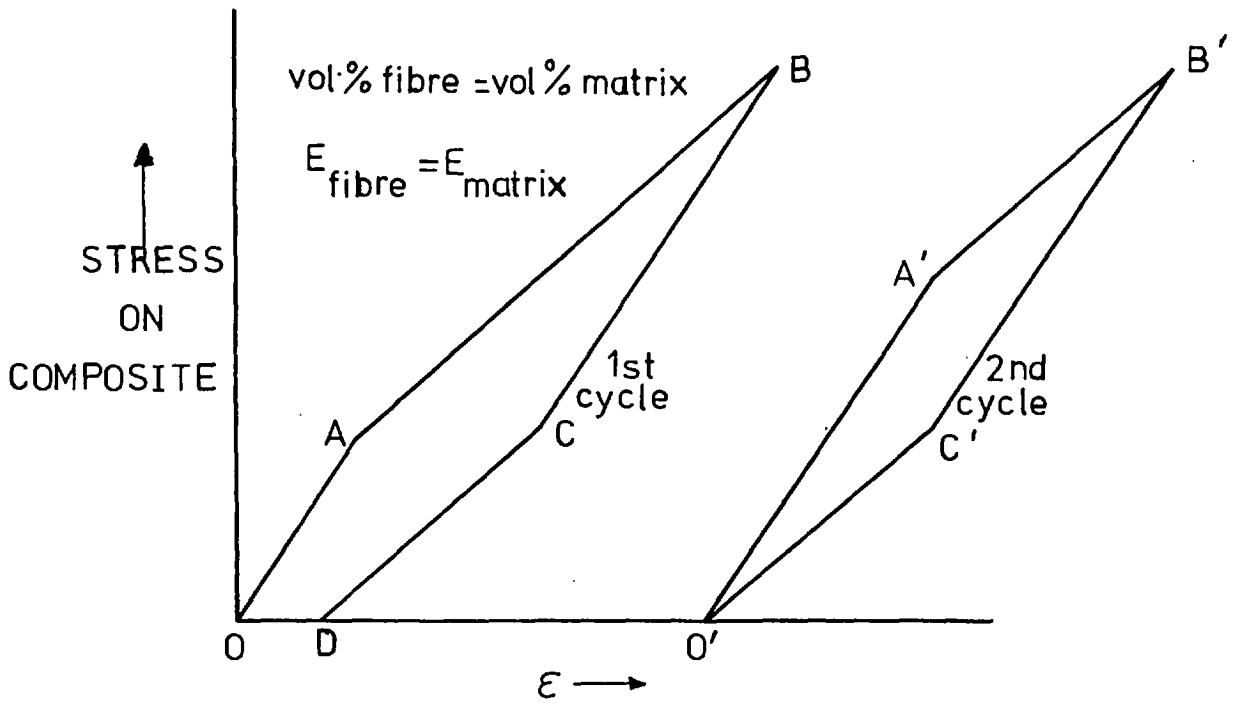


FIG. 16 Stress-strain behaviour of composite subjected to repeated loading

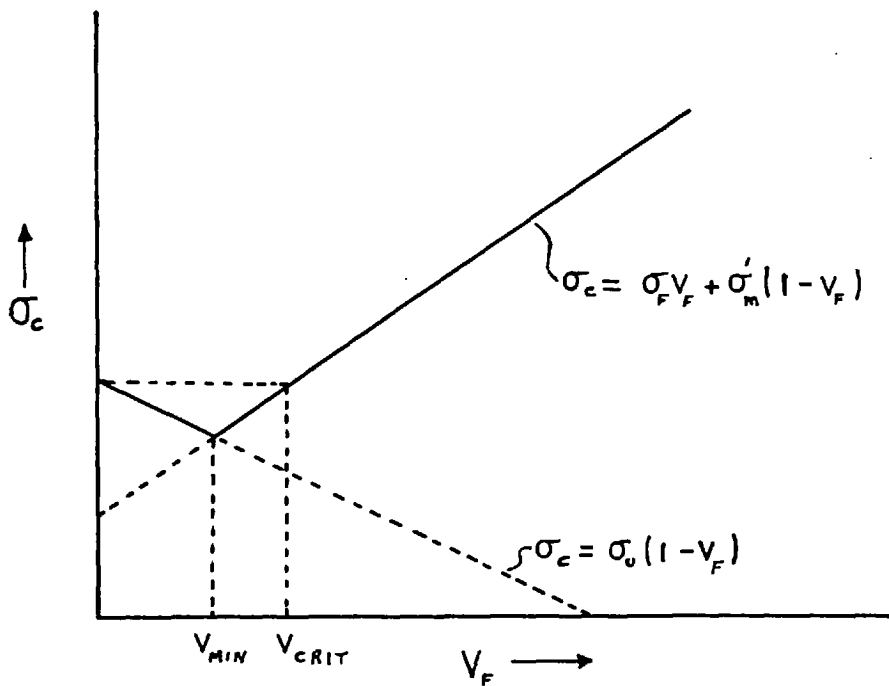


FIG. 17 Relationship between composite strength and fibre content

the volume fraction of fibres exceeds a value denoted by  $V_{crit}$ . This may be defined as:-

$$V_{crit} = \frac{\sigma_u - \sigma'_m}{\sigma_F - \sigma'_m} \quad (46)$$

(see Fig. 17). This expression is obtained by equating the right hand side of (44) with  $\sigma_u$ , the ultimate tensile strength of the bulk matrix. It should be noted that equation (44) assumes that all the fibres break in a given cross-section and that the presence of the continuous fibres does not interfere with the work hardening characteristics of the matrix material.

#### 3.7.4 Stress/strain behaviour of discontinuous fibre composites

In most practical cases, including the case of composites produced by extrusion, the fibres involved are not continuous. Therefore the previous two theory sections must be modified to allow for:-

- (1) Possible composite fracture by the "pulling out" of fibres from the matrix if the fibre aspect ratio is less than a critical value (3.7.5).
- (2) The stress concentrating effects of the fibre ends.

One additional assumption required for this modified analysis is that the fibre content, in any cross section, is constant. Stages (1), (2) and (3), mentioned previously in section 3.7.2, are once again present. For fibres with aspect ratios greatly exceeding the critical value of  $(\sigma_F/\tau_m)$  equation (42) applies.

### 3.7.5 Stresses present in discontinuous fibre composites

We must once again consider the first two stages in the behaviour of the composite under load:

- (a) Elastic matrix - elastic fibre
- (b) Plastic matrix - elastic fibre

#### (a) Elastic matrix - Elastic fibre

As the elastic moduli are similar in the aluminium -  $B_2O_3$  glass system the stresses and strains in the matrix and fibres are the same in this elastic region.

#### (b) Plastic matrix - Elastic fibre

When the load has been increased to such an extent that the stress in the matrix exceeds its flow stress  $\sigma_m$ , we can assume that the matrix everywhere starts to flow plastically. As the load continues to increase the "effective" modulus,  $d\sigma_m/d\varepsilon$ , of the matrix becomes very much smaller than  $E_F$ . This means that different displacements now occur in the bulk matrix and in the interfacial matrix. The flow of the bulk matrix along the interfacial matrix produces shear stresses causing the stress in the fibres to build up from the ends. (Fig. 18 and Fig. 20). This build up of tensile stress can be expressed thus:-

$$\frac{dP}{dZ} = 2\pi r \tau_m \quad (47)$$

where  $P$  is the tensile load in the fibre,  $r$  = fibre radius,  $\tau_m$  is the shear flow stress of the bulk matrix or of the interface, whichever is the least and  $Z$  is the distance along from the fibre end. (See Fig. 18)

If we assume that the work hardening in the matrix is very small we may integrate (47) and obtain:-

$$P = 2\pi rZ \tau_m \quad (48)$$



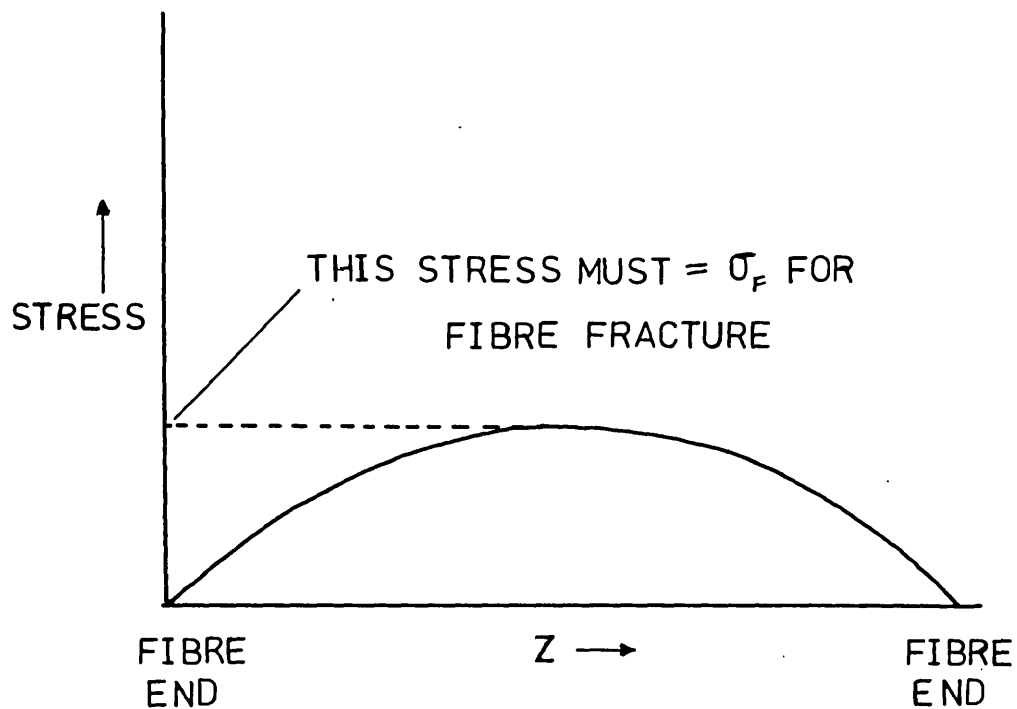
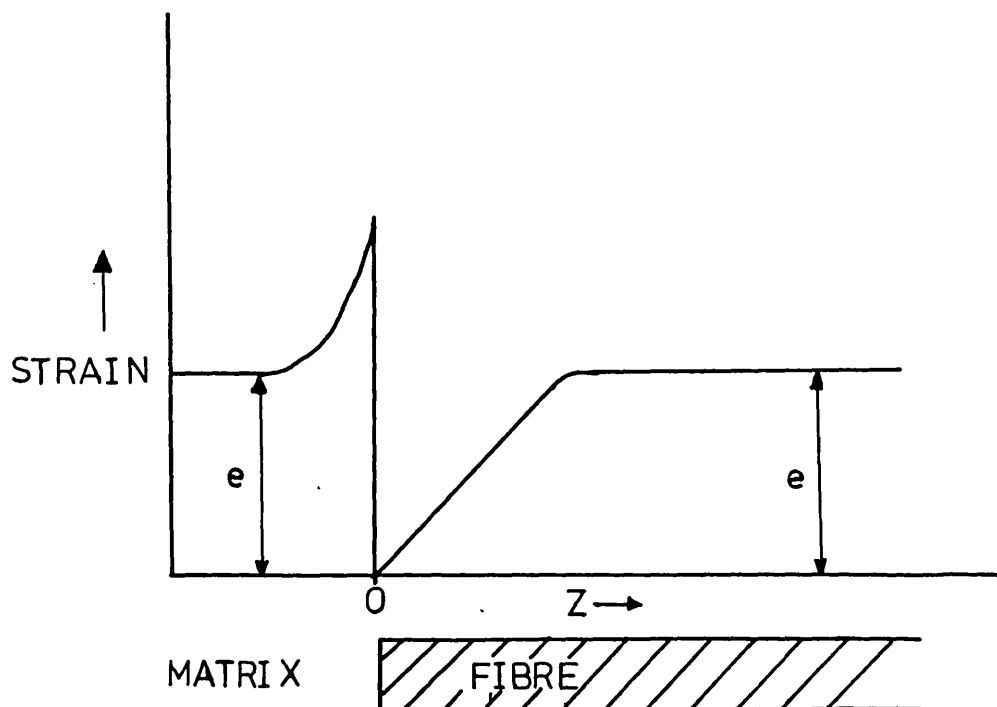


FIG. 18 Stress build up in fibre

FIG. 19 Strain distribution across matrix-fibre interface



This predicts a linear build up of tensile stress in the fibre from the ends. Now  $P$  can also be expressed as:-

$$P = \pi r^2 \sigma_{ZZ} \quad (49)$$

where  $\sigma_{ZZ}$  is the stress in the fibre at a distance  $Z$  from the end.

$$\therefore \sigma_{ZZ} = \frac{2\tau_m Z}{r} \quad (50)$$

To make full use of the strength of the fibre i.e. to make sure that composite failure occurs by the breaking of the fibres,  $\sigma_{ZZ}$  in (50) must reach  $\sigma_F$ , the U.T.S. of the fibre. For this to occur we can see that the fibre length must be greater than a certain value i.e.  $Z$  must exceed  $L_{crit}$  for a given fibre radius  $r$ . Since the fibre is loaded from both ends  $L_{crit}$  may be expressed as:-

$$L_{crit} = \frac{r \sigma_F}{\tau_m} \quad (51)$$

Equation (51) may be rewritten:

$$\frac{L_{crit}}{r} = \frac{\sigma_F}{\tau_m} \quad (52)$$

The left hand side is the limiting fibre aspect ratio which must be exceeded for efficient reinforcement to occur. When the fibre length is greater than  $L_{crit}$  the strain distribution around a fibre end is as shown in Fig. 19.

For composites formed by extrusion, clearly a minimum extrusion ratio exists for the formation of fibres having this limiting aspect ratio. This minimum extrusion ratio can be estimated approximately as shown

below.<sup>†</sup>

If the fibre length is less than  $L_{crit}$ , for a given fibre radius, fracture of the composite occurs by the failure of the matrix - fibre interface. If such a failure occurs the  $\tau_m$  value is equal to the frictional force per unit area which the matrix exerts on the fibre while sliding over it. It should be noted that if  $V_{min}$  is not exceeded failure of the interfaces will not cause instant composite fracture .

If composite failure occurs by the breaking of fibres we can see from (Fig. 18) that the average stress in the fibre is less than  $\sigma_F$  and may be expressed as

$$\sigma = \frac{1}{l} \int_0^l \sigma_{ZZ} dz \quad (53)$$

<sup>†</sup> Assuming that the glass particles are spherical with radius  $r_1$  and that after extrusion their shape is cylindrical with radius  $r_2$  and length  $l$ .

The ratio  $r_1^2/r_2^2 = R$ , the extrusion ratio. Constant volume requirements of the glass reinforcement yields:-

$$\frac{4}{3} R^{3/2} r_2 = 1$$

$$\text{The critical fibre length } l_c = \frac{r_2 \sigma_F}{\tau}$$

where  $\sigma_F$  is the stress in the fibre and  $\tau$  the fibre shear strength.

Hence for satisfactory reinforcement:-

$$\frac{4}{3} R^{3/2} r_2 > \frac{r_2 \sigma_F}{\tau}$$

Now for this system  $\sigma_F = 2412 \text{ MN/m}^2$  (assuming ideal fibres)  
 $\tau = 100/\sqrt{3} \text{ MN/m}^2$  and  $\therefore R = 9.94$ .

### 3.7.6 Strength of discontinuous fibre composites

The ultimate tensile strength of a composite containing discontinuous, uniaxially aligned, randomly distributed fibres can be expressed as:-

$$\sigma_C = \sigma_F V_F [1 - \alpha] + \sigma'_m (1 - V_F) \quad (54)$$

if  $V_F > V_{\min}$ . The term  $\alpha$  allows for the fact that the average stress in the fibres at fracture is less than  $\sigma_F$ .

### 3.7.7 Effect of fibre orientation

Fig. 21 shows a schematic representation <sup>87</sup> of a fibre reinforced composite in which the fibres are inclined at an angle  $\phi$  to the tensile axis. Even assuming that the fibre aspect ratio <sup>and the volume</sup> /fraction of fibres exceed the critical values failure may occur by one of the following modes:-

- 1) The fibres break due to the stress transfer by flow of the matrix parallel to fibres. The failure stress is then given by:-

$$\sigma_C = \sigma \cos^2 \phi \quad (55)$$

- 2) The matrix fails due to shear stresses on planes parallel to the fibres. The failure stress becomes:-

$$\tau_s = \sigma \sin \phi \cos \phi \quad (56)$$

- 3) The matrix fails by flow transverse to the fibres. The failure stress is given by:-

$$\sigma_p = \sigma \sin^2 \phi \quad (57)$$

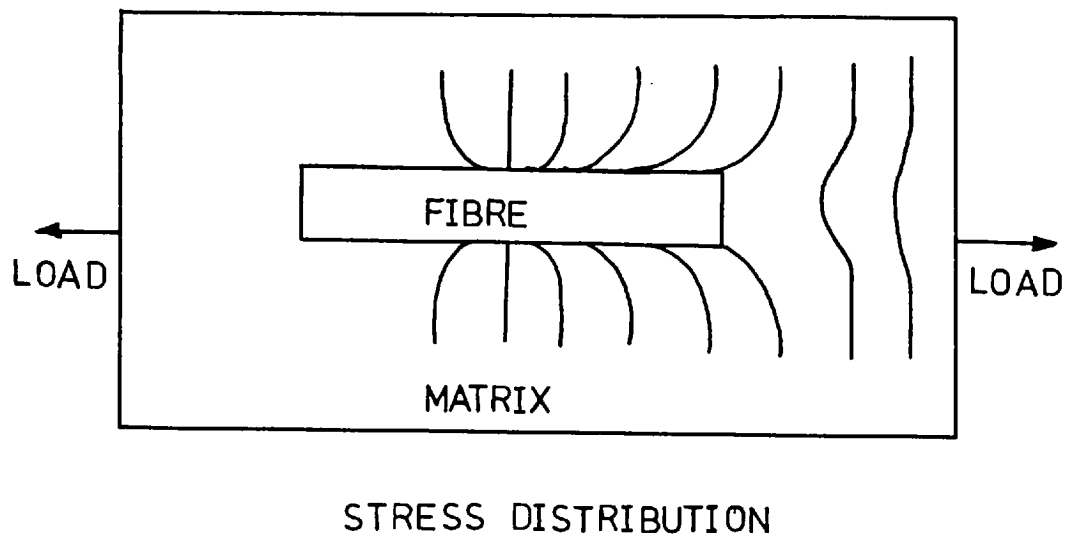


FIG. 20 Stress distribution in matrix

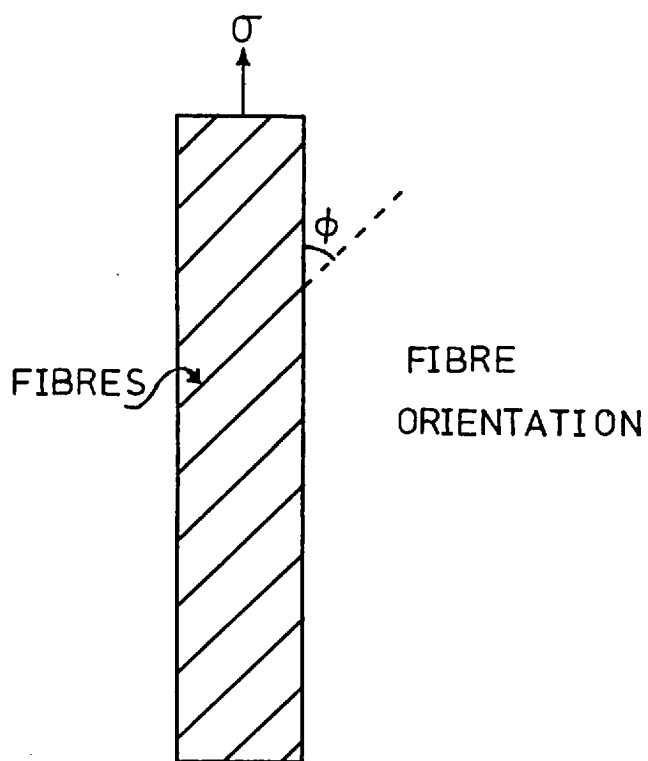


FIG. 21 Composite with fibres inclined to stress axis

The flow stress in shear for a constrained matrix e.g. when it contains fibres fairly close together, is increased by a factor of  $1.5^{87}$ . Also the value of  $\sigma_p$  in (57) is the U.T.S. of the matrix in plane strain and is therefore  $\sim 1.15$  times the value measured in a tensile test. Obviously the composite fails by the mechanism requiring the lowest value of  $\sigma$ . Fig. 22 gives the minimum value of  $\sigma$  predicted by the three equations as a function of  $\phi$ . It can be seen that when the fibres are inclined to the tensile axis at angles greater than  $6^\circ$  the strength of the composite falls drastically. The effect of random alignment of fibres was investigated by Cox<sup>104</sup>. His theory predicts that the tensile strength of a random, three-dimensional array of fibres equals one sixth of the theoretical value for the same composite containing uniaxially aligned fibres, provided that a sufficient volume fraction of fibres is present.

### 3.7.8 Fracture toughness of composites

Fig. 23 depicts fracture toughness as a function of notch depth for two types of materials:

- a) notch insensitive
- b) notch sensitive.

The straight line (a) shows that the fracture strength of an insensitive material is directly proportional to the cross sectional area of the specimen. Curve (b) can be represented by the equation:-

$$\sigma_y = \sigma_A (1 + 2 \sqrt{a/\rho}) \quad (58)$$

where  $\sigma_A$  = bulk applied stress (in y direction)

$a$  = depth of notch

$\rho$  = radius of curvature of notch

$\sigma_y$  = local tensile stress perpendicular to the notch

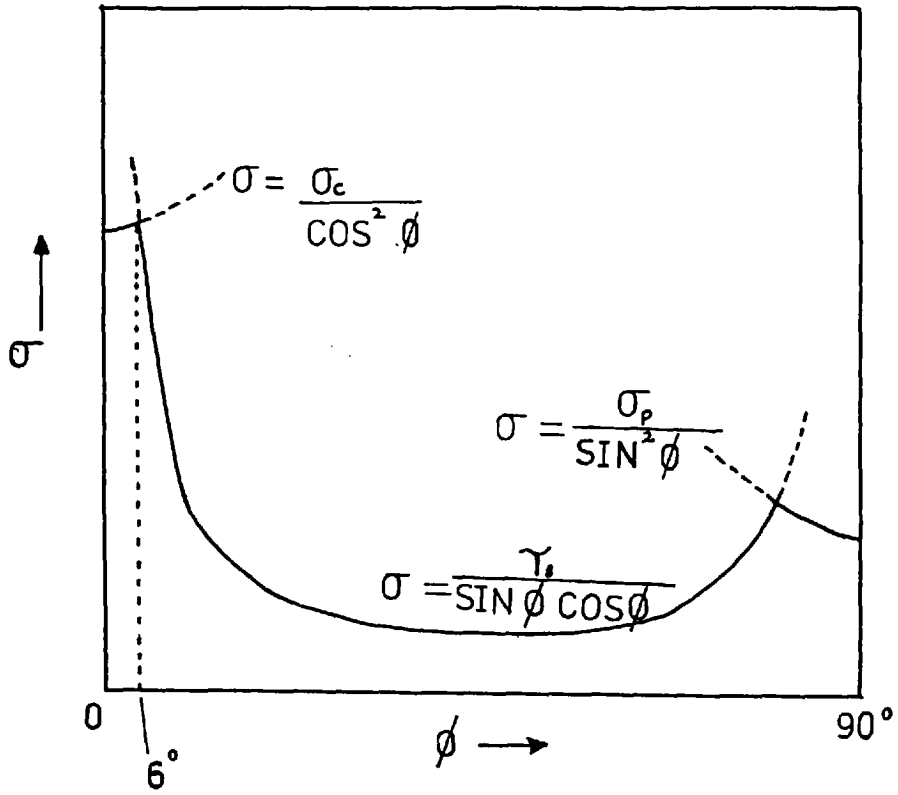


FIG. 22 Effect of fibre-stress axis inclination on composite strength

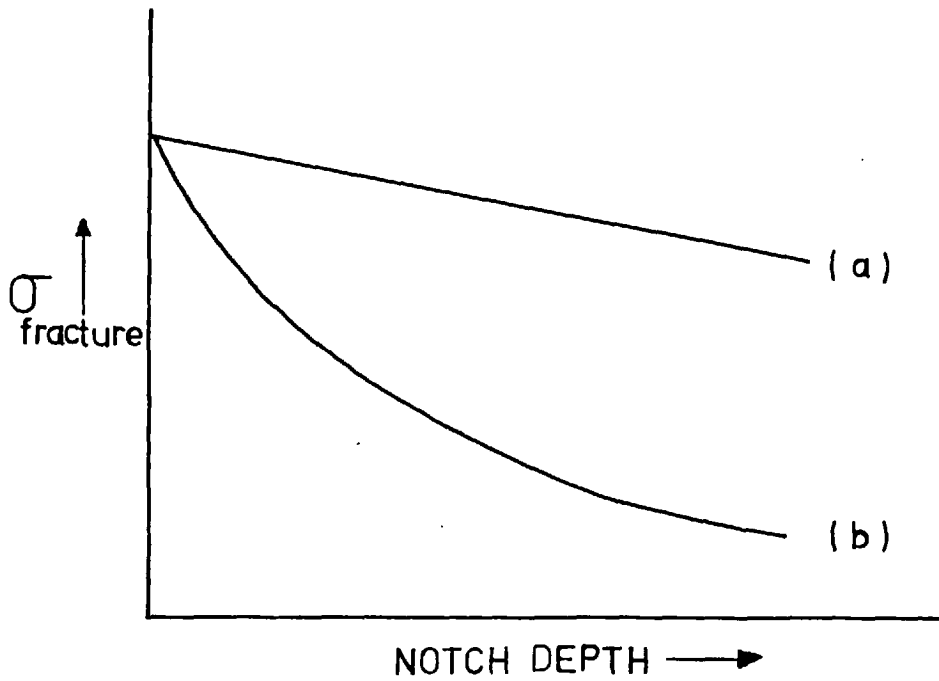


FIG. 23 Effect of notch depth on composite fracture toughness

Consider a brittle solid. We can set  $\rho \approx b$  the inter-atomic spacing and the theoretical strength as

$$\sigma_{th} = \sqrt{\frac{E\gamma}{b}} \quad (59)$$

where  $\gamma$  = surface energy/unit area

$E$  = Young's modulus

Combining (58) and (59) we find:-

$$\sigma_A \approx \frac{1}{2} \sqrt{\frac{E\gamma}{a}} \quad (60)$$

This is the Griffith crack relation.

Now for a composite to be tough it can be seen that  $\sigma_A$  and/or  $\rho$  must be large. From (60) it can be seen that  $\sigma_A$  increases with surface energy. Both of these terms,  $\sigma_A$  and  $\rho$ , increase if the crack tip is blunted i.e. the radius increases and the effective value of  $\gamma$  increases by an amount which takes into account the plastic work done. Thus one method of producing a tough composite is to employ a ductile matrix so that plastic flow at or near the tip blunts the crack tip.

Considering the crack (parallel to the x axis) as a semi-ellipse with major axis length =  $2a$  and minor axis length =  $2b$ , Fig. 24 shows  $\sigma_y$  and  $\sigma_x$  as functions of the distance from the crack tip. Now  $\sigma_x$  has a maximum value at a distance of approximately 1 crack tip radius ahead of the crack. For a composite material with relatively weak fibre-matrix bonding Fig. 25 shows how a crack may be deflected parallel to the fibre causing delamination of the fibre. Once again this produces a high fracture toughness in the composite.

Thus we can expect a composite with a ductile matrix and relatively weak bonding to exhibit reasonably high fracture toughness values.



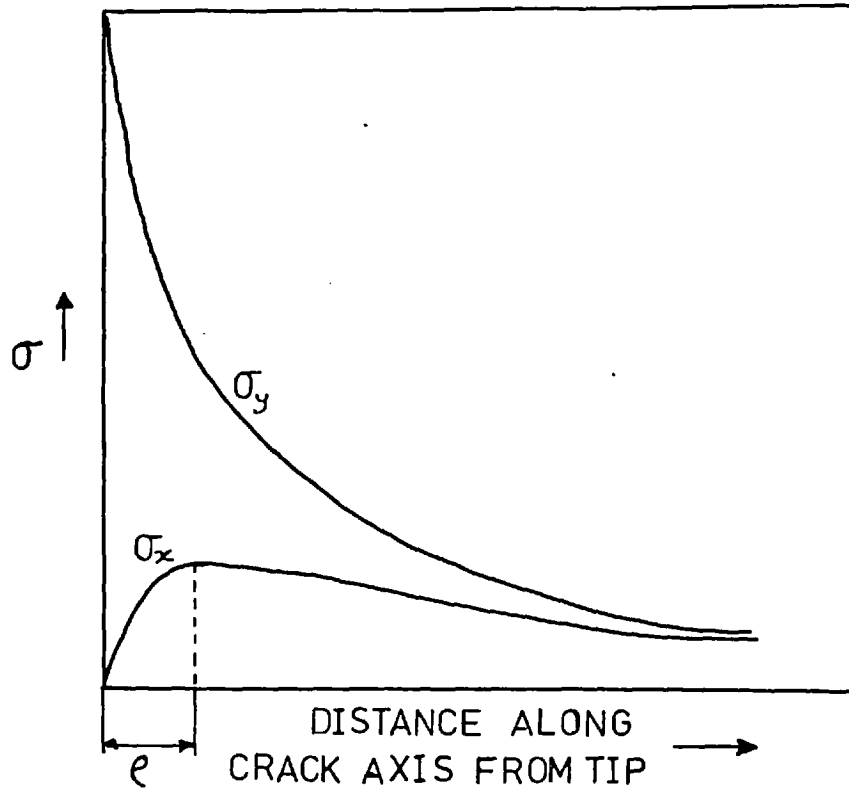


FIG. 24 Stress distribution as a function of distance from crack tip

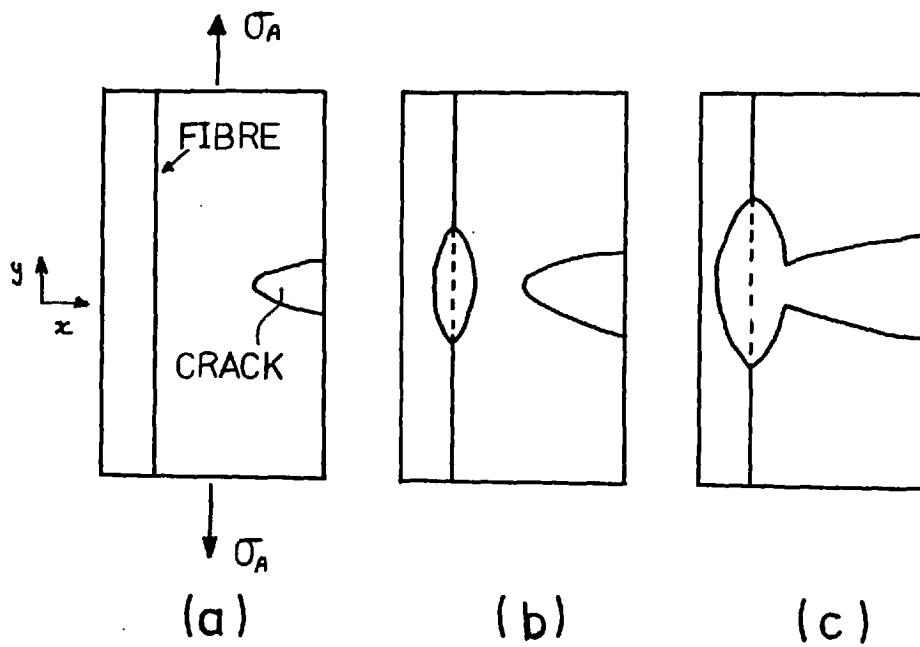


FIG. 25 Three stages in fibre delamination by crack propagation

### 3.8.1 Effect of temperature on extrude flow stress

The flow stress of most materials is made up of two components:-

- 1) The athermal component i.e. the stress needed to enable dislocations to overcome long range obstacles such as precipitate particles and other dislocations on parallel slip planes.
- 2) The thermally activated component i.e. the stress needed to overcome short range obstacles such as solute atoms and lattice friction effects.

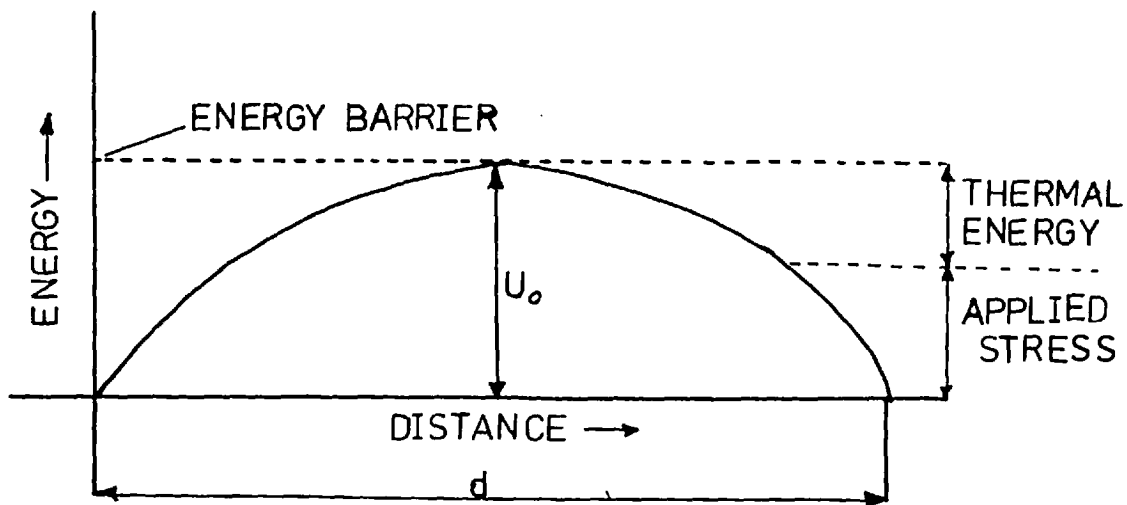
The opposition to dislocation motion may be represented as in Fig. 26. Consider a dislocation of length  $l$  and Burgers vector  $\underline{b}$ . The product  $\underline{b} \cdot d \cdot l$  is termed the activation volume,  $V$ . N.B. This is not truly a volume as  $\underline{b}$ ,  $d$  and  $l$  are coplanar but it gives an estimate of the size of the obstacle to be overcome. If the applied stress is  $K_s$  the force per unit length on the dislocation is  $K_s \underline{b}$ . Therefore the work done by the applied stress in overcoming the obstacle is  $K_s \underline{b} l d$  i.e.  $K_s V$ . We can write<sup>(109)</sup>

$$u_o = u + K_s V \quad (61)$$

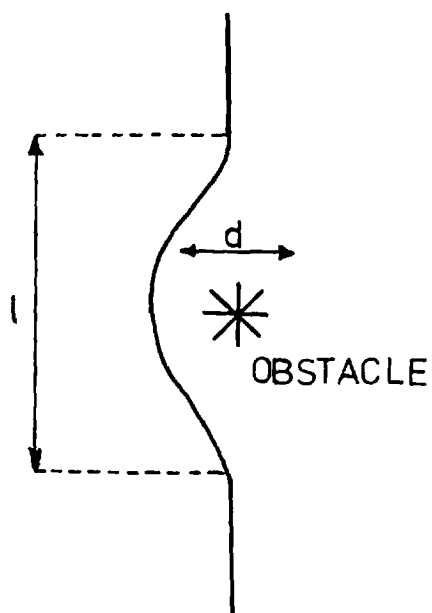
However, part of  $K_s$  is occupied in overcoming other long range obstacles which interfere with this considered obstacle. Let this part =  $K_A$ . Therefore

$$u_o = u + (K_s - K_A) V \quad (62)$$

we know that strain rate = dislocation density  $\times \underline{b} \times$  velocity of dislocation movement. Also the velocity of dislocation = rate of overcoming obstacles  $\times$  distance apart of obstacles.



FRONT VIEW



PLAN VIEW

FIG. 26 Schematic representation of obstacle to dislocation movement as an energy barrier

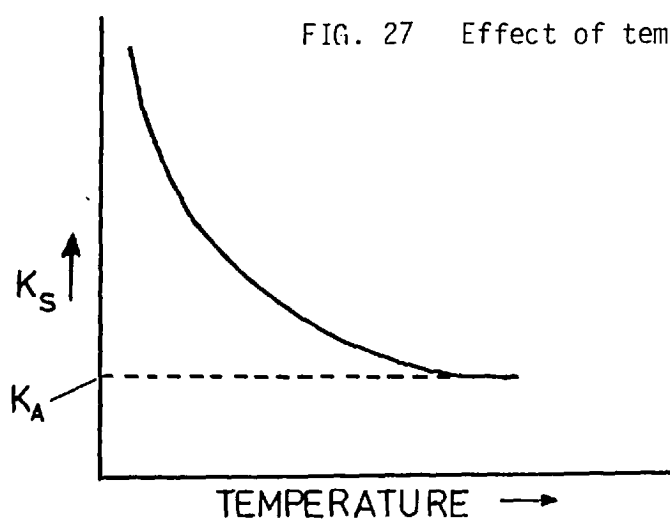


FIG. 27 Effect of temperature on flow stress

Therefore

$$\text{Velocity} = v_o \exp\left(-\frac{u}{KT}\right) \times D \quad (63)$$

where:-  $v_o$  = frequency of attempts to overcome obstacles

$$\exp\left(-\frac{u}{KT}\right) = \text{fraction of successful attempts}$$

$$D = \text{distance apart of obstacles}$$

$$\text{Hence } \dot{\epsilon} = \rho \underline{b} D v_o \exp\left(-\frac{u}{KT}\right) \quad (64)$$

$$\text{Now } u = u_o - (K_s - K_A)V \quad (65)$$

$$\text{Therefore } \dot{\epsilon} = D \rho \underline{b} v_o \exp\left[\frac{V(K_s - K_A)}{KT}\right] \exp\left(-\frac{u_o}{KT}\right) \quad (66)$$

$$\text{Let } D \rho \underline{b} v_o = \dot{\epsilon}_o$$

$$\text{Therefore } K_s = \frac{u_o - KT \ln(\dot{\epsilon}_o / \dot{\epsilon})}{V} + K_A \quad (67)$$

Fig. 27 shows the form of this relationship schematically.

### 3.9.1 Temperature change during extrusion

During extrusion the billet temperature is usually raised by the work done upon it. (Except for very high initial temperatures and slow ram speeds). It is estimated that 90% of this work is converted to heat energy. Heat flow from the billet to the container, tools and ram also occurs. A mean temperature of the billet may be predicted at any time after the beginning of extrusion by use of an integral profile method proposed by Sheppard and Raybould<sup>34</sup>, assuming an initial uniformity of temperature between billet and container. However, if the initial billet temperature is different from that of the container the theory must be modified. Given below is the modified solution proposed by Sheppard and Wood<sup>128</sup>.

The first step involves introducing a control volume around the deformation zone and equating the heat leaving this zone to the work put into it. Since the billet is also losing heat to the container outside this area, a second control volume may be used to include this heat loss in the overall volume equation. It is assumed that there is no conduction from control volume 1 to control volume 2 (see fig. 28). This is reasonable as the temperature gradient between them is small due to the high conductivity of aluminium. Back conduction into the pressure pad is considered. The heat flows are shown in (Fig. 28) and assuming 90% conversion of work done into heat we may write:-

$$0.9 \times L \times V \times t = Q_1 + Q_2 + Q_3 + Q_4 + Q_5 + Q_6 + Q_7 \quad (68)$$

The interface temperature between deforming stock and tools is calculated in the usual way:-

$$\frac{T_B - T_I}{T_I - T_S} = \sqrt{\frac{K_S \rho_S C_A}{K_A \rho_A C_S}} \quad (69)$$

Thus the temperature of the deformation zone is  $T_B + \Delta T$  and the temperature differential  $T_I - T_S$  is designated  $\Delta T_D$  whilst in the deformation zone the temperature differential is  $\Delta T + \Delta T_D$  designated  $\Delta T^*$ .  $Q_1$  to  $Q_7$  cannot be estimated until the time/temperature profiles in the container, tools and billet have been estimated. The temperature profile around a heat source may be expressed:-

$$T(x,t) = a_0 + a_1 x + a_2 x^2$$

where  $T(x,t)$  is the (temperature - datum temperature) at a point a distance  $x$  from the source at a time  $t$ . The constants  $a_0$ ,  $a_1$ ,  $a_2$  may be found by consideration

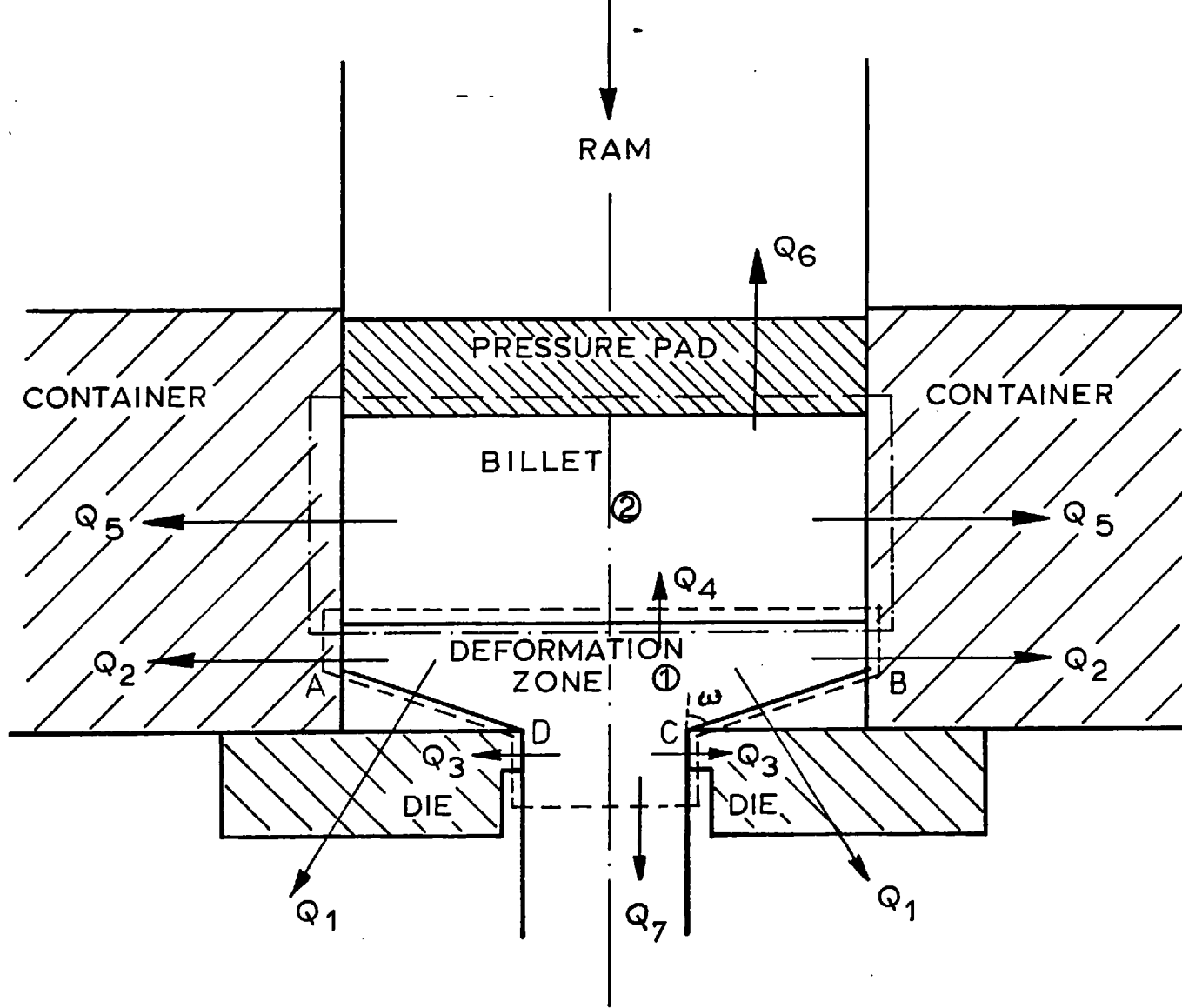


FIG. 28 Control volumes and heat losses employed in integral profile temperature rise model

of the boundary conditions.

$$\text{at } x = 0 \quad \Delta T(x, t) = \Delta T^* \quad \text{and} \quad a_0 = \Delta T^*$$

At distance  $s$ , the maximum penetration of the heat front:-

$$\Delta T(x, t) = 0$$

$$\text{and} \quad \frac{d\Delta T(x, t)}{dt} = 0$$

$$\text{hence} \quad a_1 = -2a_2 s$$

$$\text{and} \quad a_2 = \frac{\Delta T^*}{s^2}$$

$$a_1 = -\frac{2\Delta T^*}{s}$$

$$\Delta T(x, t) = \Delta T^* - \frac{2\Delta T^*}{s} x + \frac{\Delta T^*}{s^2} x^2 = \Delta T^* \left(1 - \frac{x}{s}\right)^2 \quad (70)$$

If heat flows only in the  $x$  direction then the heat accumulated may be written:-

$$\begin{aligned} Q_{\text{acc}} &= \int_0^s A C_p \Delta T^* \left(1 - \frac{x}{s}\right)^2 dx \\ &= A C_p \Delta T^* \frac{s}{3} \end{aligned} \quad (71)$$

$$\text{Hence} \quad Q_{\text{acc/sec}} = \frac{A C_p \Delta T^*}{3} \frac{ds}{dt} \quad (72)$$

From the Fourier equation:-

$$\frac{dQ}{dt} = -KA \frac{dT}{dx} = -KA \left[ \frac{d\Delta T(x, t)}{dx} \right]_{x=0} \quad (73)$$

Equating (72) and (73) gives:-  $s = \sqrt{\frac{12kt}{\rho C}}$  (74)

which will be valid where the contact area A does not vary with x.

For  $Q_2$ ,  $Q_3$  and  $Q_5$ , A will vary with x and

$$\begin{aligned} Q_{acc} &= \int_0^s 2\pi \rho C \Delta T^* \left(1 - \frac{x}{s}\right)^2 dx \\ &= \frac{\pi \rho C \Delta T^* s^2}{6} \end{aligned} \quad (75)$$

Proceeding as before

$$\frac{dQ}{dt} = \frac{\pi \rho C \Delta T^* s}{3} \frac{ds}{dt}$$

and

$$s = \sqrt[3]{\frac{18kAt}{\pi \rho C}} \quad (76)$$

The heat losses from each control volume may now be calculated

Q1 The surface contact area of the cone A B C D

$$= \frac{\pi}{\cos \omega} \left\{ \frac{D_B^2 - D_E^2}{4} \right\}$$

hence  $Q_1 = \Delta T^* \frac{\pi (D_B^2 - D_E^2)}{\cos \omega} \left( \frac{K \rho C}{12} \right)^{\frac{1}{2}} t^{\frac{1}{2}}$  (77)

Q2 The depth of the zone of working is approximately:-

$$\left( \frac{D_B - D_E}{2} \right) \tan 45^\circ$$

hence  $l_D = \left( \frac{D_B - D_E}{2} \right) (1 - \tan \omega)$

$\omega$  being obtained from an upper bound solution.



The heat accumulated in the radial direction is given by (75) which assumes the heat source to be at the centre of a large cylinder. In fact the heat source is distance  $R_B$  from the billet centre. Hence

$$Q_{\text{acc}} = \frac{\pi}{6} l_D \rho C \Delta T^* (R_B + s)^2$$

which includes the heat stored in the material. Hence heat loss is given by:-

$$Q_2 = \frac{\pi}{6} \Delta T^* l_D \left[ \left\{ 18 K_S D_B (\rho_S C_S)^{\frac{1}{2}} t \right\}^{\frac{2}{3}} + \left\{ 18 K_S D_B^4 (\rho_S C_S)^2 t \right\}^{\frac{1}{3}} \right] \quad (78)$$

Similarly

$$Q_3 = \frac{\pi}{6} \Delta T^* l_1 \left[ \left\{ 18 K_S D_E (\rho_S C_S)^{\frac{1}{2}} t \right\}^{\frac{2}{3}} + \left\{ 18 K_S D_E^4 (\rho_S C_S)^2 t \right\}^{\frac{1}{3}} \right] \quad (79)$$

The heat losses due to back conduction  $Q_4$  from volume 1 will be ignored. Tanner and Johnson<sup>44</sup> have justified this assumption and shown the losses to be negligible for normal ram speeds. The heat loss  $Q_6$  will be significant when the ram approaches the deformation zone area. Following a similar derivation to that used to find  $Q_1$  we may write:

$$\text{Surface area of contact } A = \frac{\pi D_B^2}{4}$$

$$Q_6 = \Delta T_D \pi D_B^2 \left[ \frac{K_S \rho_S C_S}{12} \right]^{\frac{1}{2}} t^{\frac{1}{2}} \quad (80)$$

Q5

If the length  $l_T$  is the depth of control volume 2 in (Fig. 28) then  $l_T$  at any time  $\Delta t$  after start of extrusion will be

$$l_T = V \Delta t$$

and  $Q_5$  may be written:-

$$Q_5 = \frac{\pi \Delta T_D}{6} \left[ \left\{ 18K_S D_B l_T^{3/2} (\rho_S C_S)^{1/2} \right\}^{2/3} \left\{ t^{2/3} - \frac{3V t^{5/3}}{5l_T} \right\} + \left\{ 18K_S D_B^4 l_T^3 (\rho_S C_S)^2 \right\}^{1/3} \left\{ t^{1/3} - \frac{3V t^{4/3}}{4l_T} \right\} \right] \quad (81)$$

The heat loss  $Q_7$  is straight forward and may be expressed:-

$$Q_7 = \Delta T \rho_{AL} C_{AL} R V t \frac{\pi D_E^2}{4} \quad (82)$$

hence

$$Q_1 = (\Delta T + \Delta T_D) (K_1 t^{1/2})$$

$$Q_2 = (\Delta T + \Delta T_D) (K_2 t^{2/3} + K_3 t^{1/3})$$

$$Q_3 = (\Delta T + \Delta T_D) (K_4 t^{2/3} + K_5 t^{1/3})$$

$$Q_4 = 0$$

$$Q_5 = \Delta T_D K_6 t^{2/3} + K_1 t^{5/3} + K_3 t^{1/3} + K_9 t^{4/3}$$

$$Q_6 = \Delta T_D K_{10} t^{1/2}$$

$$Q_7 = \Delta T K_{11} t$$

Hence work done =

$$\Delta T \{A_1 t^{\frac{1}{2}} + A_2 t^{\frac{1}{3}} + A_3 t^{\frac{2}{3}} + A_4 t\} + \Delta T_D \{B_1 t^{\frac{1}{2}} + B_2 t^{\frac{1}{3}} + B_3 t^{\frac{2}{3}} + B_4 t^{4/3} + B_5 t^{5/3}\} \quad (83)$$

$$\text{Work done} = \Delta T C_1(\text{Total}) + \Delta T_D C_2(\text{Total})$$

Hence temperature rise of extrude:-

$$\Delta T = \frac{0.9 \times L \times V t - \Delta T_D C_2 \{t\}}{C_1 \{t\}} \quad (84)$$

The program given in appendix (4) uses this method to calculate the temperature change

### 3.10.1 Torsion testing

Generally, torsion tests are carried out to gain information about the flow stress of a material at various temperatures and strain rates. The equation most commonly used to obtain flow stress information from torsion data was first proposed by Nadai<sup>105</sup>:-

$$\tau = \frac{1}{2\pi a^3} (3M + \theta \frac{dM}{d\theta}) \quad (85)$$

where  $\tau$  = surface shear stress

$M$  = torque

$\theta$  = amount of twist

$a$  = specimen radius

$\frac{dM}{d\theta}$  = slope of torque-twist curve.

The shortcoming of this equation is that it assumes the stress is not dependent upon the strain rate. This may be acceptable for some materials which are virtually strain rate insensitive but it is unacceptable for aluminium and its alloys which show a marked strain rate sensitivity. Fields and Backofen<sup>106</sup> suggested a modified equation to incorporate strain rate effects:-

$$\tau = \frac{M}{2\pi a^3} (3 + n + m) \quad (86)$$

where:-  $n$  = the gradient of  $\ln M$  v.  $\ln \dot{\theta}$  at const. strain rate

$m$  = the gradient of  $\ln M$  v.  $\ln \dot{\theta}$  at const.  $\theta$   
 ( $\dot{\theta}$  = rate of twisting).

The use of this equation permits the calculation of the flow stress from the results of the minimum number of torsion tests and removes the need for differential torsion testing (i.e. the testing of specimens of different diameter).

## CHAPTER 4

### EXPERIMENTAL PROCEDURE

#### 4.1.1. Introduction

The following chapter describes the experimental methods used to :-

- (1) Examine the starting materials.
- (2) Prepare composites by extrusion.
- (3) Monitor process conditions.
- (4) Prepare specimens for testing and examination.
- (5) Measure mechanical properties.
- (6) Examine the extrude structure microscopically.

#### 4.2.1. Metal Powder Production

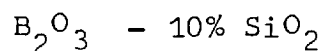
The metal powders used in this research project were produced by one of two methods :-

- (1) Air atomization. (horizontal air blast)  
(Aluminium Powder Co)
- (2) Water atomization. (horizontal water jet)  
(Davy-Lowe Ltd.)

In both cases the feedstock to the atomizer furnaces was prealloyed cast ingots. The HE9 and HE30 were produced by the former method whereas the Al-Mg and Al powders were produced by water atomization in an attempt to increase the oxygen content. It was hoped that the increased oxygen content would improve the extrude mechanical properties.

#### 4.2.2. Glass Powder Production

The glass used for reinforcement throughout this project was of nominal composition :-



This particular glass was selected for three main reasons :-

(1)  $\text{B}_2\text{O}_3$  has a low softening point which is much less than the temperatures obtained when extruding Aluminium or Aluminium Alloys. It is thus viscous at hot working temperatures.

(2)  $\text{B}_2\text{O}_3$  forms a good bond with aluminium during extrusion. This is an essential feature, necessary to ensure that the fibres do not "pull-out" of the matrix when a load is applied to the composite.

(3)  $\text{B}_2\text{O}_3$  with 10%  $\text{SiO}_2$  is a glass with good mechanical properties. This is a necessity if a strong composite is to be produced.

The glass was manufactured at the Borax Research Laboratories. It was prepared by roller mixing carefully weighed amounts of the constituent oxides which were fused in fireclay crucibles and poured into steel moulds. The product was then crushed, ground and sieved to produce a glass powder.

#### 4.2.3. Powder Size

There are a number of techniques available for the determination of the particle size distribution of a quantity of powder. The simplest technique is that of sieving. The end-point of a sieve test is specified<sup>129</sup> as either :-

(a) The end of a fixed time period.

or (b) The time at which the rate of particle passage through a sieve has reached a specified value.

Technique (a) was used to simplify matters as much as possible. Powder samples were taken from each drum after the contents had been thoroughly mixed by rolling.

The sieving was carried out on conventional 0.2M diameter B.S. wire mesh sieves. A weighed amount (0.15 - 0.2 Kg) of the powder (glass or metal) was placed on the uppermost of a stack of sieves. The stack was then placed on an "Endrock" mechanical sieve shaker. The sieving was then carried out by a mechanical vibratory and rotary motion for one hour and the separate fractions collected and weighed. Particles trapped on a particular sieve were assumed to have a mean size between that of the catching sieve and the one before. In reality a wide range of sizes in between these two mesh sizes existed.

#### 4.2.4. Powder Shape and Structure

The shapes of the individual powder particles within the powder mass were investigated using a Cambridge stereoscan 600 scanning electron microscope. Particles were distributed on small aluminium stubs which had previously been painted with an electrically conducting paint (silver Dag). The stubs were then placed in the column of the microscope and the column evacuated. The surfaces of the stubs were scanned at magnifications ranging from 50 to 5000 times.

To investigate the powder structures, powder particles were mounted in "met-set". The specimens were then ground on silicon carbide paper and polished with diamond paste and alumina powder (down to 0.05 $\mu$ ). Finally the specimens were cleaned and then etched in Keller's reagent. A Vickers' light microscope

was used to examine the specimens at magnifications of up to 400 times.

#### 4.2.5. Chemical Analysis

The analysis of the atomised powder was carried out by the "analytical services" department within the College. They employed infra-red spectrophotometry on compacted powder pellets. A wet chemical analysis was also used to determine the magnesium content of the Al-Mg alloy.

#### 4.3.1. Mixing and Compaction of Billets

Carefully weighed amounts of metal and glass powder were poured into screw-topped containers which were then rolled for two hours to ensure thorough mixing. The glass content of the billets was varied between 0 and 20 wt.%.

The process of cold compaction was carried out in a 0.075m diameter ram and die set made from boronised heat treated KEA 145 steel. The compaction set was designed so that the ram was the same length as the die to facilitate compact ejection. The ram was machined down to a push fit tolerance to ensure that little powder forced its way up between the ram and die during the compaction. Before the die was filled with powder, the walls and tool pieces were painted with a solution of stearic acid in chloroform to act as a lubricant. This ensured little pickup of aluminium on the tooling surfaces. Having filled the container with a weighed amount of powder, (0.77Kg), compaction was carried out using a "Tangye" 2MN (200 TON) press to a pressure of  $14 \text{ MN/m}^2$ . This gave a billet of 80-90 % theoretical density (depending on the alloy and glass content) of height 0.09m.

#### 4.3.2. Strength of the Compacted Billet

The strength of the compacted billet was



investigated to discover if the billet was strong enough to withstand fairly rough handling on the factory floor prior to extrusion. This "post-compaction" billet strength was deemed important if the process was to be commercially viable.

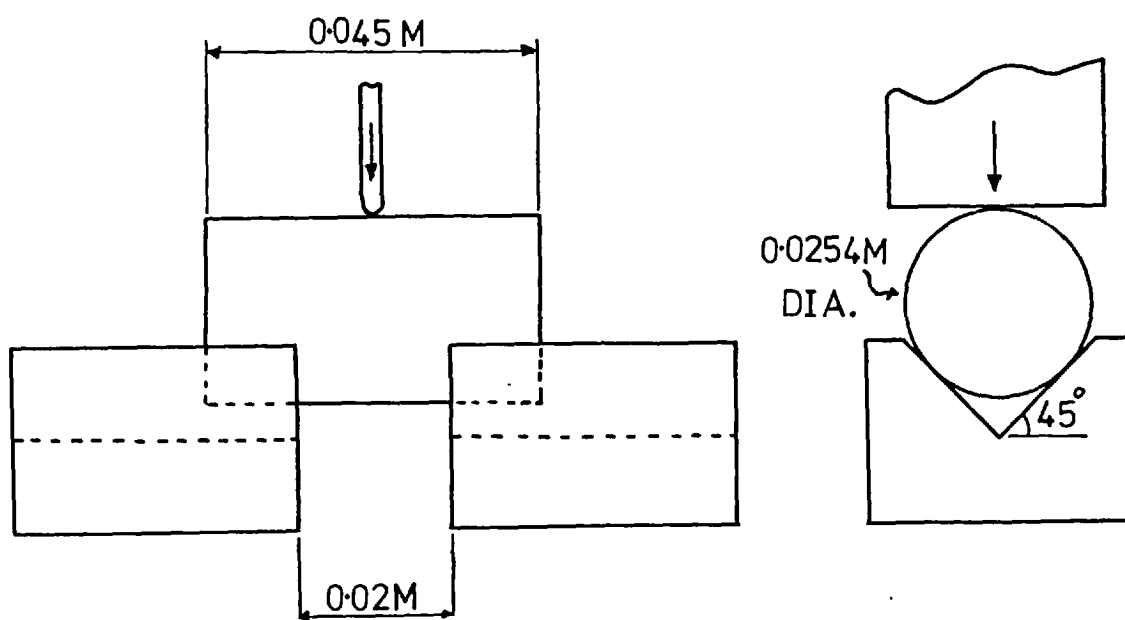
These tests were carried out in the Borax Research Laboratories. In all cases the billets tested were HE<sup>9</sup> alloy plus 10wt% of B<sub>2</sub>O<sub>3</sub> - S<sub>1</sub>O<sub>2</sub>. The billets were compacted in a 0.0445 M long X 0.0254 M diameter ram and die set, each billet weighing 0.05 Kg. These dimensions ensured that the shape factor of the billet was the same as that of the larger extrusion billet.

In theory it had been intended to study the toughness i.e. notch ductility, of the billets. Unfortunately as no impact testing equipment was available, at the time, the true toughness was not measured. From experience the billets were known to be most likely to shatter transversely, and so it was decided that the billets should be loaded transversely.

This applied load was increased gradually and continuously from zero. The experimental set up is shown in Fig.29. The billets were supported by two 45° "V" blocks with a 0.02 M gap between them. The load was applied by means of a "knife edge" ( in fact of finite radius ) to the middle of the billet. The load was produced by a sand crushing machine.

As the area of contact between block and billet was unknown, the actual fracture pressures were unknown. What was obtained was a comparison of the "relative" strengths of billets compacted at pressures ranging from 185 to 465MN/m<sup>2</sup>. The theoretical densities were found by measuring the weights and volumes of the billets and assuming a 100% density of 2580 Kg/m<sup>3</sup>. The effect of sintering the billets after compaction at temperatures ranging from 150-450°C was also

FIG. 29 Apparatus used to determine compact strength



investigated. Billets compacted from different sieve fractions were also tested to find the effect of powder particle size on compact strength.

#### 4.4.1. The Extrusion Press

The extrusion press used was a 5MN, vertically mounted, fast action ENEFCO press. It was situated over a run out pit thus permitting direct extrusion with the die fixed in position above the pit by a backing ring. The press layout is shown in plate 1 with operating controls on the right beneath the load indicating dial and oil pressure gauges.

The container, fixed onto two hydraulic rams via a cast housing, plate 2, was capable of accommodating billets of up to 0.075 m in diameter and 0.158 m in length.

An auxilliary pump enabled the ram approach speed prior to the commencement of extrusion to be increased greatly. This reduced the heat lost by the billet to its surroundings whilst the ram was being lowered to touch the pressure pad. Once the load exceeded 0.2 MN this pump "cut out" and the ram moved at the pre-set ram speed during the extrusion.

#### 4.4.2. The Heaters

The container was heated by eight inconel heating elements placed around it and held in position inside the container holder casting. These heaters, with a total rating of 12 kilo-watts, had a maximum operational temperature of 700°C. To prevent overheating of the elements thermocouples, attached to eurotherm controllers, were braised onto each element. With the elements operating near their maximum temperatures the temperature of the container liner was maintained at 300°C.

#### 4.4.3. Lubrication

Previously Chare <sup>19</sup> showed the use of graphite



PLATE 1

General layout of the extrusion press

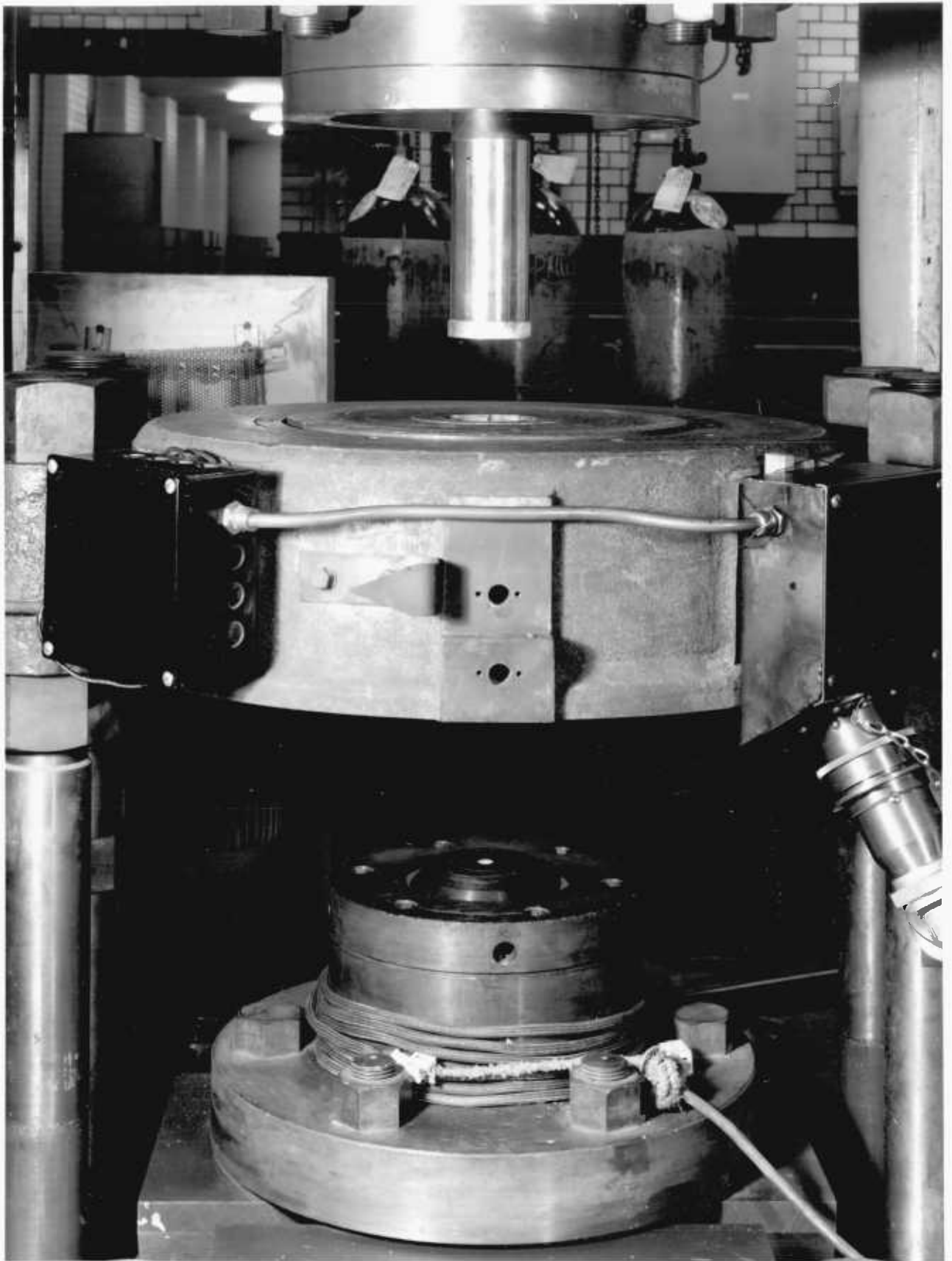


PLATE 2

Container, ram and die on extrusion press.

as a lubricant for powdered aluminium alloy extrusion to be unsuccessful, producing a severe fir tree defect due to excessive die pick up and friction. He achieved satisfactory results using circular pads of Aluminium 0.01m thick and 0.073m in diameter placed in front of the billet before extrusion. These pads formed dead metal zones for the powder billets and also covered the extrudes with "sheaths" of solid material during passage through the deformation zone and die.

In the present series of experiments a 0.01m thick pad of solid HE9 of 0.073m diameter was used for each of the HE9 powder extrusions and a similar pad of HE30 was used for each of the remaining HE30, Al-Mg and Al powder extrusions. In the case of composite extrusions the sheath produced by the pad was of even greater importance in that it protected the glass fibres from the atmosphere and prevented hydrolysis from occurring.

#### 4.4.4. Billet Preheat

The preheating of the billets to the desired extrusion temperature was performed in an air circulating furnace situated adjacent to the press. Temperatures in the range 250 - 600<sup>o</sup> C were investigated. The furnace was calibrated by means of a billet, drilled axially to the centre, containing a thermocouple which was connected to a digital voltmeter. The furnace temperature control was set at various temperatures in the range mentioned above and the furnace allowed to attain these temperatures. The billet containing the thermocouple was then placed in the furnace for 40 minutes and then the D.V.M. reading was noted and converted to the true billet temperature.

It was necessary for the heat losses, during the transfer of the billet from the air circulating furnace to the container, to be measured. The billet containing the thermocouple was removed from the furnace (at different temperatures) and transferred

to the container whilst a series of D.V.M. readings at measured time intervals were noted. This gave cooling curves for pre-heat temperatures. These curves are shown in appendix (5).

Attempts to preheat billets in an induction furnace situated above the press container proved futile due to the voids and the high oxide content of the compacted billets.

#### 4.4.5. Reduction Ratio

The reduction ratios used in this series of experiments were 5, 10, 30, 40, 60, 80 and 180:1. These ratios were obtained by using interchangeable dies in the die holder. Fig 30 shows the die design used throughout. The die, held in place by the holder and the retainer ring, is shown in Plate 2.

Changing the die involved the removal of the retaining ring, which was held in place by 6 bolts, followed by the removal of the die holder leaving the die piece and backing ring. The new die was then placed on the backing ring and the holder and the retaining ring replaced.

#### 4.4.6. Ram Speed

The ram speed was controlled by the adjustment of the pumps to give a range of extrusion speeds varying from 0.003 m/sec to 0.013 m/sec by contra flow direct pumping. Higher speeds, of the order of 0.1 - 0.2 m/sec were obtained by using the accumulator drive i.e. discharging nitrogen filled pressure bottles. The speed was measured by a rectilinear potentiometer with a 0.6m stroke which was fixed between the cross head and the base of the press. The voltage, which varied with ram position, was fed into a Hewlett Packard 136A x-y recorder and plotted against time, the slope of the line on the chart giving the average ram speed.

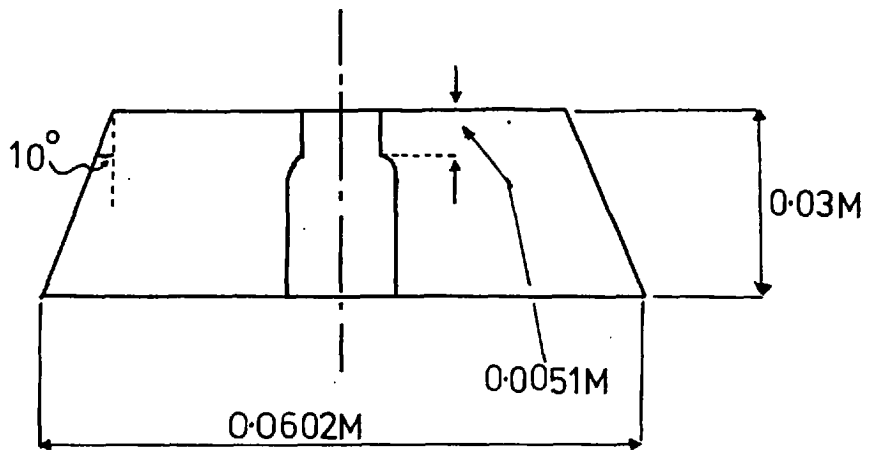
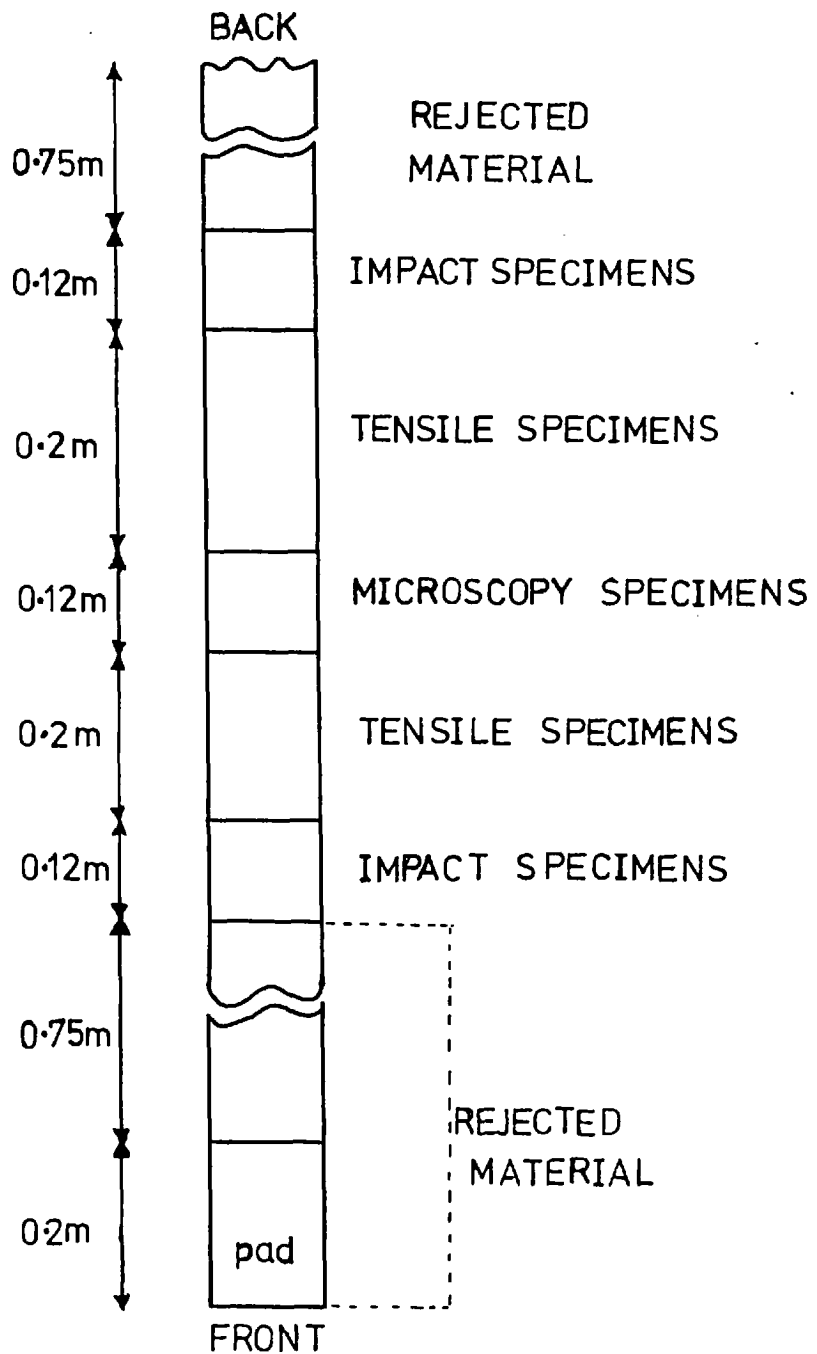


FIG. 31 Locations of samples taken from an extrude





#### 4.4.7. Extrusion Load

The extrusion load was measured by a Mayes load cell situated directly above the ram stem. The output was indicated on the dial gauge and recorded on the X-Y recorder. The X-Y recorder pen speed was fixed thus enabling a load/displacement curve to be drawn.

#### 4.4.8. Extrusion Procedure

The billet with a pad taped on top was heated in the air circulating furnace for 40 minutes at the desired temperature. The billet was then removed from the furnace with tongs, inverted so that the pad sat directly on top of the die and then dropped into the container. A pressure pad to prevent "skulling" around the ram was placed behind the billet and the extrusion initiated. The ram was lowered quickly via the fast approach mechanism until it reached the back of the billet and the load reached 0.2MN. The fast approach motor then cut out automatically and the ram continued at the pre-set, desired speed. The load and ram displacement were recorded on the X-Y recorder. The extrusion was stopped when the ram made contact with two protective steel ring segments placed on top of the container. The stem was then raised and the container lifted. The extrude was sawn through just above the die. Then, with the discard and pressure pad left in the container, the extruded rod was punched out of the die into the pit below. The container was then lowered onto two liner supports, placed on the die holder, and the discard and pressure pad pushed out by the ram, using a dummy stem. A scraper pad, having an interference fit with the liner, was then pushed through the container to clean the surface of the liner. This insured a fairly uniform liner surface condition for each extrusion and thus fairly uniform friction conditions.

#### 4.4.9. Heat Treatment

As both HE9 and HE30 are classical precipitation hardening alloys, the precipitate in question being  $Mg_2Si$ , the effect of heat treatments on the extruded rod was of some interest. Specimens cut from the extruded rod were soaked in the air circulating furnace at temperatures ranging from 500-580°C for times of 1 to 20 hours. The specimens were then quenched in water and aged at temperatures ranging from 160 to 195°C for times of 1 to 20 hours.

Some extrusions were subjected to a press quench followed by an ageing treatment to see if the preheat given to the powder billet had homogenised and dissolved the  $Mg_2Si$  phase.

#### 4.5.1. Product Properties

Specimens for mechanical testing and for optical and electron microscopy were cut from each extrude. In each case these specimens were taken from the central region for the sake of consistency. (see Fig 31) The central portion was taken because the beginning of each extrude contained pad material followed by a short length of "improperly" consolidated material and the end of each extrude might have suffered from "back-end" defect.

#### 4.5.2. Hardness Tests

These tests were carried out on the mounted specimens used for optical microscopy. A standard Vickers hardness machine was employed using a load of 2.5 Kg. Hardness traverses across the section of the extrude were performed. A sectioned, partially extruded billet was also traversed longitudinally through the deformation zone to find if there was any correlation between hardness and the extent of consolidation down through the deformation zone.

#### 4.5.3. Tensile Tests

These tests were carried out using No.12 Hounsfield specimens on an Instron Universal Testing Machine. During each test the cross head speed remained constant. The approximate strain rate was calculated from the constant cross head speed and the initial specimen length. (Initial length = length when plastic flow commences). Tests were performed at strain rates varying from 0.0005 - 0.02 sec<sup>-1</sup>, the limiting strain rates produced by the available cross head speeds.

Tests were also performed at temperatures ranging from room temperature to 400°C. For the high temperature tests the specimens and the nimonic rods attached to the cross head were surrounded by a split furnace. A thermocouple placed close to the tensile specimen controlled the heating operation. When the required temperature was reached, the specimen was maintained at this temperature for ten minutes to attain thermal equilibrium before commencing the test. In each case two specimens were tested under each set of conditions and the results averaged. (The small diameter of the 180:1 extrude precluded the manufacture of specimens for this particular extrusion ratio).

#### 4.5.4. Impact Tests

Impact tests on notched specimens were carried out using a Hounsfield balanced impact machine. The notch cross-section, method of supporting the specimen and direction of delivery of the blow were the same as in the standard Izod impact test, but specimen size and shape were different (0.008 mm dia. bar 0.0445m long, compared with 0.01m square bar 0.075m long for an Izod specimen). The energy reading given by this machine was multiplied by a factor of 2.4 in order to obtain the corresponding Izod value.

#### 4.5.5. Torsion Tests

Specimens for torsion testing were taken from extrudes of extrusion ratio 5:1 and 10:1. Plate 3 shows the torsion specimen used in these tests. The gauge length and diameter were such that the maximum strain rate attainable in the torsion machine, having a maximum motor speed of 1000 r.p.m., was  $50 \text{ s}^{-1}$ .

$$\text{Strain Rate} = \frac{2\pi rn}{L}$$

where  $r$  = specimen radius

$L$  = specimen length

$n$  = revs/sec

This was desirable as typical extrusion strain rates employed were of the order of  $10\text{--}50 \text{ s}^{-1}$ . The short gauge length used also increased the homogeneity of deformation during the test. The specimens were heated in situ by an induction heater with the temperature controlled by a thermocouple in the fixed end connected to a eurotherm. Specimens were tested at various temperatures between  $250\text{--}500^\circ\text{C}$  and at various strain rates from  $0.5 - 50 \text{ s}^{-1}$ . After testing to failure, the specimen, enclosed in a sliding, sealed container, was quenched with a water jet to achieve rapid cooling. The torque was measured by a lever arm operating a load cell, the output being fed into a U.V. recorder. A calibration curve of torque versus deflection, was obtained by hanging a known weight on a rod, connected to the load cell, at known distances from the centre of the driving shaft and reading the U.V. deflections. The calibration curve is shown in appendix 6.

#### 4.6.1. Optical Microscopy

Both longitudinal and transverse specimens were cut from the extrudes for examination. These were mounted in slow setting (24 hrs) met. set. This mounting fluid was used as it undergoes no exothermic reaction on

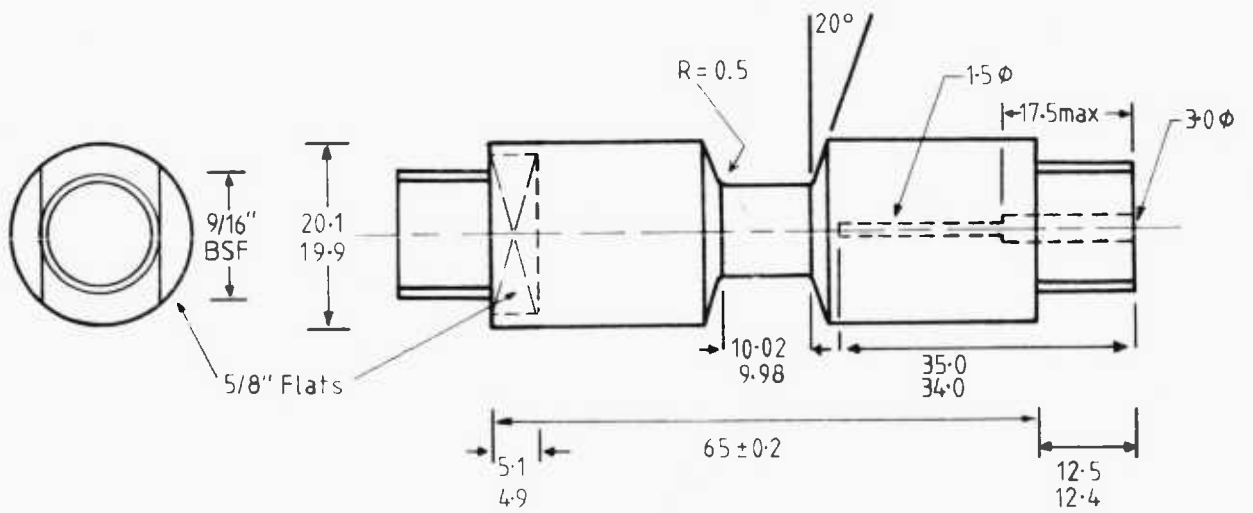
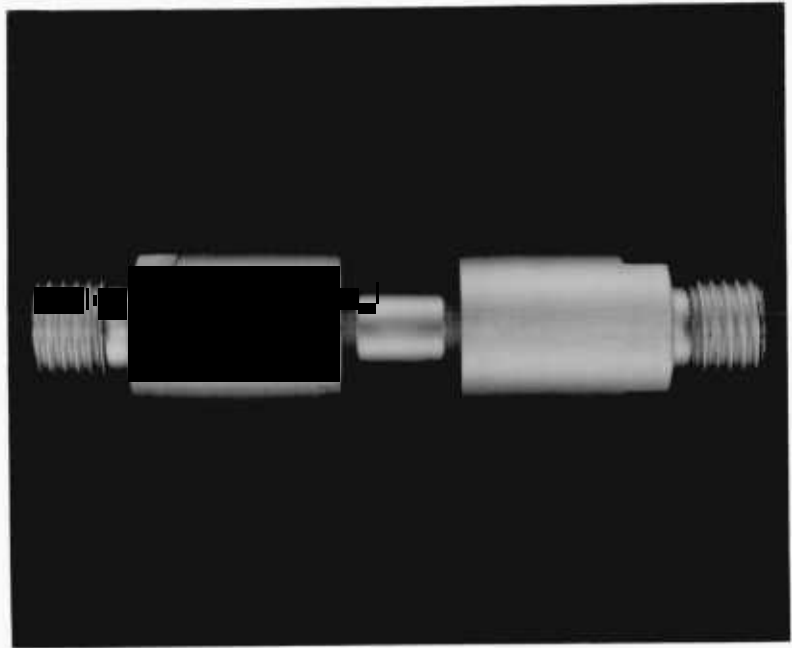


PLATE 3

Torsion Specimen

hardening. The specimens were then ground on silicon carbide paper, polished on a brasso wheel and then finished off on a vibromet automatic polisher containing a slurry of  $0.3\mu$  alumina in distilled water.

The polished specimens of composite material required no etching before examination under the optical microscope. The specimens containing no glass were etched in the usual Aluminium alloy etches i.e. Keller's reagent, Tuckers' reagent and 0.5% HF to show the  $Mg_2Si$  precipitates.

#### 4.6.2. Transmission Electron Microscopy

Longitudinal and transverse specimens were cut from the extrudes using a fine piercing saw to avoid too much heat generation. The specimens were then ground on silicon carbide paper, using a steel grinding block, to a thickness of  $0.0001m$ . A standard "window" technique was then employed to thin the specimens down to foil thickness. A solution of 30% Nitric Acid in methanol was used for the electro-polishing. A potential difference of 15V and a temperature of not greater than  $-30^{\circ}C$  were found to give the best results. A solution of perchloric acid, ethanol and glycerol was also tried under different conditions of temperature and potential difference, and proved to be unsatisfactory.

The reinforced specimens proved impossible to thin properly as the holes left by the fibres caused the foils to disintegrate. The unreinforced HE9, HE30 and Al-Mg specimens thinned reasonably well although some difficulty was experienced with the Al-Mg specimens. The Al specimens were extremely difficult to thin, probably due to their high oxide content.

The transmission microscopy was carried <sup>out</sup> on an AEI EM6 100 Kv electron microscope fitted with a tilting stage.

#### 4.6.3. Scanning Electron Microscopy

As the type of fracture of the tensile specimens was considered to be important, it was decided to examine the fracture surfaces under the scanning microscope. The fractured end of each tensile specimen was sawn off and mounted upon an aluminium stub with araldite. A spot of electrically conducting paint (silver dag) was then used to prevent the specimen from electrically charging up. The stub was then inserted in the microscope and the fracture surface examined as described in 4.2.4.

#### 4.6.4. Partially Extruded Billets

Several extrusions under different conditions werestopped after the "steady state" region of the load-time graph had been reached. These partial billets were then pushed out, sectioned, polished and etched in an attempt to observe the flow pattern. However, it was found that due to the very fine initial powder particle size no flow pattern could be easily distinguished.

To overcome this difficulty several powder billets containing copper discs placed 0.01m apart in the compaction die were compacted and partially extruded. These billets, when sectioned and polished, gave a much better representation of the flow pattern existing during extrusion. One partial extrusion of a composite billet containing no copper strips was examined after sectioning and polishing. Etching was unnecessary and the formation of the fibres through the deformation zone was clearly visible.

Several partially extruded billets were sectioned and a 0.0015m thick slice machined from one half of each for preparation of electron microscope foils.

## CHAPTER 5

## RESULTS AND DISCUSSION

5.1.1 Sieve analysis of metal powders

The sieve analyses for the HE9, HE30, Al-Mg and Al powders are given in tables 1-4 (see end of chapter for tables), together with the median particle sizes. The figures shown represent, on average, the results of six sieving operations. As the particle size distribution has such a marked effect on the subsequent extrude structure and properties, a knowledge of this distribution is vital. It must also be remembered that a meaningful comparison of results with those obtained by other workers in the powder metallurgy field is possible only if the size distribution for each powder used is available.

5.1.2 Sieve analysis of glass powder

The sieve figures for the  $B_2O_3$ -10%  $SiO_2$  glass powder used throughout the investigation are given in table 5. The median particle size of the glass is much smaller than that of the metals ( $74\mu$  compared to  $110$ - $150\mu$ ). This is advantageous as smaller glass particles have a greater tendency to elongate without fracturing in the deformation zone during extrusion.

5.1.3 Chemical analysis

The chemical analyses of the metal powders are given in table 6. The most surprising feature is the large difference in oxygen content between the Al-Mg and Al powders, both of which were produced by water atomization. Considerable differences must have existed in the atomizing conditions to produce this effect. From the sieve figures given in tables 3 and 4 it is evident that the Al powder is somewhat coarser. Now as smaller particles cool faster it is reasonable to assume that the longer "flight path" of the larger Al particles, during

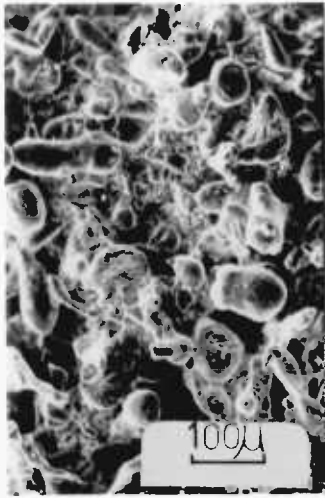


the atomization process, allows a greater amount of oxidation to occur. i.e. the larger particles cool more slowly and therefore enjoy a greater time at temperatures which permit oxidation. It should also be remembered that smaller particles have a greater surface area: volume ratio (i.e.  $4\pi r^2 / \frac{4}{3}\pi r^3 \uparrow$  as  $r \downarrow$ ) and therefore have more surface area available for oxidation. Clearly, from the figures in table 6, the surface area consideration is far out-weighed by the cooling rate effects causing the Al powder oxide content to be far larger than that of the Al-Mg. The higher melting point of the Al (60°C higher than that of Al-3 wt.% Mg) may also have necessitated a higher atomization temperature, leading to even greater oxidation.

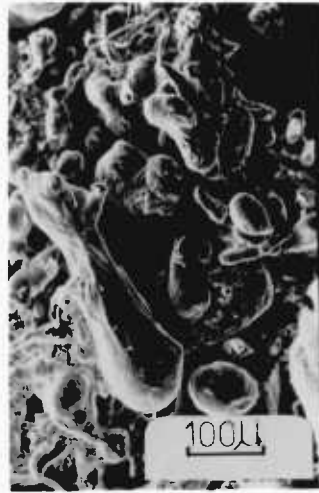
#### 5.1.4 Shape of powder particles

The scanning electron micrographs in Plate 4 show the shape of the individual powder particles of the four types of metal powder investigated. All four materials exhibit "tailing" which is a feature commonly produced by atomization processes using horizontal blasts. Chare<sup>19</sup> found this effect in aluminium powders produced in a horizontal blast air atomizer. He also noted an absence of "tailing" in powders produced using a vertical air blast.

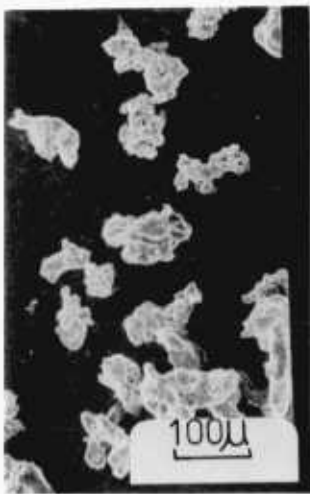
The shape and size of powder particles produced by atomization depend upon the nature of the impinging gas or water jet and on the geometrical configuration of the apparatus. Long cooling times are required to produce spherical powders. Slow cooling rates permit surface tension forces to minimize the surface area, producing spheres. However, to prevent particle coalescence, the cooling rate must be great enough to ensure sufficient solidification prior to deposition. Gurney et al<sup>24</sup> produced remarkably spherical 7075 aluminium alloy powder using a rotating electrode process. In fact, for compacting



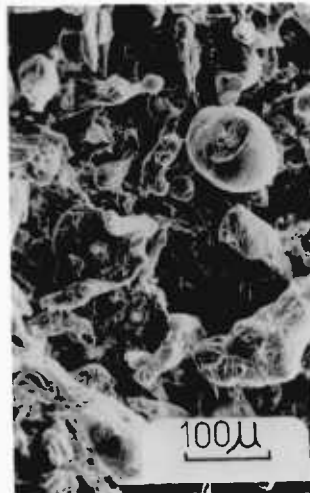
(a)  
HE9



(b)  
HE30



(c)  
Al



(d)  
Al-Mg

PLATE 4

Scanning electronmicrographs of powders

purposes, spherical particles are less desirable than irregularly shaped particles with "tails". This is because spherical particles provide the minimum surface area for interparticle bonding. Irregularly shaped particles compact to a higher density due to bridging effects i.e. particle "tails" fill irregularly shaped voids. Thus compacts made from irregular particles are stronger. On the other hand sintering rates for spherical particles are believed to be higher than for irregular particles. Thus, for compacts produced by pressing and sintering, the optimum particle shape lies somewhere between the two extremes of completely spherical and completely irregular.

#### 5.1.5 Structure of powder particles

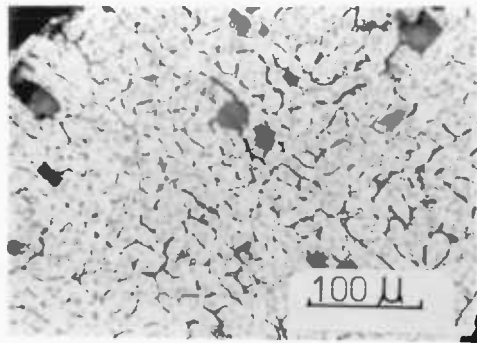
The presence of a very fine structure, dendritic in nature, in the HE9, Al-Mg and Al powders is revealed by optical micrographs (a), (b), and (c) respectively in Plate 5. This structure (also present in the HE30 powder) is caused by the high solidification rate present in the atomization process. Gurney et al<sup>24</sup> found similar structures in 7075 aluminium alloy powder produced by the rotating electrode process.

#### 5.1.6 Strength of compact

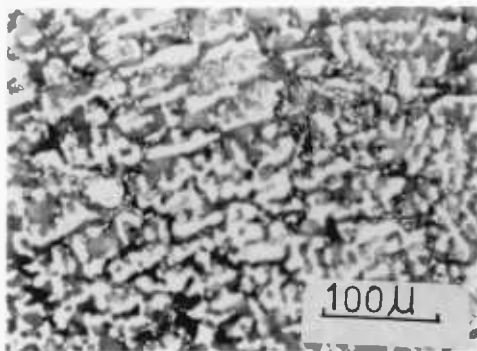
The results of the experiments to determine the effects of:-

- (1) compaction pressure
- (2) sintering temperature
- (3) powder particle size

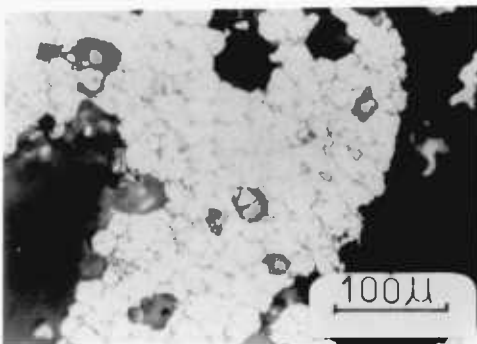
on compaction strength are given in tables 7-9. The theoretical densities mentioned in table 7 are calculated by expressing the density of the compact, found from volume and weight measurements, as a percentage of the density of cast HE9 + 10 wt.% glass ( $2850 \text{ kg m}^{-3}$ ).



(a)  
HE9



(b)  
Al-Mg



(c)  
Al

A linear relationship between theoretical density and "fracture force" (see 4.3.2) is demonstrated in Fig. 32. However, a correlation coefficient of 0.948 indicates that the experimental points fit an equation of the form

$$\text{Force} = \text{const}' + \text{density} \times \text{const}'' ,$$

only approximately. The results are replotted in Fig. 33 in a form which tests the applicability of the relationship suggested by Konopicky<sup>5</sup>:-

$$\ln \left( \frac{1}{1-D} \right) = K'P + \text{constant} \quad (1)$$

where D = relative density

P = compact pressure

K' = constant.

In this case the equation is:-

$$\ln \left( \frac{1}{1-D} \right) = 0.293 \times 10^{-2} P + 1.349$$

A correlation of 0.985 indicates that the data points fit equation (1) quite well. Over the range of pressures investigated the traditional three stages of compaction (see 2.1.1) are not discernible. Stage 1 compaction i.e. a decrease in volume without particle deformation, is not present as this involves lower pressures than those investigated. Chare<sup>19</sup> encountered stage 1 compaction only at pressures of less than 50 MN/m<sup>2</sup>, for Al-Fe alloy powder. The absence of stage 3 compaction i.e. bulk compression of the compact, is due to the relatively low pressures used. Thus Fig. 33 represents stage 2 compaction only i.e. the elastic-plastic deformation of particles.

Several billets, compacted at the same pressure, were sintered at various temperatures. The effect of sintering temperature on compact strength is shown in

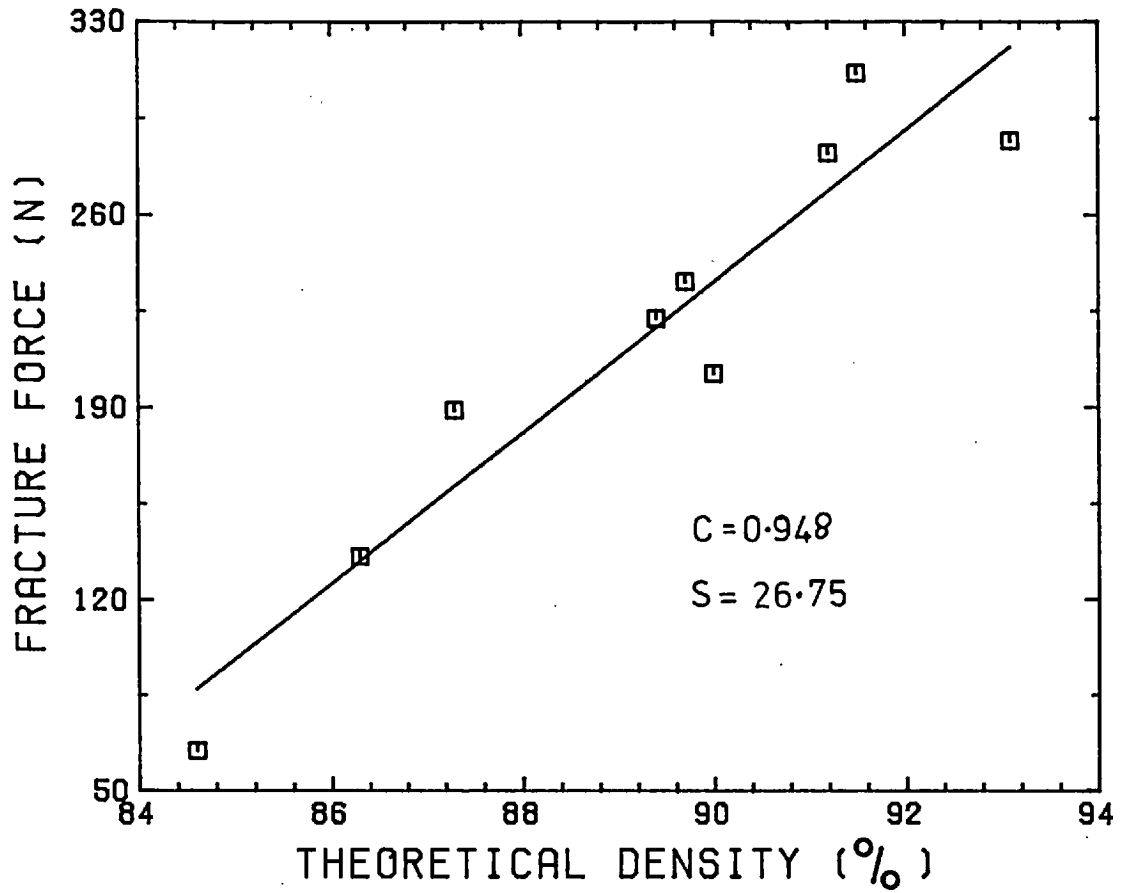


FIG. 32 Compact fracture force as a function of theoretical density

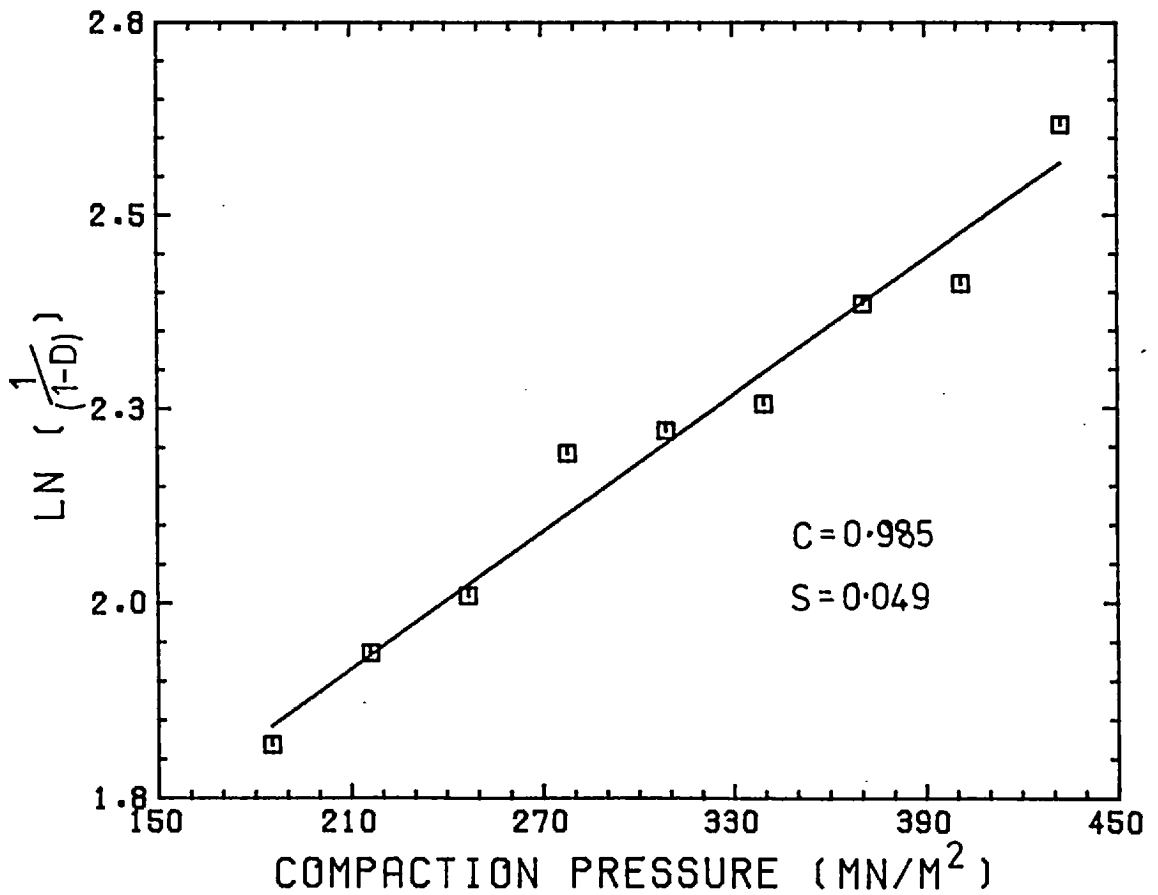


FIG. 33 Relative density of compact as a function of compaction pressure

Fig. 34. Billet strength increases rapidly with sintering temperature in the range 250-450°C. Below 250°C the rate of increase is considerably less. This temperature dependence is to be expected as the rate of diffusion increases rapidly with temperature according to Darken's equation<sup>108</sup>:-

$$J_i = -B_i kT \frac{dC_i}{dx}$$

where T = temperature  
 $J_i$  = rate of self-diffusion  
 k = Boltzman's constant  
 $B_i$  = mobility  
 $\frac{dC_i}{dx}$  = concentration gradient

The effect of particle size on compact strength was also investigated. A decrease in compact strength with increasing particle size is evident in Fig. 35. The rate of decrease in strength diminishes with increasing particle size. This behaviour is fairly easily explained. As the particle size decreases the surface area/volume ratio increases thus providing more surface area for interparticle contact. Thus the total interparticle bonding area increases, improving the compact strength.

As mentioned in Chapter 2, previous investigators have not attempted to relate compact strength directly to compaction pressure. In the present work although only a limited amount of data was available, it was decided that an attempt to relate fracture force to compaction pressure would be useful. Fig. 36 reveals that a second degree polynomial fits the data points reasonably well. The equation of the line is:-

$$F = -326 + 2.77P - 0.003P^2$$

where F = fracture force  
 P = compaction pressure

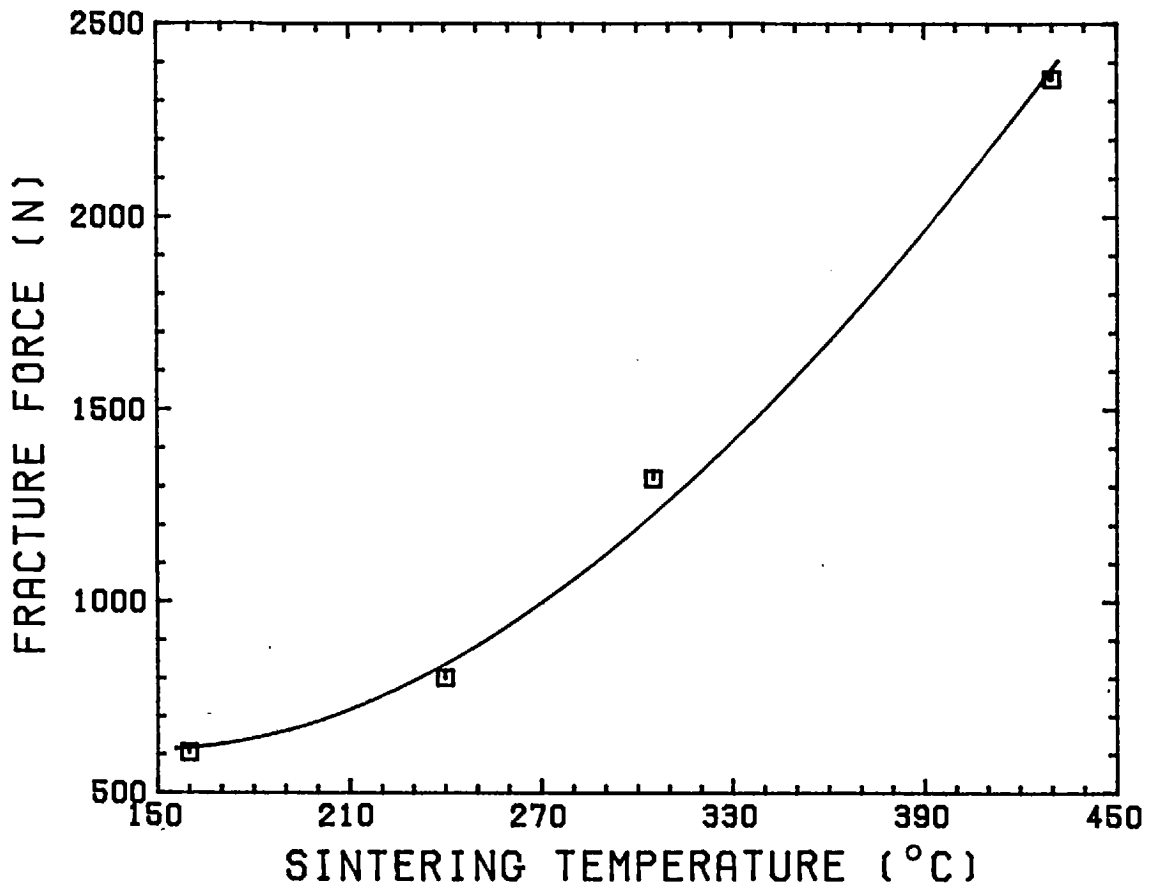


FIG. 34 Relationship between compact fracture force and sintering temperature

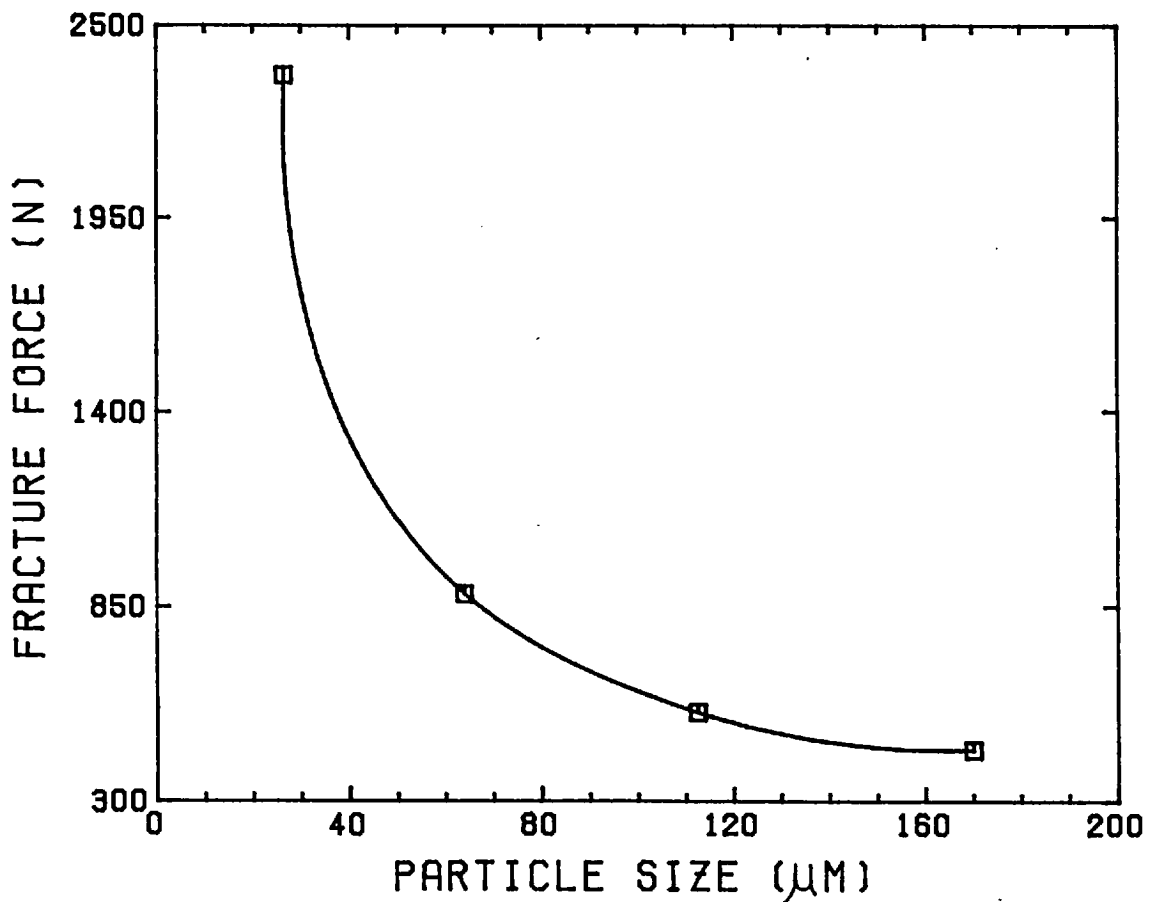


FIG. 35 Dependence of compact fracture force on powder particle size



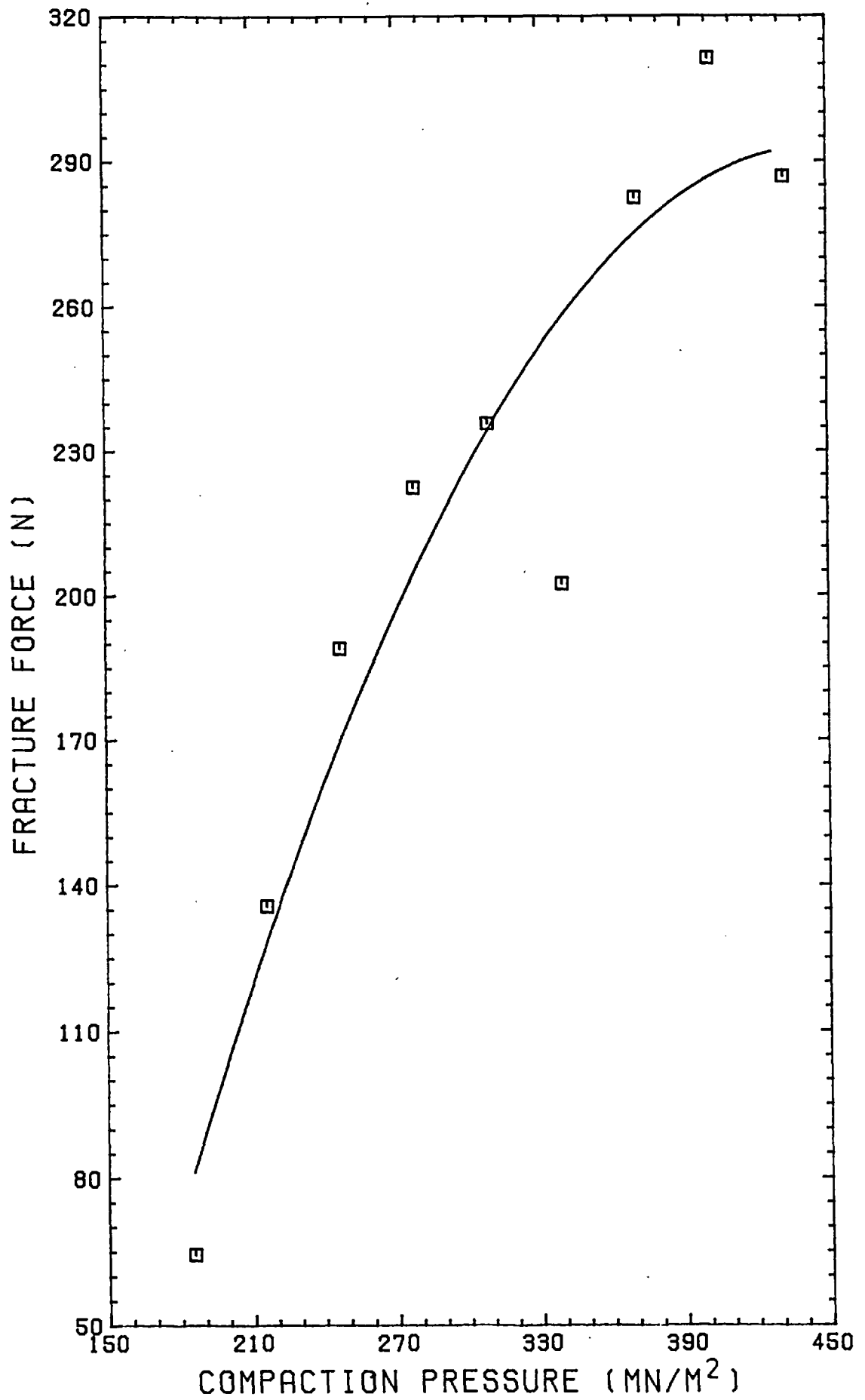


FIG. 36 Relationship between compact fracture force and compaction pressure

It must be emphasised that this equation is the result of only seven compactions carried out at room temperature.

### 5.2.1 Theoretical prediction of extrusion loads

The theory discussed in (3.2.3) enables the extrusion load to be predicted by the use of one of two solutions:-

either (a) a minimised plane strain upper bound solution  
or (b) a minimised plane strain solution modified for axisymmetric extrusion.

From the results of torsion tests (5.5.1) and tensile tests (5.9.1, 5.9.3 and 5.9.4) equation (17) becomes:-

$$K_{ij} = \frac{40}{\sqrt{3}} \frac{\dot{\epsilon}}{\epsilon} 0.1 \frac{\dot{\epsilon}}{\epsilon} 0.21$$

Extrusion pressure plotted as a function of extrusion ratio, in Fig. 37, enables the loads predicted by the two solutions mentioned above to be compared to the experimental results for a series of HE9 extrusions. A very large discrepancy exists between the pressures predicted by the modified solution for axisymmetric extrusion and those observed in practice. The predicted values are generally 50% less than the experimental values. Clearly this method is of little use. In fact the plane strain solutions give more reasonable results. The predicted slope of this curve is, however, manifestly incorrect. For both high (> 55:1) and low (< 25:1) extrusion ratios the theory is unacceptable. When extruding cast billets this method usually predicts pressures which are ~20% above those actually obtaining. One of the main reasons why the two methods mentioned above give such unsatisfactory results is that although the general form of equation (17) is correct, the strain and strain rate exponents cannot be accurately determined by simulated tensile and/or torsion tests in the case of powder extrusion. This is because:-

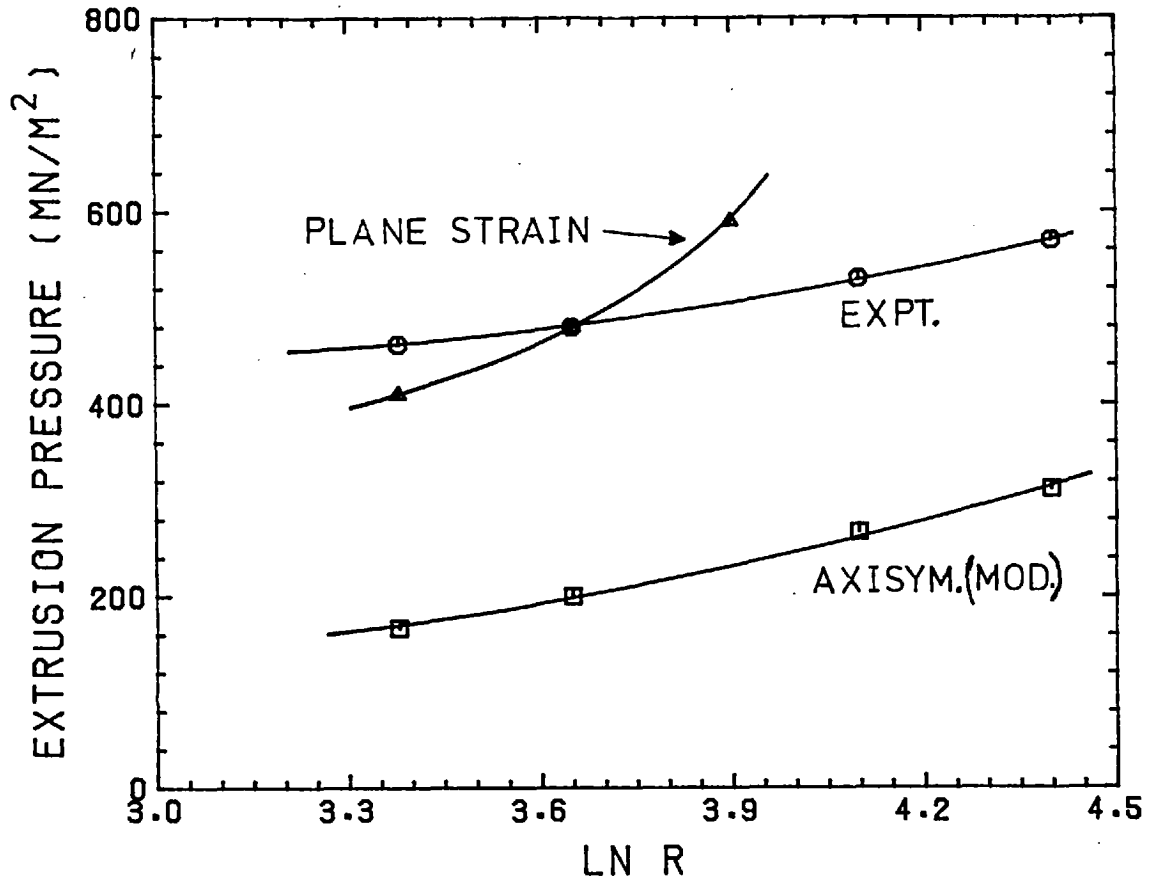


FIG. 37 Comparison between experimentally measured HE9 powder extrusion pressures and theoretically predicted values (plane strain upper bound)

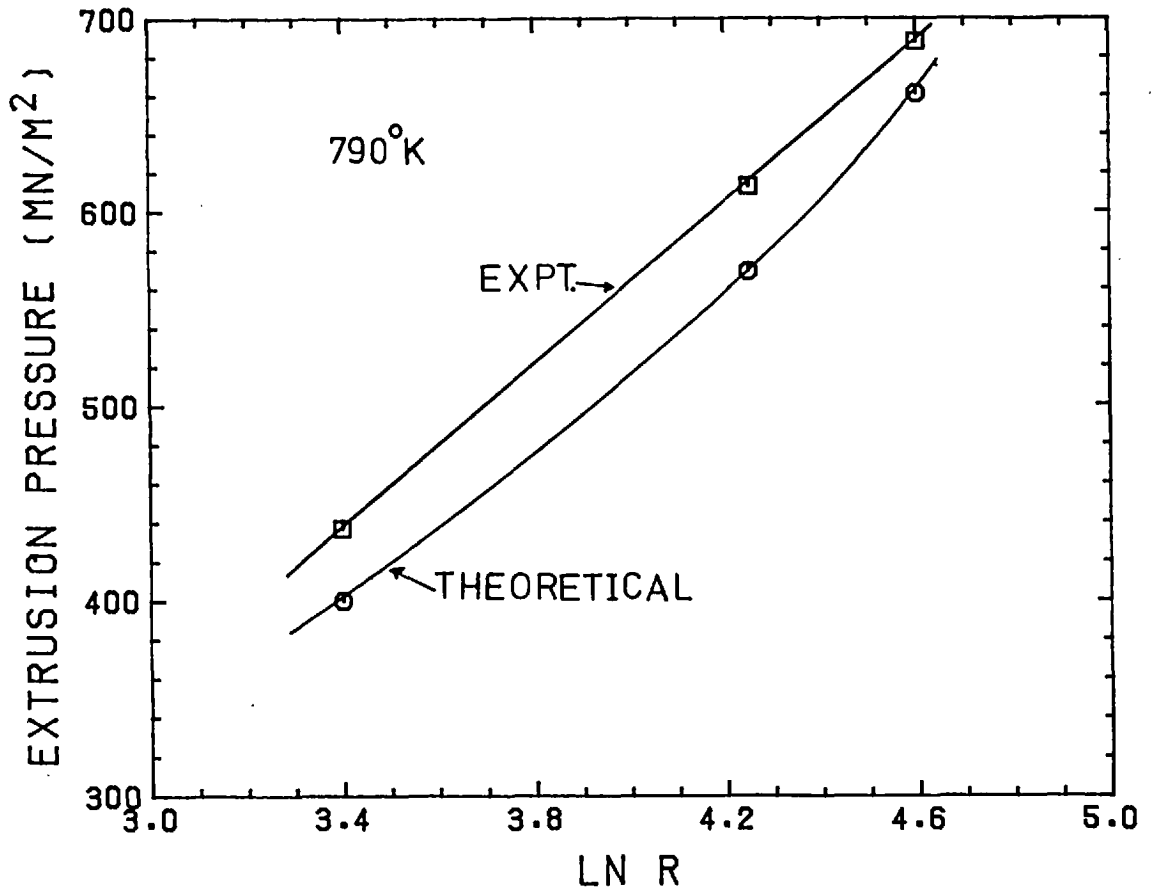


FIG. 38 Comparison between measured extrusion pressures for cast aluminium alloy and theoretically predicted values (elemental axisymmetric upper bound)

- (1) The compressive and shear stress must be simultaneously present in order to achieve the welding and rewelding processes associated with consolidation of the material.
- (2) The material used in laboratory tests is the extruded product and possesses properties different to those of the powder compact before it negotiates the deformation zone.

For these reasons the exponent values in (17) must be calculated as part of the optimization procedure associated with the upper bound approach.

This is possible using the theory in 3.4.1. Before applying this theory to powder compact extrusion it is necessary to establish that it adequately predicts extrusion pressures for known flow stress conditions. Wood and Sheppard<sup>128</sup> have shown that for an as-cast aluminium copper alloy subjected to torsion testing the flow stress is given by

$$\bar{\sigma} = 62.5 \ln \left[ (0.087\dot{\epsilon})^{0.23} + \left\{ (0.087\dot{\epsilon})^{0.46} + 1 \right\}^{\frac{1}{2}} \right]$$

(at 790°K)

This equation was substituted in the program given in appendix 3 to calculate the flow stress. The predicted pressures thus obtained are compared with the experimental results of Wood in Fig. 38. It can be seen that the axisymmetric upper bound approach, when used in conjunction with a suitable optimization process, predicts pressures which are fairly near the experimental values.

Earlier workers<sup>35,36,117</sup> have assumed a constant mean flow stress when evaluating the rate of internal energy dissipation although Halling and Mitchell<sup>36</sup> suggested a method enabling the effects of work hardening to be considered. In the present study the rate of energy

dissipation is determined for each element since the flow stress is considered to be both strain and strain rate sensitive. The deforming region was divided into 50 elements representing a 10 x 5 matrix after ascertaining the fact that a 800 element matrix improved the answer by only 1.2%. The use of the coarser matrix results in a considerable saving in computer time.

The experimental powder extrusion pressures for HE9 are compared with those predicted by this theory over a range of extrusion ratios from 10:1 to 180:1 in Fig. 39. The optimum values of A, n and m (equation 17) are 45, 0.02 and 0.05 (at 780°K). Clearly excellent agreement exists between predicted and experimental pressures over a wide range of extrusion ratios (5:1 to 180:1). For most aluminium alloys the value of the strain rate exponent, n, varies from 0.18 to 0.25 in the hot working region. The present value of 0.02 indicates that powder compacts are very much less strain rate sensitive than their cast counterparts. This is further evidence in support of the thesis of Sheppard and Chare that the predominant consolidation mechanism in the deformation zone is welding of particle necks, shearing and rewelding as a continuous process resulting in final product properties being achieved only near the exit of the zone. Fig. 40 demonstrates that the theory works equally well for HE30. In this case the optimum values of A, n and m are 55, 0.03 and 0.05 respectively (at 800°K). The low n value once again re-emphasies the argument concerning the consolidation mechanism.

Other workers have shown that for Al powder<sup>123</sup> and 7075 Al alloy powder<sup>24</sup> extrusion a natural strain of 2.3 is required for complete densification and consolidation of the product. The computer program which calculates the theoretical extrusion pressure also predicts that 70% of the deformation zone experiences a total strain of less than 2.3, even at an extrusion ratio of 80:1 (Fig. 41). This also indicates that final product properties

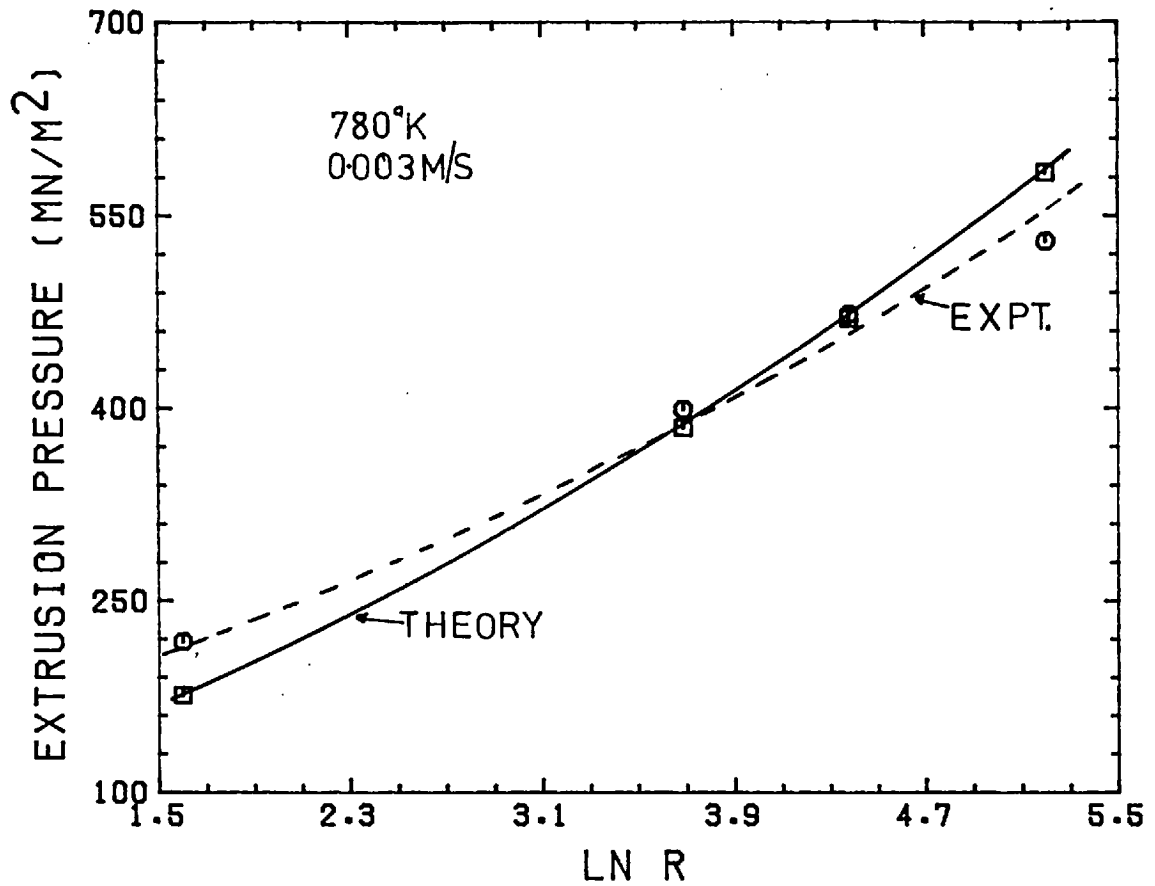


FIG. 39 Comparison between measured HE9 powder extrusion pressures and theoretically predicted values (elemental axisymmetric upper bound)

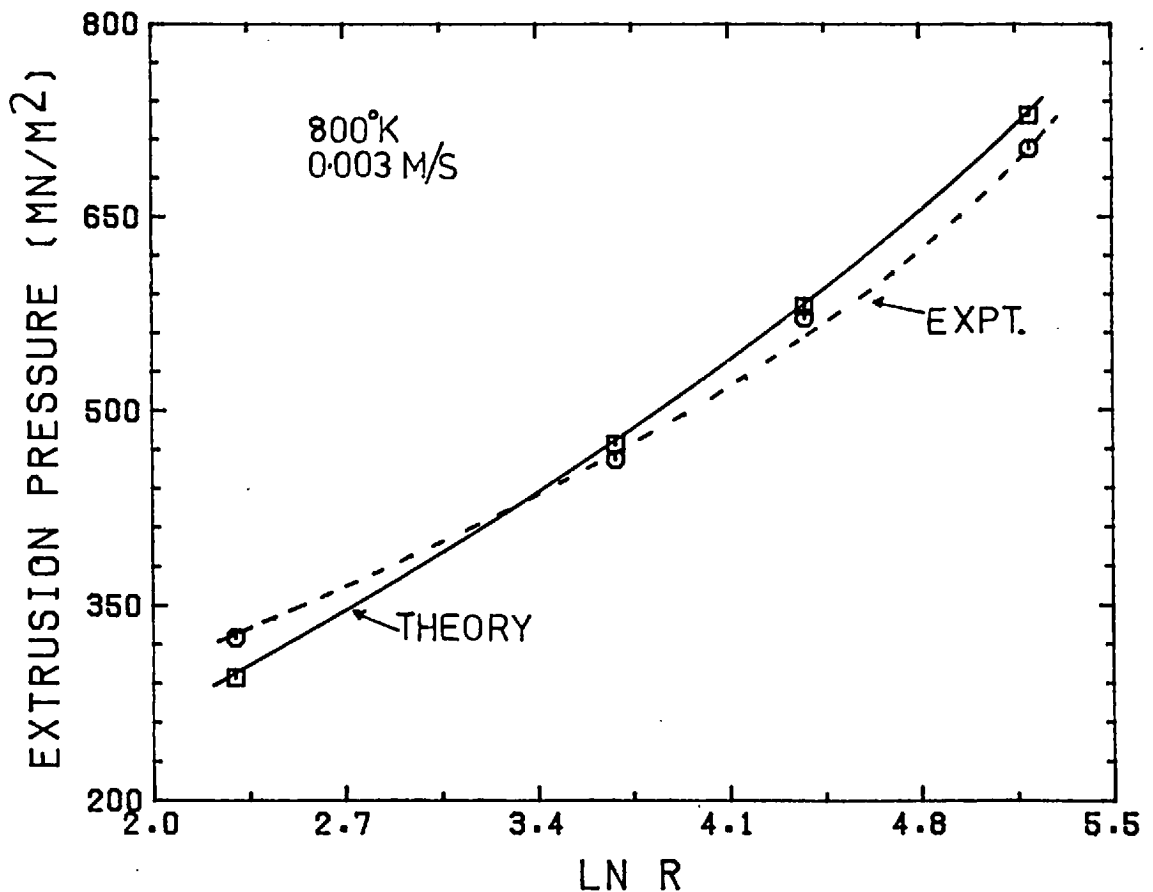


FIG. 40 Comparison between measured HE30 powder extrusion pressures and theoretically predicted values (elemental axisymmetric upper bound)

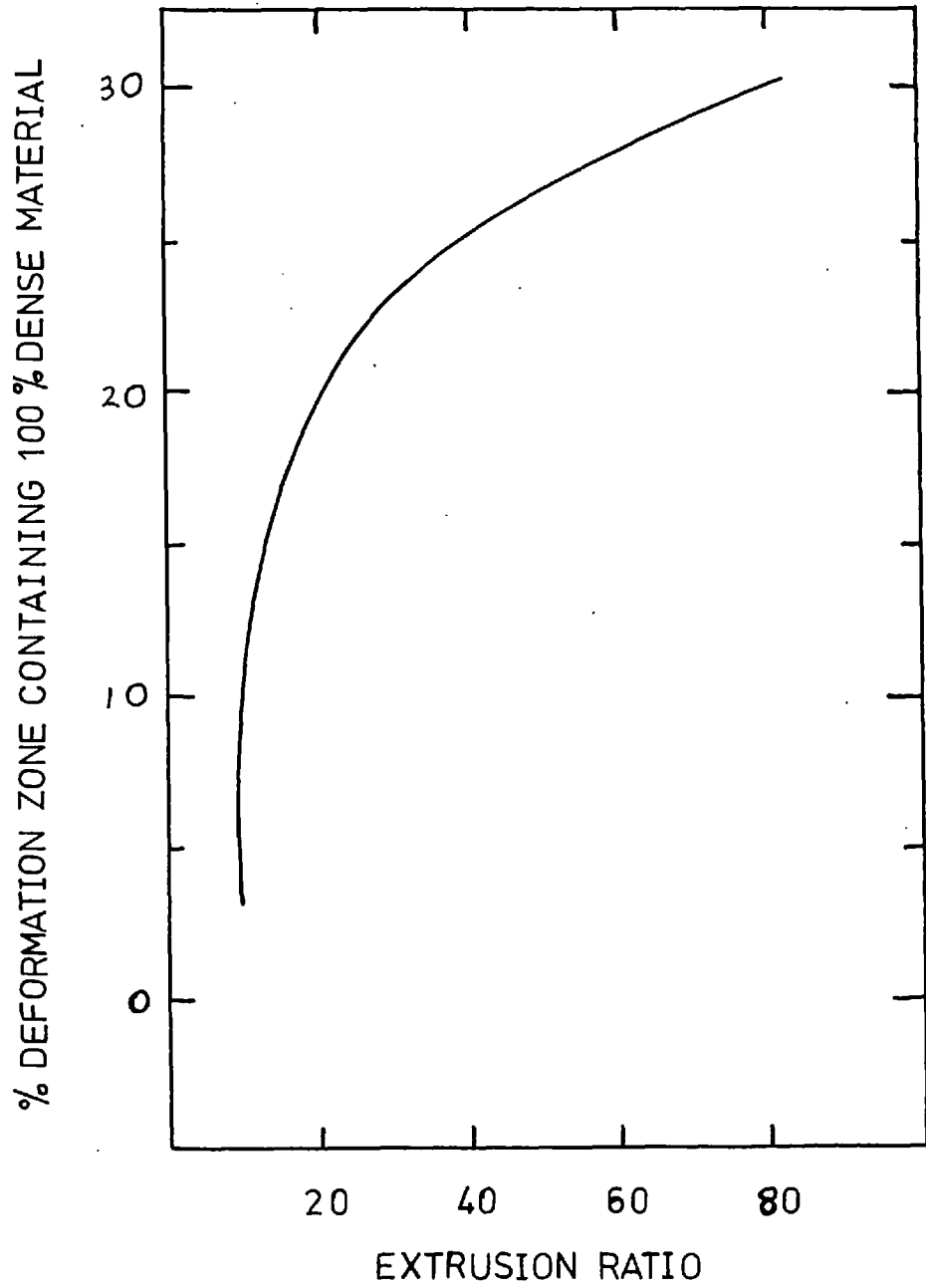


FIG. 41 Theoretically predicted effect of extrusion ratio on densification

are not achieved until far into the deformation zone. Fig. 42 shows how the predicted values of angles  $\alpha$  and  $\delta$  (Fig. 11) vary with extrusion ratio. The value of  $\delta$  increases continuously with extrusion ratio but  $\alpha$  behaves in a more mysterious way, falling to a minimum value at 40:1 and then increasing again.

The program may also be used to predict P/K values at various extrusion ratios by equating A, n and m to  $\sqrt{3}$ , 0 and 0 respectively. In Fig. 43 the values thus predicted are compared with those produced by Halling and Mitchell's single triangle field axisymmetric solution and with the values produced by the plane strain, double triangle field solution. Clearly the P/K values given by the present analysis are slightly better than those of the axisymmetric analysis, over the entire range of ratios from 5:1 to 180:1. The plane strain solution does yield lower P/K values at ratios below 20:1. However, above this ratio the P/K values predicted increase alarmingly, until at 180:1, the value is 100% larger than the answers given by the other two analyses.

### 5.3.1 Strain rate prediction (from upper bound solution)

The program in appendix 3 calculates the strain rate within each element of the deformation zone (in addition to the strain rate at each discontinuity). Therefore by introducing print statements into the program the strain rate at the centroid of each element may be obtained. For a 10x5 matrix, an average strain rate acting along each of five stream lines can be calculated. (The stream line is assumed to pass through the centroids of a row of elements). i.e.

$$\bar{\epsilon}_1 = \sum_1^{10} \epsilon_i d_{l_i} / L_1 \quad (\text{see fig. 44})$$

A mean overall strain rate,  $\bar{\epsilon}_T$ , may then be obtained thus:-

$$\bar{\epsilon}_T = \sum_1^5 2\pi r_i d_r \bar{\epsilon}_i / \pi R_B^2$$



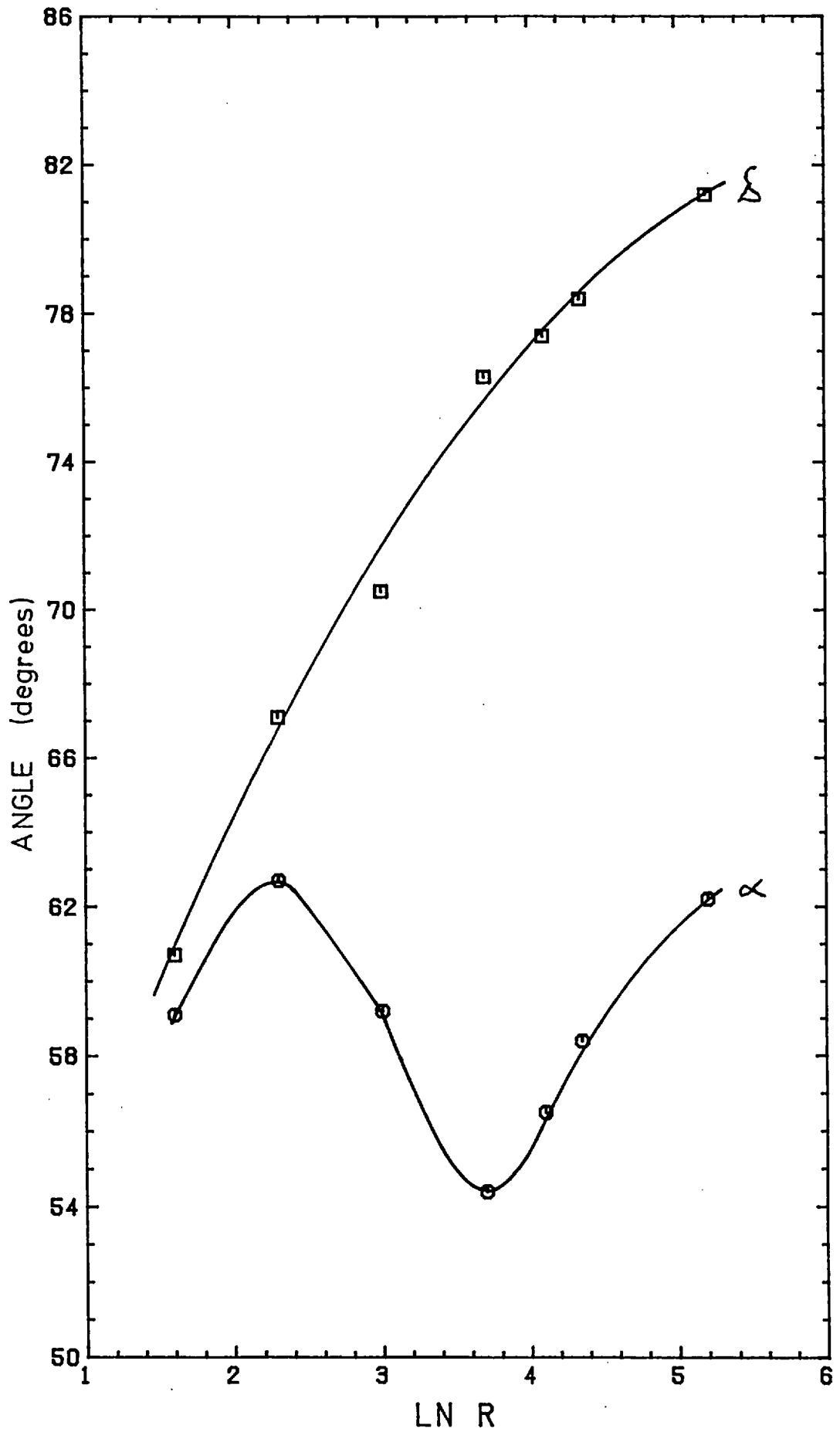


FIG. 42 Theoretically predicted variation in velocity field angles with extrusion ratio

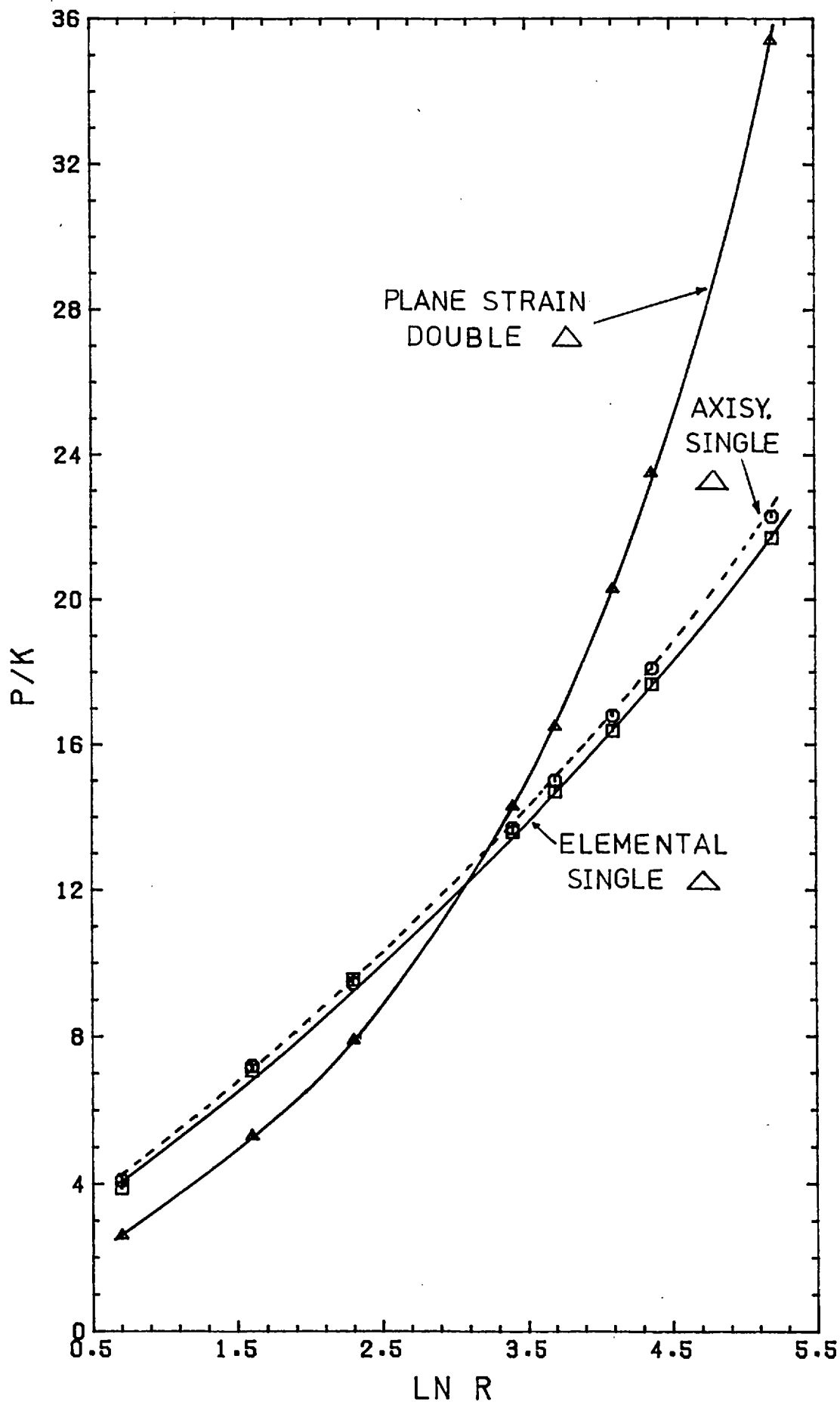


FIG. 43 Variation in predicted P/K values with extrusion ratio for the three upper bound solutions

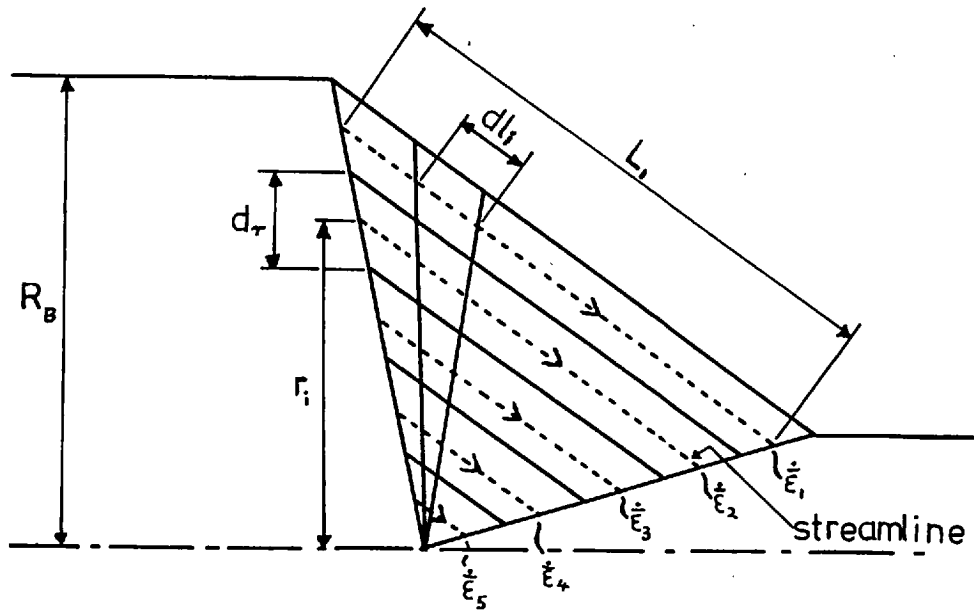


FIG. 44 Upper bound model for strain rate prediction

The results obtained by this method are plotted against  $\ln R$  in Fig. 45. They are compared with those predicted by Feltham's equation at two ram speeds, 0.003 m/sec and 0.013 m/sec. At both speeds the strain rate predicted by the upper bound solution is smaller at all ratios below 180:1. The agreement between the two theories seems to be relatively independent of the ram speed but clearly improves with increasing extrusion ratio. (At some ratio in excess of 180:1 the curves seem destined to intersect).

#### 5.4.1 Extrusion parameter measurements

The following parameters were measured for each extrusion and are given in table 10:-

- 1) Extrusion ratio
- 2) Ram speed
- 3) Billet temperature
- 4) Glass content
- 5) Extrusion pressure

The calculated values of flow stress, strain rate and final extrude temperature are also listed.

Typical load-displacement curves for two HE9 powder billets and a solid billet of the same alloy composition (excluding  $\text{Al}_2\text{O}_3$  content) are shown in Fig. 46. The curves differ significantly in the initial stages of the process i.e. while the quasi-static deformation zone is being established. The cast billet, B, exhibits the normal linear region during upsetting prior to the achievement of the peak load. The powder billets, A and C, show non-linear compaction zones. Sheppard<sup>123</sup> reasoned that this corresponded to the compact obtaining 100% density. It should be clearly understood that the attainment of the theoretical density does not coincide with the attainment of optimum mechanical properties. In fact, at this stage, the billet is brittle and has practically no resistance to

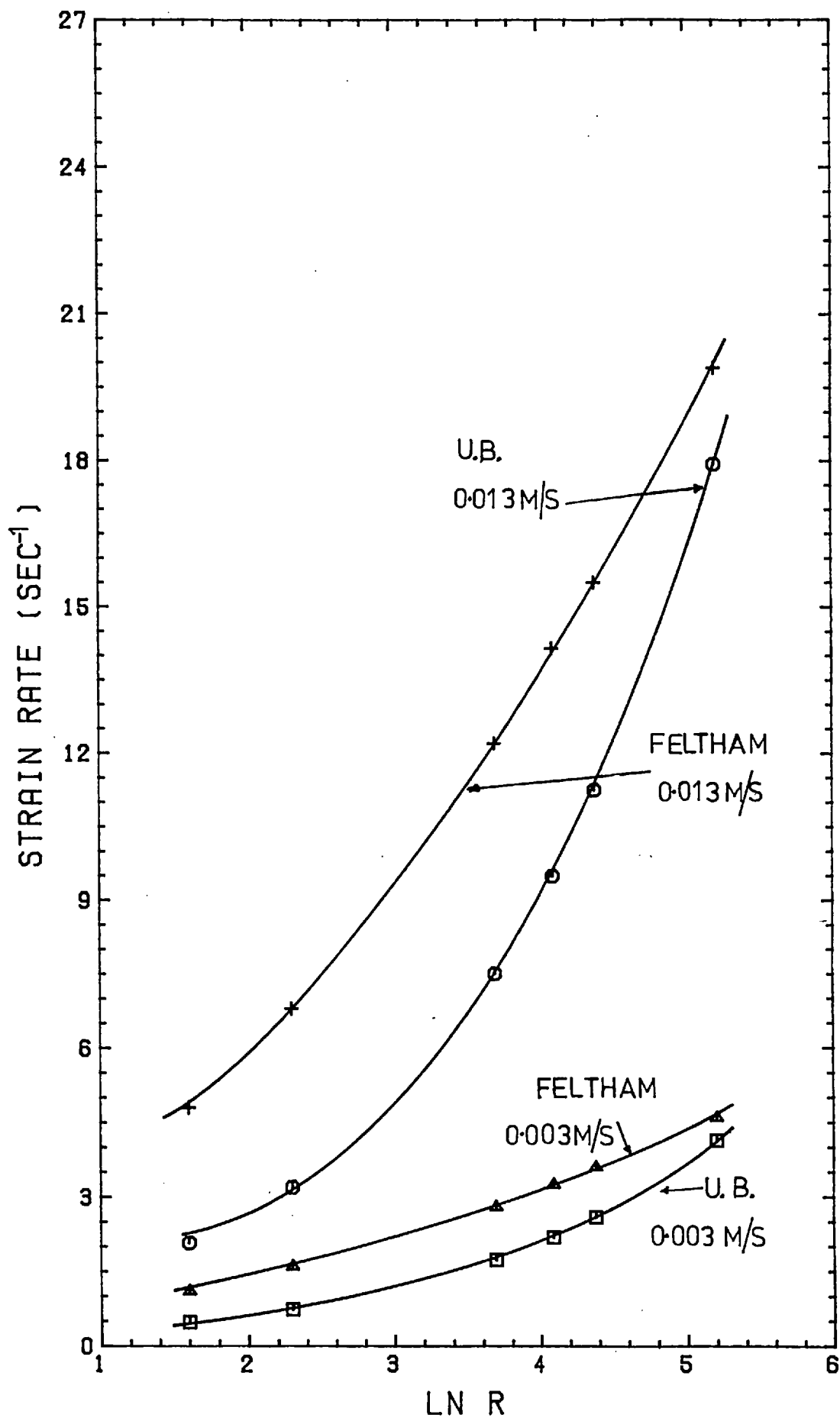


FIG. 45 Comparison between upper bound and Feltham strain rates predicted for various ram speeds and extrusion ratios

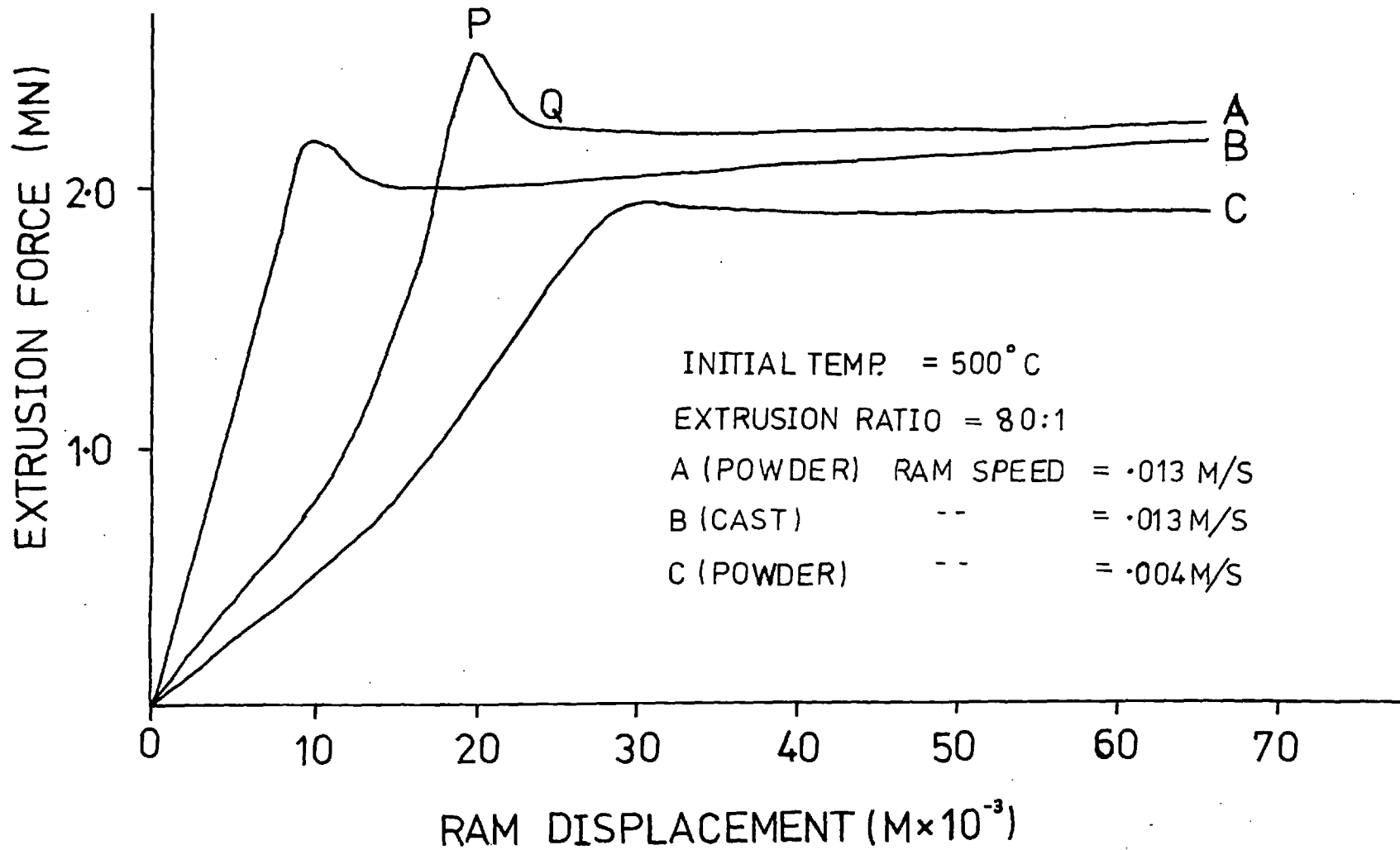


FIG. 46 Experimentally recorded extrusion load vs. ram displacement curves

shear. The mechanical properties of forged<sup>120</sup> metal powder billets are generally much inferior to those of extruded powder billets. This evidence is clearly in agreement with the thesis<sup>135</sup> that deviatoric deformation (only a limited amount of which is present in forging) is a prerequisite for the achievement of acceptable properties and the powder billet cannot obtain optimum properties until it has completely traversed the deformation zone present during extrusion.

The fact that a peak exists on curve A but not on curve C is interesting and is discussed in some detail in the next section (5.4.2).

On first examining the curves A and B, in Fig. 46, the hypothesis that powder compacts require lower extrusion loads than cast billets of the same composition seems improbable. However, it must be remembered that the cast billet, B, does not possess the high  $Al_2O_3$  content of the powder compact. The flow stress of extruded material containing this oxide dispersion is increased considerably by its presence. In Fig. 47 the curves A and B are re-plotted in a different form to allow for this variation in flow stress. The extrusion pressure readings are made dimensionless by dividing them by the 0.2% proof stress values. The "dimensionless pressure" required to extrude the cast billet is clearly much higher. This tends to support the thesis that a cast billet with the same oxide content as the powder would require much higher extrusion loads than those shown by curve A in Fig. 46.

#### 5.4.2 Effect of extrusion parameters on load

The most widely used relationship between load and extrusion ratio was first suggested by Johnson:-

$$P/2K = A + B \ln R \quad (3)$$

where P = pressure                      K = shear flow stress  
        R = extrusion ratio                A, B = constants.

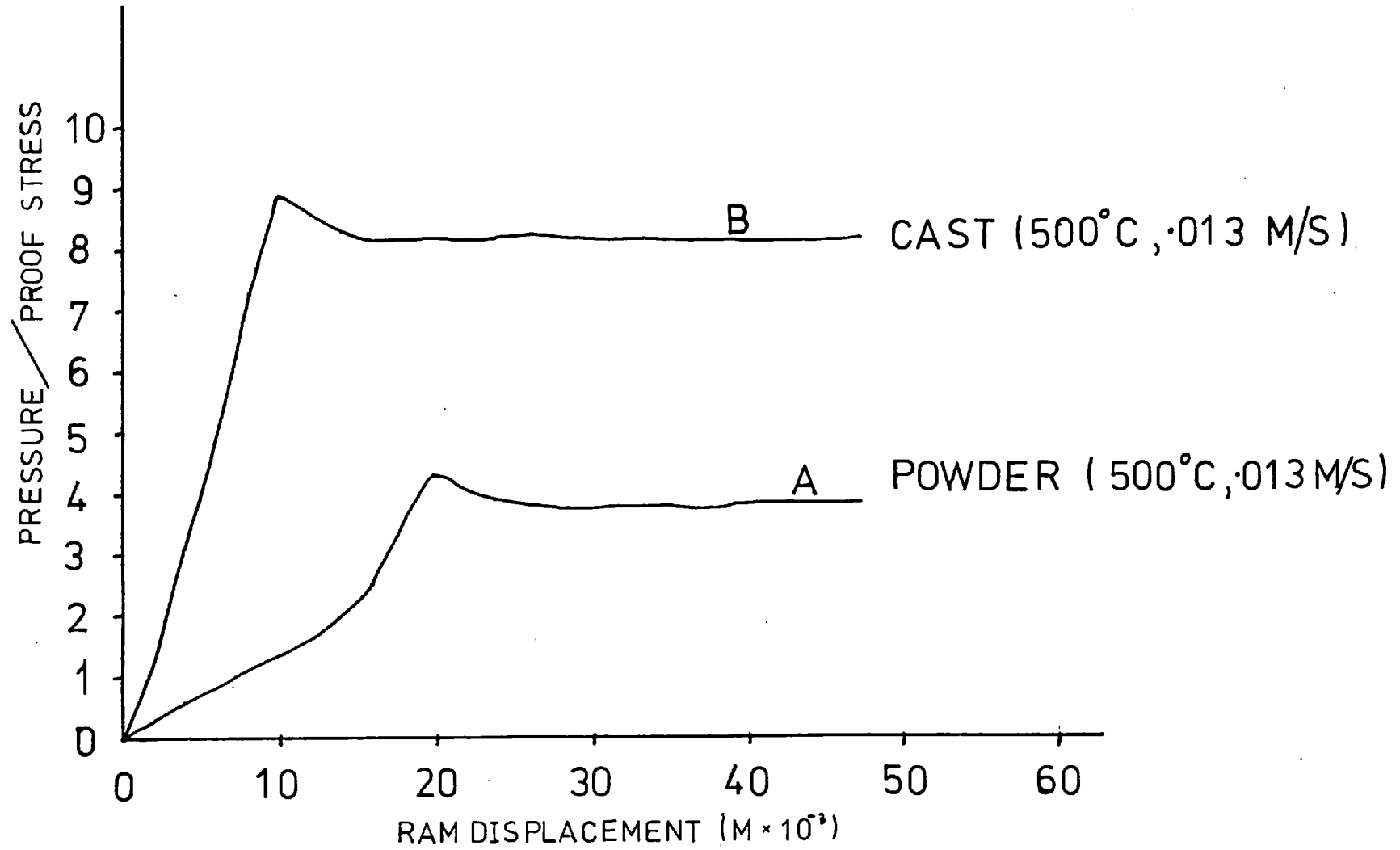


FIG. 47 Dimensionless extrusion pressures vs. ram displacement curves for powder and cast billets



This relationship has been shown to hold for solid billet extrusion of many alloys. However, in the case of powder extrusion, the value of K varies throughout the deformation zone. In order to use equation (3) a mean value of K must be employed. The equation may be written:-

$$P = A' + B' \ln R$$

where A', B' contain a mean shear stress term.

Figs. 48-50 demonstrate that linear relationships between extrusion pressure and extrusion ratio do exist for the extrusion of HE9, HE30 and HE9-5% glass powders, at various temperatures, over the extrusion ratio range 5:1 to 180:1. Volmer<sup>121</sup> discovered a similar relationship for iron powder compact extrusion. Various workers have found that this relationship breaks down at ratios of less than ~5:1 for aluminium alloy powders<sup>123,124</sup> and for high speed steel powders<sup>22</sup>. This breakdown is due to incomplete consolidation of the extrude at these low ratios.

For cast billets of HE9 alloy the relation between pressure and extrusion ratio at 540°C is:-

$$P/\sigma = 0.52 + 1.32 \ln R \quad (87)$$

The equation for HE9 powder compact extrusion, at 540°C, is:-

$$P = 71.8 + 82.5 \ln R \quad (88)$$

For comparative purposes it is convenient to write equations (87) and (88) in the form:-

$$P/B' = 0.39 + \ln R \quad \text{cast billet} \quad (89)$$

$$P/B' = 0.87 + \ln R \quad \text{powder billet} \quad (90)$$

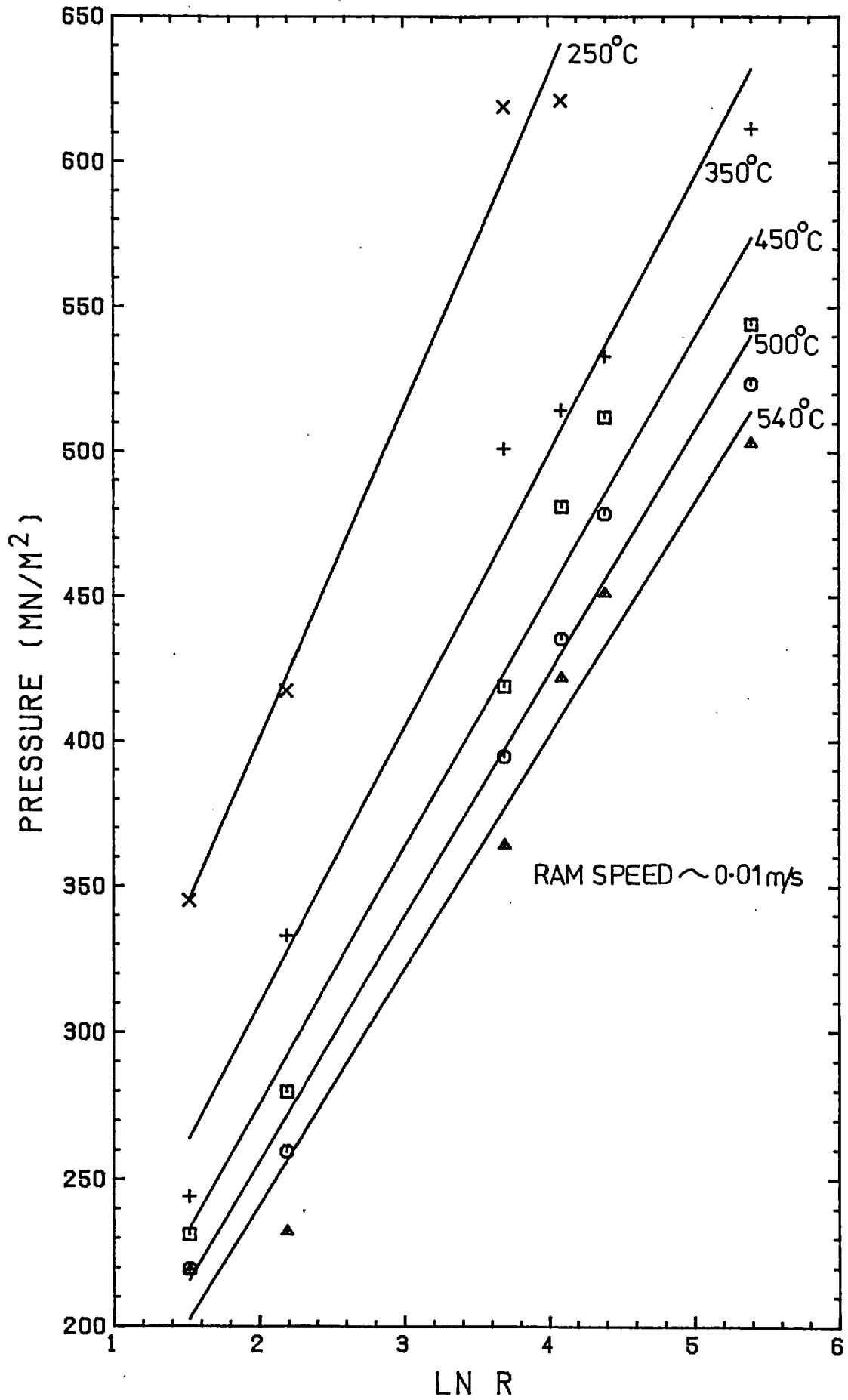


FIG. 48 Effect of temperature on extrusion pressure vs.  $\ln R$  relationships for HE9 powder extrusion

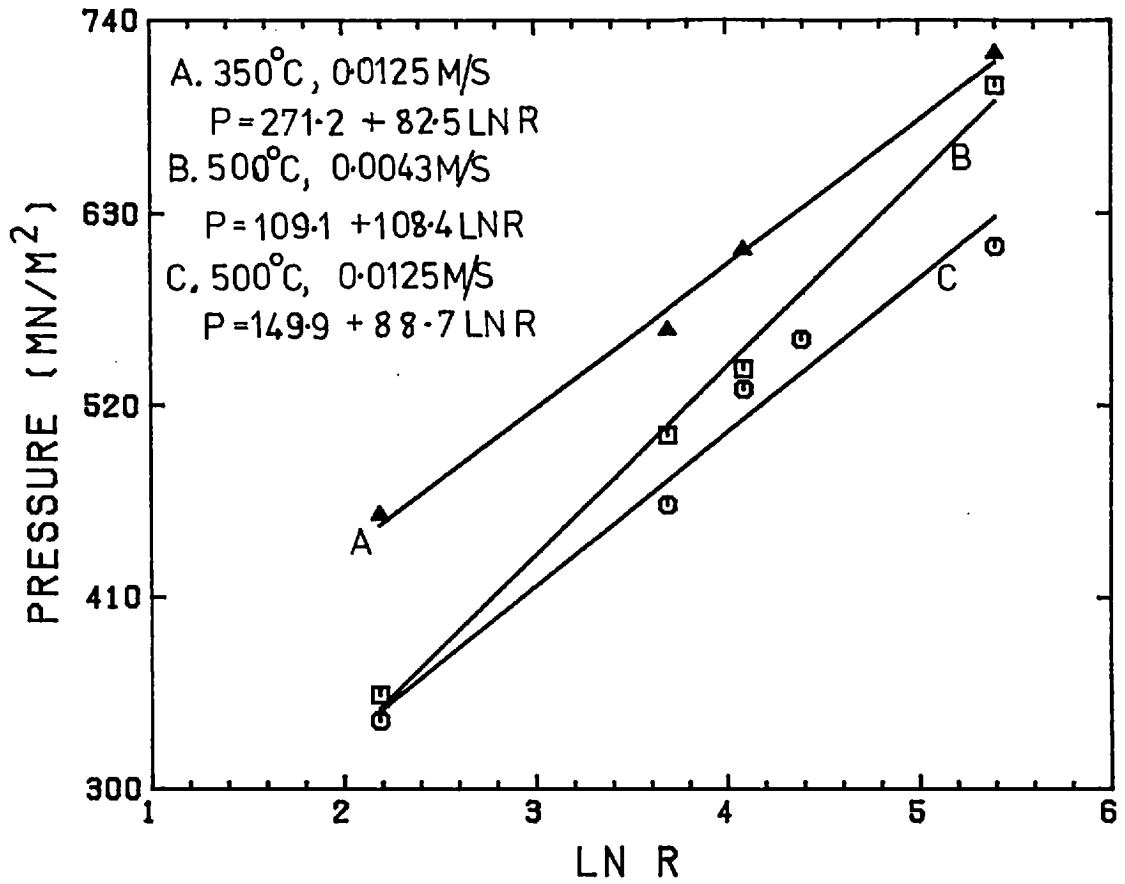


FIG. 49 Extrusion pressure vs.  $\ln R$  relationships for HE30 powder extrusions

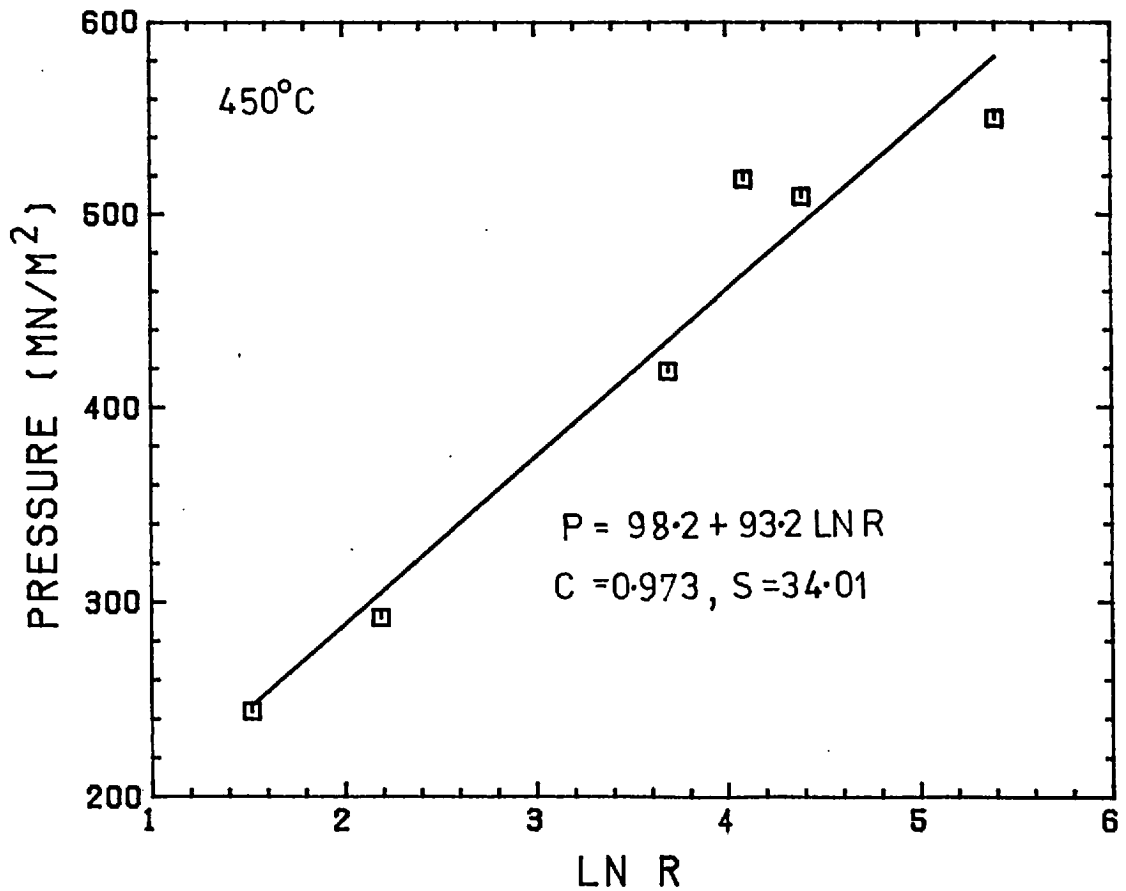


FIG. 50 Extrusion pressure vs.  $\ln R$  relationship for HE9 + 5wt.% glass billet extrusion

The significant feature here is the relative magnitudes of the constants on the right hand sides of these equations. This constant is generally recognised to be a measure of the so-called 'redundant' work i.e. work not directly contributing to a shape change. This constant represents the redundant portion of the total energy of extrusion. At a ratio of 40:1 this redundant work represents 19% of the total for powder extrusion compared to only 10% in the case of cast extrusion. This is consistent with the concept of 'building' the extrude as the powder traverses the deformation zone; weld necks being continuously broken and rewelded until the compact as a whole can withstand the applied deviatoric stresses. Thus the actual construction of the product (normally achieved by sintering) is performed by the portion of the extrusion pressure normally termed redundant - clearly a misnomer for powder extrusion processes.

Equations similar to (88) and (90) may be written for HE9 at all the temperatures investigated in the range 250-540°C. These equations are listed in table 11. The effect of extrusion temperature on this 'redundant work' term is shown in Fig. 51. A rapid decrease in this term with increasing extrusion temperature is apparent. The results of Ashcroft and Lawson<sup>112</sup> exhibit a similar trend for commercially pure aluminium extrusion. However, their results for an Al-Mg-Zn-Cu alloy do not exhibit any correlation between redundant work and extrusion temperature. These results of Ashcroft and Lawson together with the present findings suggest that the correlation between temperature and redundant work depends on the alloy content. The temperature dependence suggests that extrusion is more efficient at higher temperatures as less 'redundant work' is required for consolidation. In fact the 'redundant work' term represents 30.4% of the total work done at 250°C (for a 40:1 ratio) decreasing to 15.4% of the total at 500°C. This improvement in efficiency with increasing temperature is probably due to enhanced softening at the higher temperatures producing the familiar decrease in

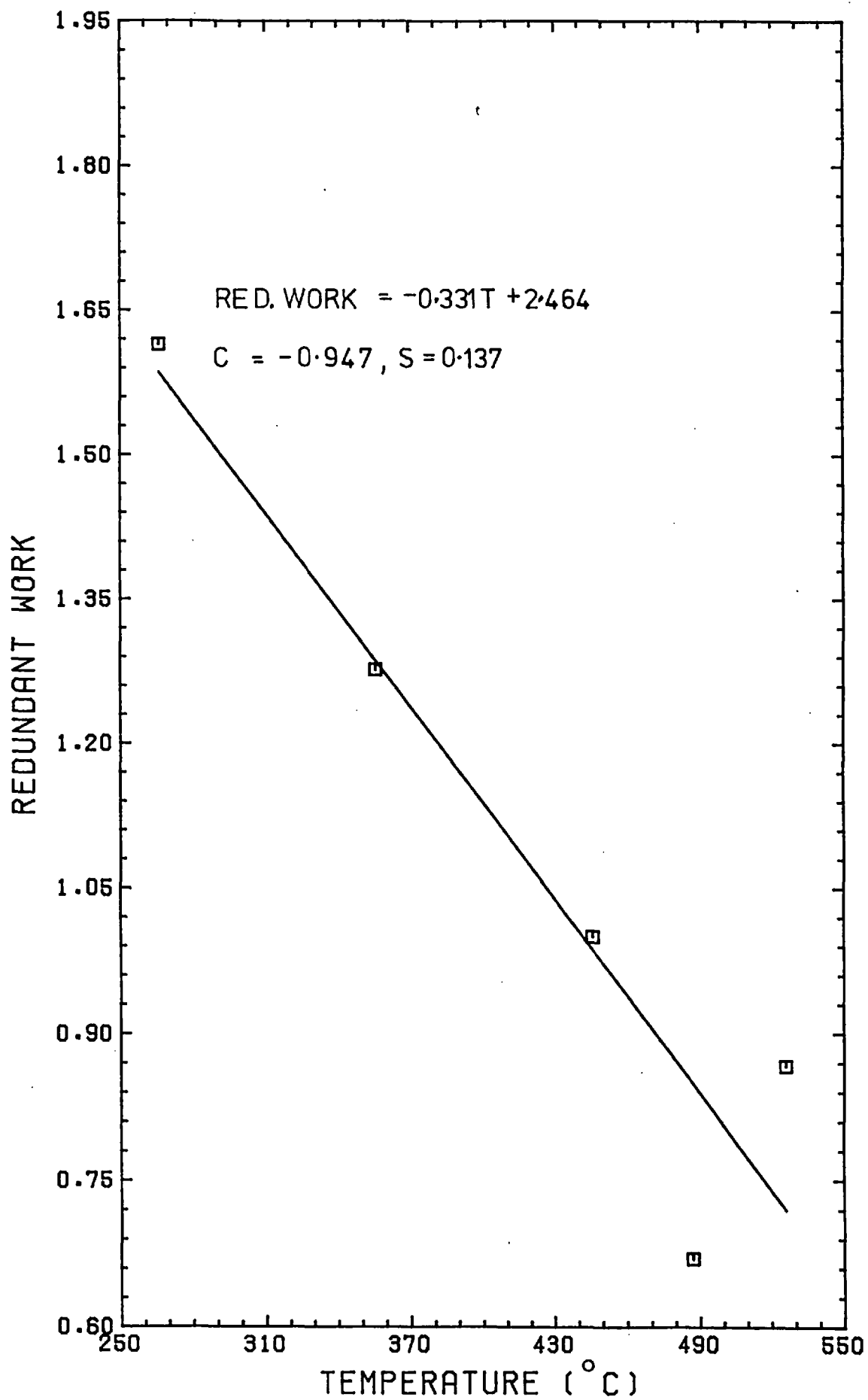


FIG. 51 Effect of extrusion temperature on redundant work term (For HE9)

flow stress. It would seem reasonable to assume the consolidation of a powder during extrusion to be a thermally activated process, as in sintering. If this is the case the 'redundant work', employed in consolidating the powder, and the temperature ought to obey an Arrhenius type of relation i.e. redundant work  $\propto \exp(1/T)$ . Fig. 52 shows such a relationship to exist. The correlation coefficient of 0.954 indicates that the experimental points fit the relationship quite well. The effect of a 5% glass content on the extrusion pressure vs.  $\ln R$  relationship is very small indeed (Fig. 50). The redundant work term only changes from 1.03 to 1.05.

Peak extrusion pressure is plotted against temperature in Fig. 53, for the four metals investigated. The correlation coefficients, given in Fig. 53, indicate that linear relationships definitely exist for these materials. This suggests that the flow stress is directly related to the extrusion temperature.

The existence of a peak (denoted by PQ in Fig. 46) in one of the powder billet load-ram displacement curves is contrary to evidence reported on the extrusion of Ni-rich alloys<sup>113</sup> but agrees with work carried out on high speed steel<sup>122</sup> powder extrusions. During the latter investigation the removal of the peak was achieved by using a soft, iron powder "nose" placed in front of the billet. This break through pressure is quite obviously more than that necessary to overcome friction between the compact and the container liner and has been attributed to temperature quenching at the die/billet interface<sup>114</sup>.

In the present series of experiments billets extruded at the same temperature as the tools also exhibited peaks in the load-displacement curve, proving the quenching theory to be inadequate. Sheppard and Castle<sup>13</sup> have presented an explanation based on dislocation dynamics but for cast billets their experimental evidence suggests

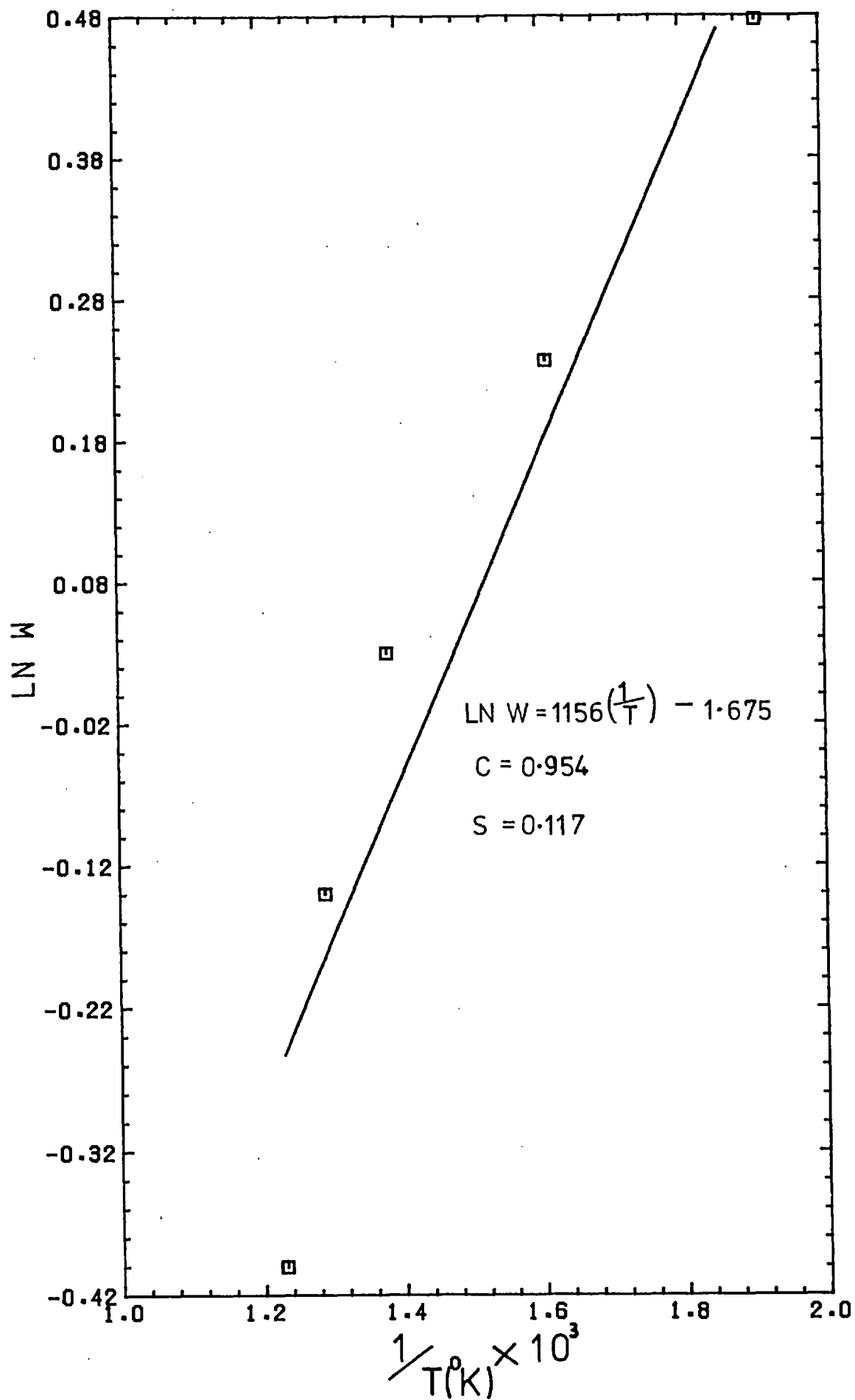


FIG. 52 Logarithm of redundant work as a function of reciprocal of extrusion temperature

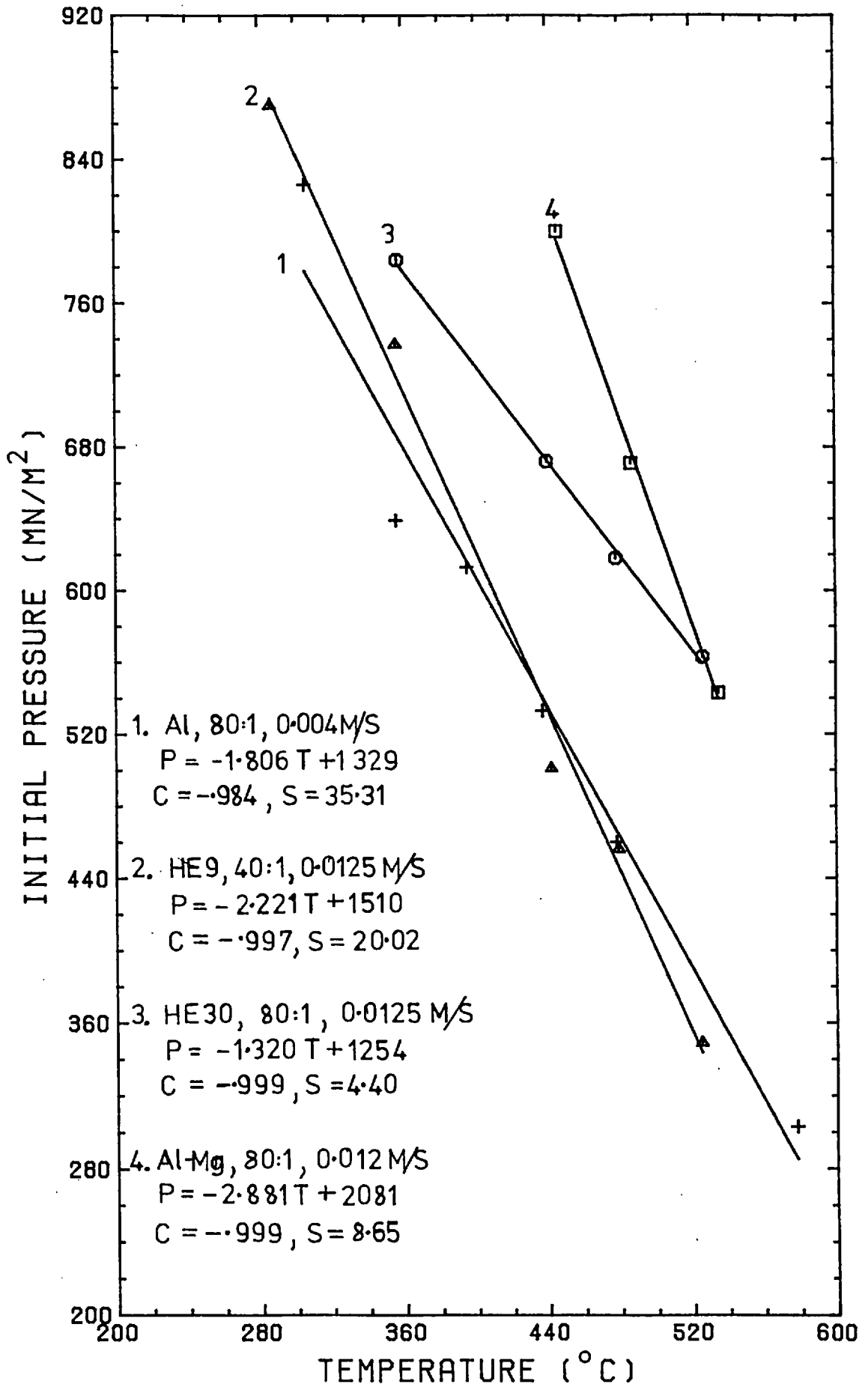


FIG. 53 Initial extrusion pressure as a function of extrusion temperature



that the strain rate has little effect. Fig. 46 is in disagreement with this finding since the only variation in extrusion conditions between A and C is in the strain rate. (It should be noted that during extrusion, with a nominally constant ram speed, the ram in fact slows down while the load reaches a peak and then accelerates until the steady state phase begins). However, the maximum load in extrusion C is also lower in this experiment and existing evidence suggests the existence of a threshold load below which these pressure peaks either do not occur or cannot be detected. (In the present series of experiments several compacts, extruded without lubricating pads, also exhibited peaks).

The evidence, then, supports the view that this phenomenon is connected with a dynamic process. At high strain rates an excess of dislocations is generated immediately after the compaction phase and before dynamic softening processes, such as recovery, commence operating. At this early stage in the extrusion cycle the continuous oxide coatings of the powder particles act as barriers to dislocation motion and annihilation causing high dislocation densities within the particles. With fine powders (as in the present case) many particles of 2-3 $\mu$ m exist. Thus the actual sub-grain size is limited, in many cases, by the size of the particles. Limiting the subgrain size increases the flow stress and thus the extrusion load i.e.

$$\sigma \propto (\text{subgrain size})^{1/x}$$

As the extrusion proceeds the oxide layers are fragmented allowing the dislocations to move more freely and reduce their number by annihilation. Thus the dislocation density decreases and the flow stress is lowered causing a drop in extrusion load.

When lower strain rates are employed in extrusion the rate of dislocation production is balanced by the rate of the dynamic softening processes. The same argument

could be applied to those extrusions in which the maximum load is low. This thesis is supported by the experimental points in Fig. 54. The difference between peak pressure and steady state pressure increases with strain rate in HE9, HE30 and HE9 + 5 wt.% glass compact extrusions. From this argument it would follow that extruding at higher temperatures should reduce both the magnitude and the dependence of peak height on strain rate due to increased dynamic softening rates. The curves for HE9 at 350°C and 540°C (in Fig. 54) support this thesis. It should be recalled, however, that increasing the strain rate increases the temperature rise obtaining as well as increasing the flow stress in the absence of a temperature rise. Hence the pressure required may be greater or less depending upon the balance of these variables.

#### 5.5.1 Torsion test results

The primary reason for performing torsion tests was to obtain flow stress-strain rate relationships over a range of temperatures. From such information the values of  $\alpha$ , the hot working equation constant and  $\Delta H$ , the activation energy of the material may be calculated.

Fig. 55 depicts a schematic representation of the shape of torque-twist curves obtained, at various temperatures, for HE9 and HE30 extrudes. As the temperature decreases the maximum in the torque-twist curve occurs at larger strains. Jonas<sup>115</sup> and Sellars<sup>131</sup> encountered similar behaviour in commercially pure aluminium. The maximum becomes less well defined with decreasing temperature until, at 300°C and below, no maximum exists.

During deformation the flow stress increases as the dislocations multiply until softening processes begin to operate. The flow stress is then governed by a balance between the rates of work hardening and dynamic softening. If these rates are equal in magnitude (i.e. rate of dislocation multiplication = rate of dislocation annihilation) a steady

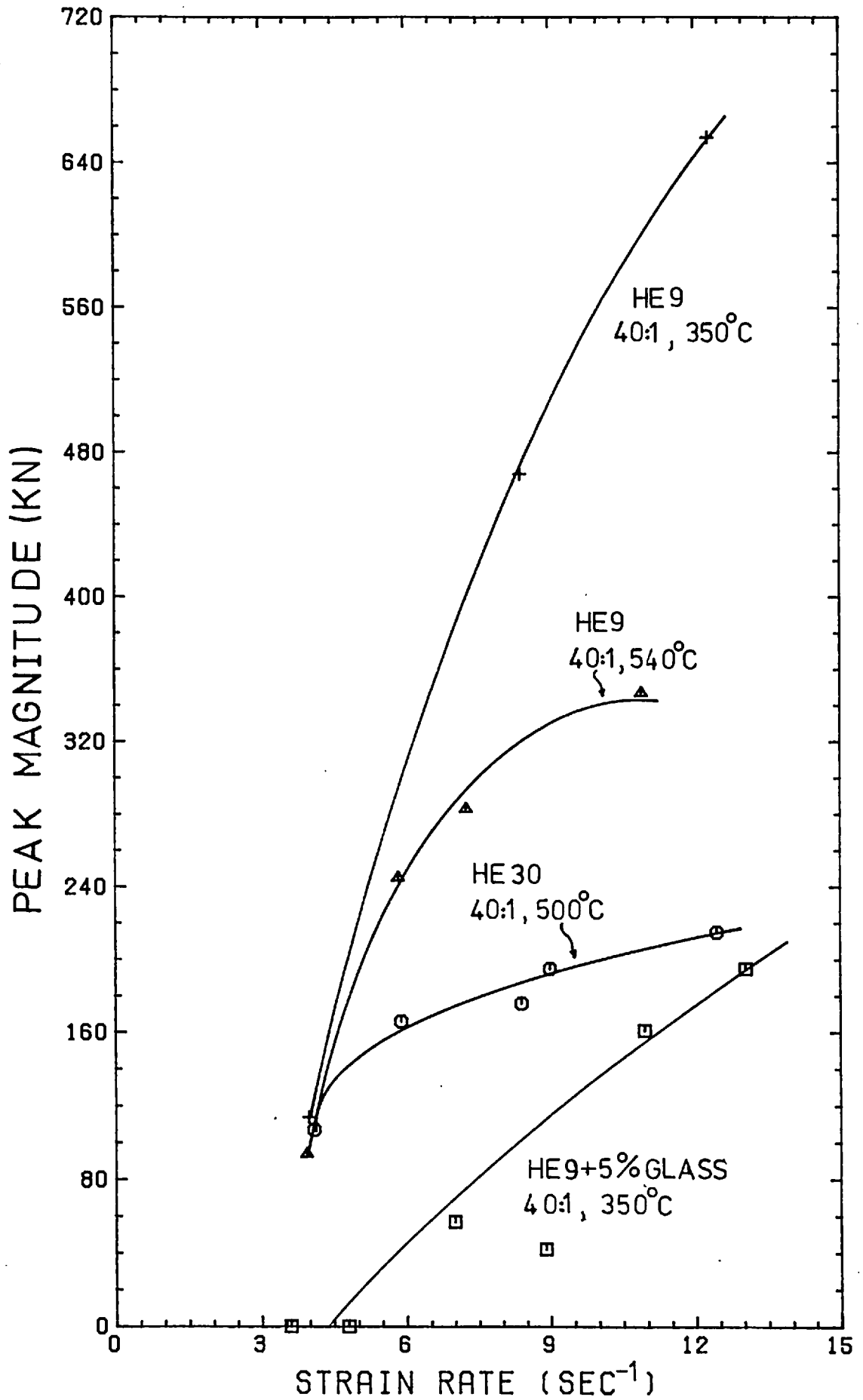


FIG. 54 Effect of strain rate on magnitude of extrusion load curve peak

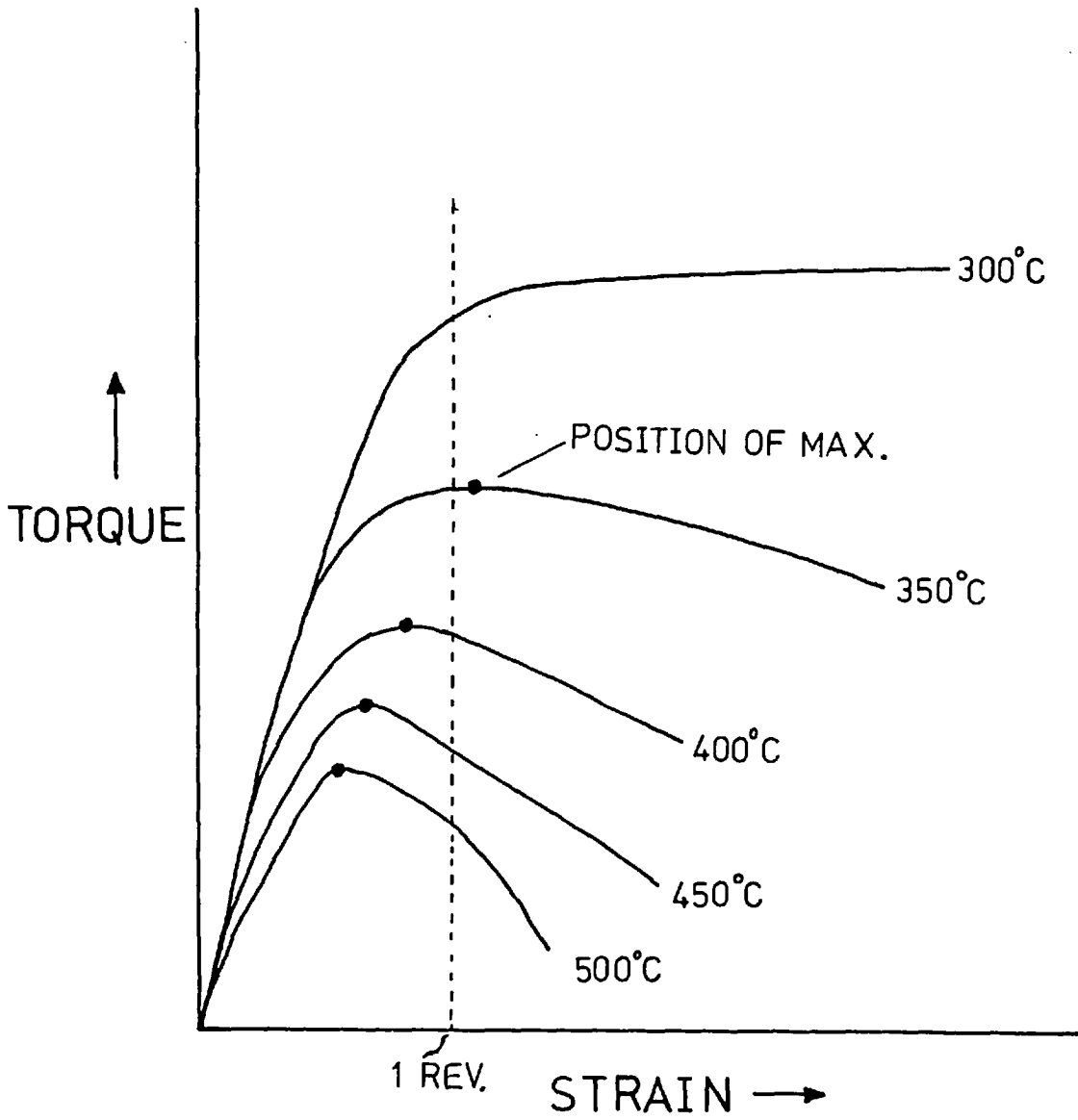


FIG. 55 Schematic representation of torsion curves at various temperatures

state is reached with the flow stress remaining constant. If the softening rate is predominant a decrease in flow stress occurs. In Fig. 55, the position of the maximum marks the strain at which the softening rate becomes dominant. As the test temperature is increased, the critical strain required to initiate softening processes, such as dynamic recovery, decreases. This causes a shift in the maximum towards smaller strains.

Tables 12 and 13 give the maximum torque value for each HE9 and HE30 specimen together with the twist rate. Plots of maximum torque vs.  $\ln$  Twist rate (revs/sec) are shown in Figs. 56 and 57. From the correlation coefficients given it is clear that linear relationships definitely exist for both materials at all the temperatures investigated. Sellars also found similar linear relationships for several aluminium alloys as did Ormerod<sup>139</sup> for high purity aluminium.

The Backofen analysis detailed in 3.10.1 was used to determine flow stresses from the torque values measured. The  $m$  and  $n$  values used were calculated and are listed in tables 14 and 15. The shear flow stress was calculated (at a constant strain  $\sim 1/4$  rev.) for each specimen and is listed in tables 16 and 17. (At such an early stage in the test the effects of temperature rise may be discounted).

Figs. 58 and 59 show the linear relationships between  $\ln$  shear flow stress and  $\ln$  shear strain rate at the various temperatures. From the correlation coefficients given it is obvious that both HE9 and HE30 obey a law of the form:-

$$\text{Flow stress} = \text{constant} \times (\text{strain rate})^n$$

$$\text{i.e. } \sigma = C \dot{\epsilon}^n$$

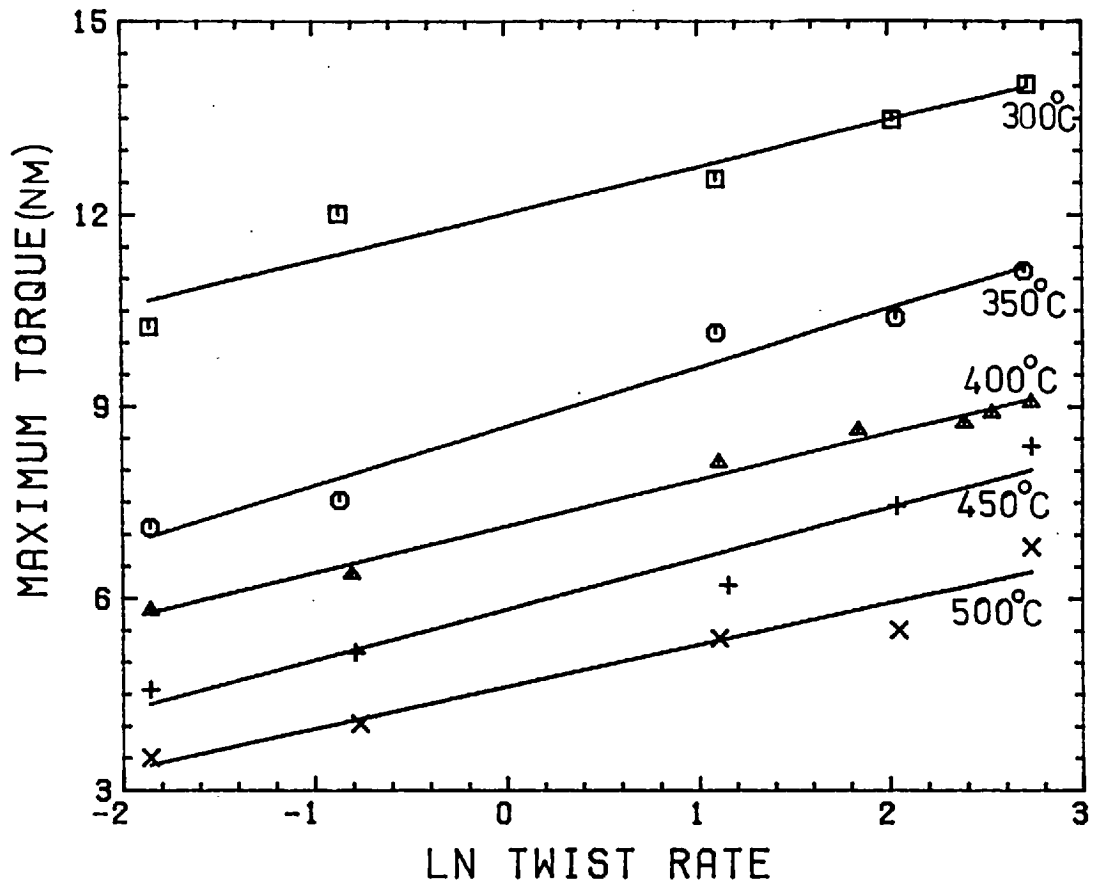


FIG. 56 Maximum torque as a function of twist rate at various temperatures (HE9)

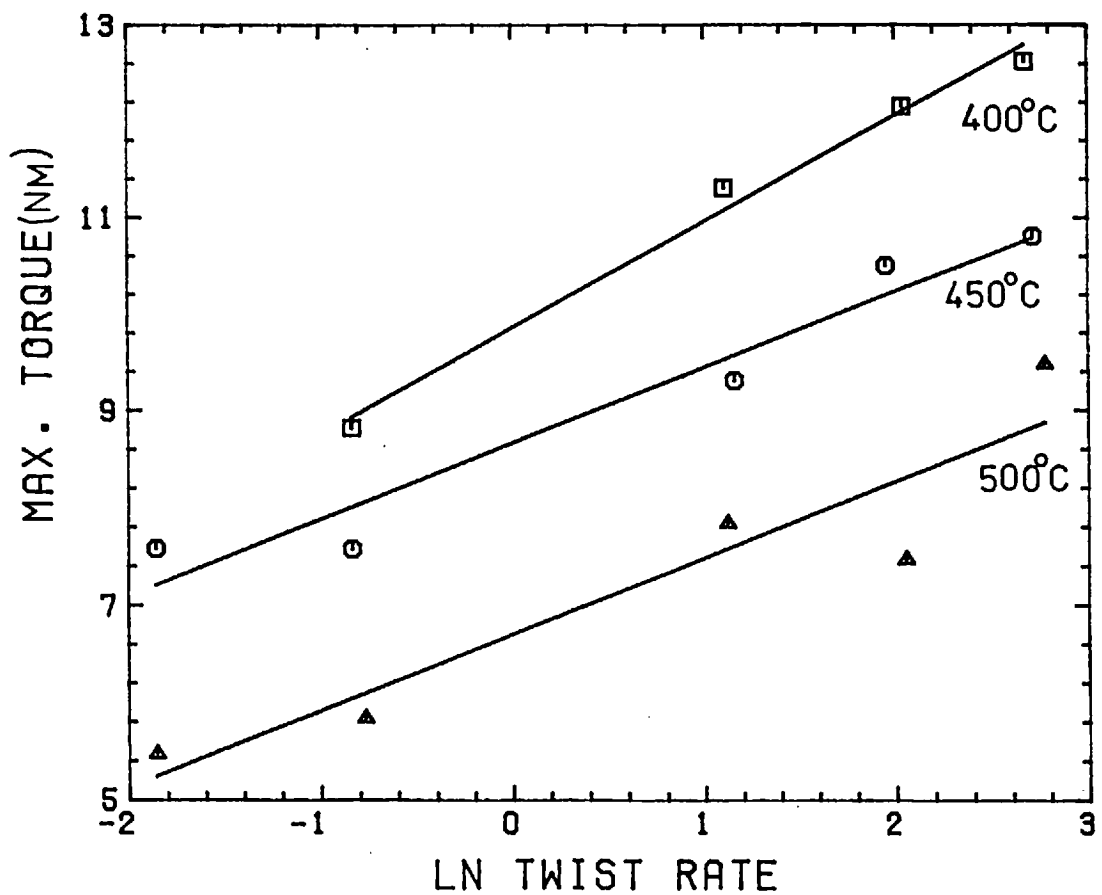


FIG. 57 Maximum torque as a function of twist rate at various temperatures (HE30)

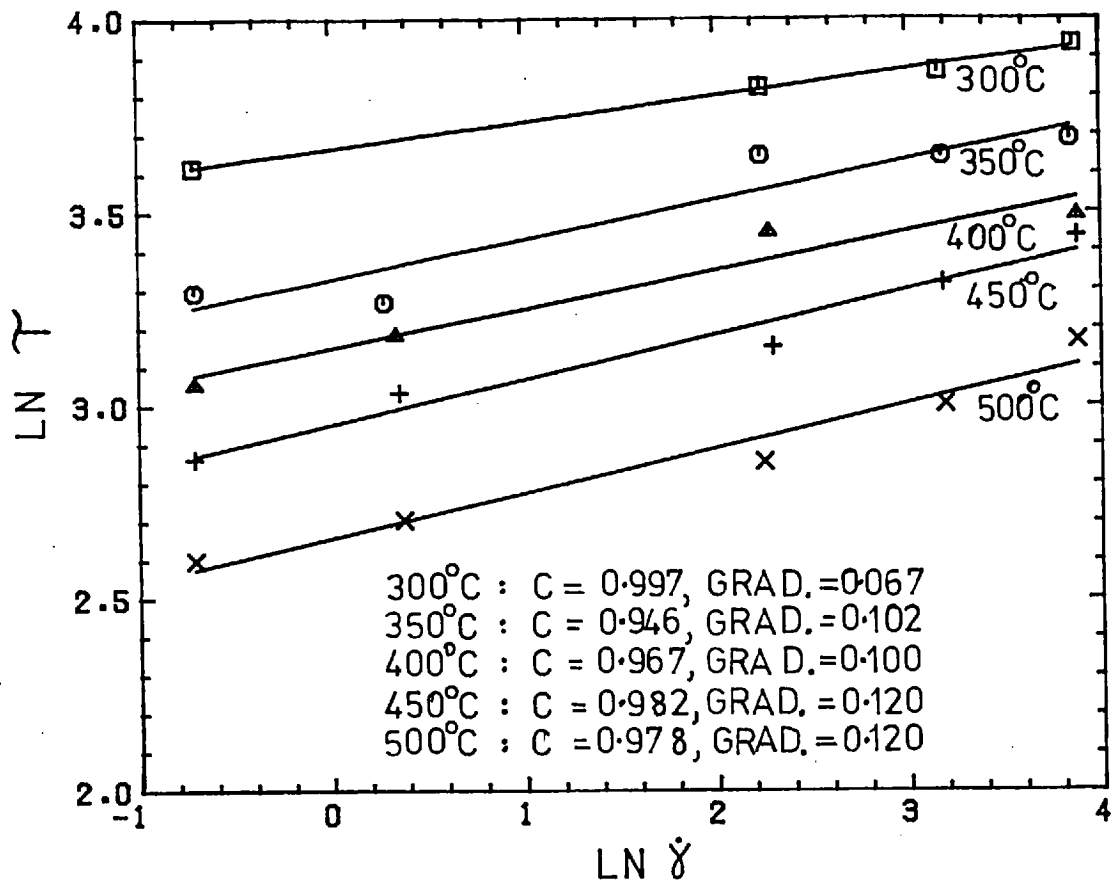


FIG. 58 Relationship between  $\ln \tau$  and  $\ln \dot{\gamma}$  at different temperatures (HE9)

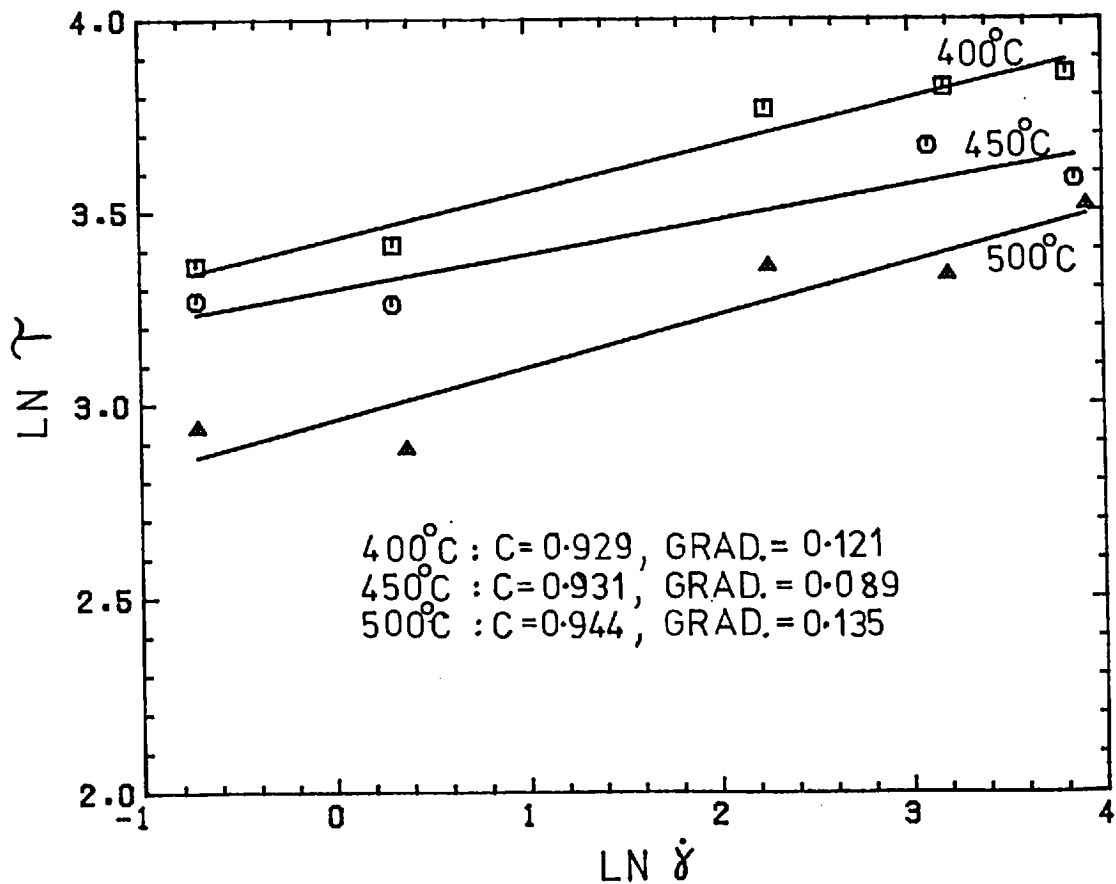


FIG. 59 Relationship between  $\ln \tau$  and  $\ln \dot{\gamma}$  at different temperatures (HE30)

For HE9 the strain rate exponent  $n$ , given in Figs. 58 and 59, varies from 0.07 at 300°C to 0.12 at 500°C. Lack of data prevents conclusions being drawn about the effect of temperature on the strain rate exponent for HE30.

Alder and Philips<sup>134</sup> found that the strain rate exponent of aluminium decreased dramatically with temperature from 550-20°C (for compression to 40% reduction). The strain rate exponent,  $n$ , was found to vary from 0.16 to 0.02 over this temperature range. Thus it can be seen that the present torsion results agree well with the compression results of Alder and Philips.

#### 5.6.1 Determination of the hot working constants and the activation energy

According to the theory in 3.6.2 the constant,  $\alpha$ , may be obtained from the gradients of  $\ln \sigma$  vs.  $\ln \dot{\epsilon}$  plots at high stress levels and  $\ln \sigma$  vs.  $\dot{\epsilon}$  plots at low stress levels. i.e.  $\beta$  and  $n_2$  values. Values of  $\beta$ ,  $n$  and  $\alpha$ , for HE9 and HE30, at each torsion test temperature are listed in tables 18 and 19. Apparently  $\alpha$  varies significantly with temperature. (Fig. 60 shows that  $\alpha$  increases with temperature.) As  $\alpha$ , in fact, is defined as a temperature independent constant it is quite obvious that this method is unsatisfactory. In order to use this method properly, high and low stress levels must be defined clearly. For this purpose a value of  $\alpha$  is required, which defeats the object. An alternative approach must be adopted.

$$\dot{\epsilon} = A_1 (\sinh \alpha \sigma)^n \exp\left(-\frac{\Delta H}{RT}\right) \quad (32)$$

by definition:  $Z = A_1 (\sinh \alpha \sigma)^n$

substituting:  $\left(\frac{Z}{A_1}\right)^{1/n} = \sinh \alpha \sigma$

∴  $\alpha \sigma = \sinh^{-1} \left(\frac{Z}{A_1}\right)^{1/n}$



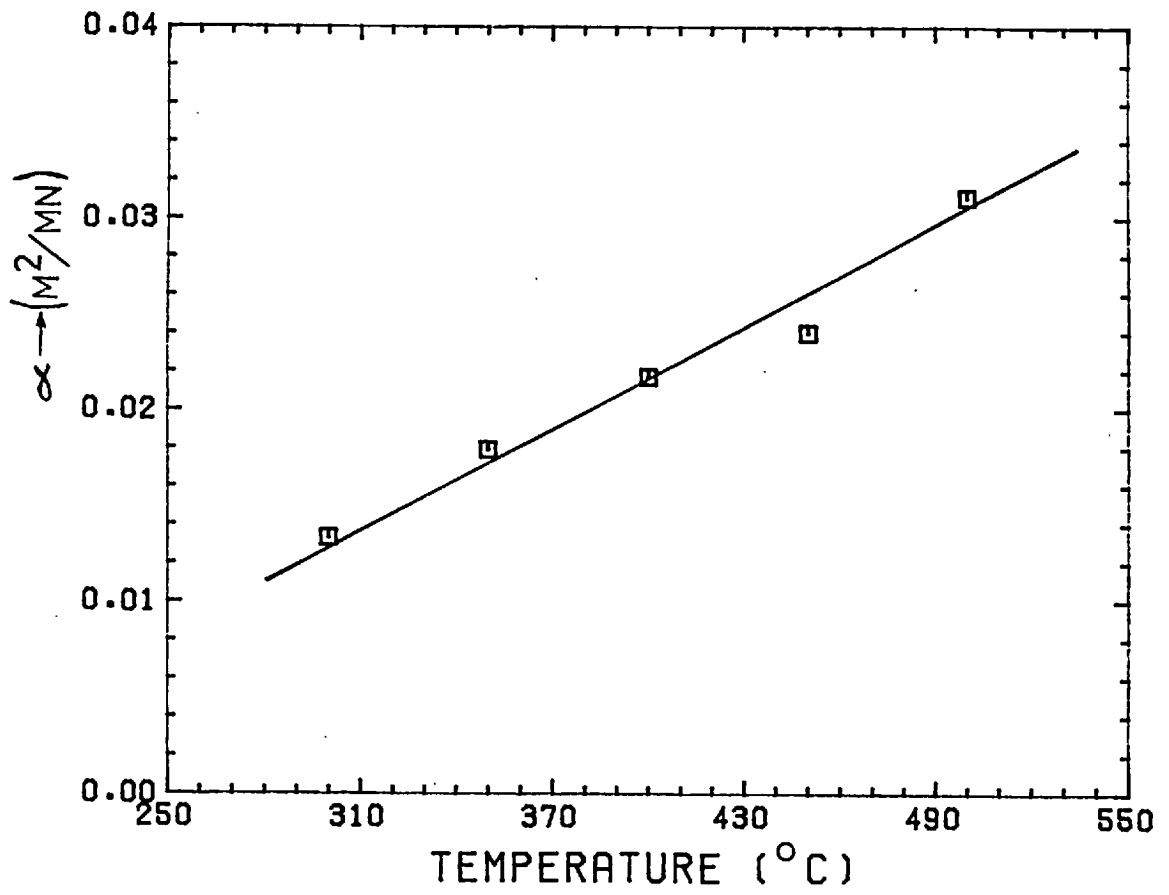


FIG. 60 Effect of temperature on alpha

substituting for  $\sinh^{-1}$

$$\alpha\sigma = \ln \left[ \left(\frac{Z}{A_1}\right)^{1/n} + \sqrt{\left(\frac{Z}{A_1}\right)^{2/n} + 1} \right]$$

$$\therefore \sigma = \frac{1}{\alpha} \ln \left[ \left(\frac{Z}{A_1}\right)^{1/n} + \sqrt{\left(\frac{Z}{A_1}\right)^{2/n} + 1} \right] \quad (91)$$

Equation 91 contains four variables:  $\alpha$ ,  $\Delta H$ ,  $A_1$  and  $n$ . If guesses of  $\alpha$ ,  $\Delta H$ ,  $A_1$ , and  $n$  are supplied, the minimization routine described in 3.4.1 can be used to minimize the difference between experimental flow stresses and theoretical values calculated using (91) by changing  $\alpha$ ,  $\Delta H$ ,  $A_1$  and  $n$ . The values of these variables which give the minimum difference between theoretical and experimental stresses are the best which can be obtained from this data.

The results for the HE9 and HE30 torsion test data are listed in table 20. The activation energies of 167, 224 and 170,881 J/mole for these alloys are somewhat higher than the value for extruded commercially pure aluminium powder of 155,000 found by Sheppard and Chare<sup>135</sup> and the value of 156,000 found by Wong and Jonas<sup>68</sup>, also for aluminium. (The values obtained by these other investigators are equivalent to the energy of self diffusion for aluminium). This difference is probably caused by the alloy content of the HE9 and HE30 together with the oxide dispersion obstructing dislocation motion and therefore increasing the energy barrier to be overcome for recovery processes to occur. The  $\alpha$  values found in the present investigation (0.038 and 0.040) compare very well with those found by Wong and Jonas (0.040) and by Sheppard and Chare (0.031-0.042). The  $n$  values of 5.16 and 3.87 also compare well with those of Wong and Jonas (4.2). Figs 61 and 62 show  $\ln Z$  vs.  $\ln \sinh \alpha\sigma$  plots for the HE9 and HE30 torsion data. The correlation coefficients of 0.988 and 0.976 respectively, indicate that the general hot working equation fits the experimental data rather well. Using the activation energy and  $\alpha$  values obtained from the torsion results, plots of  $\ln Z$  vs.  $\ln \sinh(\alpha\sigma)$ ,

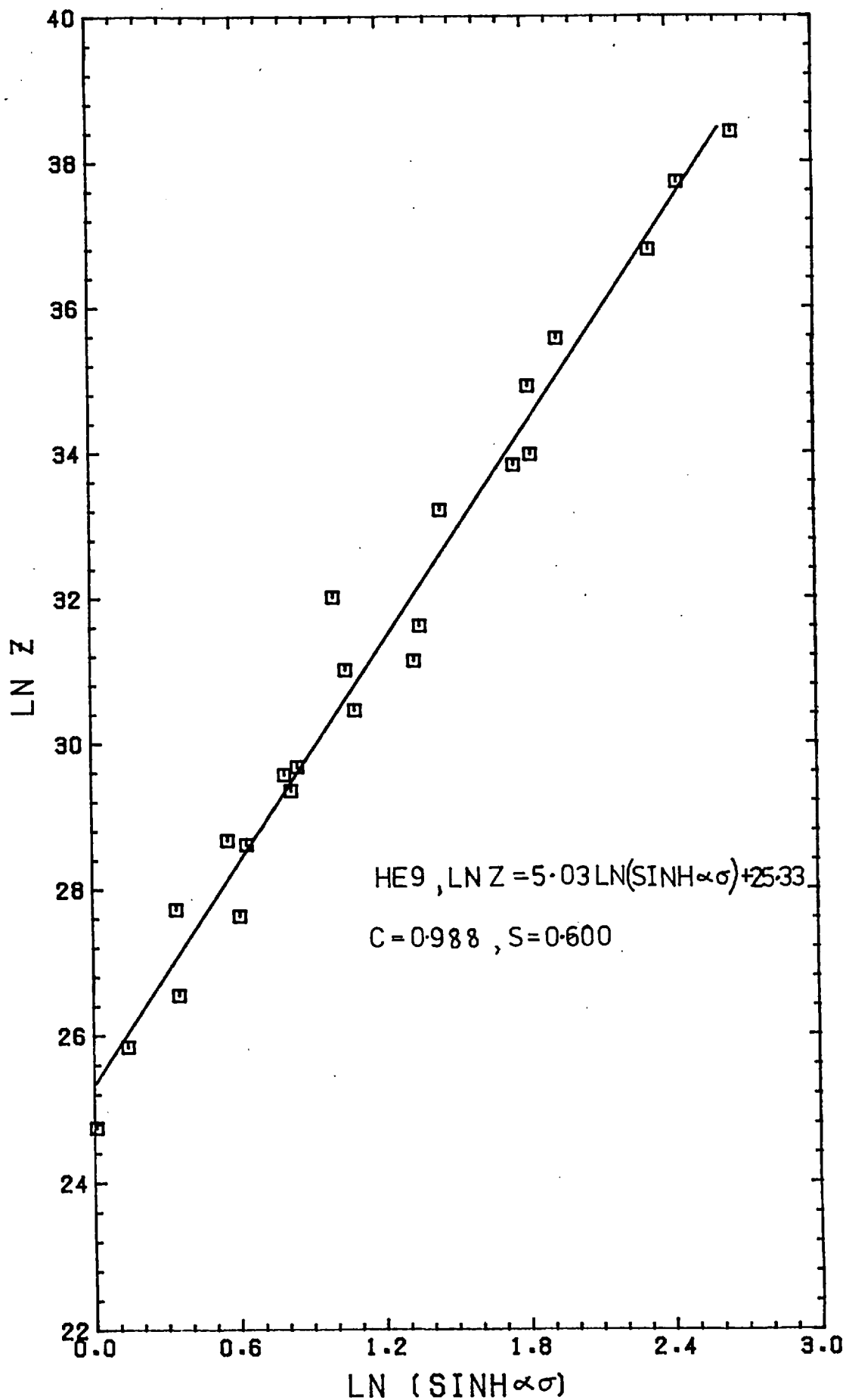


FIG. 61  $\ln \sinh(\alpha\sigma)$  vs.  $\ln Z$  (HE9 Torsion Tests)

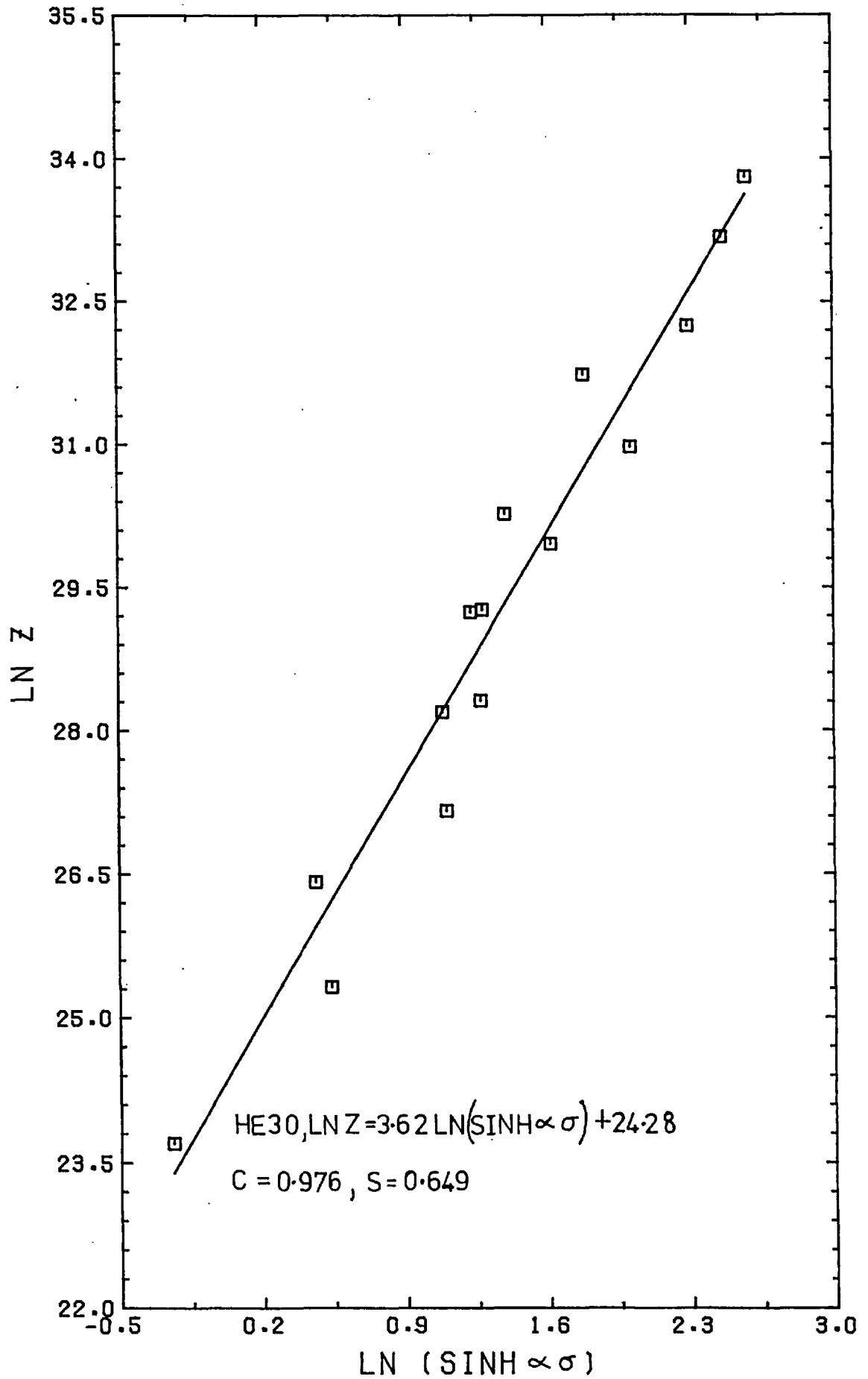


FIG. 62  $\ln \sinh(\alpha \sigma)$  vs.  $\ln Z$  (HE30 Torsion Tests)

for extrusion data, are shown in Figs. 63, 64 for HE9 and Fig. 65 for HE30 (The extrusion temperatures were calculated using the integral profile technique). Clearly, the extrusion data points do not fit the hot working theory nearly as well as the torsion results. Obviously, the flow stress, strain rate and extrude temperature values derived from extrusion results are much less accurate than values derived from torsion data. Therefore greater inaccuracies must be expected when working with extrusion data. The gradients of the plots in these three figures give  $n$  values of 3.74 and 2.98 for HE9 and 5.07 for HE30, respectively, compared to the values of 5.16 and 3.87 obtained from the torsion results. Now the strain rate sensitivity of a material is proportional to  $1/n$ . Therefore the higher the  $n$  value the lower the strain rate sensitivity. If the thesis that powder compacts are less strain rate sensitive than solid billets is correct one would expect  $n$  values derived from the extrusion of powder billets to be higher than  $n$  values derived from torsion tests on the consolidated extrude. This appears to be the case for HE30 but not for HE9. Thus some doubt must exist as to the relative magnitudes of the strain rate sensitivities of powder and cast material. One possible explanation for this discrepancy between the two powders is outlined below. The flow stress values obtained from the torsion tests were calculated from the torque-twist curves after  $1/4$  of a revolution. The readings were taken at this early stage on the assumption that the temperature rise of the specimen could be neglected. This is a reasonable assumption at the higher temperatures where the flow stress is small. During the low temperature experiments, especially at high strain rates, the temperature rise may not be negligible, due to the larger flow stress of the material. Thus data points, in Figs 61 and 62 at the top end of the  $\ln \sinh \alpha \sigma$  scale, should

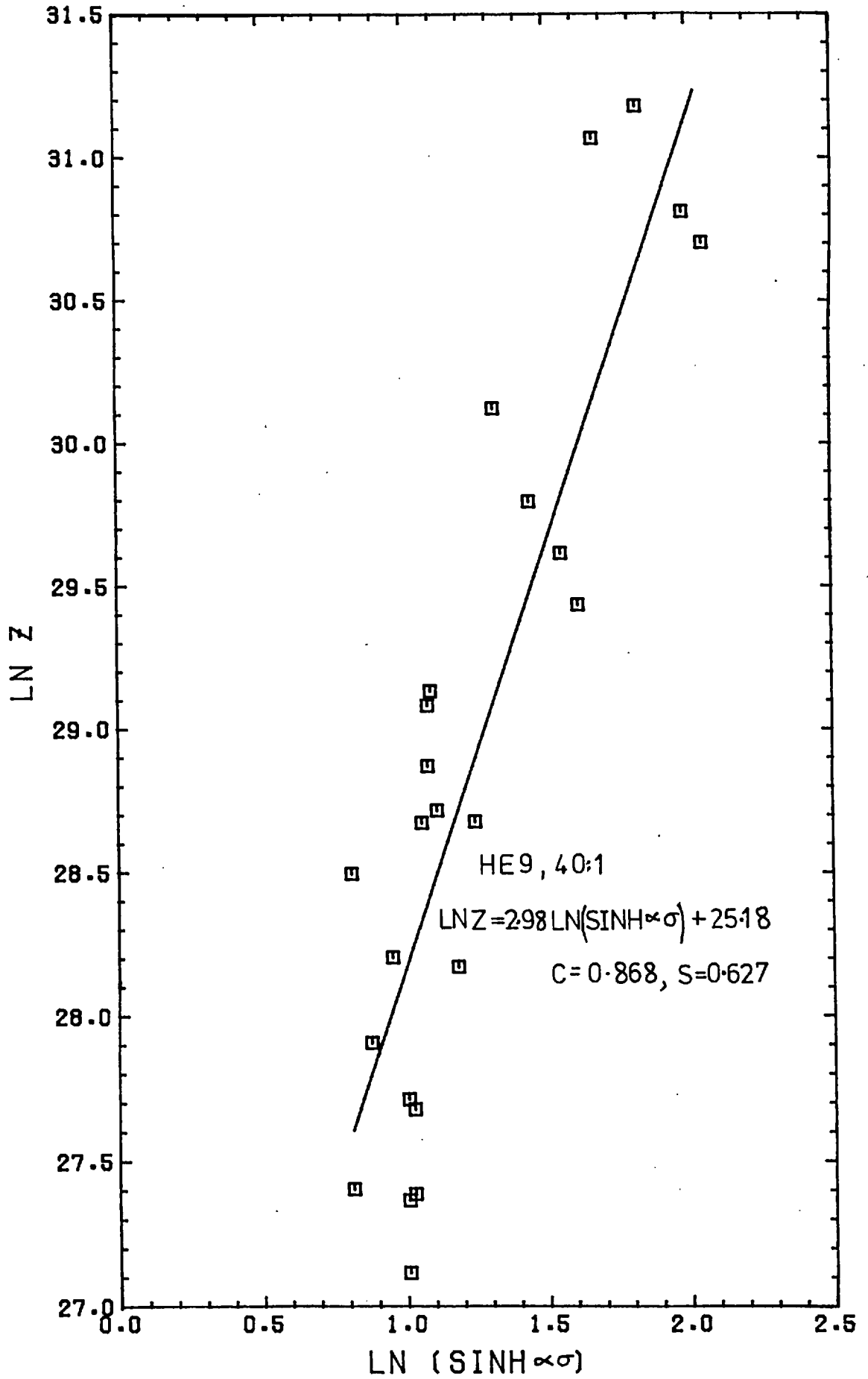


FIG. 63  $\ln \sinh(\alpha\sigma)$  vs.  $\ln Z$  (HE9 40:1 Extrusions)

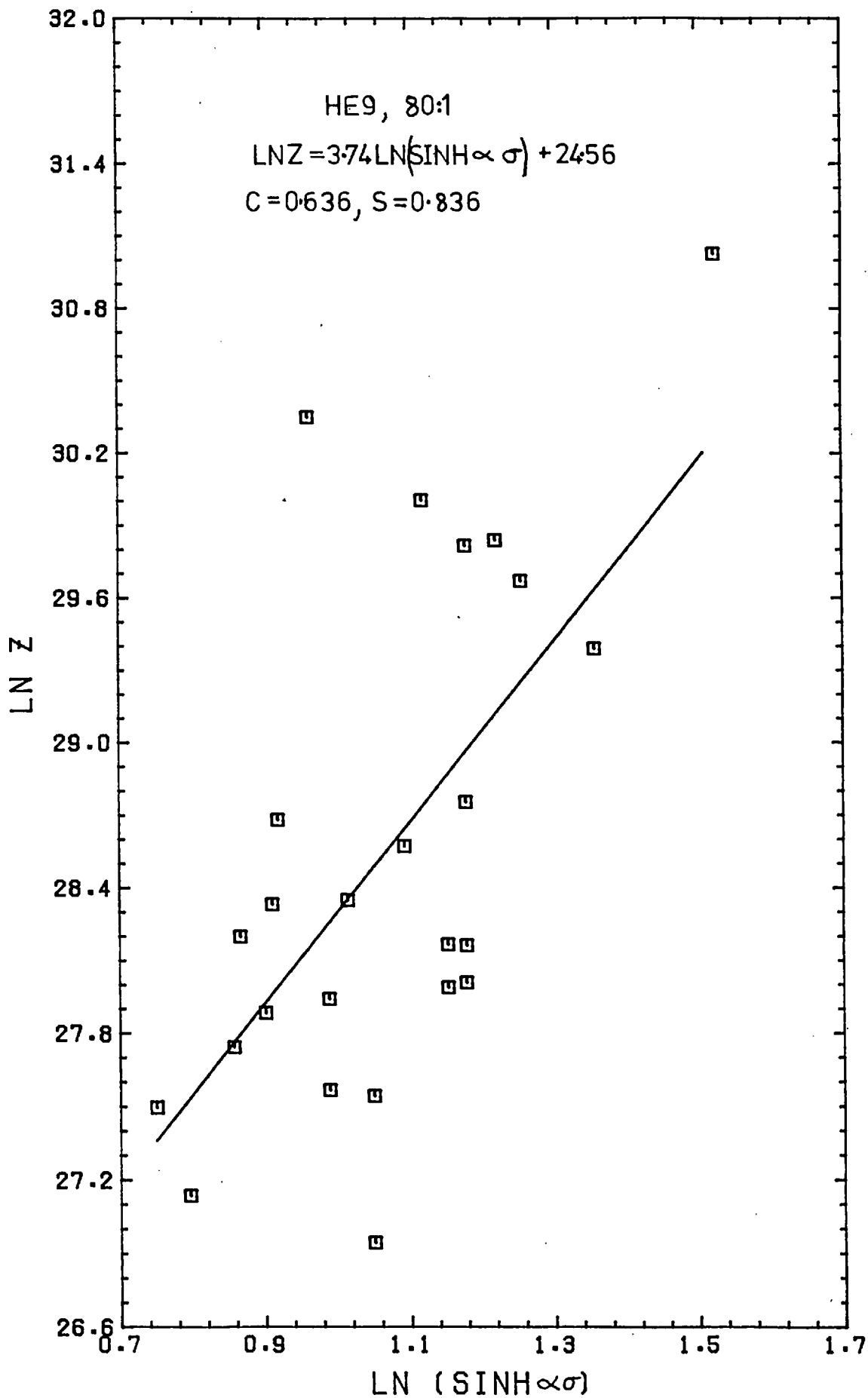


FIG. 64  $\ln \sinh(\alpha \bar{\sigma})$  vs.  $\ln Z$  (HE9 80:1 extrusions)

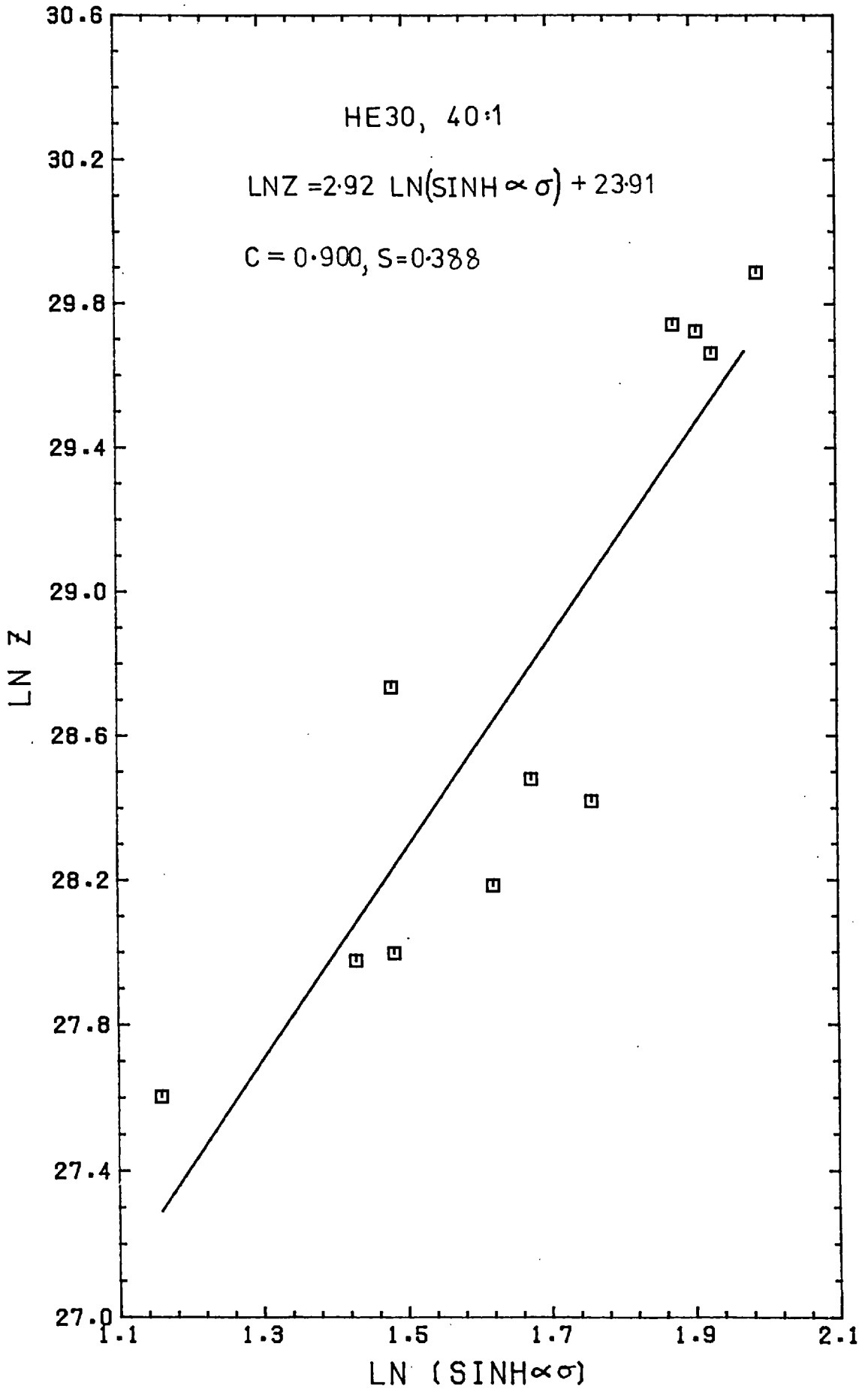


FIG. 65  $\ln \sinh(\alpha\sigma)$  vs.  $\ln Z$  (HE30 40:1 extrusions)

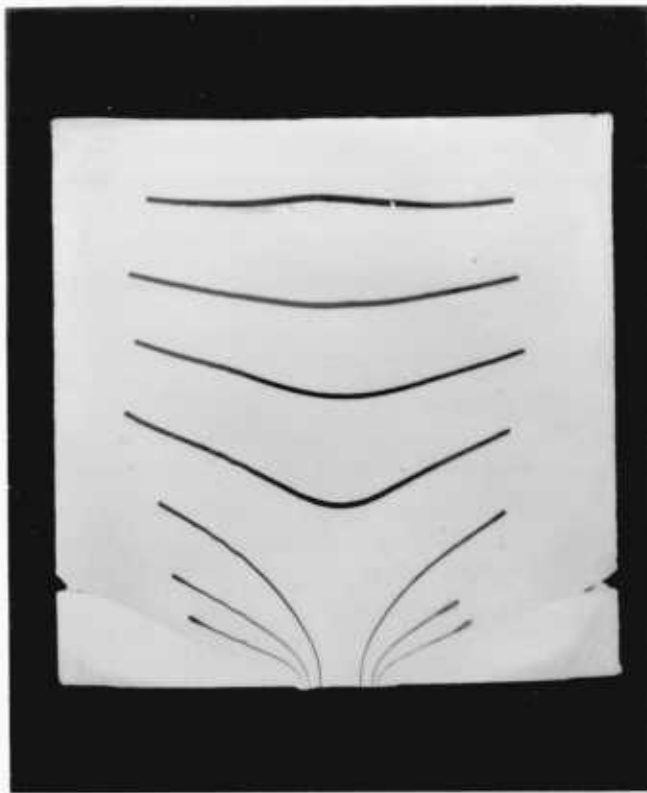


really be moved to slightly lower  $\ln Z$  values. This in turn would produce a lower gradient and thus a lower  $n$  value, indicating a larger strain rate sensitivity in the consolidated extrude used in the torsion tests. Thus the strain rate sensitivities of consolidated HE9 and HE30 may be more than  $\sim 1/5.16$  and  $1/3.87$  respectively.

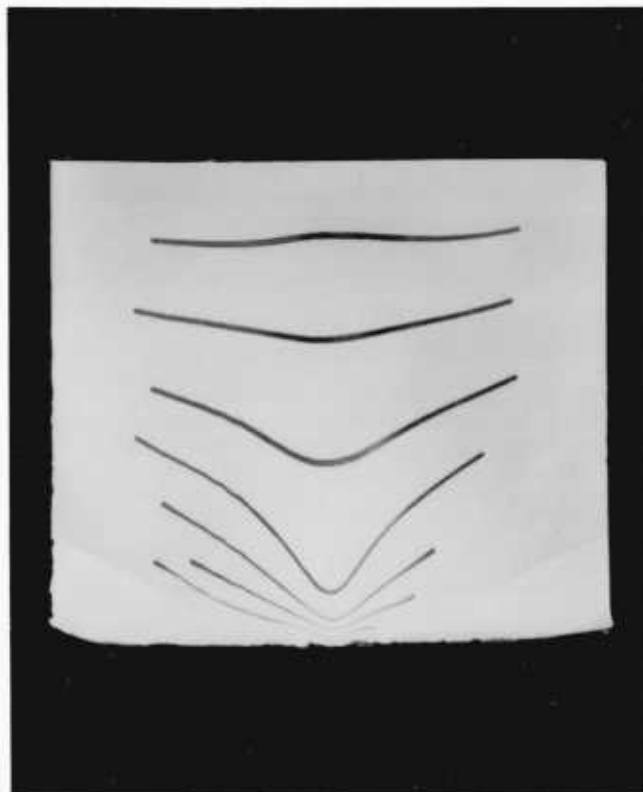
#### 5.7.1 Partial extrusions - flow patterns

The flow patterns obtained for extrusion ratios of 40:1 and 180:1 (both at 450°C) are shown in Plate 6(a) and (b) respectively. In both cases the dead metal zone, consisting of the pad material, is clearly visible. Conical deformation zones including semi-angles of 66° and 69° respectively are fairly distinct. For ratios of 40:1 and 180:1 the minimized axisymmetric single triangle upper bound solution predicts semi-angles of 54° and 56°, an error of approximately 17%. Several workers including Childs<sup>136</sup> have used grids machined on split billet faces to study flow patterns and to estimate strain rates during extrusion. Obviously this technique cannot be applied to powders.

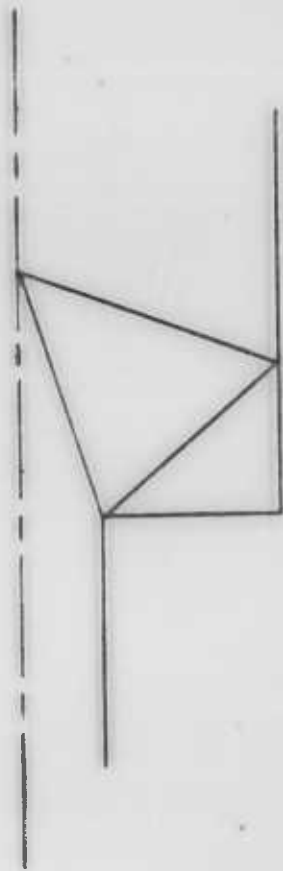
Plate 7 shows a fibre composite billet, partially extruded at 450°C ( $\sim 170^\circ\text{C}$  above the softening point temperature) at a ratio of 10:1. The formation of the fibres in the deformation zone is clearly shown. The elongation of the fibres near the extrude surface is much greater than that of the central fibres. This confirms the concept of greater deformation occurring near the billet surface during extrusion. As previously mentioned in 2.3.1 this greater deformation can produce a surface layer of recrystallised material in the extrude giving undesirable product properties. The degree of fibre misalignment in the extrude in Plate 7 is negligible and so an assumption of uniaxially aligned fibres is quite reasonable. Drawn on the micrograph, in Plate 7, is one half of the upper bound field predicted, for a 10:1 extrusion ratio, by the axisymmetric solution discussed in 3.3.1. Clearly

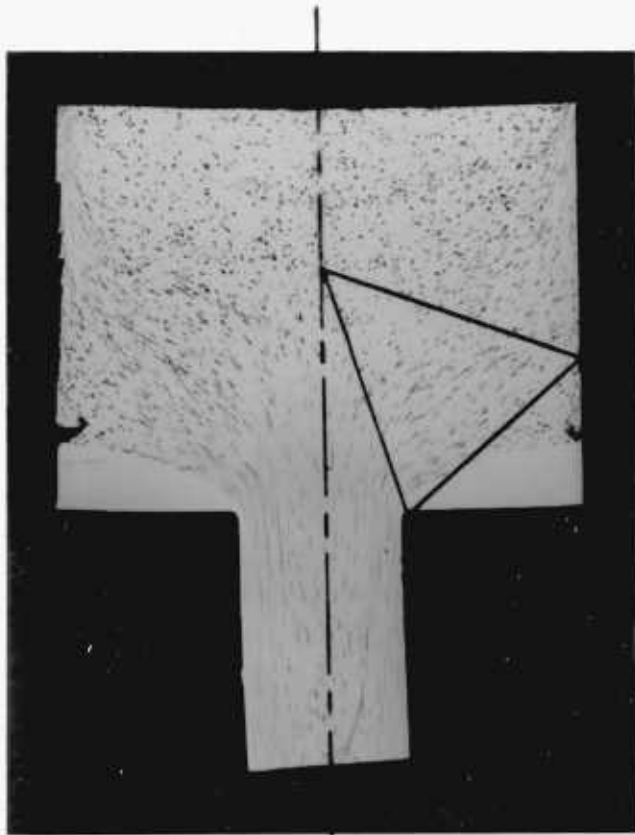


(a)  
(40:1)



(b)  
(180:1)

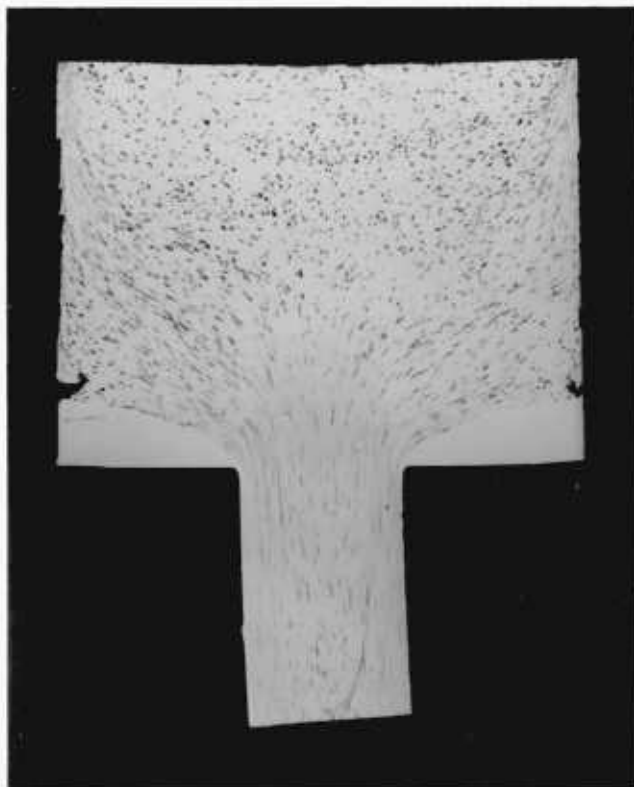




(10:1)

PLATE 7

Partial extrusion showing fibre formation



(10:1)

PLATE 7

Partial extrusion showing fibre formation

the theoretically predicted field agrees quite well with the actual deformation obtaining and the fibres are not fully elongated until they have almost reached the final (theoretical) velocity discontinuity AB. The calculation in 3.7.5 predicted a critical aspect ratio i.e. (fibre length/fibre diameter) of  $\sim 40$  and a minimum extrusion ratio of 10:1, for ideal fibres of  $2412 \text{ MN/m}^2$  tensile strength. In practice the fibres are far from ideal and so their tensile strength is considerably less than  $2412 \text{ MN/m}^2$ . Thus the true critical aspect ratio is  $\ll 40$ . (In 5.9.6 the tensile strength is estimated  $\sim 890 \text{ MN/m}^2$  giving a critical aspect ratio  $\sim 15$ ). Thus in practice the fibres attain the critical amount of elongation some time before reaching AB. Due to variations in deformation across the section, the outer fibres attain this critical aspect ratio at an earlier stage than the central fibres.

#### 5.8.1 Effect of extrusion conditions on extrude surface finish

A good product surface finish is essential if any extrusion process is to be commercially successful. Surface defects can provide sites for crack initiation, leading to reduced mechanical strength, and also lower resistance to degradation by the elements. If the extrude is to be successfully anodized a homogeneous, good quality surface<sup>119</sup> is vital. Even if the extruded product is not subjected to loading or exposed to the elements, the surface finish must have an acceptable appearance.

Table 21 used in conjunction with Plates 8 and 9 summarises the effects of extrusion temperature, ram speed and glass content on the surface finish of the various materials. Each combination of ram speed (or glass content) and extrusion temperature is represented by a letter which refers to a micrograph in Plate 8 or 9.

HE9 extrude, with a good surface finish, is fairly easy to produce over a wide range of extrusion conditions. The only defects occurring are scoring, due to die pick-up, (Plate 8b), and disintegration of the surface layer of pad material (Plate 8c). Generally the surface finish improves with increasing ram speed and temperature. For ram speeds from 3-8 mm/sec temperatures in excess of 500°C must be employed. However, at 13 mm/sec any temperature, between 250 and 540°C, is satisfactory.

HE30 behaves in a similar manner. However, the surface finish appears to be more dependent on ram speed and less on temperature. A satisfactory finish is produced using any ram speed from 8-13 mm/sec over the temperature range 350-540°C. Pearson<sup>116</sup> recommends a temperature of 450-490°C and a ram speed of approximately 10 mm/sec for cast 51S alloy (similar in composition to HE30). Neither HE9 or HE30 suffer from "hot shortness" at the higher extrusion temperatures. Although, generally, the ductility of aluminium increases with temperature its flow stress decreases and thus high speed - high temperature deformation tends to cause tearing in the weakened aluminium. Many aluminium alloys are especially susceptible to hot shortness. A typical example of such an alloy is any of the strong Al-Mg-Cu-Zn alloys which contain the Al-CuAl<sub>2</sub> eutectic. During extrusion the surface "layers" of the extrude heat up more than the central core. In many cases the eutectic temperature is exceeded and "regions" of eutectic composition begin to melt i.e. incipient melting. (Even if no actual melting occurs, these regions become so weak that their flow stress is ~ zero). Secondary tensile stresses acting in the die neck, elastic expansion stresses exerted on emergence from the die and thermal stress due to cooling cause cracking and tearing in the extrude surface. This is the defect known as "hot-shortness". The use of the powder extrusion process ensures a fine, homogeneous distribution of second phase particles minimizing the occurrence of segregation. This, in turn,

reduces the likelihood of hot shortness cracking.

In the case of Al-Mg the surface finish - ram speed dependency is reversed. A satisfactory extrude is only obtained with lower ram speeds of 3-8 mm/sec. The effect of temperature over the range investigated, 400-500°C, appears to be very slight. Al-Mg alloys are notoriously strain rate sensitive during extrusion. Extruding too rapidly often produces a poor quality extrude due to surface checking and/or die pick-up.

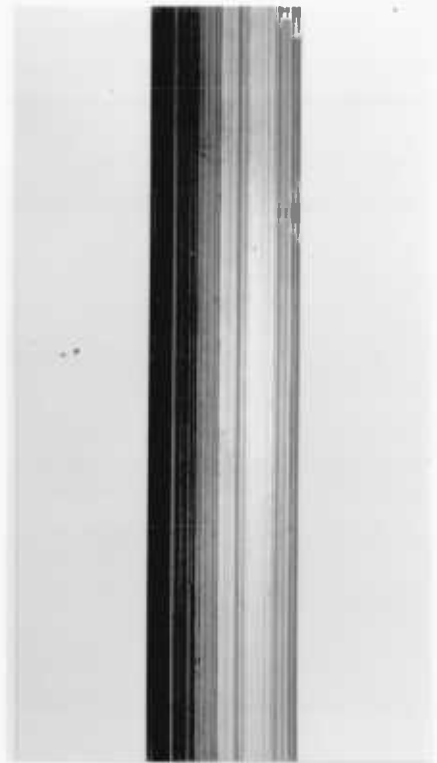
Extrusion of the Al powder compacts is more difficult. A satisfactory surface finish is only obtained if high temperatures,  $\sim 600^\circ\text{C}$ , and fast ram speeds,  $\sim 13\text{mm/sec}$ , are used. Other extrusion conditions produce a "fir-tree" type defect (Plate 8d). This type of defect is usually associated<sup>11</sup> with hot shortness. In this case, however, this defect occurs when low temperatures and ram speeds are used. Remembering the absence of lubricating pads, the defect may be caused by die pick-up. Secondary tensile stresses, generated in the region of the die, may also play a part. At the lower temperatures the flow stress of the Al is higher and it becomes brittle and may thus suffer cracking due to these stresses. Thus the extrude might be said to suffer from "cold shortness". Wilcox and Whitton<sup>132</sup>, while extruding duralumin and Parsons et al<sup>133</sup>, while extruding aluminium<sup>133</sup> (both at low temperatures and ram speeds) encountered a "fir-tree" defect due to "cold shortness".

Additions of 5 wt.% glass have little effect on the surface finish of HE9 extrudes. An acceptable product is obtained over a wide range of extrusion conditions. Once again the faster ram speeds, 8-13 mm/sec, give the best results in the temperature range 350-550°C. The maximum amount of glass which can be successfully incorporated in an extrude is approximately 6-7 wt.%. Extrudes





(a)



(b)



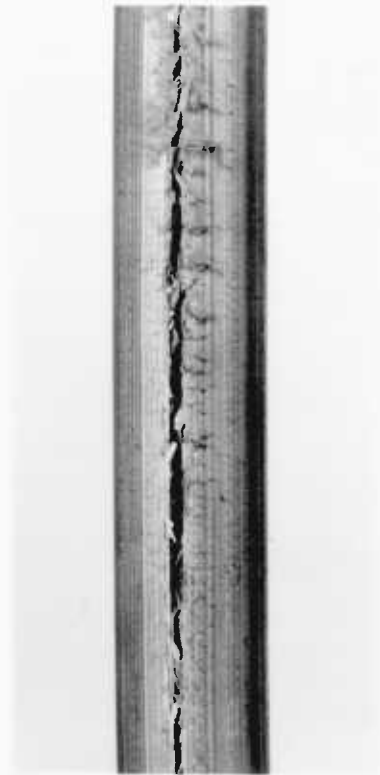
(c)



(d)



(e)



(f)



(g)



(h)

containing more than this amount suffer from splitting (Plate 9 (g) and (h)) due to elastic expansion of the glass.

The limited number of glass reinforced Al-Mg extrusions produced also exhibit an improvement in surface finish with increasing ram speed. This trend is the reverse of that exhibited by the unreinforced Al-Mg extrudes.

Over the temperature range 375-550°C all of the reinforced aluminium extrudes are unsatisfactory. A "fir-tree" defect due to cold shortness and die pick up is common at the lower ram speeds. Increasing the ram speed produces only a very slight improvement.

#### 5.9.1 Stress-strain behaviour of the extrude

The results of the tensile tests are given in table 22. Great difficulty was experienced in testing the glass reinforced specimens at elevated temperatures due to a tendency for the specimens to fracture in the grips. The shapes of the load-extension curves for the different materials are represented schematically in Figs. 66 and 67.

Unreinforced HE9 and HE30 specimens display a fairly linear elastic region followed by an extended plastic region in which the load increases steadily up to a maximum value at which point necking occurs. The load then gradually decreases until fracture occurs, usually after quite large elongations. The glass reinforced HE9 behaves similarly in the elastic region. However, during plastic deformation the curve is significantly steeper (in this region the glass still behaves elastically) as predicted in 3.7.2. A very limited amount of necking occurs, after the maximum load is reached, quickly followed by fracture. The elongation to fracture is noticeably reduced by the presence of the brittle glass.

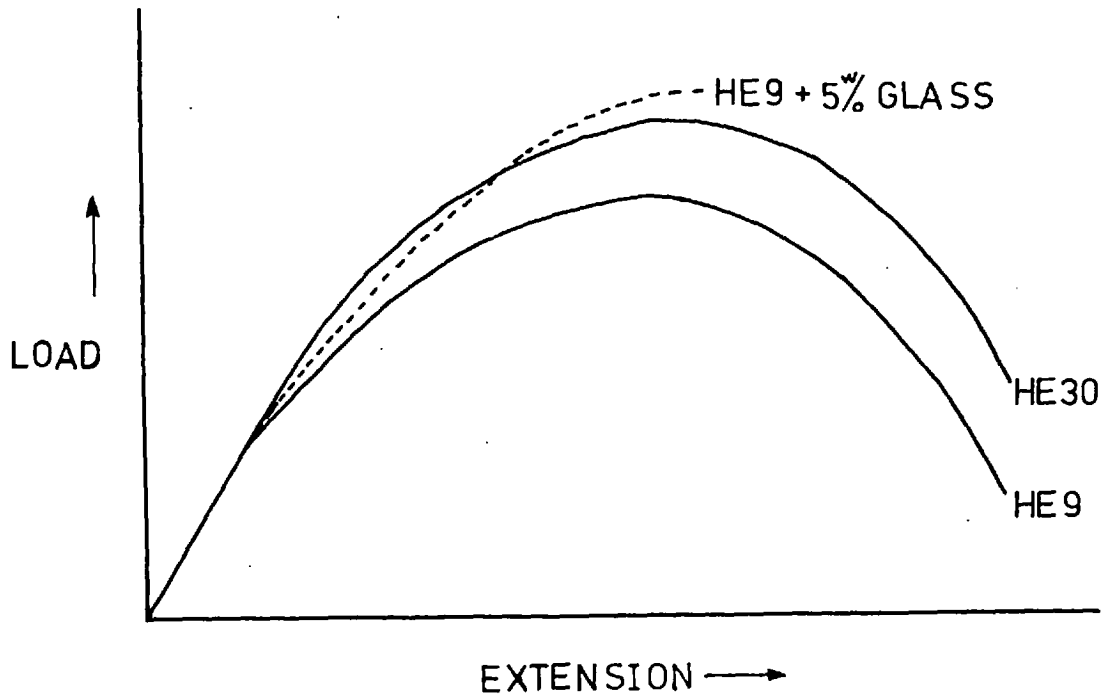


FIG. 66 Load vs. extension curves from tensile tests (HE9, HE30, HE9 + 5wt.% Glass)

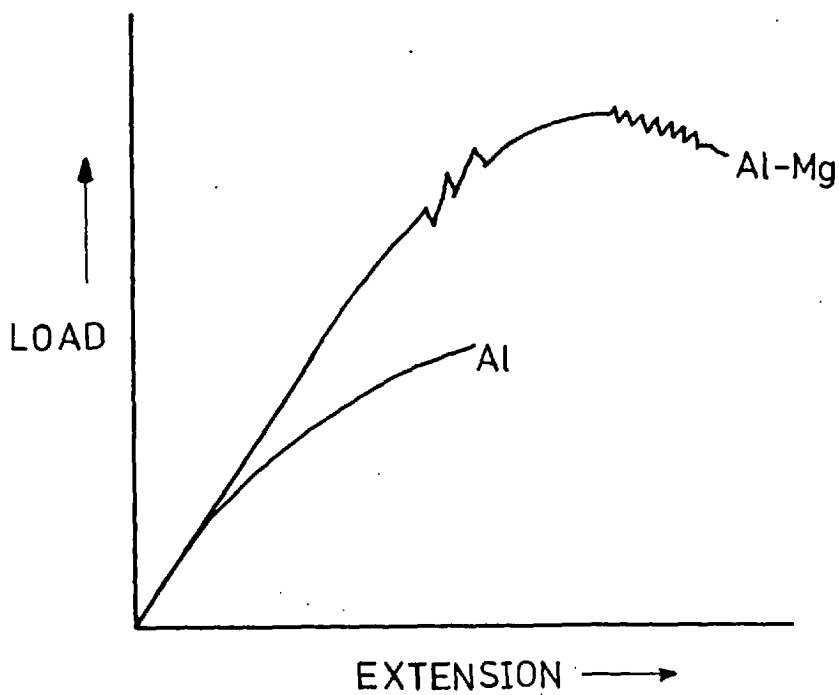


FIG. 67 Load vs. extension curves from tensile tests (Al, Al-Mg)

Several tests were performed on larger HE9, HE9 + 5 wt.% glass and HE30 tensile specimens using a standard 50mm strain gauge, to obtain values of elastic moduli. The results are given below

	HE9	HE9+5 wt.% glass	HE30	Al (Published)
Elastic Modulus (MN/m <sup>2</sup> )	67.3	61.2	72.8	71

The values for HE9 and HE30 are in good agreement with the published value for aluminium. The elastic modulus of the reinforced HE9 is about 13% less than that of aluminium. Theoretically the modulus of the material should remain unaffected by the addition of fibres of similar modulus. However, cracks introduced by the glass addition may well cause this reduction in modulus. Although the presence of such cracks in the matrix reduces the modulus of the composite, the U.T.S. is still higher than that of unreinforced material. This is because the tensile strength of the fibres has a much larger influence on the U.T.S. than the strength of the matrix does, within reason. i.e. as long as the matrix does not disintegrate.

The Al specimens (Fig. 67) possess a very limited elastic region. Brittle fracture occurs after small plastic strains before the gradient of the curve ever reaches zero. i.e. no necking occurs.

The Al-Mg alloy exhibits a fairly well defined elastic region. During plastic deformation (room temperature tests only - not at 400°C) the phenomenon of serrated yielding occurs (manifested by the saw-tooth curve in Fig. 67). Al-Mg alloys are prime examples of metals subject to this strain ageing phenomenon which, basically, involves three stages (See 5.9.4):-

- 1) dislocations are pinned by solute atoms
- 2) the flow stress increases until the dislocations are torn free
- 3) the flow stress decreases again.

Necking occurs in the Al-Mg specimens leading to fracture, usually at fairly high strains.

Listed below are the optimum tensile properties of the various materials investigated together with the properties of some commercially produced alloys.

(all as extruded)	Extru- sion Temp °C	0.2% P.S.	U.T.S.	% Elongation
HE9 (powder)	250	132	160	23
HE9+8wt.% glass	450	92	223	28.4
HE30 (powder)	350	141	193	32
Al-Mg (powder)	400	176	308	22
Al-Mg+1wt.% glass	450	158	307	20
Al (powder)	600	74	160	10.5
Al+9wt.% glass	550	73	140	4
25 Alcan Aluminium (Solid)		--	60	18
Al-3Mg-Mn (solid)		100	215	14
Al-4.5Mg-Mn (solid)		130	280	11
HE9 (solid)		55	100	12

(For unreinforced HE9, HE30 and Al-Mg the extrusion temperature giving the optimum properties is fairly low i.e. dynamic softening is minimized at low temperatures. Reinforced HE9, Al-Mg and Al require higher extrusion temperatures i.e. the glass viscosity is reduced at high temperatures and fibre formation improves).

From the figures the advantages of glass reinforcement and the powder route are fairly obvious. Unreinforced HE9 powder extrude has a proof stress  $2\frac{1}{2}$  times that of conventional "solid" HE9. The U.T.S. is also 60% greater and the elongation twice as large as the corresponding "solid" extrude value. The U.T.S. of the Al powder extrude is  $2\frac{1}{2}$  times that of its conventional "solid" counterpart

although its elongation is significantly less due to the high oxide content of the powder material. HE9 + 8wt.% glass extrude has proof stress and U.T.S. values which are the equal of Al-3%Mg-Mn "solid" extrude, plus a much better elongation. The properties of unreinforced HE30 are comparable to those of Al-3%Mg-Mn "solid" although once again, the powder derived material has a much better elongation value. Finally, the properties of Al-3%Mg powder extrude are superior in everyway to those of Al-4.5%Mg-Mn "solid" extrude.

It must be stated that the temperatures employed during the extrusion of the reinforced materials were probably higher than necessary for good fibre formation. Thus the figures given for the reinforced materials are probably not the optimum values attainable. i.e. HE9 + 8 wt.% glass could probably be extruded at 350°C giving even higher proof stress and U.T.S. values.

#### 5.9.2 Analysis of fracture behaviour

The type of fracture experienced by the various materials during tensile testing at room temperature and 400°C is listed in table 34.

Both the HE9 and HE30 specimens exhibit necking prior to fracture. At room temperature and 400°C transparticle, cup and cone, ductile fracture occurs (Plate 19(a) and (b) ). Glass additions cause embrittlement of the HE9 extrude although a cup and cone type of fracture still takes place (much less necking occurs prior to fracture). Plate 19(e) shows the fracture surface of a glass reinforced HE9 extrude. The ends of the fibre channels are clearly visible.

The Al and Al+glass extrudes experience a very brittle type of fracture with a complete absence of necking. On examination of the surfaces the fracture appears interparticle in nature, probably due to the high oxide content present.

(Plate 19(c) and (d)). A large crack, probably initiated at an oxide fragment, is also visible in 19(c).

The Al-Mg alloy undergoes a fairly ductile type of fracture at both test temperatures. At room temperature, some necking precedes a  $45^\circ$  shear, transparticle, ductile fracture. At  $400^\circ\text{C}$  transparticle, cup and cone, ductile fracture occurs. Once again, the addition of reinforcing glass causes embrittlement.

### 5.9.3 Effect on extrude properties of variations in extrusion conditions

The variation in mechanical properties, with initial billet temperature, is shown for the various materials in Figs 68-72.

In the case of HE9 (Fig. 68) the room temperature proof and ultimate stresses show the expected decrease with increasing billet temperature (in the range  $250\text{--}480^\circ\text{C}$ ) which is due to softening, probably by dynamic recovery. However, from  $480$  to  $540^\circ\text{C}$  the trend is reversed: the material becomes stronger and elongation continues to improve. This may be because, at the higher temperatures, a limited amount of magnesium silicide ( $\text{Mg}_2\text{Si}$ ) is taken into solution and on subsequent air cooling after extrusion a supersaturated solution with a non-equilibrium vacancy concentration is formed. A natural<sup>73</sup> ageing process at room temperature might then produce a fine precipitate of  $\text{Mg}_2\text{Si}$ . The room temperature proof stress varies from  $75\text{--}125\text{ MN/m}^2$  over the temperature range investigated which compares with an as-extruded strength of  $50\text{--}55\text{ MN/m}^2$  for the normal cast alloy. In fact this strength compares well with that of the heat treated alloy, quoted as  $130\text{--}160\text{ MN/m}^2$ . The elongation varies from approximately 22-27%, which is remarkable since the normal as-extruded product is assigned a value of 12% for this property and the heat-treated alloy only 7%. These excellent elongation figures are most



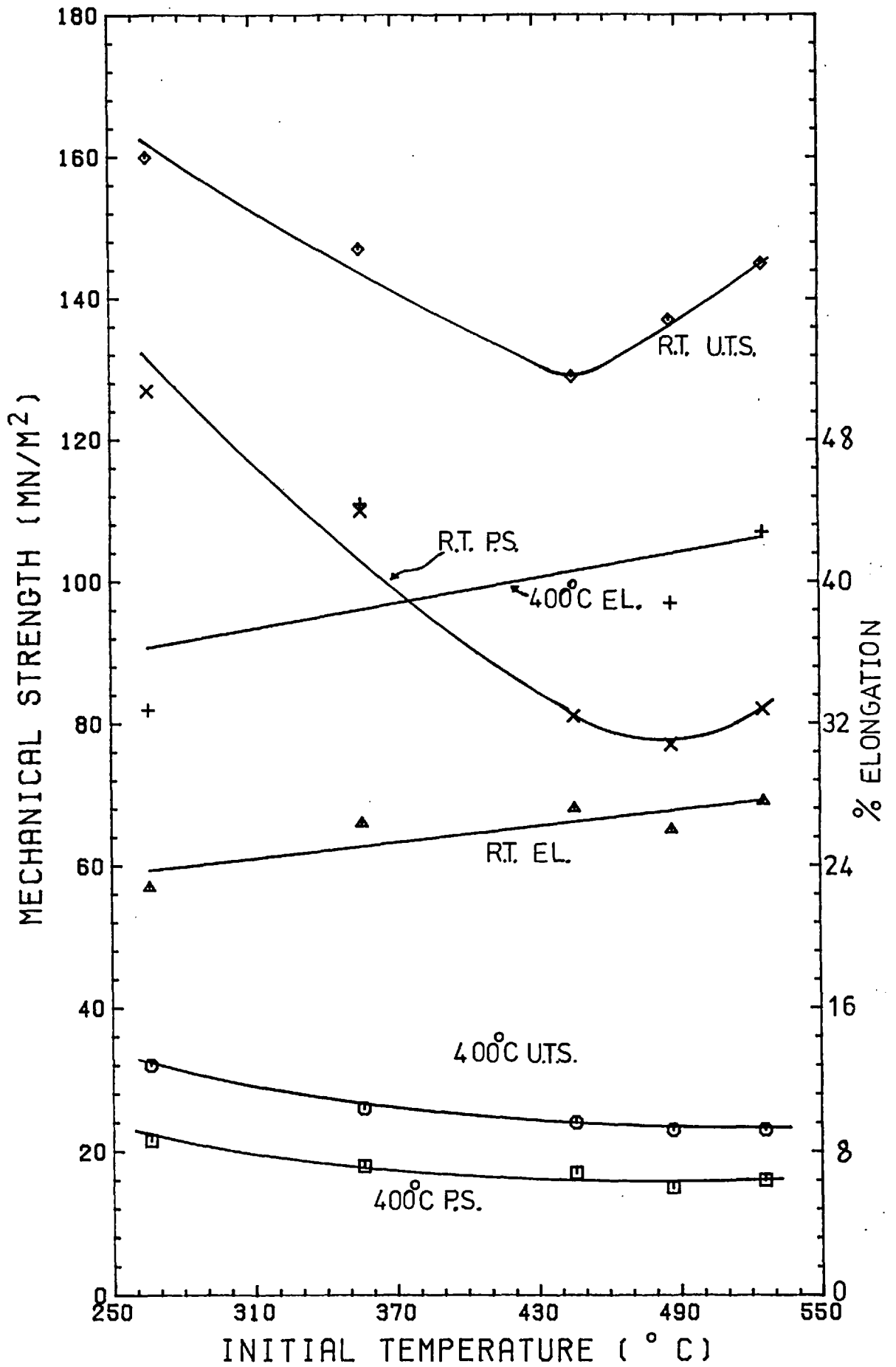


FIG. 68 Effect of extrusion temperature on HE9 mechanical properties

probably due to the inherently fine structure and small particle size of the atomized powder. The increase in elongation with billet temperature is also due to enhanced recovery at the higher temperatures causing more efficient dislocation elimination.

The strength of the extrude at 400°C decreases slightly with increasing billet temperature. This indicates that at the higher test temperature the form and distribution of precipitate have little effect on resistance to deformation. This alloy is therefore unsuitable for high-temperature applications.

Fig. 69 shows the effect of billet temperature variation on the properties of HE30. Generally, the behaviour is similar to that of HE9. However, the room temperature proof and ultimate stresses also decrease, with increasing billet temperature, from 480-540°C. This behaviour is the reverse of that exhibited by HE9 in the same temperature range. This is somewhat surprising as HE30 is more responsive to conventional heat treatments. The excellent elongation values (32-44%) may once again be attributed to the structure and particle size of the atomized powder. In fact the HE30 elongation values are even better than those of HE9. This is probably due to the HE30 powder median particle size (122 $\mu$ ) being finer than the HE9 powder (141 $\mu$ ). Fig. 70 clearly demonstrates that the strength of extruded Al-Mg decreases with increasing billet temperature over the range 400-550°C. (The minimum temperature investigated was dictated by the press capacity). The elongation is little affected by this parameter and remains approximately 21-22%.

In the case of the Al powder extrusions (Fig. 71) the proof and ultimate tensile stresses decrease with temperature from 300-400°C and then increase as the temperature is raised from 400-600°C. This increase in U.T.S. at temperatures above 400°C is particularly marked.

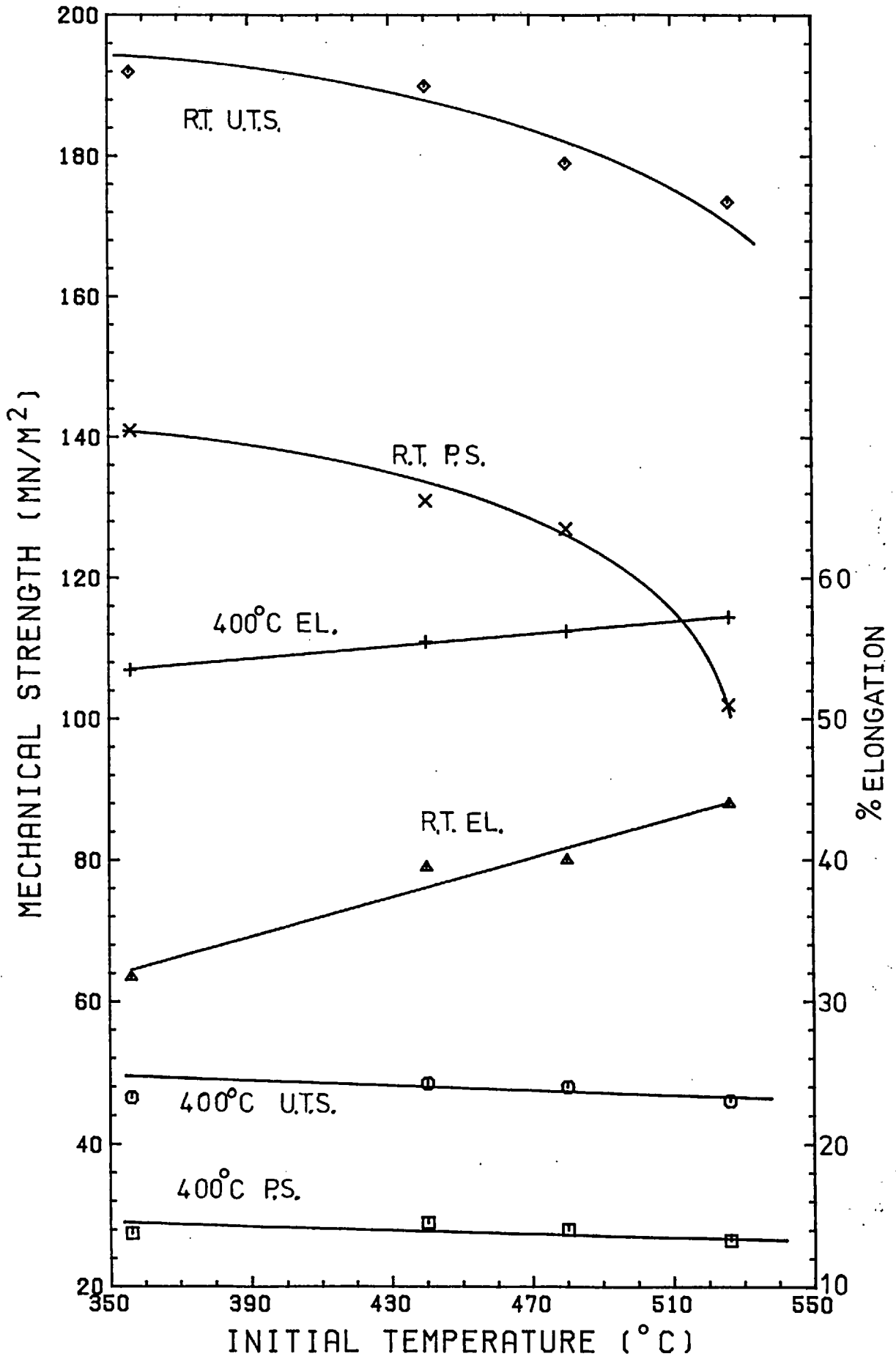


FIG. 69 Effect of extrusion temperature on HE30 mechanical properties

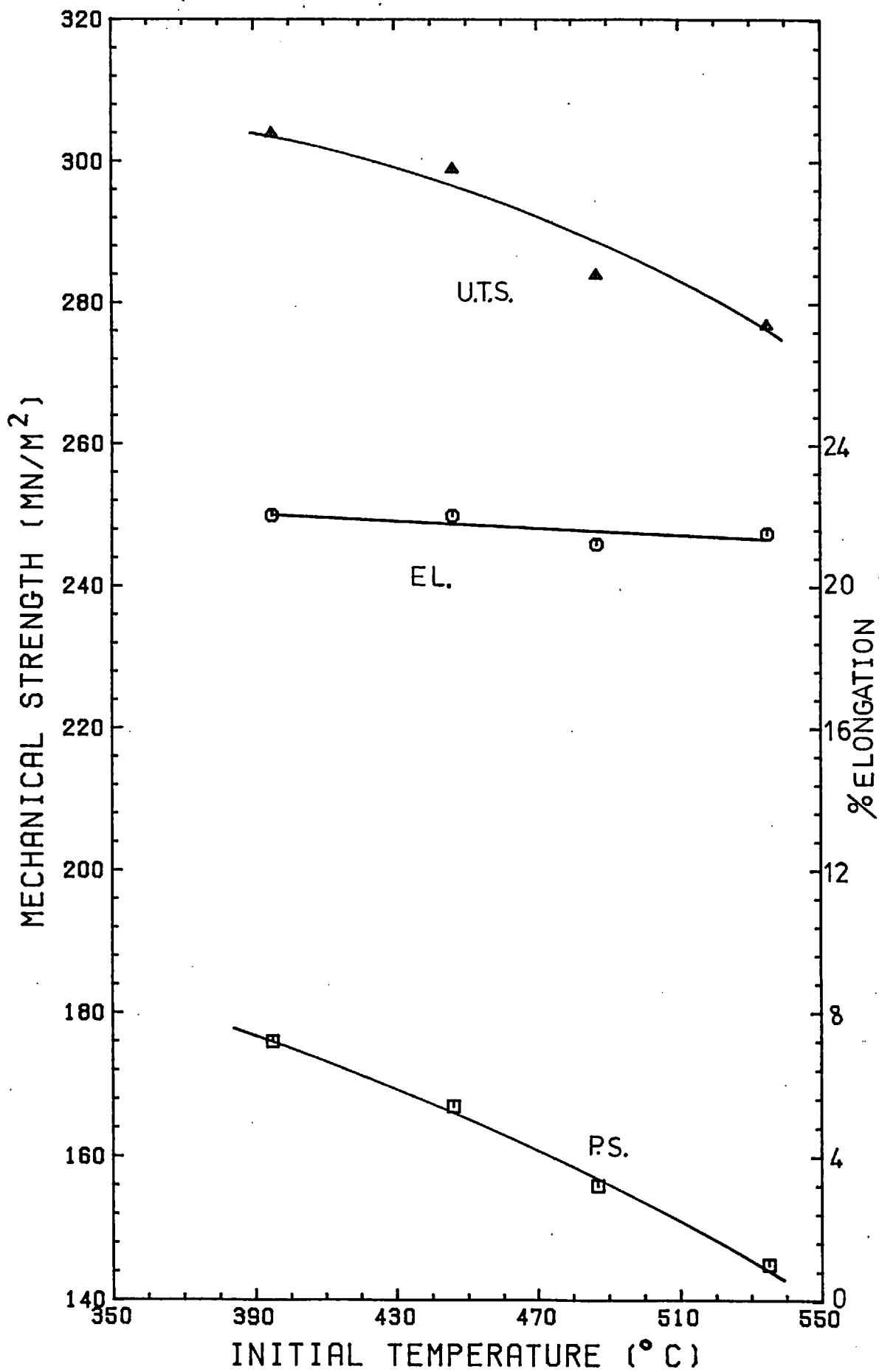


FIG. 70 Effect of extrusion temperature on Al-Mg mechanical properties

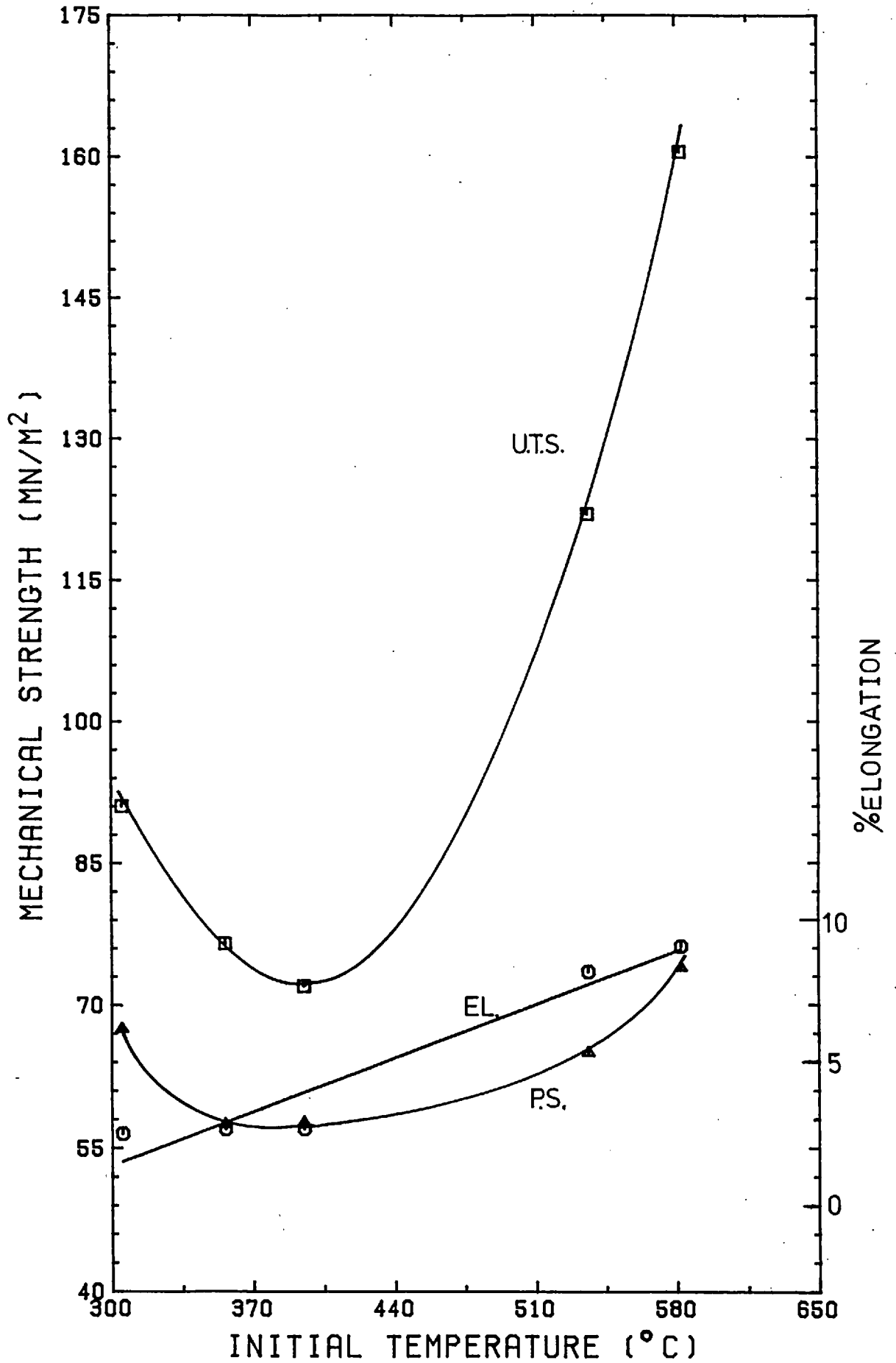


FIG. 71 Effect of extrusion temperature on Al mechanical properties

The decrease in strength with increasing temperature over the initial range may once again be due to improved dynamic softening. As mentioned in 5.6.1 increasing the temperature up to approximately 600°C increases the ductility and improves the extrude quality by preventing cracking due to secondary tensile stresses. This may in turn increase the extrude strength. In general the elongation is much lower than that of HE9, HE30 or Al-Mg due to the high oxide content of the Al powder ( $\sim 7\text{wt.}\%$ ).

The effect of varying the billet temperature on the properties of a HE9 + 5 wt.% glass composite is shown in Fig. 72. The U.T.S. increases, almost linearly with temperature from 400–550°C. This increase in U.T.S. is caused by improved matrix-fibre bonding and by an increase in fibre perfection. (At higher temperatures the glass viscosity is lower making fibre formation easier). This effect more than compensates for the softening of the metal matrix by dynamic recovery. The proof stress decreases from 90–70 MN/m<sup>2</sup>, with increasing temperature, due to enhanced softening mechanisms. As fibre reinforcement theory predicts, the proof stress is little affected by the addition of the glass. This is because the metal flows plastically before any effective reinforcement occurs. (The very slight decrease in proof stress is probably due to stress concentrating effects and is also within the range of experimental error). Once again the elongation increases with temperature.

The effect of varying the extrusion ratio is shown in Fig. 73. Over the range from 5:1 to 80:1 the proof stress increases steadily for both HE9 and HE30. Gurney<sup>24</sup> found that for extrusion ratios above 10:1 the mechanical strength of 7075 powder extrudes deteriorated. He suggested that this was due to cracking caused by excessive deformation and to the large grain size produced by recrystallisation. He further proposed that extrusion ratios above 10:1 produced cracks resulting from excessive amounts of shear during the extrusion

(continued...)

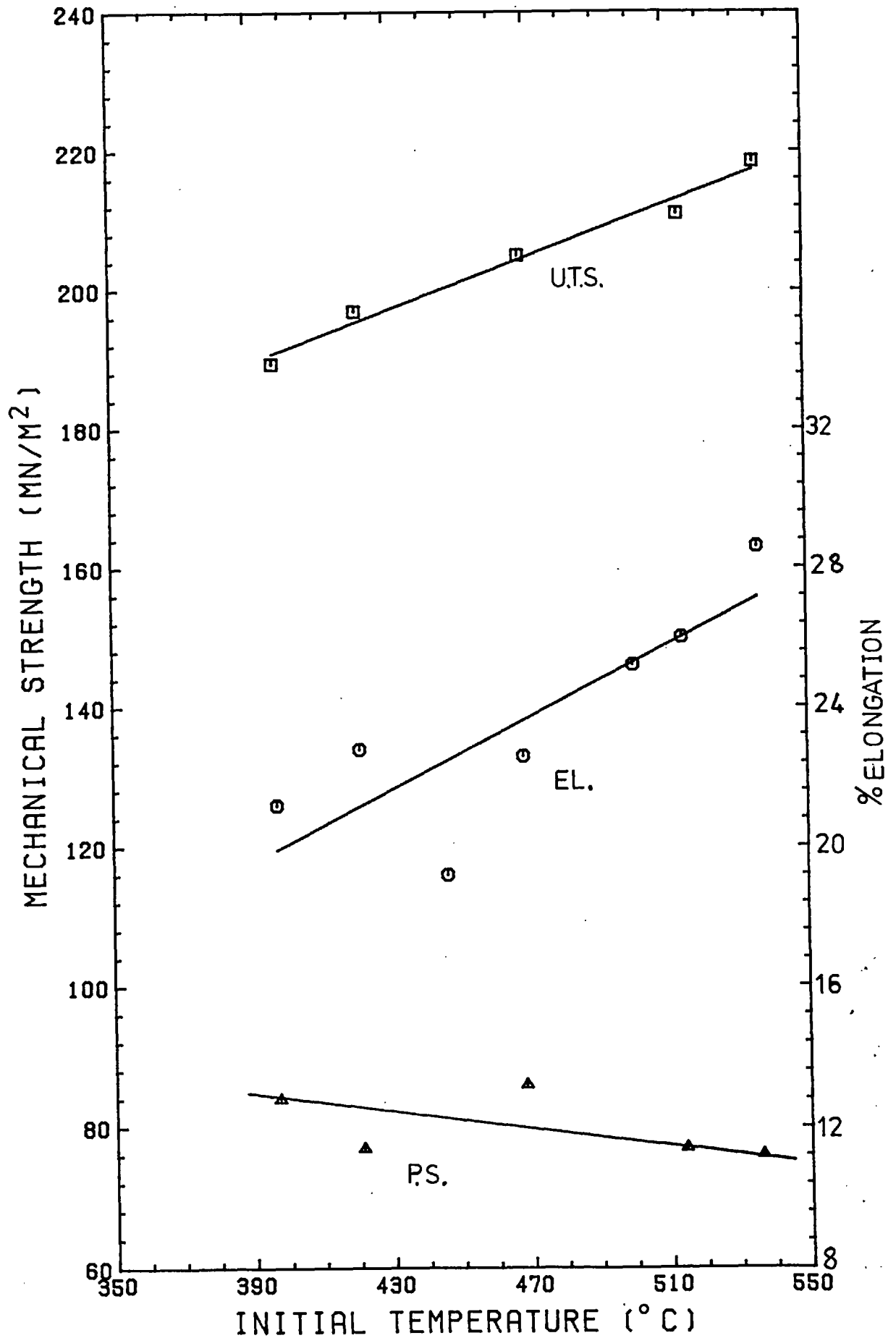


FIG. 72 Effect of extrusion temperature on HE9 + 5wt.% glass mechanical properties

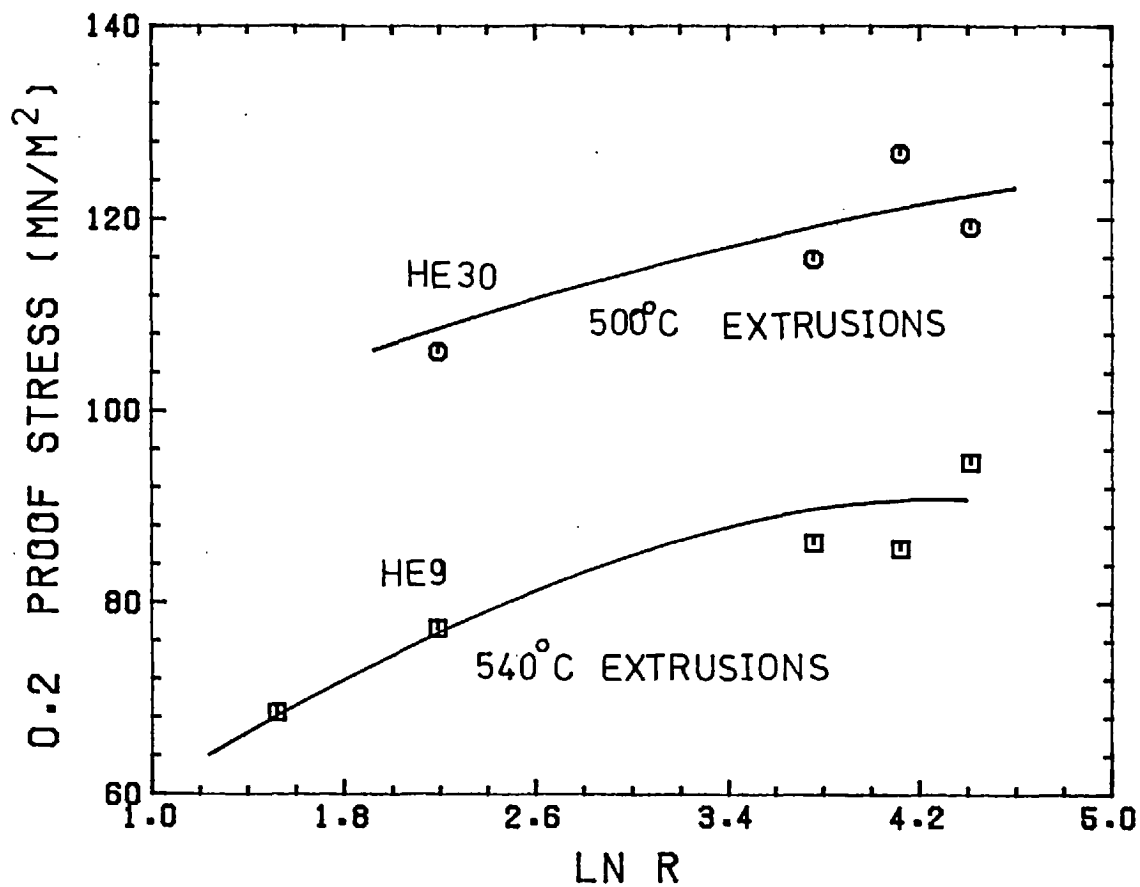


FIG. 73 Effect of extrusion ratio variation on the strength of HE9 and HE30 extrudes

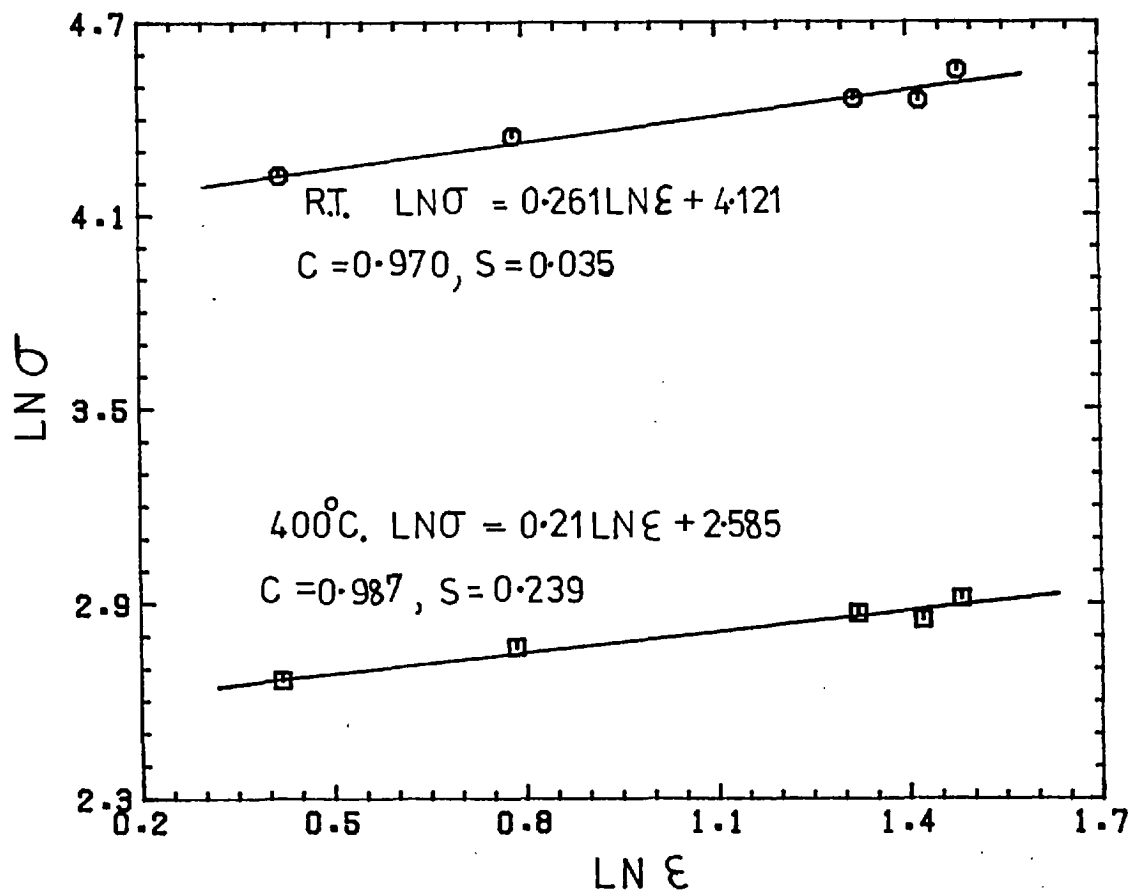


FIG. 74 Tensile test  $\ln \sigma$  vs.  $\ln \epsilon$  relationships at different temperatures (HE9)



process which could not be accommodated in the matrix near the hard particles of fragmented oxide. As no such cracks were found in the present investigation, or by other workers such as Chare<sup>23</sup>, this theory must be questioned. Fig. 74 demonstrates the applicability of a law of the form:-

$$\sigma = \text{const. } \epsilon^m$$

The relatively high correlation coefficients indicate that the experimental points fit the law quite well. The strain or work hardening exponent,  $m$ , varies from 0.26 at RT. to 0.21 at 400°C. This also indicates that an extrusion ratio of 5:1 produces fully consolidated material i.e. if a 5:1 extrude were not fully consolidated  $m$  would decrease with increasing extrusion ratio over the range 5:1 to 80:1.

#### 5.9.4 Dependence of extrude properties on test conditions

The variation in the proof stress of HE9 with tensile test temperature is shown in Fig. 75. As expected, the strength decreases rapidly with increasing temperature. This curve very closely resembles that depicted in Fig. 25. (predicted by Seeger's<sup>109</sup> analysis 3.8.1). Therefore the curve may be represented by an equation of the form:-

$$K_s = \frac{[U_o - KT \ln (\frac{\dot{\epsilon}}{\epsilon_o})]}{V} + K_A \quad (\text{See 3.8.1 for symbols}) \quad (67)$$

The athermal component of the flow stress, due to long range obstacles such as oxide dispersions, precipitates etc. is designated as  $K_A$  in (67). From Fig. 75 the value of  $K_A$  is 16 MN/m<sup>2</sup>. For the normal cast alloy  $K_A$  is quoted as 7-8 MN/m<sup>2</sup>. The discrepancy is caused by the presence of fragmented oxide particles in the powder extrude.

Fig. 76 indicates the effect of strain rate on the proof stress of HE9 and HE30. Both alloys obey a law of

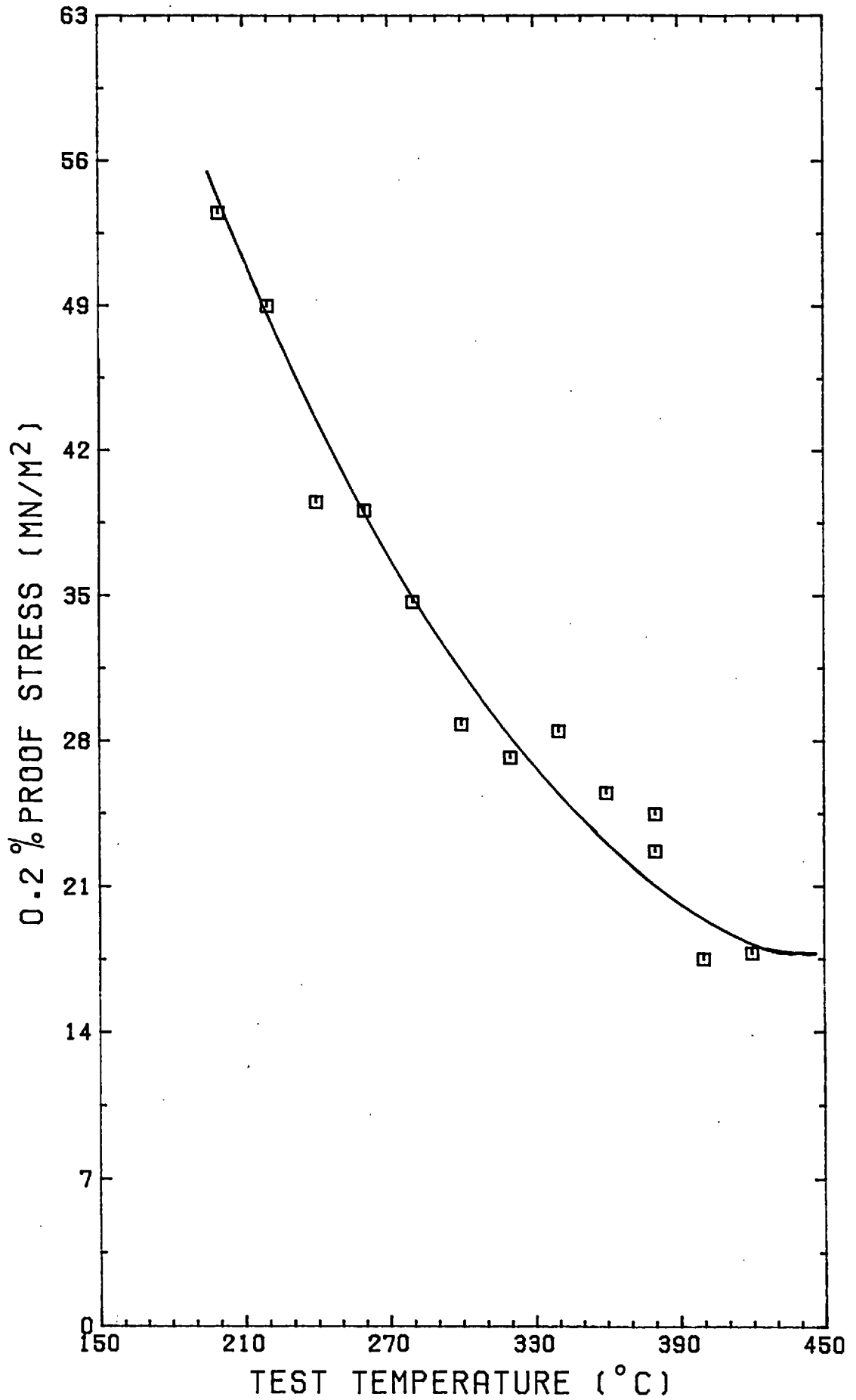


FIG. 75 HE9 proof stress as a function of temperature

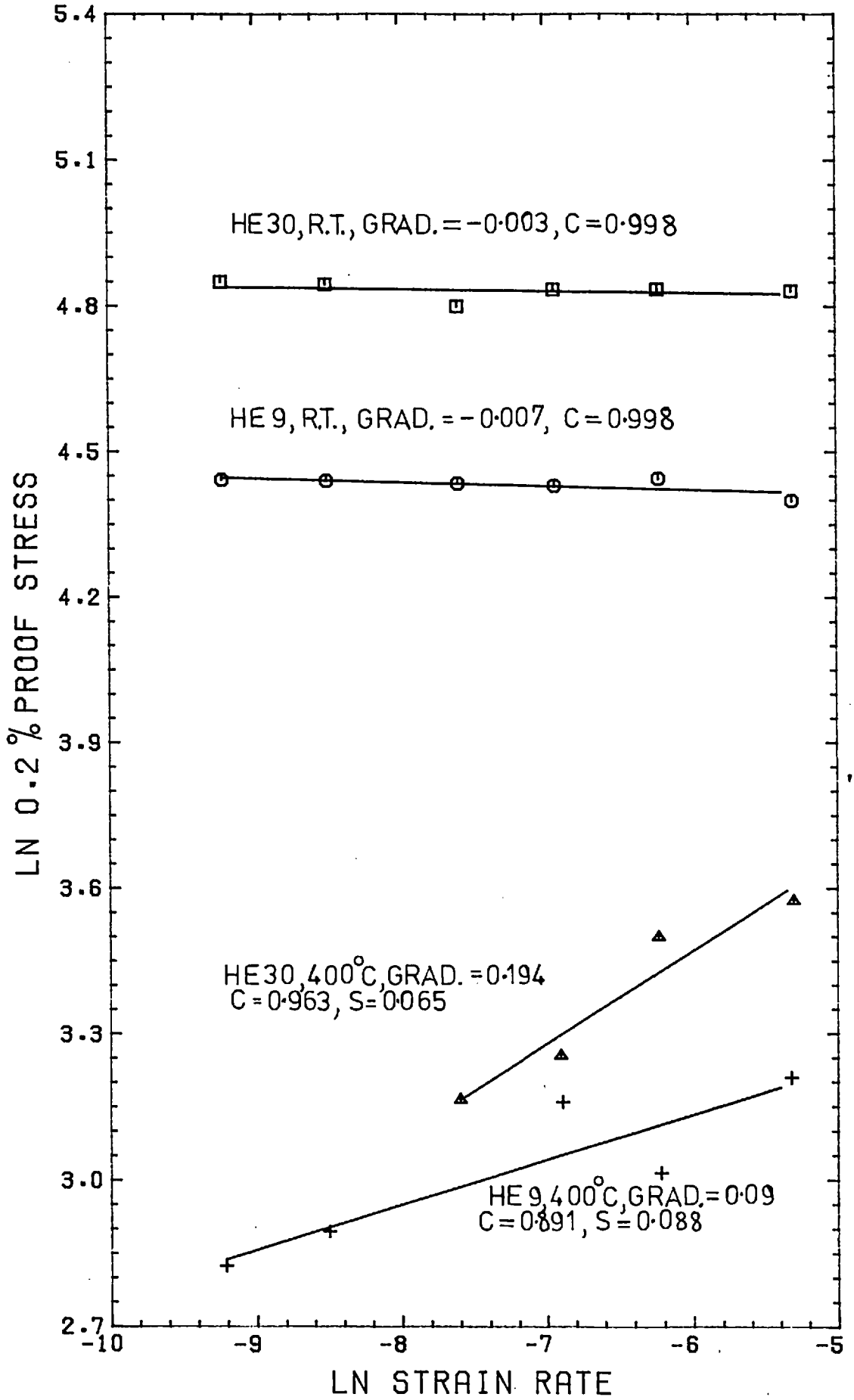


FIG. 76 Effect of strain rate on proof stress of HE9 and HE30

the form:-

$$\sigma = \text{const. } \dot{\epsilon}^n$$

The reasonably high correlation coefficients indicate a good fit between the experimental points and the theory. Raising the test temperature from room temperature to 400°C has a dramatic effect on the strain rate exponent of both alloys. The value of  $n$  changes from -0.007 to +0.09 for HE9 and from -0.003 to 0.19 for HE30. This trend was also found in 7075 aluminium alloy by D'Antonio et al.<sup>110</sup> and by Alder and Philips<sup>134</sup> in commercially pure aluminium. In particular D'Antonio discovered variations in the strain rate exponent over similar temperature and strain rate ranges (i.e.  $< 10^{-2}$ /sec) to those investigated in the present work. The increase in proof stress with strain rate at 400°C occurs because the work hardening rate exceeds the recovery rate by an ever increasing amount as the strain rate rises. The negative exponent at room temperature is not so easily explained. Lubahn<sup>111</sup> has reported that the strain rate exponent of 61S alloy (Al-0.6%Mg-Si) attains a minimum, negative value at room temperature. He suggests that this phenomenon is due to strain ageing which is most prevalent in 61S at room temperature. Some materials, during plastic deformation, suffer dislocation pinning by solute atom clusters. As the load increases the stress acting on the dislocations builds up and eventually the dislocations are torn free. The stress needed to maintain dislocation motion is less than that needed to initiate it and so the load falls slightly. However, the solute atoms diffuse rapidly and "repin" the dislocations. Thus the cycle is complete. Repetitions of this cycle cause the "saw tooth" effect in the load extension curve (Fig. 67). If the strain rate is increased a stage is reached when the diffusion rate of the solute atoms is not great enough to permit this "repinning" to occur and this in turn lowers the flow stress. This mechanism may be operating in the HE9 and HE30 alloys although no serrated

yielding is apparent in their tensile test curves. Strain ageing does not occur at higher temperatures (i.e. 400°C) because the increased solute atom diffusion rate affords the atoms insufficient time to repin dislocations.

This n value of 0.09 for HE9 at 400°C agrees well with the value of 0.1 found from torsion results (5.5.1). This is quite interesting considering that the torsion tests were performed at strain rates several orders of magnitude higher than those used in the tensile tests.

#### 5.9.5 Dependence of extrude properties on powder particle size

The mechanical properties of extruded HE9 greatly depend on the initial powder particle size, as seen in Fig. 77. The U.T.S., 0.2% proof stress and elongation all deteriorate with increasing powder particle size. Chare<sup>23</sup> found, for Al powder, that the proof stress increased linearly with (initial particle diameter)<sup>1/2</sup>, the effect being more pronounced at room temperature than at 400°C. (The greater amount of thermal energy possessed by dislocations at 400°C enables them to overcome obstacles, such as subgrain boundaries, more easily). Al-Fe, Al-Mn and Al-Mg-Zn alloy powders were found to behave in a similar manner although the overall strengthening effect, produced by decreasing the particle size, was much smaller. Chare also found the extrude subgrain size to increase linearly with the initial powder particle size. Therefore he suggested that the proof stress was also proportional to (subgrain size)<sup>1/2</sup>. Hansen<sup>84</sup> extruded aluminium powder and found that the Al<sub>2</sub>O<sub>3</sub> behaved like a three-dimensional network of dispersoids. He also suggested a Hall-Petch equation of the form:-

$$\tau = \tau_s + \text{const. } \lambda^{-1/2} \quad (9)$$

to relate flow stress to oxide interparticle spacing .

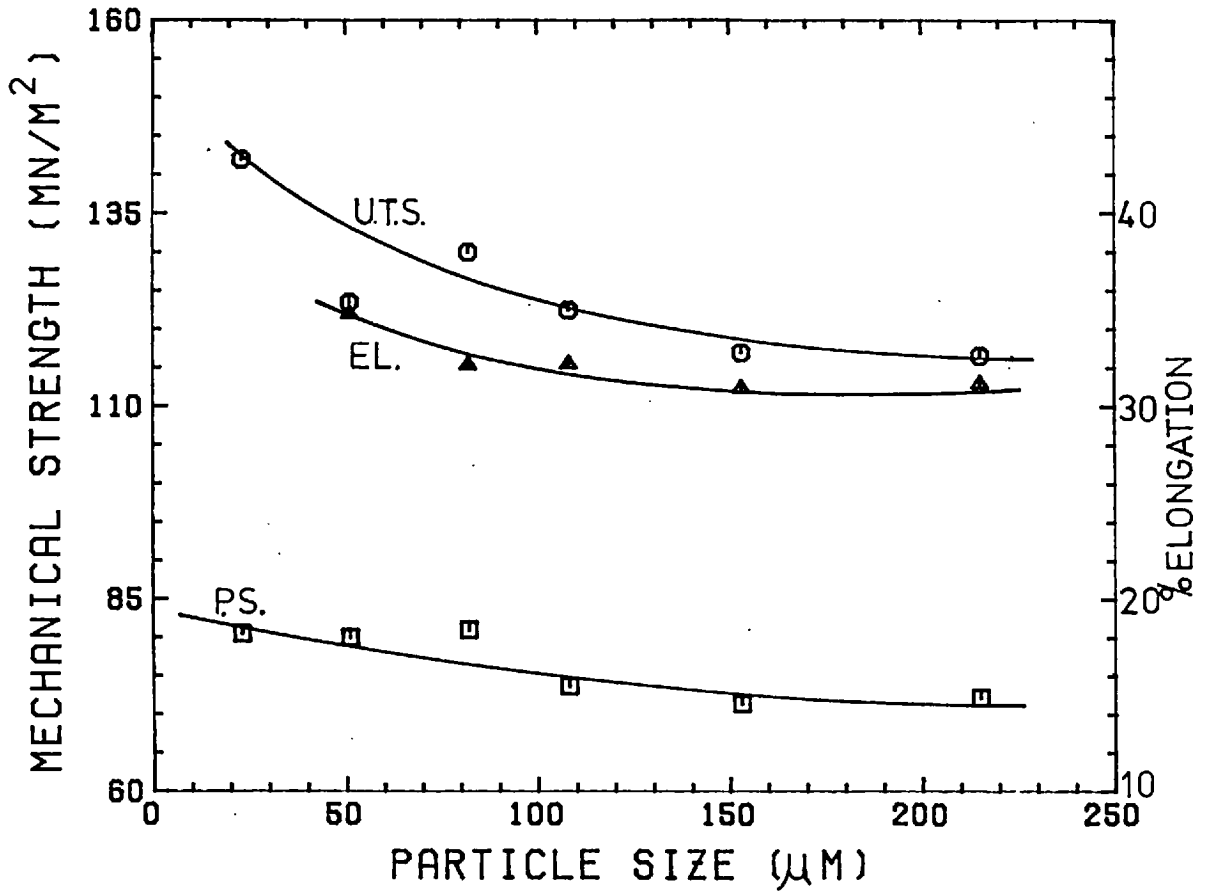
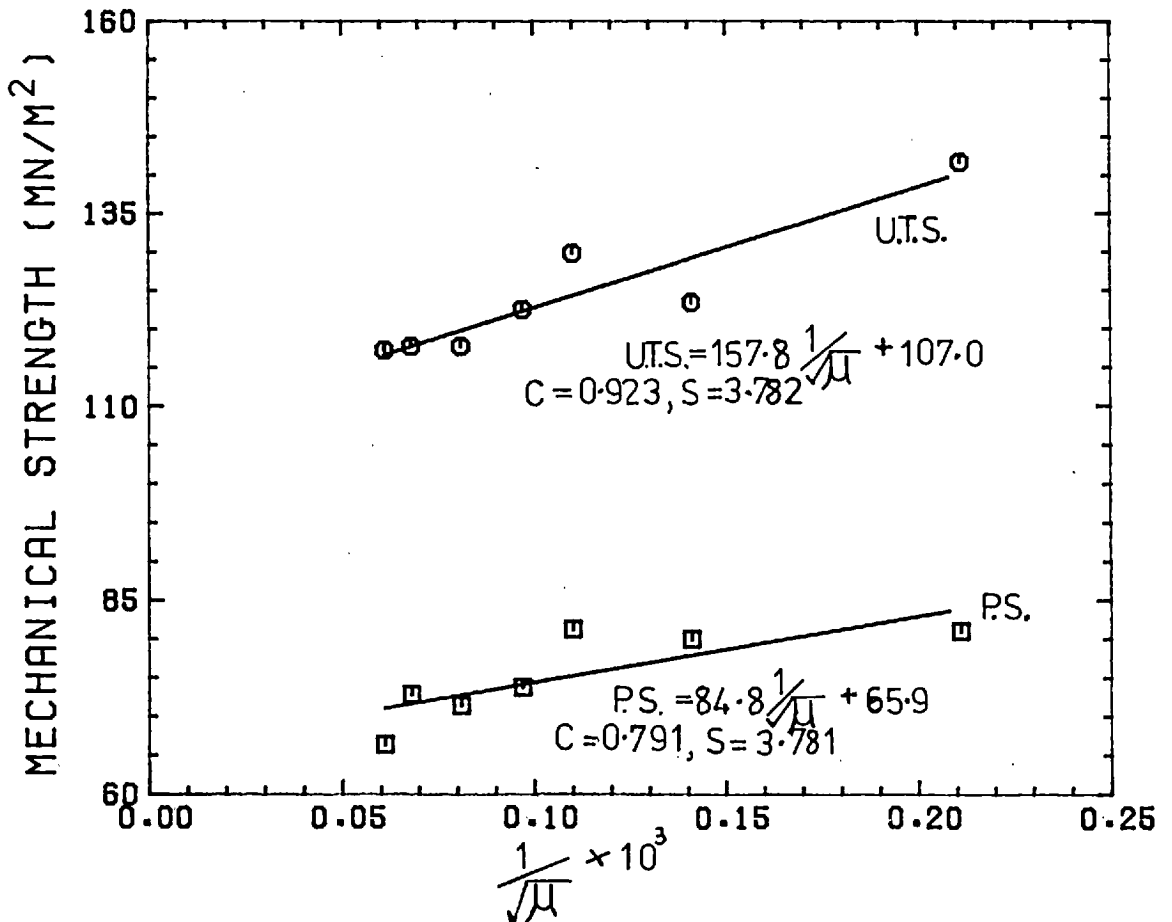


FIG. 77 Mechanical properties of HE9 extrude as a function of powder particle size

FIG. 78 Relationship between HE9 extrude strength and (powder particle size)<sup>1/2</sup>



In fact  $\lambda$  is approximately equal to the mean powder particle diameter.

Fig. 78 shows the application of this relationship to the HE9 results giving an equation:-

$$\tau = 65.9 + 84.8 \lambda^{-\frac{1}{2}} \quad (87)$$

However, the low correlation coefficient of 0.79 casts some doubt on the applicability of the relationship. More experiments are required before definite conclusions can be drawn. (In fact the U.T.S. data fits the relationship rather better giving an equation:-

$$\tau = 1070 + 157.8 \lambda^{-\frac{1}{2}} \quad (88)$$

correlation coefficient = 0.92.)

This Hall-Petch type relation between powder particle size (or indeed subgrain size) and proof stress corresponds to the relation in solid materials between grain size and flow stress.

This latter relation is often expressed in the equation:-

$$\tau_y = \tau_i + k D^{-\frac{1}{2}}$$

where  $D$  = grain size

$\tau_y$  = flow stress

The term  $\tau_i$  is the lattice friction stress and  $k$  is a term, the magnitude of which is proportional to the difficulty a dislocation experiences in traversing a grain boundary.

### 5.9.6 Effect on extrude properties of variations in glass content

The significant effect of glass content on mechanical properties is illustrated in Figs. 79-81 for HE9, Al-Mg and Al respectively. Equation (44) in 3.7.3 i.e.

$\sigma_c = \sigma_m (1 - V_F) + \sigma_F V_F$ , predicts a linear increase in U.T.S. with volume fraction of glass. This is observed for HE9 over the range 0-8 wt.% glass. Further increases in the glass content rapidly weaken the composite. By applying equation (44) the U.T.S. of the glass fibres may be obtained from a knowledge of the composite strength. e.g. at 5 wt.% glass i.e. 9.2 vol % glass (Density of glass =  $1410 \text{ kg/m}^3$ )

$$\sigma_c = \sigma_m V_m + \sigma_F (1 - V_m) \quad (44)$$

$$\therefore 200 = 130 \times 0.91 + \sigma_F (1 - 0.91)$$

$$\therefore \sigma_F = 891 \text{ MN/m}^2.$$

This value is much lower than quoted values for commercially available glasses such as E - glass ( $3700 \text{ MN/m}^2$ ) silica ( $4600 \text{ MN/m}^2$ ) and  $\text{B}_2\text{O}_3$ -10%  $\text{SiO}_2$  ( $2412 \text{ MN/m}^2$ ). This indicates the poor quality of the fibres formed during extrusion.

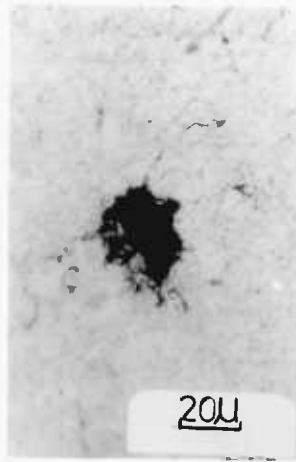
The decrease in strength with increasing glass content above 8 wt.% is probably due to the difference in bulk modulus between the glass and metal.

( $K_{\text{matrix}} = 26.2 \times 10^3 \text{ MN/m}^2$ ,  $K_{\text{glass}} = 13.8 \times 10^3 \text{ MN/m}^2$ ).

The elastic expansion of the glass fibres is much greater than that of the metal matrix. At typical extrusion temperatures the matrix is weak and the force exerted by the glass, on emerging from the extrusion die, produces cracks leading to drastic weakening of the composite.

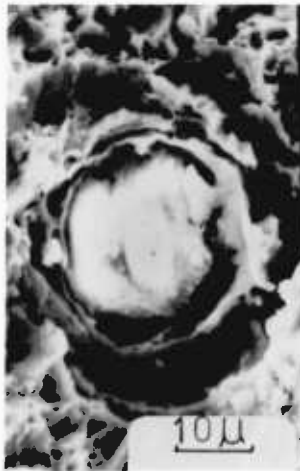
Plate 10(a) is a transverse section of a HE9 + 5 wt.% glass extrude showing cracks in the matrix surrounding a





(a)

Transverse optical micrograph  
of fibre channel



(b)

Transverse scanning electron  
micrograph of fibre

PLATE 10 Micrographs of HE9 + 5 wt.% glass  
extruded at 450°C

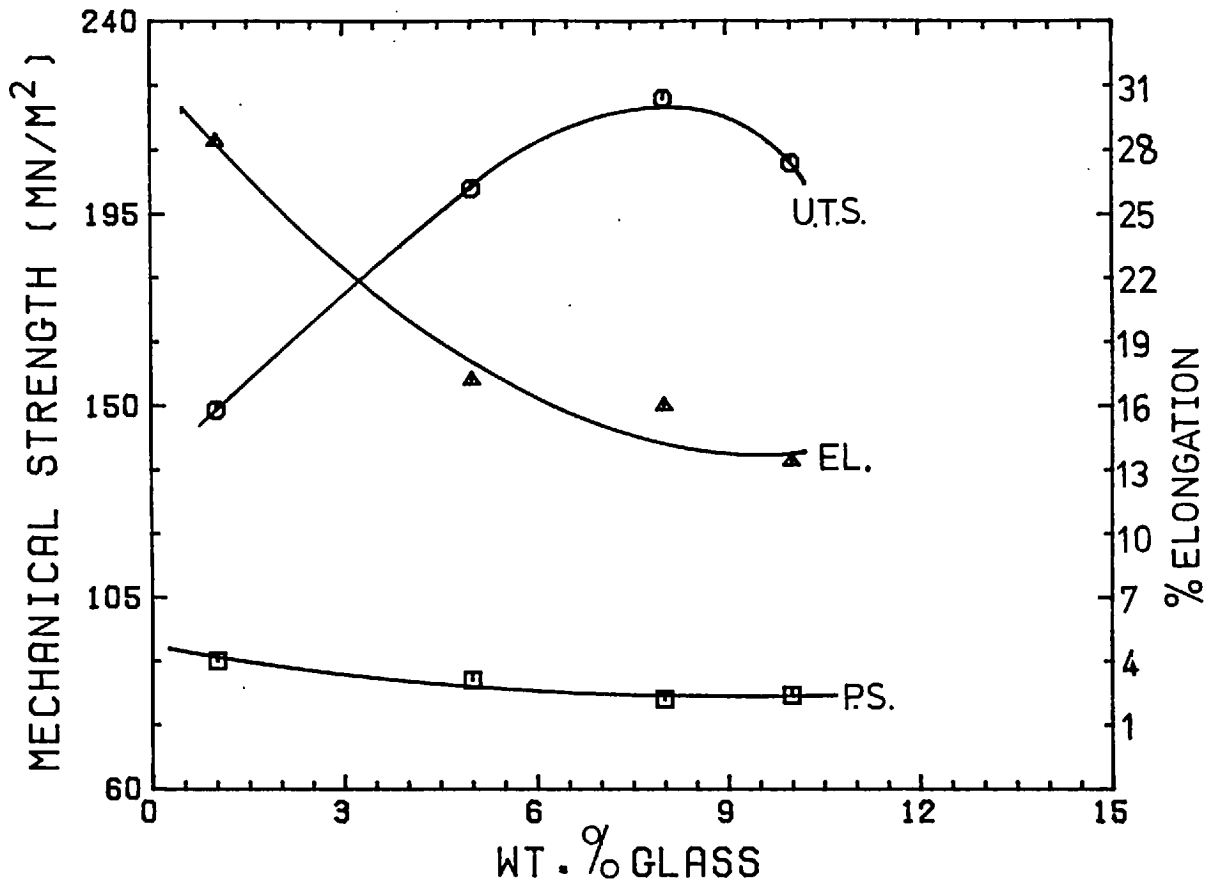


FIG. 79 Effect of variations in glass content on mechanical properties of HE9-glass extrudes

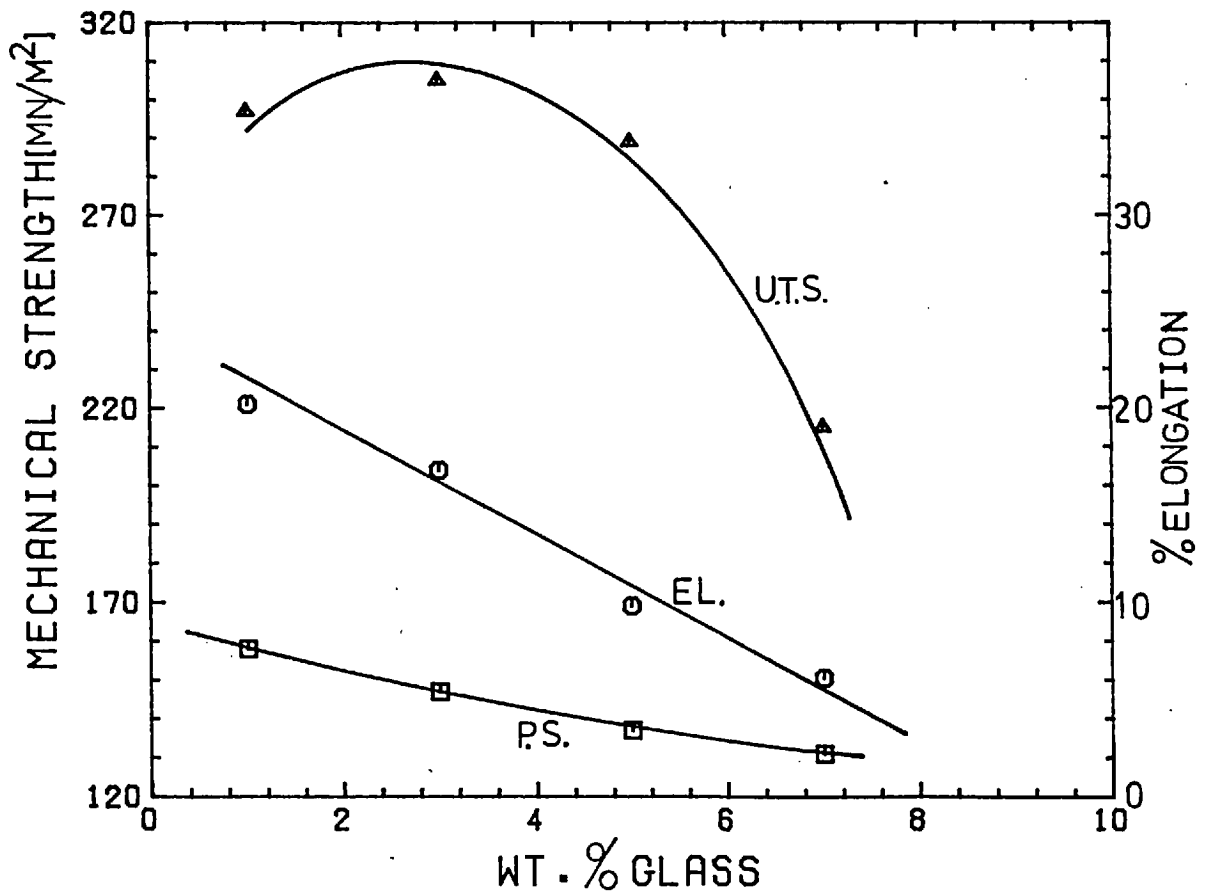


FIG. 80 Effect of variations in glass content on mechanical properties of Al-Mg-Glass extrudes

fibre. When the glass content is raised to 12-15 wt.% disintegration of the extrude results (Plate 9(h)). The optimum glass content of 8 wt.% improves the U.T.S. of the HE9, extruded at the same temperature, by 71%. The proof stress decreases with glass content due to matrix cracking and the stress concentrating effect of fibre ends. (In theory the proof stress should be unaffected by glass fibres because the metal yields before any reinforcement occurs). The addition of glass reduces the elongation value because of the inherent brittleness of the glass and the presence of matrix cracks.

Similar behaviour is observed for Al-Mg glass composites in Fig. 80. As Al-3wt.%Mg is a much stronger alloy than HE9 the peak in the U.T.S. curve might be expected to occur at a glass content in excess of 8 wt.%. In fact the peak U.T.S. occurs at only 3 wt.% glass. Further additions reduce the strength significantly. This shift in peak U.T.S. is probably due to adverse effects on the fibre strength, of chemical reactions between the glass and the abundant supply of Mg present in this alloy. As in the case of HE9 both the proof stress and the elongation decrease significantly with increasing glass content. (Extrudes containing more than 7 wt.% glass are impossible to machine due to disintegration). Overall, the properties of Al-Mg-Glass composites are very disappointing.

The U.T.S. of the Al-glass composite increases continuously, though non-linearly, with glass content as depicted in Fig. 81. Extrudes containing more than 9 wt.% glass disintegrate on machining and so it must be assumed that 9 wt.% is the optimum glass content. The U.T.S. of the 9 wt.% is only 14% greater than that of the unreinforced aluminium (extruded at the same temperature, 550°C). In fact at glass contents below

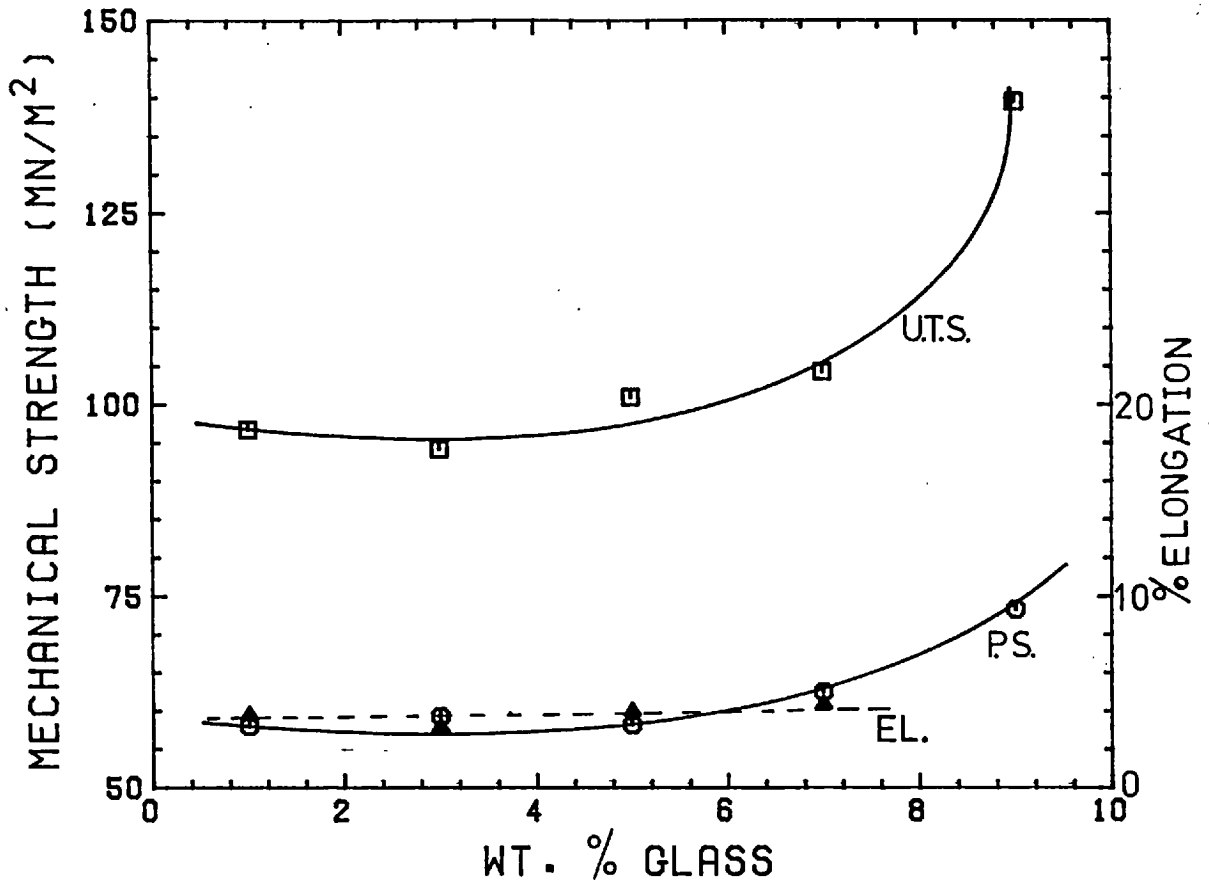
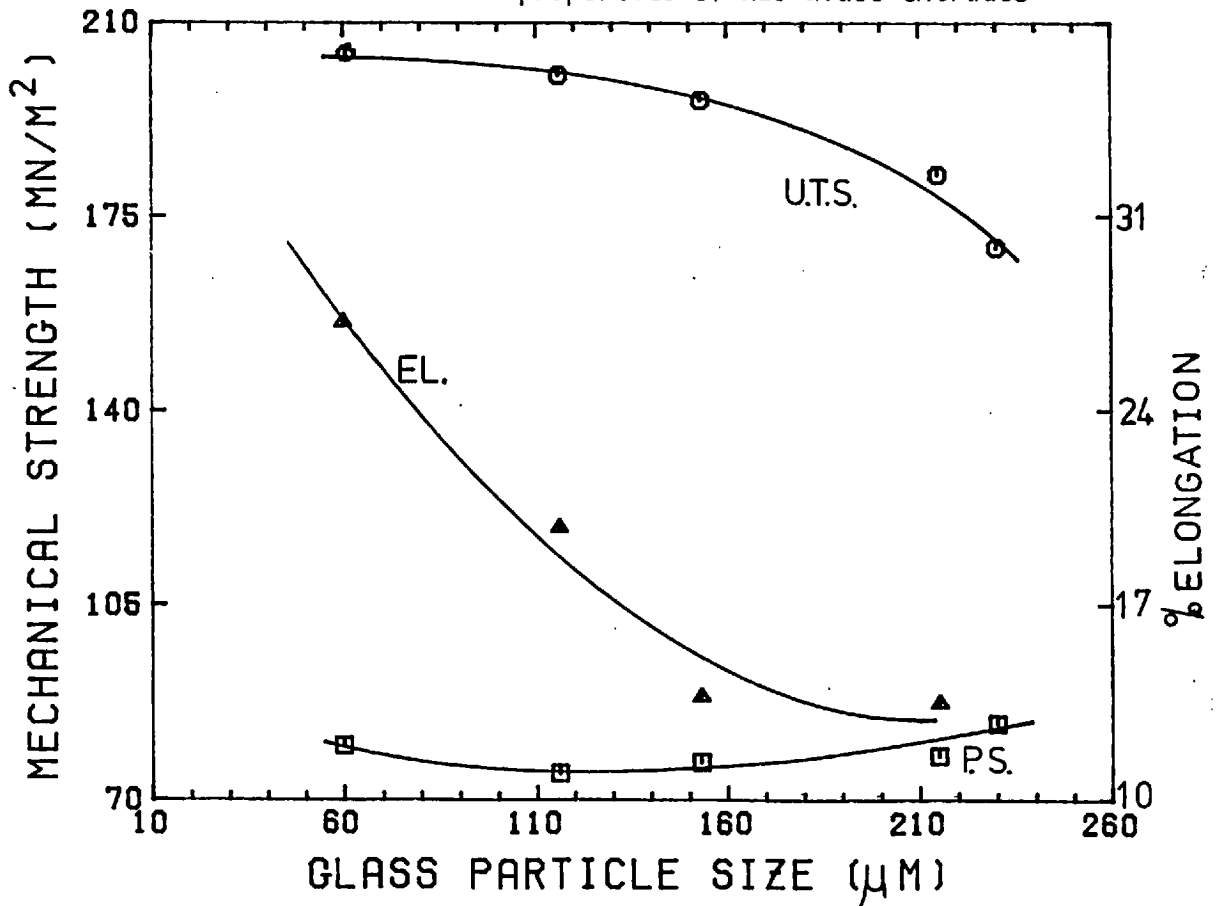


FIG. 81 Effect of variations in glass content on mechanical properties of Al-Glass extrudes

FIG. 82 Effect of variations in glass particle size on mechanical properties of HE9-Glass extrudes



~9wt.% the composite is weaker than the unreinforced matrix. The elongation value of about 4% remains largely unaffected by the glass content although the Al is very brittle due to its large oxide content and the measurement accuracy of such small values must be in some doubt. The proof stress appears to increase with glass content although this conclusion is based on only one data point at 9 wt.% glass. Thus it is more likely that no significant change, in fact, occurs in proof stress. Overall, the results for Al-glass composites are disappointing.

The effect of varying the initial glass particle size is shown in Fig. 82. The U.T.S. decreases rapidly with increasing particle size. This is fairly easy to explain. For a given weight of glass a reduction in particle size increases the surface area/volume ratio producing more metal-glass interfacial area. This leads to better bonding, better load transfer and higher U.T.S. values. As expected, the proof stress is largely unaffected by the glass particle size. The elongation decreases rapidly with increasing glass particle size because large fibres suffer more damage, during formation in the die, than small ones. (This may also contribute to the reduction in U.T.S.).

#### 5.10.1 Hardness results

The results of hardness tests carried out on the various extruded materials are given in table 23. Generally the hardness is governed by the alloy content such that the materials, in order of hardness are:

- |               |        |
|---------------|--------|
| 1) Al-3wt.%Mg | 3) HE9 |
| 2) HE30       | 4) Al  |

(This is also the order in terms of tensile strength).  
No correlation exists between extrude hardness and extrusion

speed. Some of the results indicate a trend towards a decrease in hardness with increasing extrusion temperature. Chare<sup>19</sup> found a definite decrease in the hardness of extruded Al powder with increasing extrusion temperature. The only definite trend in the present results involves an increase in hardness with decreasing glass content. This is shown clearly in Fig. 83. Once again this is due to expansion of the glass causing cracking in the matrix. Increasing the glass content increases the number and size of these cracks thus lowering the overall hardness of the extrude.

A series of hardness measurements taken longitudinally across the deformation zone of a sectioned partially extruded billet revealed only a 5% variation in hardness and no correlation between hardness and position. This is to be expected because although the strength of the compact increases as it passes through the quasi-static deformation zone by a process of building and rebuilding interparticle welds, the hardness of the individual particles is almost unaffected. (Dynamic recovery eliminates work hardening effects). For this reason a danger lies in predicting tensile properties from hardness results for materials fabricated from powder.

#### 5.11.1 Fracture toughness

Extruded HE9 and HE30 powders exhibit no correlation between extrusion process parameters and notch ductility (IZOD) values. The results from over 30 tests show little scatter (Tables 24 and 25) and give an average impact energy value of 42.2 J for HE9 which is almost identical to that reported for the cast and wrought material (42.0 J nominal). The average value for HE30 is 38.7 J. In all cases the fracture surfaces are fibrous in nature. (None of the unreinforced HE9 and HE30 specimens fractured completely in any of the tests). Ansell and Kim<sup>137</sup> encountered similar fracture behaviour in Al-Al<sub>2</sub>O<sub>3</sub> S.A.P. alloys tested at temperatures in the range 20 to -170°C

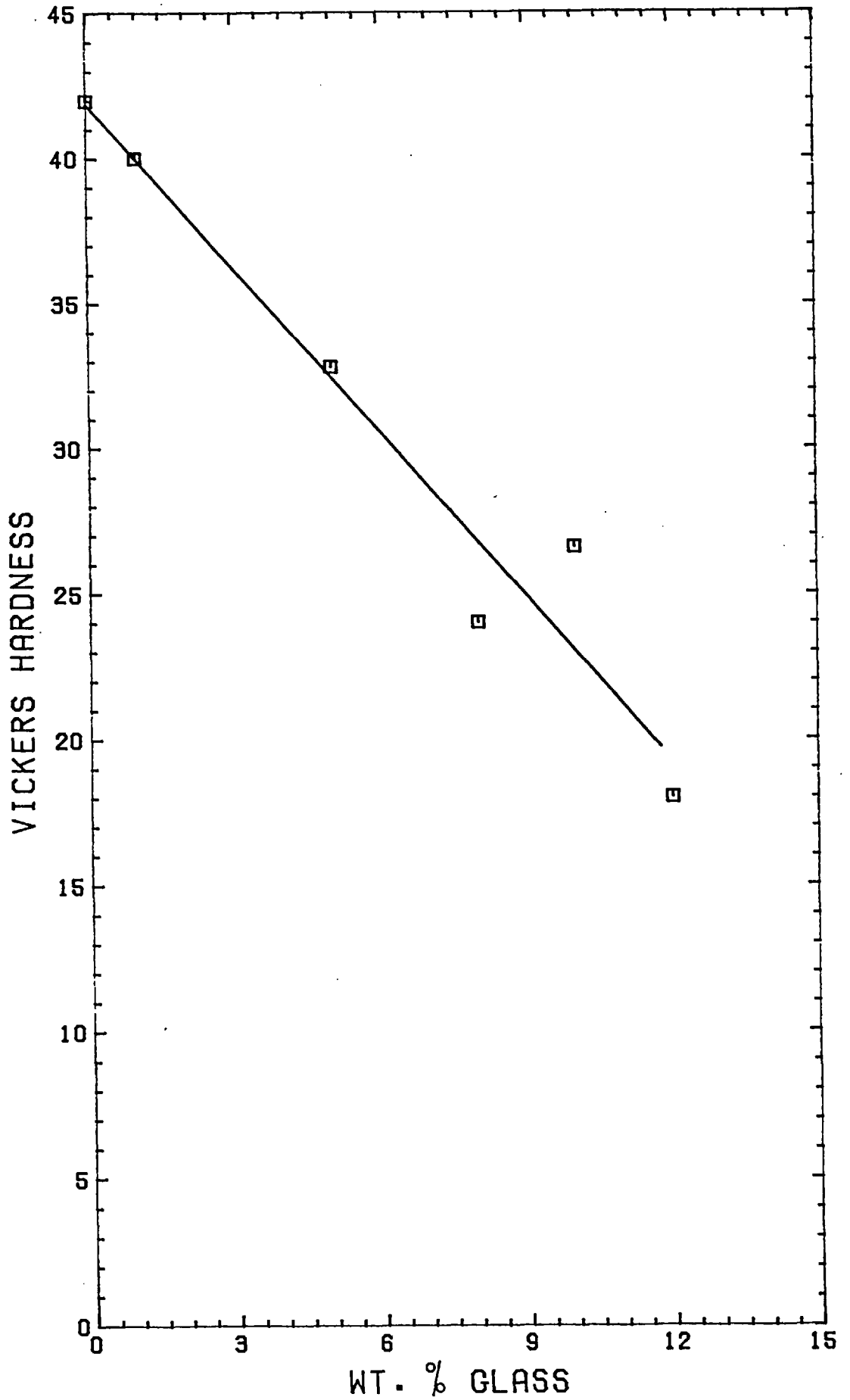


FIG. 83 Hardness of HE9 + glass extrudes as a function of glass content

Bryant<sup>141</sup> found a marked decrease in the toughness of H19 (Al, 0.4-1.4Mg, 0.6-1.3Si) with increasing extrusion temperature (for cast extrusion). He suggested that this was due to large grain boundary  $Mg_2Si$  precipitates in the recrystallised structure causing intergranular, brittle fracture. On adding 0.4-1.0 wt.% Mn (in effect changing H19 to HE30) the extrude structure changed from recrystallised to fibrous and the sharp decrease in toughness was prevented. Therefore it is to be expected that the extrusion temperature should not significantly affect the extrude toughness in the present investigation as the HE9 extrudes (containing Mn) exhibit a fibrous, cold worked structure.

The theory in 3.7.8 predicts quite high fracture toughness values for composites having ductile matrices. It predicts the occurrence of crack tip blunting which consequently prevents inter-fibre crack propagation. On the basis of this theory the results shown in Fig. 84 are rather disappointing. The fracture toughness decreases rapidly with increasing glass content. In fact an addition of only 3 wt.% glass (6 vol %) lowers the toughness by 50%. Once again these low toughness values may be partly attributed to excessive cracking in the matrix caused by elastic expansion of the fibres. In addition a chemical reaction between the glass and metal at the interface may also have a deleterious effect on the extrude toughness. The Mg present in the HE9 reacts with the glass forming  $Mg Al_2O_4$  spinel at the interface (see 5.13.1). The presence of this spinel may well cause embrittlement of the matrix. Fig. 84 also shows the effect, on the fracture toughness, of heating the test specimens to 200°C. A very slight decrease (smaller than the experimental error) is the result.



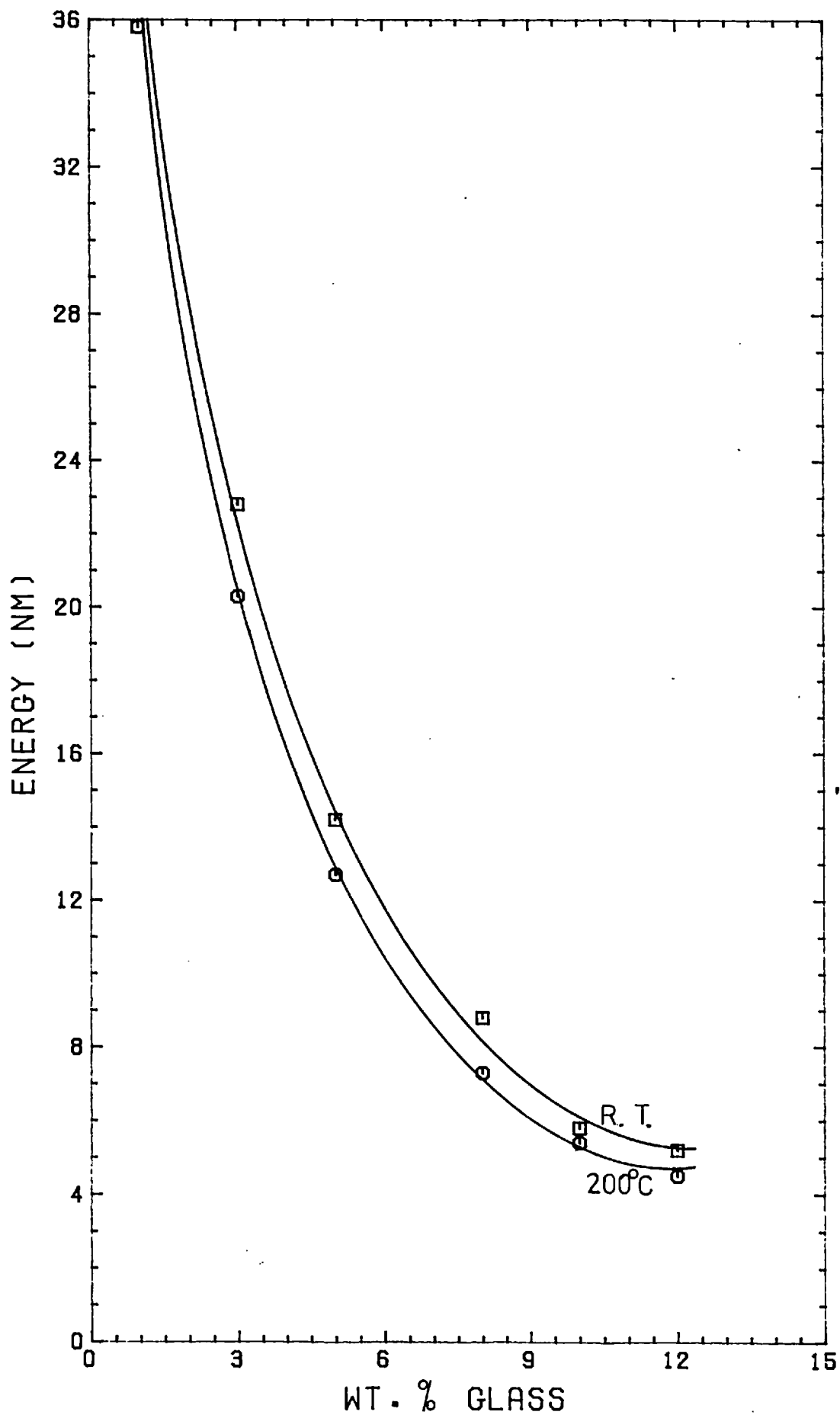


FIG. 34 Fracture toughness of HE9 + glass extrudes as a function of glass content

### 5.12.1 Heat treatment results

The results of tensile tests carried out on heat treated HE9 and HE30 extrudes are given in tables 26-31. Listed in these tables are the various heat treatments employed.

These alloys are the most widely used precipitation hardening alloys and are frequently subjected to one of three differing heat treatments designated:-

T<sub>B</sub>: solution heat treated only i.e. soaked at 500-560°C and quenched

T<sub>E</sub>: solution heat treated, quenched, cold worked and naturally aged

T<sub>F</sub>: solution heat treated, quenched and subsequently aged for several hours at 180 -200°C. A transformation of the type: needle-shaped zones → rods → platelets of equilibrium Mg<sub>2</sub>Si occurs with increasing ageing time (see 2.9.1).

Typical properties for the cast and wrought alloy subjected to the differing heat treatments are given in Table 32.

The most commonly used industrial heat treatment involves a compromise. The extrude is press-quenched (i.e. the heating and extrusion of the billet replaces the solution treatment stage) and subsequently aged. A homogenisation treatment before extrusion serves two main purposes:-

- 1) It organises the precipitate ensuring that it is taken into solution during the extrusion cycle.
- 2) It helps minimize the die pick up.

In the case of HE9 the cast billet is first heat treated to refine the  $Mg_2Si$  particles to the optimum size of  $\sim 0.5\mu$ . The billet is then reheated quickly to the extrusion temperature of  $450-480^\circ C$ . The reheat operation must be rapid to avoid coarsening of the  $Mg_2Si$  particles. The best method of reheating the billet involves the use of an induction heater. A typical heater can heat a 0.18 m diameter -0.7 m long billet to  $450^\circ C$  in 3 minutes. In this short time precipitate coarsening is negligible. During the actual extrusion procedure the work done on the metal raises the temperature in the die neck to  $490-530^\circ C$ . In this temperature range all of the  $Mg_2Si$  redissolves. The extrude is then cooled at approximately  $60^\circ C$  per minute to avoid rejection of the  $Mg_2Si$ . Finally the extrude is artificially aged for 6 hours at  $175^\circ C$  to give a fine, homogeneous precipitate ( $< 0.25 \mu m$ ).

On comparing the results for HE9 and HE30 powder extrudes subjected to a  $T_F$  type of heat treatment (tables 26-31) with those for cast and wrought 50S (table 32) it can be seen that, given the correct heat treatment, the powder extrudes are superior in U.T.S., proof stress and elongation. (The following sections 5.12.2 and 5.12.3 describe the effect of variations in the  $T_F$  type of heat treatments). Some powder billets were heated at  $450^\circ C$ , extruded and artificially aged at  $180^\circ C$  i.e. a press-quench operation. The results in table 31 show that this procedure actually weakens the product. This is almost certainly due to the lack of success of the homogenisation treatment, before extrusion i.e. very little of the silicide precipitate is dissolved. Thus only a small percentage of  $Mg_2Si$  precipitate is available for strengthening purposes. This low level of activity is indicated by the total variation in strength shown over the range of ageing times, from 2 to 6 hours, in table 31. Unfortunately this problem is not easily solved by subjecting the billets to different homogenisation treatments because the  $Al_2O_3$

layers surrounding the particles greatly hinder silicide dissolution. It might be possible to overcome this effect by compacting to higher densities (thus, it is hoped, breaking up the  $\text{Al}_2\text{O}_3$  layer) and carrying out a pre-extrusion solution soak. However, it is very doubtful whether sufficient disintegration of the oxide layers would occur. Even if such action succeeded it might prove difficult to prevent reoxidation during the soak.

The results of attempts to heat treat HE9-5wt.% glass reinforced extrudes are given in table 33. (It had been hoped to combine fibre reinforcement with precipitation hardening). Unfortunately, these results are very disappointing, with the heat treatments having little effect on the product properties. This is due to chemical reactions occurring between the  $\text{B}_2\text{O}_3$  and the magnesium. Most of the magnesium reacts with the  $\text{B}_2\text{O}_3$  leaving very little to combine with silicon for precipitation strengthening. Indeed the  $\text{B}_2\text{O}_3$ -Mg reaction degrades the fibre-metal interface<sup>138</sup>, in some cases actually weakening the product.

#### 5.12.2 Effect of soaking temperature variation on extrude properties

The important effect on the mechanical properties of HE9 and HE30, of varying the soaking temperature is shown in Fig. 85. (and table 26). The specimen extrudes were given a  $T_P$ -type heat treatment. The U.T.S. and proof stress increase with temperature up to about  $555^\circ\text{C}$  and then begin to deteriorate. At the optimum temperature,  $555^\circ\text{C}$ , the U.T.S. is increased by 28% and the proof stress by 60%. Increasing the soaking temperature increases the amount of  $\text{Mg}_2\text{Si}$  taken into solution which, in turn, produces more precipitate on ageing. This produces the increase in strength. Eventually, at about  $555^\circ\text{C}$ , a 5 hour soak effectively dissolves all the  $\text{Mg}_2\text{Si}$ . Above this temperature a slight weakening occurs probably due to subgrain growth. (At elevated temperatures the subgrains tend to grow to minimize the total subgrain boundary area and thus the

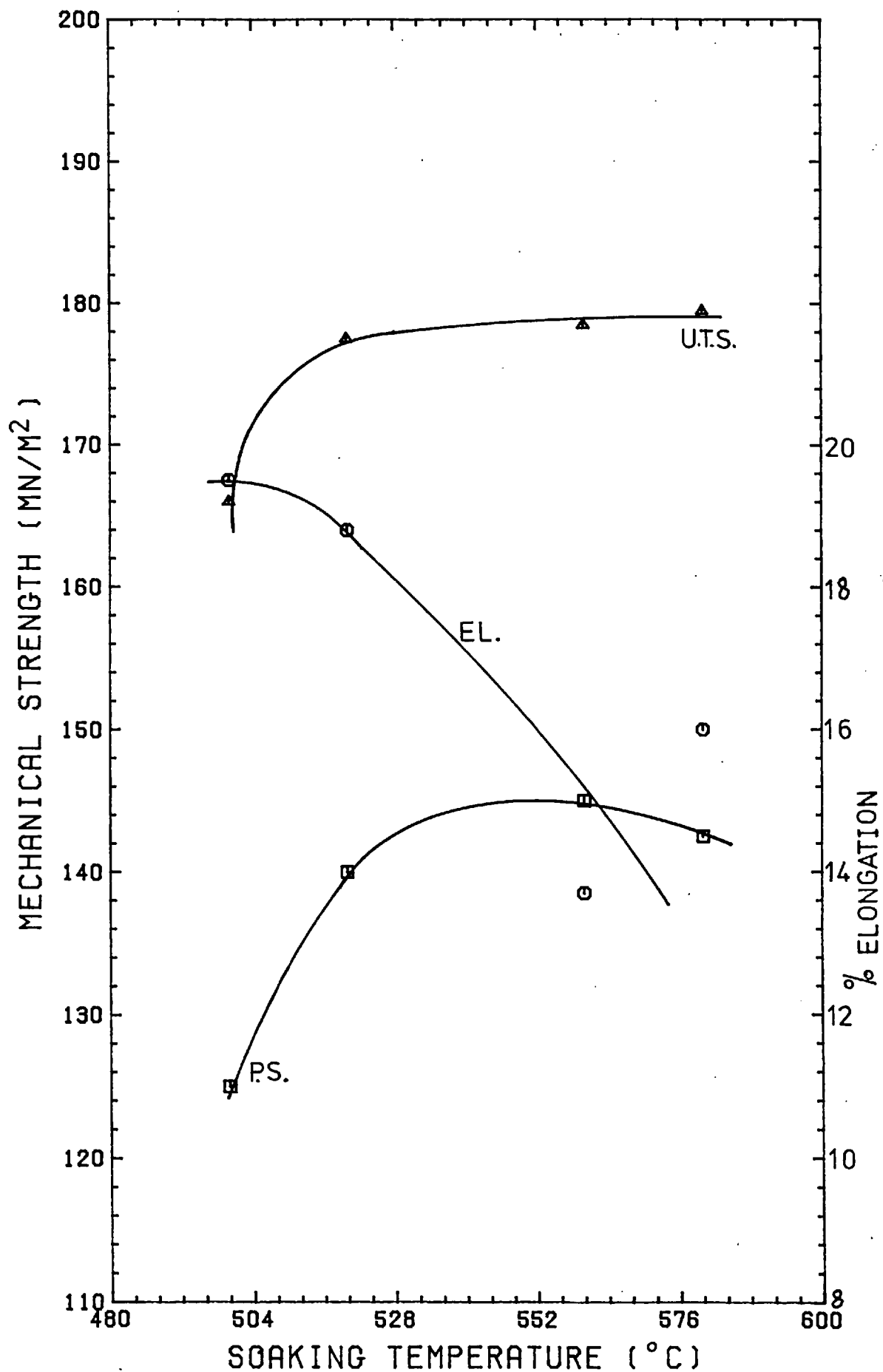


FIG. 85 Effect of variations in soaking temperature on mechanical properties of heat treated HE9 extrude

total free energy). The elongation decreases steadily with increasing soaking temperature due to the increased amount of precipitate and to the subgrain growth.

### 5.12.3 Effect of ageing time/temperature variation on extrude properties

The results from tables 27 and 28 are plotted in Figs. 86 and 87. These demonstrate the effects of ageing temperature and time variations on HE9 powder extrudes subjected to a T-F type of heat treatment. The optimum ageing temperature (for a 6 hour treatment) appears to be  $\sim 160^{\circ}\text{C}$  (Fig. 86). The recommended ageing temperature for conventionally extruded HE9 is  $175^{\circ}\text{C}$ . Using this ageing temperature a 35% increase in U.T.S. and a 65% increase in proof stress are obtained. Ageing for 6 hours at temperatures above  $\sim 160^{\circ}\text{C}$  causes overaging i.e. the precipitate particles grow larger than the optimum size. The interparticle spacing therefore increases and the mechanical strength deteriorates. The behaviour of the HE9 powder extrude at different ageing temperatures is represented schematically in Fig. 88, which explains the relative magnitudes of points A, B and C in Fig. 86. The decrease in elongation to a minimum of 13%, at an ageing temperature of  $\sim 167^{\circ}\text{C}$  coincides with an increase in the volume fraction of  $\text{Mg}_2\text{Si}$  precipitate. However, after precipitation ceases and the existing particles begin to grow, the elongation value increases again to about 21% at an ageing temperature of  $220^{\circ}\text{C}$ . It appears that:

- 1) Elongation  $\downarrow$  as volume fraction of precipitate  $\uparrow$
- 2) Elongation  $\uparrow$  as precipitate particle size  $\uparrow$

The effect of varying the ageing time, at a constant temperature of  $180^{\circ}\text{C}$ , is shown in Fig. 87. Initially the U.T.S. and proof stresses increase and the elongation decreases with ageing time as expected. However, from Fig. 86 and 88 we would expected the peak strength to correspond to an ageing time of less than 6 hours.

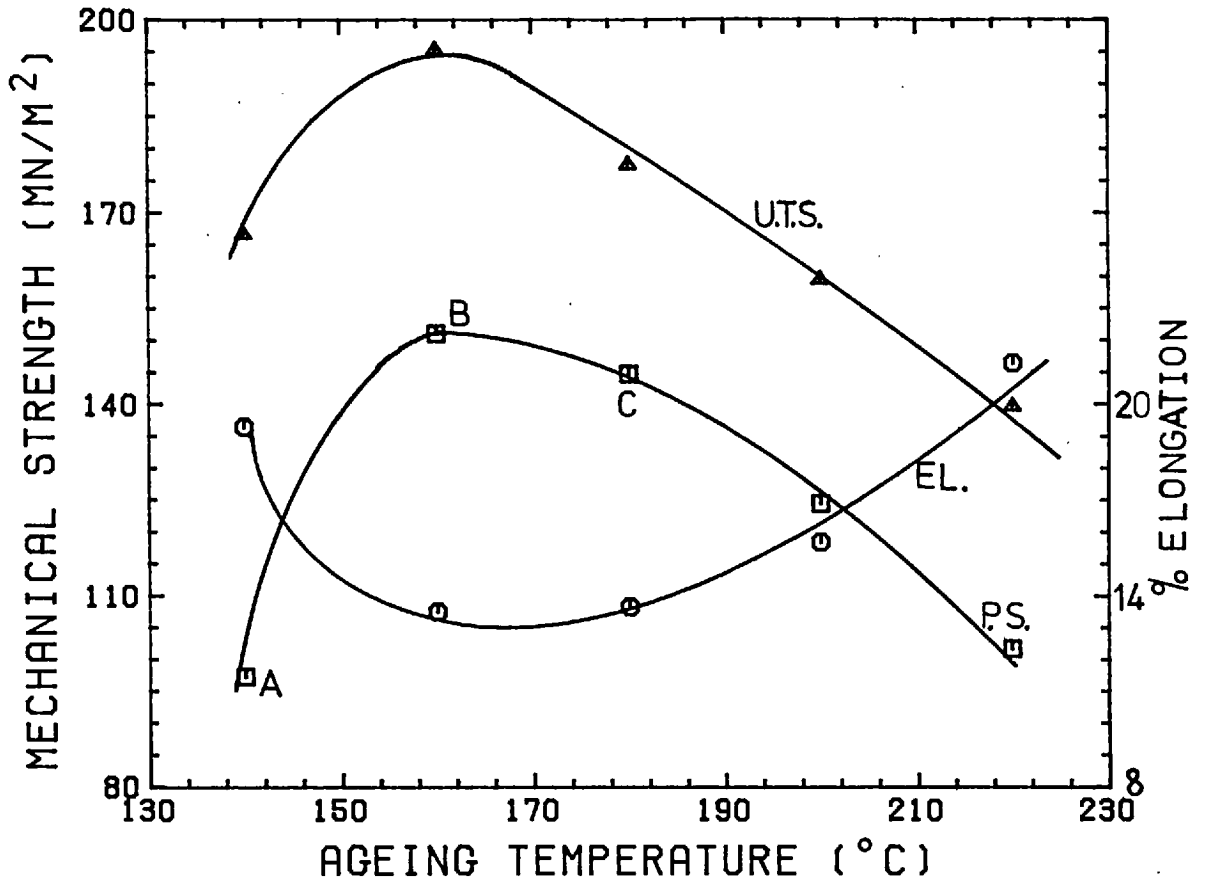


FIG. 86 Effect of variations in ageing temperature on mechanical properties of heat treated HE9 extrude

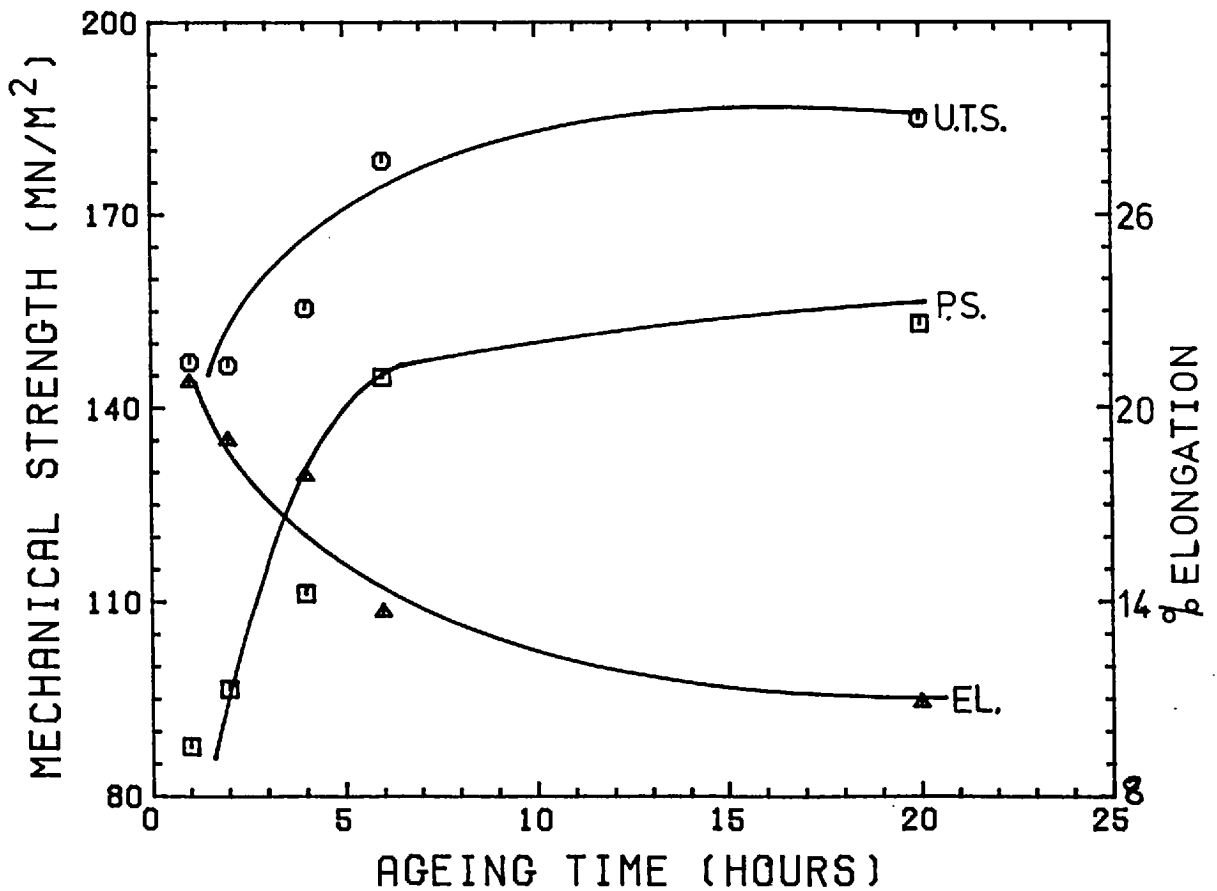


FIG. 87 Effect of variations in ageing time on mechanical properties of heat treated HE9 extrude

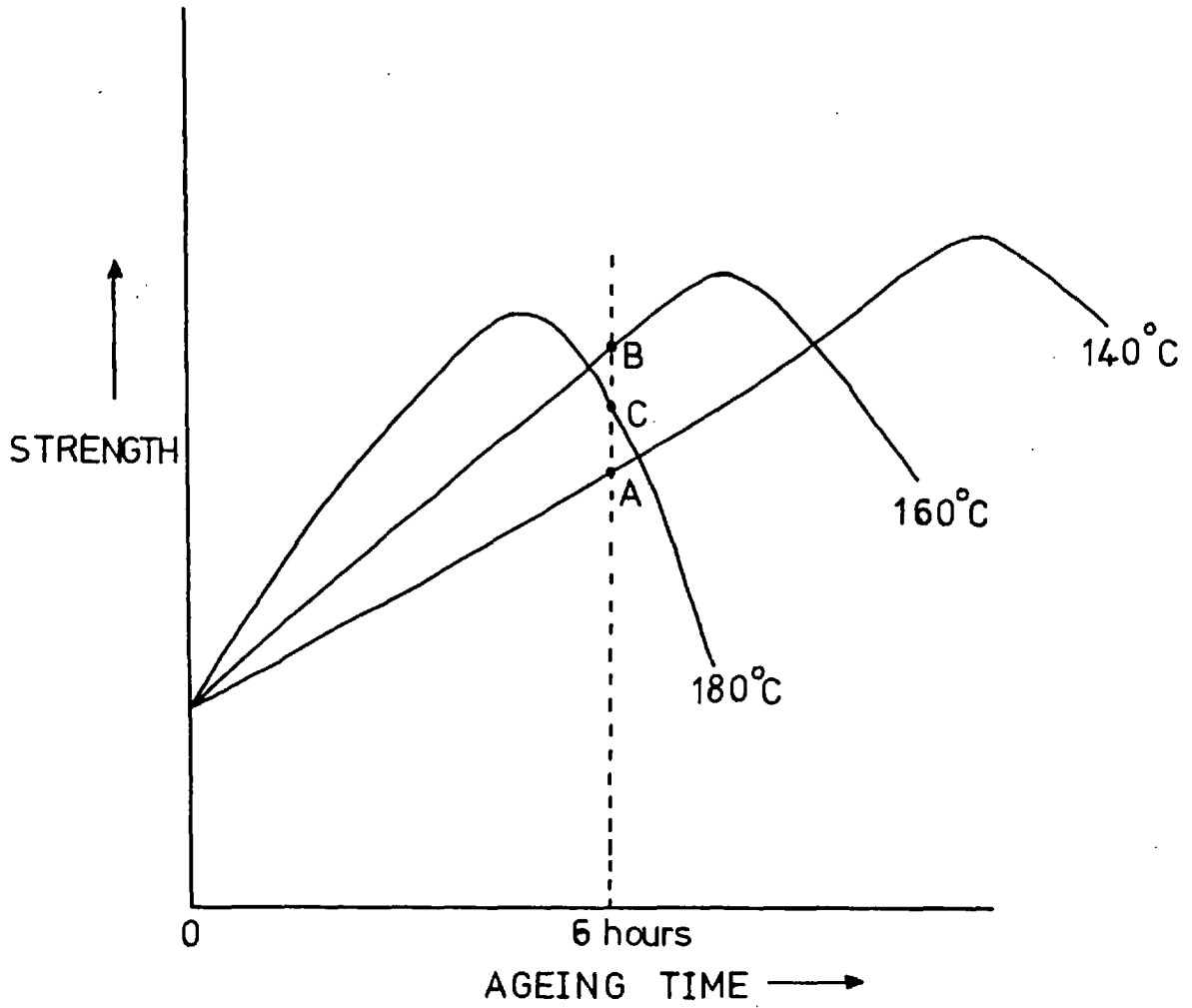


FIG. 88 Schematic representation of HE9 ageing curves at different temperatures

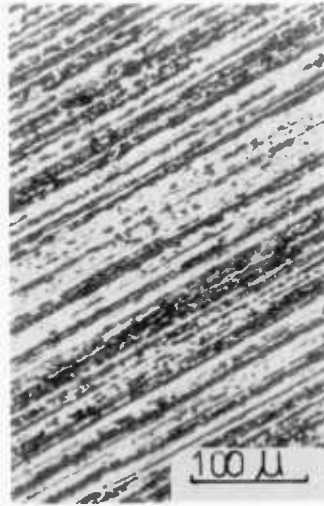


Fig. 87 indicates that the optimum strength may not be obtained for ageing times of less than 20 hours. However, this anomaly is based on the data points of only one specimen. (i.e. at 20 hours). Therefore it is more likely that the results for this specimen are erroneous.

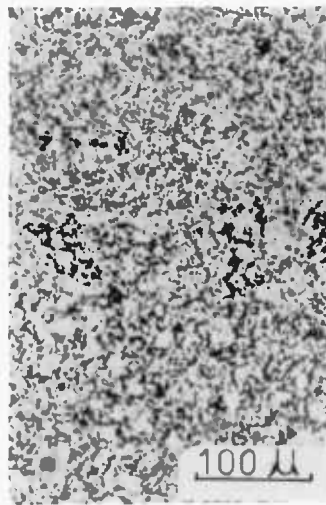
HE30 behaves similarly to HE9 as can be seen in Figs. 89 and 90. In this case, however, the optimum ageing temperature for a 6 hour treatment is  $180^{\circ}\text{C}$  (Fig. 89). The response to heat treatment is even better than that of HE9, the U.T.S. increasing by 40% and the proof stress by 85% on ageing for 6 hours at  $180^{\circ}\text{C}$ . The improved response is caused by the greater quantity of  $\text{Mg}_2\text{Si}$  available for precipitation hardening. The temperature sensitivity is greater than the time sensitivity as manifested by the curve in Fig. 90 being "flatter" than that in Fig. 89. Overageing does not seem to occur, at  $180^{\circ}\text{C}$ , until heating times of approximately 10 hours are employed. The elongation of HE30 appears to be much less affected by heat treatment than that of HE9 although prolonged ageing (16-24 hours) at  $180^{\circ}\text{C}$  does cause a noticeable decrease. The reason why the elongation of HE30 powder extrude is less sensitive to heat treatment than that of HE9 is not obvious. Clearly the elongation of HE30 might be expected to decrease even more than that of HE9 because of the greater quantity of  $\text{Mg}_2\text{Si}$  available for precipitation. However, the fact that the initial powder particle size of the HE30 powder (median =  $122\mu$ ) is smaller than that of the HE9 powder (median =  $141\mu$ ) may have some bearing on the matter.

#### 5.13.1 Structure of extrudes (optical micrographs)

The optical micrographs shown in Plate 11 reveal the structure of a typical HE9 extrude. The longitudinal micrograph (11(a)) shows a fibrous, cold-worked structure. The original powder particles have been elongated in the direction of extrusion. This "cold-worked" microstructure is the result of extruding at a temperature ( $450^{\circ}\text{C}$ ) above



(a)  
Longitudinal



(b)  
Transverse

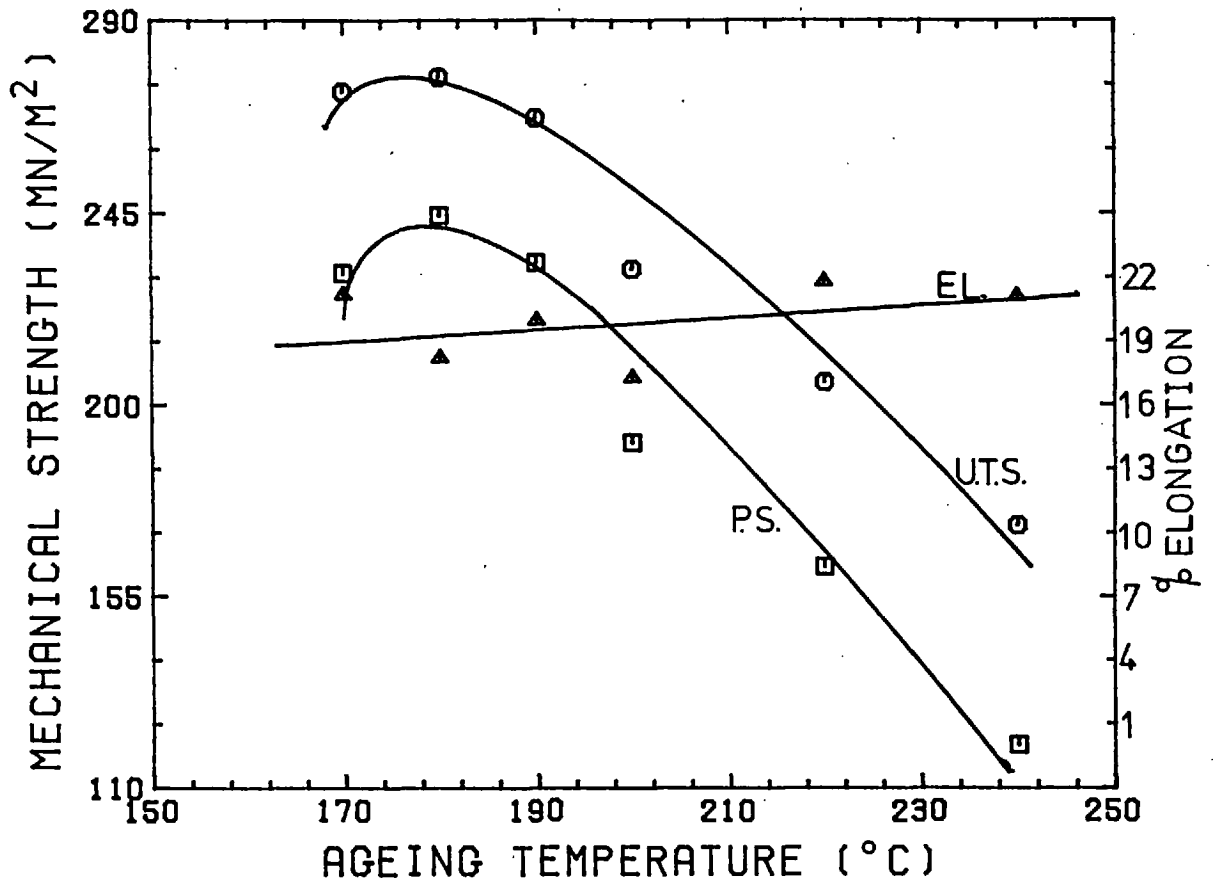


FIG. 89 Effect of variations in ageing temperature on mechanical properties of heat treated HE30 extrude

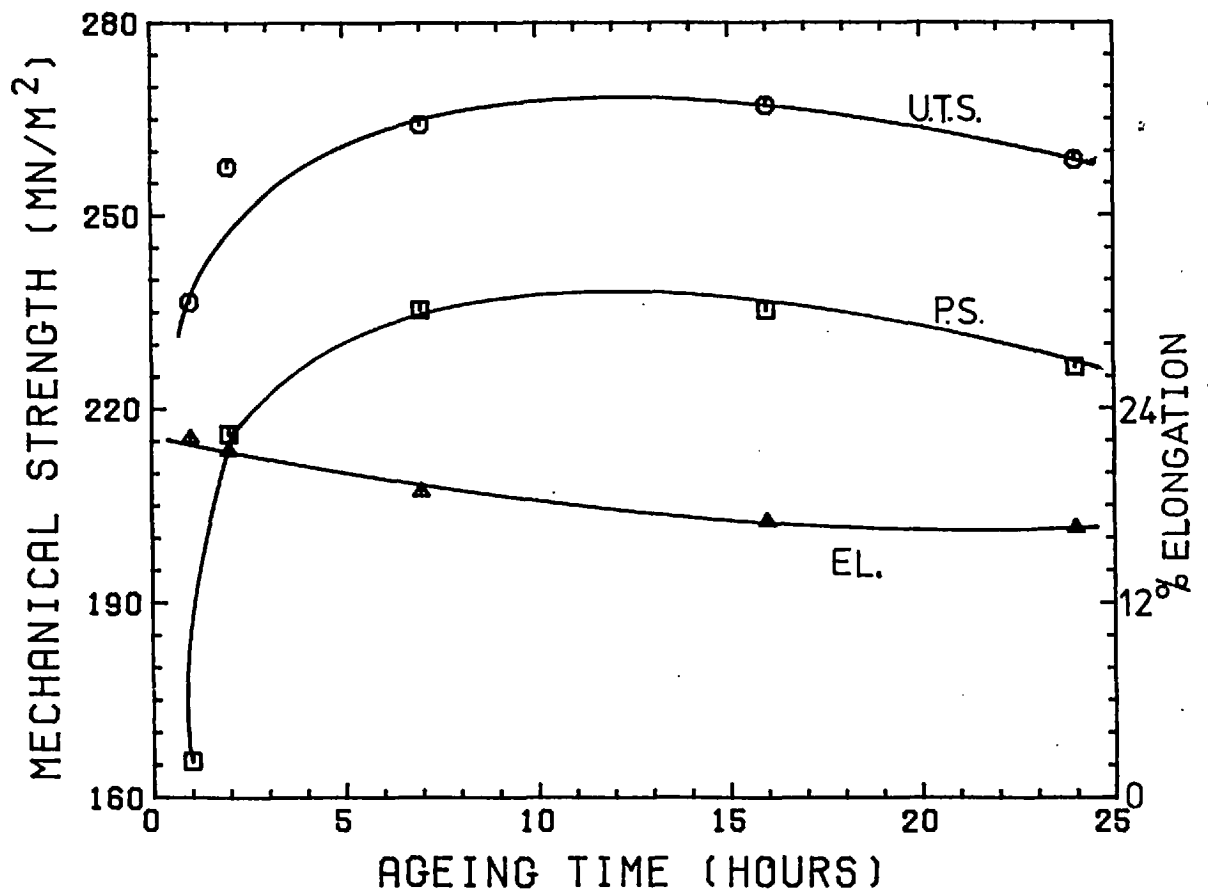


FIG. 90 Effect of variations in ageing time on mechanical properties of heat treated HE30 extrude

the nominal static recrystallisation temperature ( $350^{\circ}\text{C}$ ). The transverse micrograph (11b) reveals the equiaxed nature of the structure. This structure is typical of extrusions produced over the entire range of temperatures and strain rates investigated. Optical microscopy reveals no evidence of recrystallisation in any of the extrudes. Chare<sup>19</sup> and Ansell and Kim<sup>137</sup> found no evidence of recrystallisation in Al, Al-Fe& Al-Mn and S.A.P. powder extrudes, respectively. This evidence supports the thesis that dynamic recovery is probably the prevailing softening mechanism although the presence of sub-grains cannot be detected at these magnifications, except by the use of polarized light. Jonas<sup>49</sup> found a similar "cold-worked", fibrous structure in aluminium extrudes produced at  $400^{\circ}\text{C}$  i.e.  $50^{\circ}\text{C}$  above the recrystallisation temperature. However, he discovered that extrusions produced at  $500^{\circ}\text{C}$  and above were fully recrystallised. Plumtree<sup>58</sup> has suggested that the ability of aluminium to recrystallise is directly related to its purity. The question that must now be answered is: if impurities retard recrystallisation in Aluminium why then do many of the stronger aluminium alloys i.e. the 7000 and 5000 series recrystallise? Humphreys<sup>140</sup> found that second phase particles, such as  $\text{CuAl}_2$  particles, help to nucleate recrystallisation if they are larger than a critical size, which is strain dependent. He states that the critical size, for a 45% reduction, is  $1-2\ \mu\text{m}$ . Thus the second phase particles present in the stronger aluminium alloys actually aid recrystallisation which originates within zones of high dislocation density adjacent to the particles. Now in the present series of experiments no second phase particles of  $1-2\ \mu\text{m}$  size exist (see electron micrographs in next section 5.13.2). In fact the precipitate and fragmented oxide particles are  $\ll 1-2\ \mu\text{m}$ . Thus the high oxide content of the HE9 powder together with the Mn<sup>141</sup> alloying addition actually prevent recrystallisation, even at the highest extrusion temperatures; the oxide particles together with solute atom clusters preventing new, high angle grain boundaries from sweeping through the material.

All the HE30, Al-Mg and Al extrudes exhibit similar structures for the same reasons. Plate 12(a) and (b) show longitudinal and transverse micrographs of a typical Al extrusion (550°C, 90:1). Again the longitudinal micrograph shows a fibrous structure consisting of elongated particles; the transverse micrographs revealing an equi-axed structure. The micrographs in Plate 12(c) and (d), show the structure of an Al 80:1 extrude produced at 300°C. The obvious cracking is due to the lack of ductility of the Al at this lower temperature. At this low temperature the material is fairly rigid and difficult to work due to the low rate of dynamic softening.

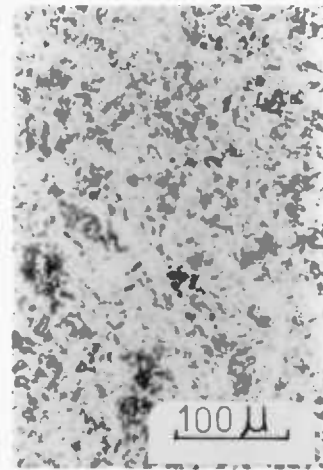
The imperfection of the fibres formed during extrusion is demonstrated in Plate 13. Micrographs (a) and (b) represent, respectively, longitudinal sections near the surface and at the centre of a HE9 + 5 wt.% glass extrude. The fibres near the surface are much more elongated than those at the centre. This is a manifestation of the variation in strain which occurs across the section of an extrusion. Micrographs (c) and (d) show the corresponding transverse structures. Clearly the fibres, in practice, are far removed from the theoretically ideal, uniform cylinders discussed in 3.7.1. This is the reason that the fibre strengths, estimated in 5.9.6, are so much less than values commonly available from industrially drawn glasses.

Extrusion of Al-glass and Al-Mg-glass composites produce similar fibres. Plate 14(a) and (b) show longitudinal sections, near the surface and at the centre of an Al-Mg-5 wt.% glass extrude, produced at 450°C (80:1). Micrographs (c) and (d) show the corresponding sections of an extrude of the same composition produced at 500°C, (80:1). Clearly the increase in temperature produces larger, better quality fibres. This is because the glass flows more easily, in the deformation zone, at the higher temperature.

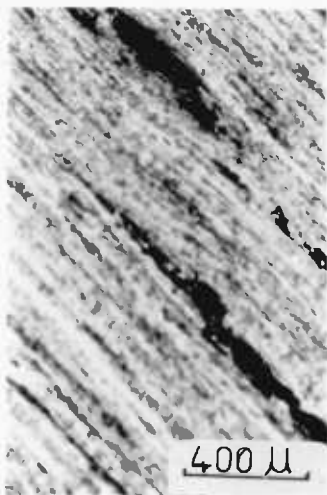


(a)  
Longitudinal

(550°C)

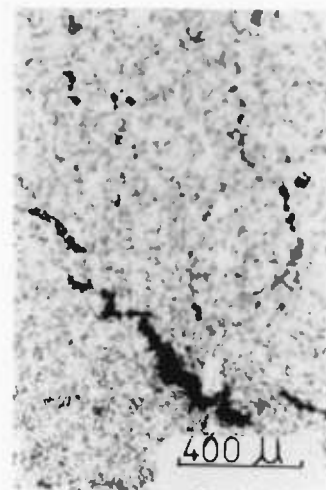


(b)  
Transverse



(c)  
Longitudinal

(300°C)



(d)  
Transverse



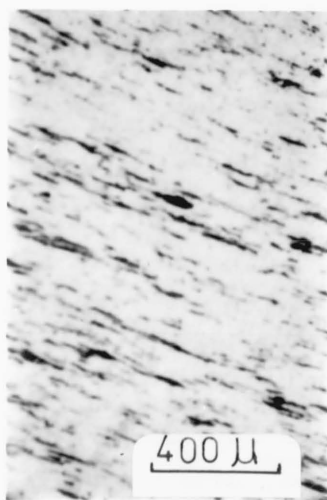
(a)  
Longitudinal  
(near surface)

(450°C)

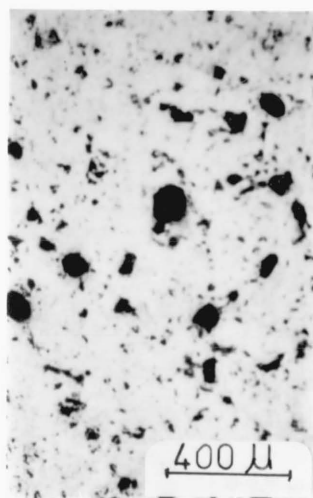
(40:1)



(b)  
Longitudinal  
(at centre)



(c)  
Transverse  
(near surface)



(d)  
Transverse  
(at centre)

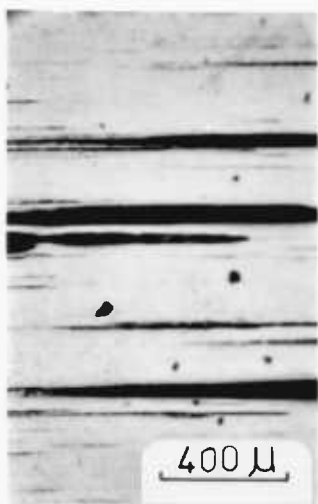


(a)  
Longitudinal  
(at centre)

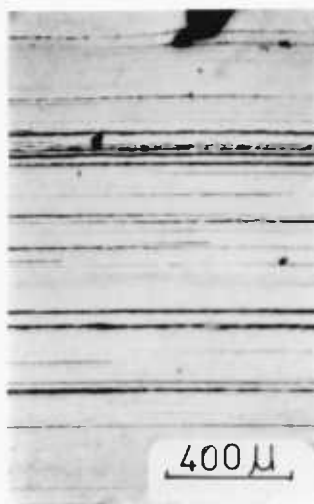
(450°C)



(b)  
Longitudinal  
(near surface)



(c)  
Longitudinal  
(at centre)



(d)  
Longitudinal  
(near surface)

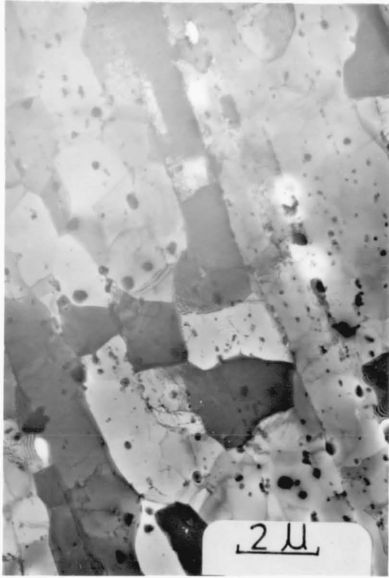


### 5.13.2 Structure of extrudes (transmission electron micrographs)

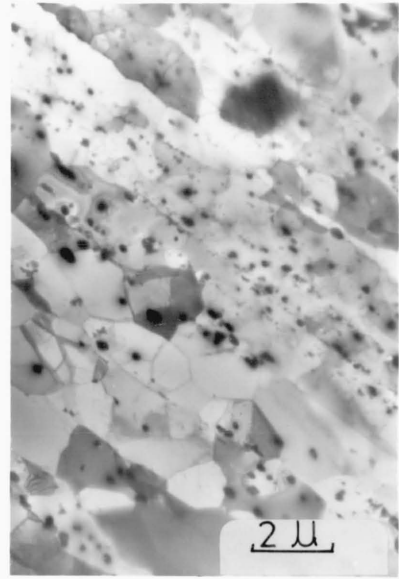
The electron micrographs in Plates 15-18 show clearly that dynamic recovery occurs during the extrusion of HE9, HE30, and Al-Mg alloys. (The absence of electron micrographs of reinforced extrudes and Al powder extrudes is due to the difficulty encountered in thinning these materials). Subgrains, elongated in the extrusion direction, constitute the substructure in the longitudinal sections of HE9 40:1 extrudes formed at 250<sup>o</sup>, 350<sup>o</sup> and 450<sup>o</sup>C. (Plate 15 (a), (b) and (c) respectively). Transverse micrographs (a) and (b) in Plate 16 reveal the "equi-axed" nature of the subgrains in this plane, in 40:1 extrusions produced at 450 and 540<sup>o</sup>C respectively. The fact that the subgrains are elongated in the extrusion direction but "equi-axed" in the plane perpendicular to this is evidence that the operating softening mechanism is dynamic recovery. The structure of HE30 extrudes is very similar to that of HE9. Plate 17(a) and (b) (350<sup>o</sup>C, 60:1) demonstrate the dynamically recovered structure. Once again the subgrains are elongated in the direction of extrusion (a) and "equi-axed" in the perpendicular plane (b). A similar substructure also exists in the Al-Mg extrudes (500<sup>o</sup>C, 80:1) as shown in Plate 18(a). The main variation in structure over the temperature range investigated is an increase in subgrain size and perfection with temperature. In addition, at the higher extrusion temperatures the subgrains tend to be "equi-axed" in three-dimensions i.e. the elongation in the extrusion direction disappears (Plates 15(a) and (c). At the higher extrusion temperatures the dislocation mobility increases and cell walls migrate tending to form "equiaxed" subgrains. Once again the driving force for this migration is the need to minimize the total grain boundary area and thus reduce the free energy of the structure.

Fig. 91 demonstrates an approximately linear relationship between subgrain size and extrusion temperature. Wong et al.<sup>118</sup> discovered a similar relationship in extruded, commercially pure Al. They also noticed a tendency for the subgrains to become elongated in the extrusion direction at the lower temperatures. Raybould and Sheppard<sup>32</sup> found an approximately linear relationship between  $\ln Z$  and subgrain size<sup>-1</sup> for Al-7%Zn (for subgrain sizes  $> 2.5\mu$ ). The present results, plotted in this form (Fig. 92), do not fit a linear relationship. (N.B. A linear relationship cannot be totally ruled out due to the lack of data points). However, a smooth curve may be drawn through the points giving quite a good fit. The divergence from linearity occurs at the higher  $Z$  values i.e. high strain rate, low temperature extrusions. Plate 15(d) shows a S.A.D.P. for a typical HE9 extrude (250°C, 40:1). A  $\langle 111 \rangle$  orientation is associated with the fibrous texture produced by dynamic recovery during aluminium extrusion.

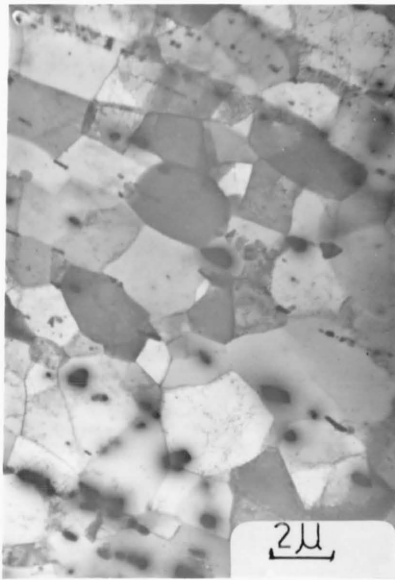
Oxide particles, remnants of the layers covering the original powder particles, are clearly visible in Plate 15(a) and (b). These fragments indicate the original powder particle boundaries. (Some of the particles which appear very close together represent boundaries parallel to the foil surface). Chare<sup>14</sup> found his original particle boundaries to be similarly outlined. The history of the formation of these oxide particles is unfolded in Plate 18 (b) and (c). Micrograph (b) shows the structure of the compact at position Y (Fig. 93) during the extrusion of an Al-Mg powder billet (350°C, 80:1). (The extrusion was stopped before completion, the billet sectioned and specimens taken). At this early stage in the deformation process the oxide layers between two adjacent particles are still almost completely continuous. Micrograph (c) shows the structure at position Z (Fig. 93) of an HE30 partial extrude (450°C, 80:1). At this later stage the oxide layers have been broken up and the original boundaries are clearly revealed by "chains" of oxide fragments.



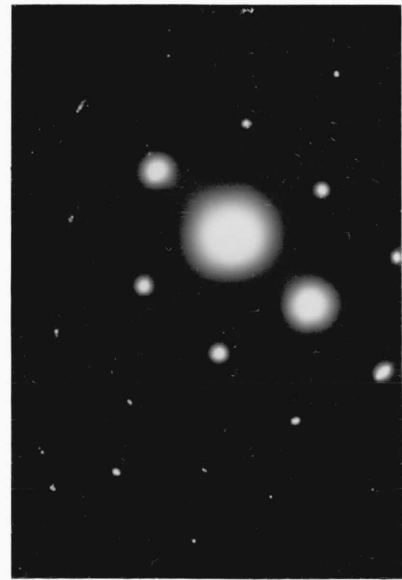
(a)  
250°C



(b)  
350°C



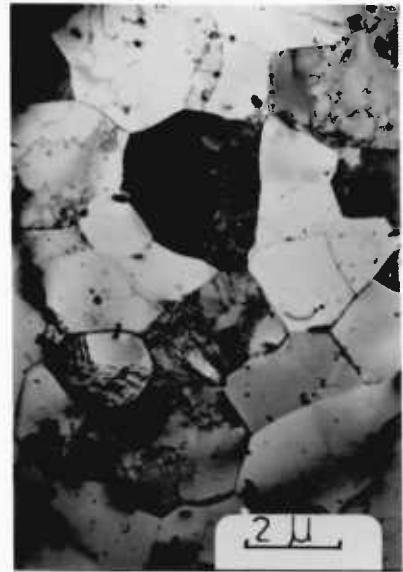
(c)  
450°C



(d)  
diffraction pattern



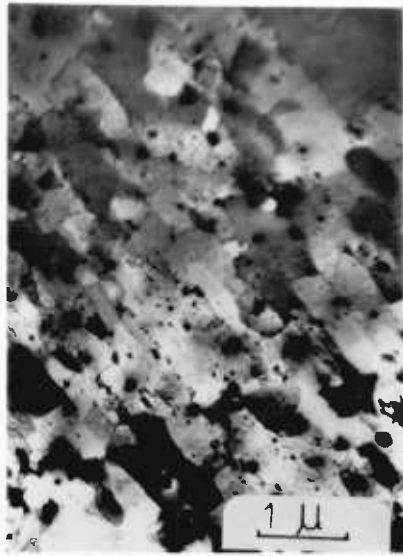
(a)  
(450°C)



(b)  
(540°C)

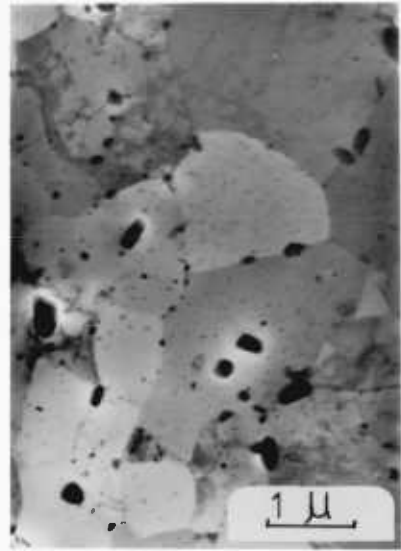
PLATE 16

Transverse electron micrographs of HE9 extrudes



(a)  
Longitudinal

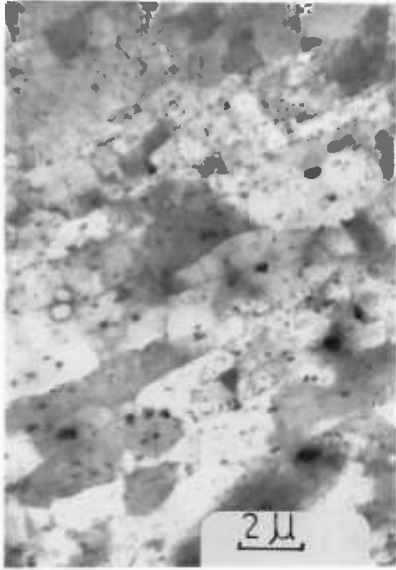
(350°C)



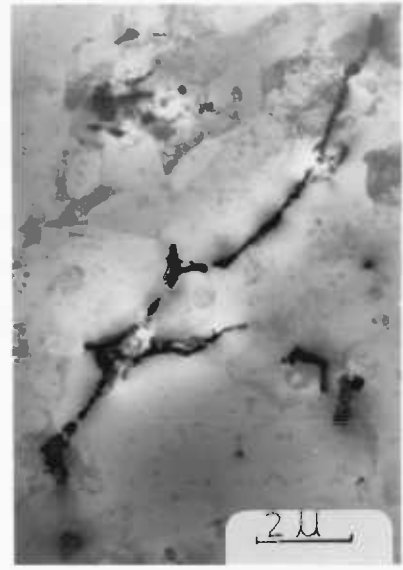
(b)  
Transverse



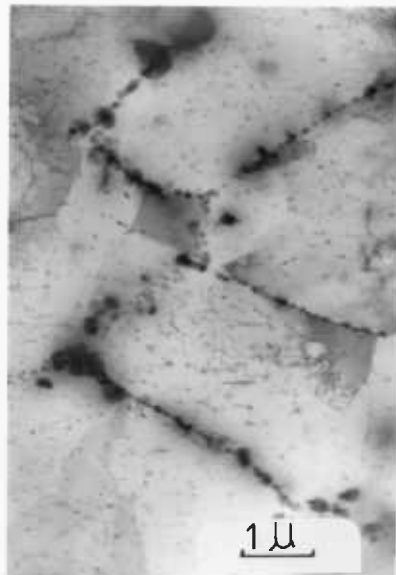
(c)  
(550°C)  
(taken from partial extrusion)



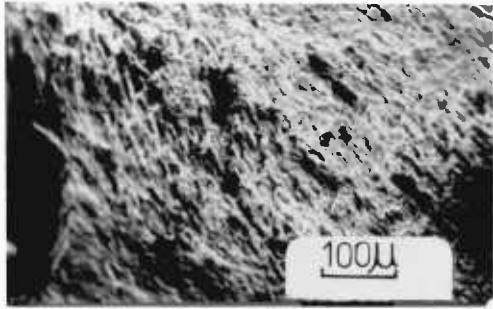
(a)  
Al-Mg (500°C)  
Longitudinal



(b)  
Al-Mg (350°C)  
(Taken from partial extrusion)



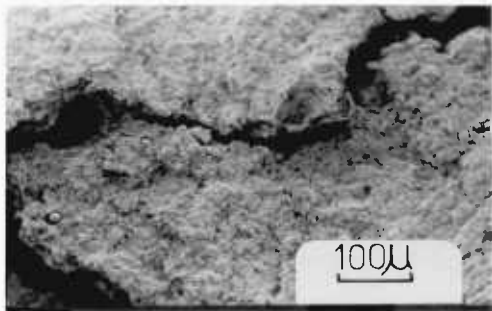
(c)  
HE30 (450°C)  
(Taken from a partial extrusion)



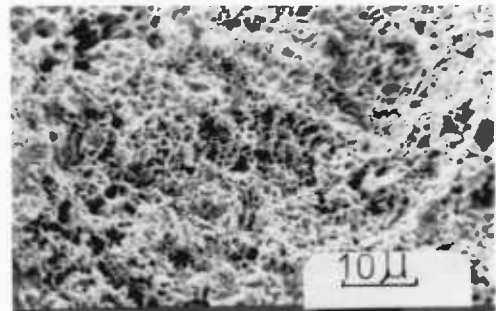
(a)  
Room temperature test



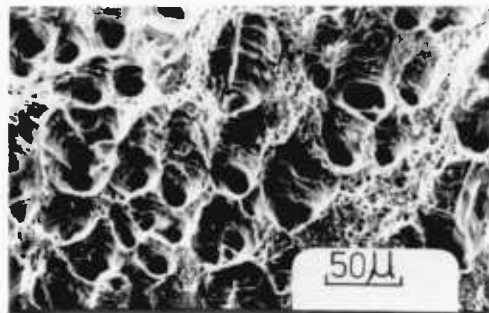
(b)  
HE9  
400°C test



(c)  
Room temperature test



(d)  
Al  
Room temperature test



(e)  
HE9 + 5 wt.% glass  
Room temperature test

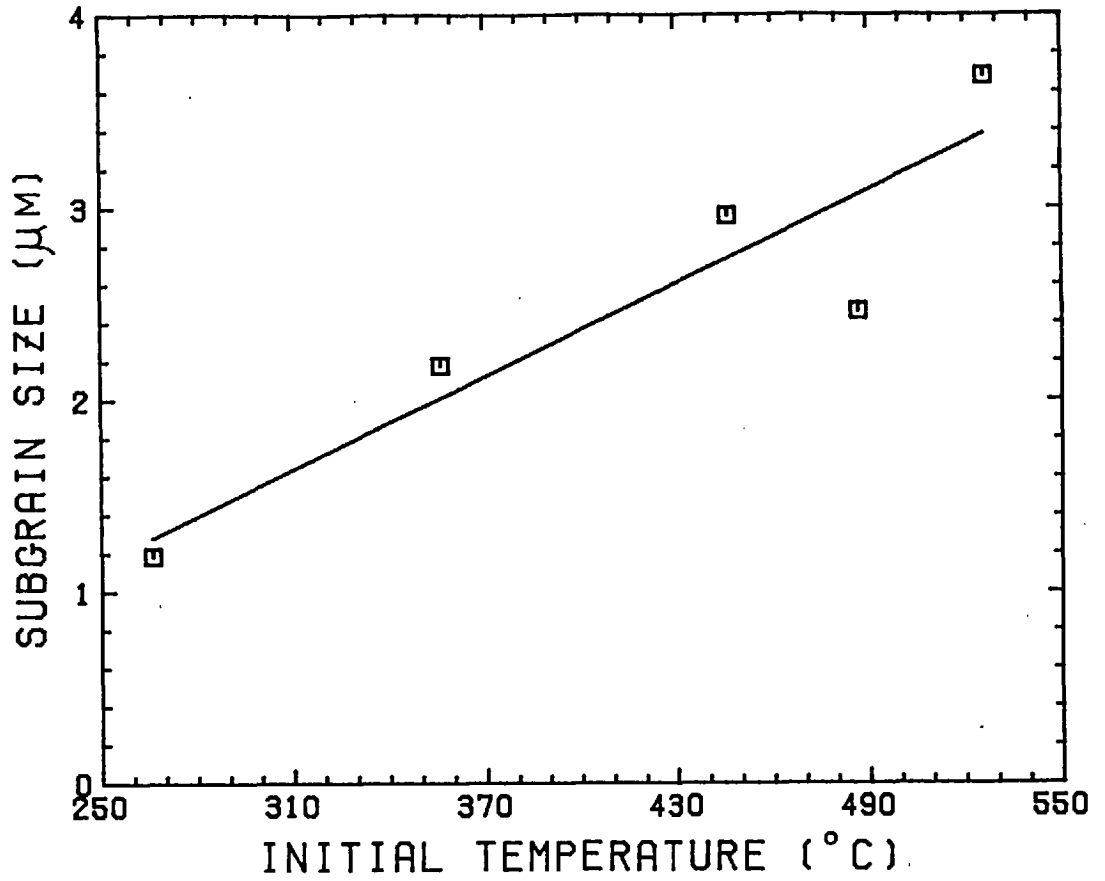


FIG. 91 HE9 extrude subgrain size as a function of extrusion temperature

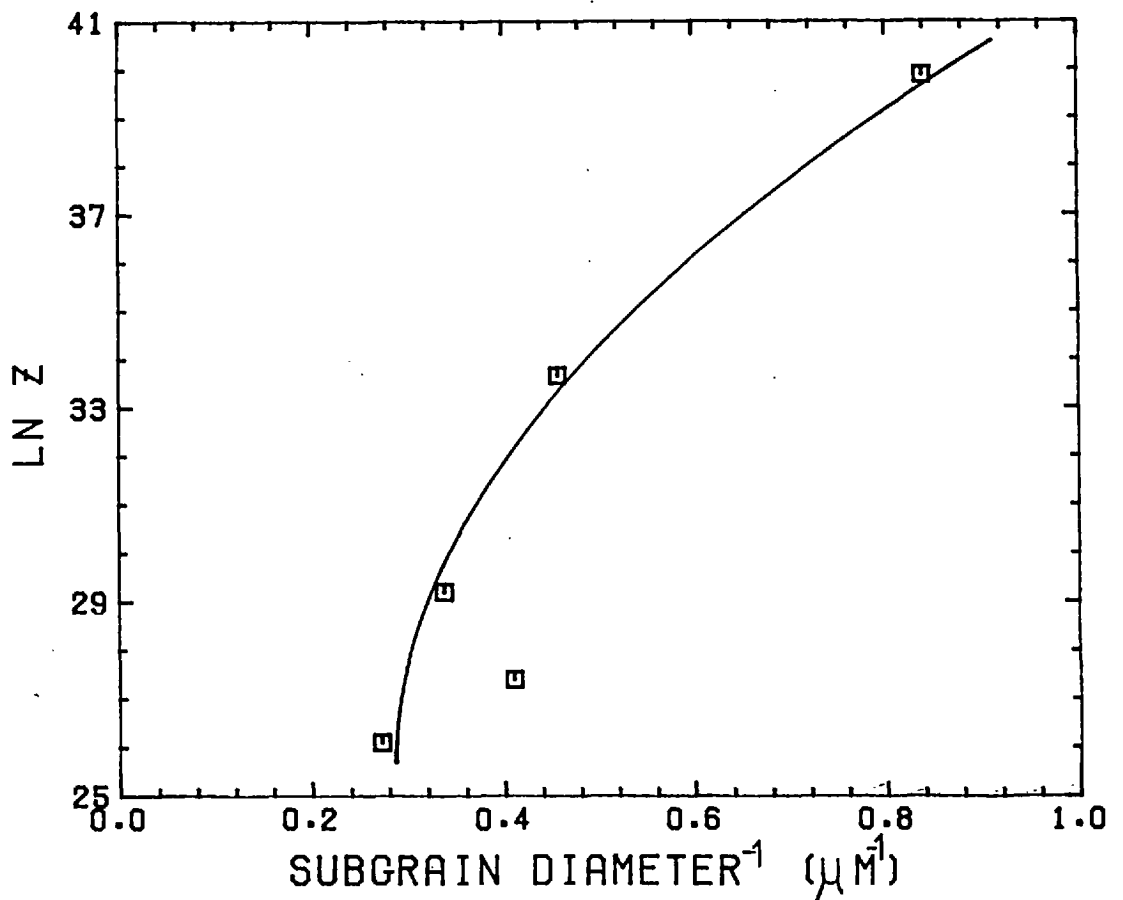


FIG. 92 ln Z vs. reciprocal subgrain size relationship for HE9 extrudes



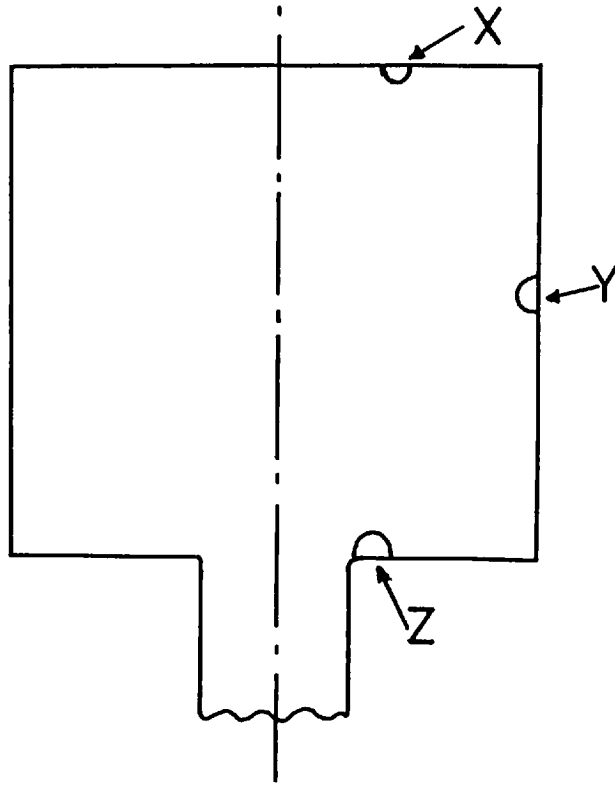


FIG. 93 Locations of electron microscope foils taken from partial extrusions

Thomas<sup>72</sup> found  $Mg_2Si$  precipitate particles, in Al-Mg-Si alloys, occurring as rods along the  $\langle 100 \rangle$  directions in the aluminium matrix. Plates 17(c), taken from position x in a partial extrusion (See Fig. 93) and 18(c) reveal rod like particles which are mutually perpendicular and thus may be considered as evidence in support of Thomas's findings.

#### 5.14.1 Nature of the glass-metal interface

Plate 10(b) is a scanning electron micrograph of the end of a fibre channel in a fractured HE9 + 5 wt.% glass tensile specimen produced from a 80:1, 450°C extrusion. The shell around the crater is fairly distinct. This shell consists of the products of the glass-metal chemical reaction. Research into the composition of these products was carried out at Borax Consolidated Limited.<sup>138</sup> (A knowledge of the composition of these products is important as the composite strength is dependent on the strength of the metal-glass interface). HE30 + 5 wt.% glass extrude specimens were dissolved in a NaOH solution leaving behind the glass-metal interaction products as a very fine suspension. Precipitates were produced by centrifuging this suspension. An x-ray diffraction examination revealed the presence of the spinel  $MgAl_2O_4$ . Several extrudes were also heated at 550°C for several hours before dissolution in the NaOH solution. The x.R.D. pattern revealed the presence of a magnesium aluminosilicate, probably  $4MgO \cdot 5Al_2O_3 \cdot 2SiO_2$ . No magnesium silicide was detected. This explains the lack of response to heat treatments (see 5.12.1) shown by the reinforced HE9 extrudes. (The lack of  $Mg_2Si$  in the matrix prevents precipitation hardening from occurring).

<u>HE9</u>		<u>HE30</u>	
<u>Particle size,</u> <u>μm</u>	<u>wt. per cent</u>	<u>Particle size,</u> <u>μm</u>	<u>wt. per cent</u>
+355	2.87	+355	2.91
+180	36.42	+180	29.26
+106	63.56	+106	55.61
+90	68.91	+90	61.88
+75	73.80	+75	67.77
+63	79.06	+63	74.54
+53	83.55	+53	80.38
-53	16.26	-53	19.51
Median Particle Size = 143 μm		Median Particle size = 121.8 μm	

TABLE 1

Sieve analysis of HE9 powder

TABLE 2

Sieve analysis of HE30 powder

<u>Al-3 wt.% Mg</u>		<u>Al</u>	
<u>Particle size,</u> <u>μm</u>	<u>wt. per cent</u>	<u>Particle size,</u> <u>μm</u>	<u>wt. per cent</u>
+355	2.35	+355	13.31
+250	15.77	+250	31.25
+180	26.52	+180	42.26
+125	42.72	+125	58.52
+106	52.18	+106	67.41
+75	65.49	+75	77.93
+45	81.59	+45	88.67
-45	18.21	-45	11.28
Median Particle Size = 110.4 μm		Median Particle Size = 153.8 μm	

TABLE 3

Sieve analysis of Al-3wt.% Mg  
powder

TABLE 4

Sieve analysis of Al powder

<u>B<sub>2</sub>O<sub>3</sub>-10% SiO<sub>2</sub></u>	
<u>Particle Size, μm</u>	<u>wt. per cent</u>
+250	8.13
+180	17.67
+125	30.09
+90	46.71
+75	49.29
+53	60.38
+45	64.65
-45	32.61

Median Particle Size = 74 μm

TABLE 5

Sieve analysis of B<sub>2</sub>O<sub>3</sub>-10% SiO<sub>2</sub> glass

	O	Si	Mg	Zn	Cu	Ni	Fe	Mn	Cr	Ti
HE9	0.062	0.120	0.330	0.025	0.014	0.004	0.150	0.100	0.010	0.014
HE30	0.062	0.330	0.350	0.020	0.040	---	0.310	0.530	0.013	0.019
Al-3wt.% Mg	0.120	---	3.000	0.100	0.078	0.006	0.300	0.510	0.040	0.040
Al	3.300	---	---	0.041	0.040	0.003	0.200	0.009	0.006	0.006

WEIGHT PERCENTAGE OF ALLOYING ADDITIONS AND IMPURITIES

TABLE 6

Chemical analysis of metal powders

<u>Compaction Pressure (MN/m )</u>	<u>Theoretical Density (%)</u>	<u>Fracture Force (N)</u>	<u><math>\ln\left(\frac{1}{1-D}\right)</math></u>
185.3	84.6	64.5	1.8689
216.2	86.3	135.7	1.9863
247.1	87.3	189.1	2.0596
278.0	89.4	222.4	2.2424
308.9	89.7	235.8	2.2721
339.83	90.0	202.4	2.3066
370.7	91.2	282.5	2.4350
401.6	91.5	311.4	2.4616
432.4	93.1	286.9	2.6679

TABLE 7

Results of mechanical tests on cold compacts

<u>Sintering Temperature (°C)</u>	<u>Fracture Force (N)</u>
500	+2358
430	2358
375	+2358
305	1321
240	801
160	605

TABLE 8

Results of mechanical tests on sintered compacts

<u>HE9</u>	<u>PARTICLE SIZE</u> <u>μm</u>	<u>FRACTURE</u> <u>FORCE (N)</u>
	26.5	2358
	64	885
	112	552
	+150	445

TABLE 9

Results of mechanical tests on compacts produced from different sieve fractions

Extrusion parameters

No	Extrusion Ratio	(°C)	(°C)	(m/sec)	(MN/m <sup>2</sup> )	(MN/m <sup>2</sup> )	P/K	(GN/m <sup>2</sup> )	(MN/m <sup>2</sup> )	(sec <sup>-1</sup> )	(sec <sup>-1</sup> )
		Initial Temp.	Final Temp.	Run Speed	Peak Load	Final Load		Flow stress	Flow Stress	STRAIN Rate	$\times 10^{12}$
(HE9)											
1	30	526	536.3	0.009	350.7	417.3	13.767	44.01	52.50	7.59	0.4648
2	30	526	509.7	0.005	345.1	408.2	13.767	43.42	51.36	4.64	0.6711
3	30	526	534.3	0.0135	394.7	338.3	13.767	49.66	42.56	11.39	0.7539
4	30	526	514.6	0.007	313.5	376.7	13.767	39.44	47.39	5.91	0.7285
5	30	526	472.3	0.003	313.5	394.7	13.767	39.08	49.66	--	--
6	30	526	--	--	310.6	394.7	13.767	39.08	49.66	--	--
7	40	432	482.3	0.0010	446.6	410.5	14.996	51.58	47.41	9.41	3.4590
8	40	426	464.5	0.0065	475.9	410.5	14.996	54.98	47.41	6.11	4.2716
9	40	439	491.3	0.0014	466.9	412.8	14.996	55.92	47.68	10.72	4.4906
10	80	437	517.2	0.0126	550.4	516.5	18.176	52.45	49.22	15.08	1.7091
11	80	438.5	493.8	0.0097	570.7	455.6	18.176	54.39	45.42	11.61	2.8605
12	80	432	459.9	0.0031	545.9	523.3	18.176	52.01	49.87	3.71	3.0770
13	80	432	483.1	0.0067	527.8	500.7	18.176	50.30	47.72	7.18	2.5650
14	80	437	--	0.101	545.6	--	18.176	51.79	--	120.9	--
15	80	356	461.2	0.0094	581.9	523.3	18.176	55.46	49.87	11.25	8.8905
16	80	356	427.5	0.0043	577.4	466.9	18.176	55.03	44.56	5.14	15.1648
17	80	356	434.3	0.004	581.9	507.5	18.176	55.44	48.37	4.79	10.7293
18	80	355.5	472.9	0.0125	620.3	543.6	18.176	59.12	51.81	14.96	7.6905
19	80	355	--	0.094	660.0	--	18.176	62.98	--	112.51	--
20	80	265.5	432.4	0.0103	821	615.8	18.176	78.24	58.68	12.35	29.8140
21	80	265.33	--	--	--	--	18.176	--	--	--	Stuck
22	80	356	474.5	0.010	669	570.7	18.176	63.84	54.39	11.97	5.8065
23	40	356	454.5	0.0091	514.3	489.5	14.996	59.24	56.42	8.56	8.7057
24	40	478	478.2	0.0043	376.7	433.1	14.996	43.51	50.02	4.04	1.7177
25	80	471	489.8	0.0042	469.1	516.5	18.176	44.70	49.22	5.06	1.4304
26	80	520	501.8	0.0042	388	473.7	18.176	36.98	45.14	4.99	0.9380
27	40	526	490.8	0.0042	387.8	394.7	14.996	44.79	45.59	3.98	1.0876
28	5	526	490.8	0.0042	--	221	7.083	--	54.04	1.57	1.1639
29	10	527	469.8	0.0044	232.3	259.4	9.507	42.33	47.27	2.32	1.3343
30	60	527	493.5	0.0043	365.4	406	16.818	37.64	41.81	4.72	1.1752
31	180	526	506.5	0.0041	597.7	503	22.304	46.42	39.06	6.18	0.9933
32	40	526	485.7	0.0043	354.1	367.7	14.996	40.89	42.47	4.05	1.3206
33	80	526	496.1	0.0041	383.4	439.8	18.176	36.55	41.92	4.91	1.1185
34	40	525	492.3	0.0043	428.5	399.2	14.996	49.49	46.11	4.05	1.0915
35	80	479	480.5	0.0043	453.4	453.4	18.176	43.22	43.22	5.14	2.0135
36	80	477	504.3	0.0066	475.9	473.7	18.176	45.35	45.14	7.90	1.3661
37	80	478	495.3	0.0063	421.8	442.1	18.176	40.20	42.12	7.54	1.7665
38	40	525	511.9	0.0062	417.3	399.2	14.996	48.20	46.11	5.83	0.7850
40	40	479	495.8	0.0083	421.8	383.4	14.996	48.72	44.29	7.71	1.7747
41	40	478	485.5	0.0077	372.2	354.1	14.996	42.99	40.89	7.24	2.4108
42	80	526	521	0.0073	394.7	412.8	18.176	37.62	39.34	8.74	0.8737
43	80	526	537.8	0.0071	484.9	489.5	18.176	46.21	46.64	8.50	0.5046
44	80	480	536	0.0122	545.8	489.5	18.176	52.01	46.64	14.60	0.9157
45	80	478	525.3	0.0123	462.4	451.1	18.176	44.06	42.99	14.72	1.2892
46	40	526	530.7	0.116	349.6	354.1	14.996	40.37	40.89	10.90	0.7895
47	40	525	539.8	0.0114	457.9	394.7	14.996	52.90	45.59	10.72	0.5897
48	180	479	528.4	0.0109	611.2	489.4	22.304	47.46	38.00	16.64	1.3215
49	10	527	511.7	0.0109	611.2	259.4	1.507	47.27	47.27	5.75	0.7762
50	60	527	551.6	0.0129	415.5	421.8	16.818	43.18	43.44	14.04	0.5503
51	5	527	511	0.0125	205.3	218.8	7.083	50.21	55.50	4.64	0.6431
52	10	526	510.6	0.0130	230.1	232.3	9.507	41.92	42.33	6.84	0.9605
53	180	528	569.6	0.0130	618	503	22.304	48.00	39.06	--	--
54	180	--	--	--	--	--	--	--	--	--	--
55	80	--	--	--	--	--	--	--	--	--	--
56	60	--	--	--	--	--	--	--	--	--	--
57	40	--	--	--	--	--	--	--	--	--	--
58	10	--	--	--	--	--	--	--	--	--	--
59	5	--	--	--	--	--	--	--	--	--	--
60	40	--	--	--	--	--	--	--	--	--	--
61	40	--	--	--	--	--	--	--	--	--	--
62	40	--	--	--	--	--	--	--	--	--	--
63	40	526	--	0.137	360.9	--	14.996	41.69	--	128.85	--
64	80	479	--	0.141	435.3	--	14.996	50.28	--	132.62	--
65	80	526	--	0.128	473.7	--	18.176	45.14	--	155.20	--
66	40	440	--	0.137	475.9	--	14.996	54.98	--	128.85	--
67	40	442	462.3	0.0043	523.3	435.3	14.996	60.45	50.28	4.05	3.0702
68	40	356	429	0.0043	631.5	462.4	14.996	72.91	53.42	4.05	12.0731
69	40	266	406	0.0045	857	541.3	14.996	98.99	62.51	4.23	31.000
70	40	266.5	442.2	0.014	902.2	627	14.996	104.2	72.42	13.17	21.000
71	40	266	436.2	0.0123	895.4	611.2	14.996	103.42	70.60	11.57	24.000
72	80	266	--	--	--	--	--	--	--	--	Stuck
73	80	265.5	--	--	--	--	--	--	--	--	Stuck
74	40	266	426.7	0.0121	--	575.2	14.996	--	66.44	11.38	34.7083
75	40	266	--	--	--	--	--	--	--	--	Stuck
76	40	356	463.5	0.0089	715	527.8	14.996	82.58	60.97	8.37	6.6677
77	40	356	469.1	0.0131	737.6	514.3	14.996	85.20	59.41	12.82	7.2716
78	40	412	495.6	0.013	500.7	406	14.996	57.83	46.89	12.23	2.8348
79	40	441	464.3	0.0043	550.3	446.3	14.996	63.57	51.58	4.05	2.8519
80	40	442	472.8	0.0061	496.2	417.3	18.176	57.31	48.20	5.74	2.9623
81	80	441	499.9	0.0071	530.3	523.3	18.176	52.45	49.87	8.50	1.7040
82	80	442	523.1	0.0130	577.4	523.3	18.176	55.03	49.87	15.56	3.4608



No	Extrusion Ratio	(°C) Initial Temp.	(°C) Final Temp.	(m/sec) Ram Speed	(MN/m) Peak Load	(MN/m) Final Load	P/K	(MN/m <sup>2</sup> ) Peak Flow Stress	(MN/m <sup>2</sup> ) Final Flow Stress	(sec <sup>-1</sup> ) Strain Rate	(sec <sup>-1</sup> ) $\dot{\epsilon} \times 10^3$
<b>(HE9)</b>											
83	80	535	539.7	0.0091	381.2	424.0	18.176	36.32	40.41	10.89	0.6102
84	80	446	501.1	0.0089	469.	480.4	18.176	44.70	45.78	10.65	2.0503
85	80	350	459.1	0.0089	575.2	534.5	18.176	54.82	50.94	10.65	9.0973
86	60	350	455.8	0.0091	575.2	514.3	16.818	59.24	52.97	9.90	9.5785
87	60	446	504.4	0.0089	--	481	16.818	--	50.40	9.69	1.6699
88	60	258	422.7	0.0091	805.2	621	16.818	82.93	61.56	9.90	35.6082
89	60	487	514.8	0.0089	421.8	435.3	16.818	43.44	49.99	9.69	1.1867
90	180	487	532.6	0.0089	487.2	523.3	22.304	37.85	40.63	13.59	0.9468
91	180	446	511.7	0.0091	552.6	544.2	22.304	42.92	41.15	13.89	1.8816
92	130	350	472.9	0.0091	648.8	611.5	22.304	50.39	46.42	13.89	7.1582
93	10	350	418.1	0.0094	388.0	333.8	9.507	70.69	60.81	4.94	21.5385
94	10	258	384.2	0.0091	595.5	417.3	9.507	108.50	76.02	4.79	93.7130
95	10	446	461.0	0.0091	293.2	279.7	9.507	53.42	50.96	4.79	3.8114
96	10	487	480.2	0.0091	248.1	259.4	9.507	45.21	47.27	4.79	1.8955
97	5	487	487.1	0.0091	259.4	219.5	7.083	63.43	65.09	3.37	1.0465
98	5	446	453.1	0.0093	236.8	231.2	7.083	57.91	56.53	3.45	3.6987
99	5	350	397.4	0.0093	309.0	243.6	7.083	75.57	59.57	3.45	36.9470
100	5	258	368.4	0.0091	558.2	345.1	7.083	136.30	84.39	3.37	140.1234
<b>(HE30)</b>											
1A	80	440	540.3	0.0128	672.1	597.7	18.176	64.05	56.95	15.52	1.4477
2A	80	478	554.7	0.0129	618	559.4	18.176	58.89	53.31	15.44	0.9399
3A	80	526	594.7	0.0131	563.9	516.5	18.176	53.73	49.23	15.68	0.5315
4A	40	479	534.3	0.0132	516.5	462.4	14.996	59.65	53.42	12.41	1.4148
5A	60	479	550.2	0.0133	584.2	530	16.818	60.17	54.58	14.48	1.0097
6A	180	479	565.9	0.0129	726.3	611.2	22.304	56.40	47.46	19.69	0.8605
7A	180	479	529.2	0.0041	834.5	703.7	22.304	64.81	54.65	6.26	0.8392
8A	10	480	468.1	0.0042	387	354.1	9.507	70.51	64.52	1.28	1.4187
9A	10	479	509.2	0.0132	345.1	338.3	9.507	62.87	61.63	6.95	1.7938
10A	40	479	521.4	0.0089	530	473.7	14.996	61.21	51.72	8.37	1.4434
11A	40	480	493.6	0.0042	577.4	503	14.996	66.68	58.09	3.95	1.7042
12A	60	479	490.7	0.0038	581.9	541.3	16.818	59.93	55.76	4.13	1.7756
13A	60	356	492.	0.00131	744.3	609	16.818	76.66	62.76	14.26	6.6451
14A	60	356	463.1	0.0039	654.1	638.3	16.818	67.36	65.73	4.24	5.6736
15A	40	356.5	453.4	0.004	703.7	581.9	14.996	81.29	76.20	4.92	9.5542
16A	40	356	482.7	0.0133	739.8	563.9	14.996	85.44	65.13	12.51	8.1090
17A	80	356.5	--	0.0133	875.1	--	18.176	83.40	--	15.92	30.9790
18A	80	356.5	468.7	0.0039	764.6	669.9	18.176	72.87	63.84	4.67	5.0601
19A	180	356.5	--	--	--	--	22.304	--	--	--	Stuck
20A	180	357	517.5	0.0129	836.8	721.8	22.304	64.99	56.05	19.70	3.8587
21A	10	356	460.5	0.0127	600	457.9	9.507	109.31	83.42	6.68	9.8685
22A	10	--	--	--	--	--	--	--	--	--	--
23A	40	266.5	--	--	--	--	--	--	--	--	stuck
24A	80	266.5	436.7	0.0043	850.3	694.7	18.176	81.03	66.20	5.14	72.4351
25A	80	356.5	471.0	0.0044	872.9	660.9	18.176	83.17	62.98	5.27	5.2435
26A	40	356.5	462.4	0.0059	730.8	568.4	14.996	84.40	65.65	5.55	7.6215
27A	40	441	499.0	0.0068	611.2	514.3	14.996	70.60	59.41	6.40	2.3373
28A	80	440	514.6	0.0069	636.1	593.2	18.176	60.62	56.53	8.26	1.7807
29A	80	480	530.0	0.0068	581.9	570.6	18.176	55.46	54.37	8.14	1.0639
30A	40	479	513.1	0.0063	593.2	503	14.996	68.42	58.09	5.92	2.1990
31A	80	357	498.5	0.012	784.9	645.1	18.176	74.79	61.47	14.37	5.3387
32A	80	266.5	--	--	--	--	--	--	--	--	stuck
33A	40	266.5	--	--	--	--	--	--	--	--	stuck
34A	40	357	473.0	0.0095	755.6	557.1	14.996	87.28	64.35	8.94	8.2563
35A	40	440	502.8	0.010	581.9	473.1	14.996	67.20	54.62	9.41	3.0161
36A	40	480	530.8	0.0096	568.4	494	14.996	65.65	57.05	9.03	1.1688
37A	40	527	536.1	0.0096	475.9	406	14.996	54.98	46.89	9.03	0.9724
38A	80	479	547.1	0.0106	611.2	559.4	18.176	58.25	53.31	12.69	0.9824
39A	80	442	536.1	0.0106	694.7	604.5	18.176	66.20	57.61	12.69	1.3675
<b>(Al-Ng)</b>											
1N	80	258	--	--	--	--	18.176	--	--	--	stuck
2N	80	305	--	--	--	--	18.176	--	--	--	stuck
3N	80	350	--	--	--	--	18.176	--	--	--	stuck
4N	80	395	529.4	0.0086	--	721.8	18.176	--	68.78	10.30	--
5N	80	446	559.2	0.0099	800.7	699.2	18.176	76.30	66.63	11.85	--
6N	80	487	567.2	0.0096	671	639.4	18.176	63.95	60.93	11.49	--
7N	80	535	581.1	0.010	543.6	57.29	18.176	51.81	54.59	12.00	--
8N	80	487	566.9	0.0133	658.6	579.7	18.176	62.77	55.24	15.92	--
9N	80	446	569.8	0.0131	823.3	694.7	18.176	78.46	66.20	15.68	--
10N	80	446	546.5	0.0083	783.8	678.9	18.176	74.69	64.69	9.94	--
11N	80	446	516.7	0.0043	817.6	687.9	18.176	77.91	65.56	2.97	--
12N	80	446	519.7	0.0043	814.2	701.5	18.176	77.60	66.86	2.97	--
13N	80	446	522.8	0.0041	--	727.4	18.176	--	69.32	4.91	--
14N	80	446	551.3	0.0086	796.2	692.4	18.176	75.88	65.97	10.30	--
15N	80	446	549.5	0.0090	775.9	676.7	18.176	73.94	64.48	10.77	--
16N	80	487	561.0	0.0086	654.1	636.1	18.176	62.34	60.62	10.30	--
17N	80	487	557.4	0.0080	647.3	636.1	18.176	61.68	60.62	9.57	--
18N	80	487	531.6	0.0047	651.8	617.3	18.176	62.11	61.68	5.63	--
19N	80	487	529.4	0.0043	665.4	660.9	18.176	63.41	62.98	5.11	--
20N	80	487	534.5	0.0041	761.2	697.0	18.176	72.54	66.47	4.91	--

No	Extrusion Ratio	(°C)	(°C)	(m/sec)	(MN/m <sup>2</sup> )	(MN/m <sup>2</sup> )	P/E	(MN/m <sup>2</sup> )	(MN/m <sup>2</sup> )	(sec <sup>-1</sup> )	(sec <sup>-1</sup> )
		Initial Temp.	Final Temp.	Ram Speed	Peak Load	Final Load		Peak Flow Stress	Final Flow Stress	Strain Rate	$\times 10^{11}$
(A1)											
1A1	80	478	492.3	0.0043	439.8	509.7	18.176	41.92	48.57	5.14	
2A1	80	480	495.2	0.0041	469.1	530.0	18.176	44.70	50.51	4.91	
3A1	80	478	489.8	0.0057	475.9	534.5	18.176	45.35	50.94	4.43	
4A1	80	478	526.4	0.0083	180.4	521.0	18.176	45.78	49.64	9.94	
5A1	80	478	536.6	0.0106	475.9	507.5	18.176	45.35	48.56	12.68	
6A1	80	476	548.9	0.0159	536.8	530.0	18.176	51.15	50.51	16.64	
7A1	80	437	519.2	0.0123	534.5	527.8	18.176	50.94	50.30	14.72	
8A1	80	437	518.3	0.0107	536.8	543.6	18.176	51.15	51.81	12.81	
9A1	80	437	515.1	0.009	557.1	557.1	18.176	53.09	53.09	10.77	
10A1	80	437	491.0	0.0043	543.6	590.9	18.176	51.81	56.31	5.14	
11A1	80	437	466.2	0.0041	527.8	484.9	18.176	50.28	46.21	4.91	
12A1	80	437	483.7	0.004	530.0	570.6	18.176	50.51	54.37	4.79	
13A1	80	395	471.9	0.0041	609.0	593.2	18.176	58.04	56.53	4.91	
14A1	80	265.5	--	--	--	--	18.176	--	--	--	stuck
15A1	80	305	--	--	--	--	18.176	--	--	--	stuck
16A1	80	305	447.3	0.0054	827.7	639.3	18.176	78.88	60.83	6.46	
17A1	80	356	463.4	0.0041	782.6	658.3	18.176	74.58	60.83	4.91	
18A1	80	356	502.0	0.00154	787.1	649.6	18.176	75.0	61.90	16.04	
19A1	80	395	514.1	0.0157	671.0	597.7	18.176	63.95	56.95	16.40	
20A1	80	395	762.4	0.0044	615.5	541.3	18.176	58.46	51.58	5.27	
21A1	80	533	532.1	0.0043	489.4	577.4	18.176	46.64	55.03	5.14	
22A1	80	536	567.0	0.0124	417.3	471.4	18.176	39.77	44.93	14.84	
23A1	80	582	589.0	0.0126	333.8	442.1	18.176	31.82	42.12	15.08	
24A1	80	582	534.7	0.0044	309.0	480.4	18.176	29.45	45.78	5.27	

No	% Glass	Extrusion Ratio	(°C)	(°C)	(M/sec)	(MN/m)	P/E	(MN/m)	(sec <sup>-1</sup> )
			Initial Temp.	Final Temp.	Ram Speed	Final Load		Final Flow Stress	Strain Rate
(HE9 + Glass)									
1C	1	80	446	482.4	0.005	500.7	18.176	47.72	5.96
2C	3	80	446	472.6	0.0044	480.4	18.176	45.78	5.29
3C	5	80	446	482.4	0.0047	512.0	18.176	48.79	5.63
4C	8	80	445	494.8	0.005	557.1	18.176	53.09	5.98
5C	10	80	447	487.0	0.0047	530.0	18.176	50.51	5.63
6C	12	80	446	488.1	0.0047	536.8	18.176	51.15	5.63
7C	15	80	446	499.8	0.0049	581.9	18.176	55.46	5.81
8C	17	80	446	502.5	0.0044	613.5	18.176	58.46	5.29
9C	15	80	446	494.2	0.0047	563.9	18.176	53.73	5.63
10C	20	80	444	496.7	0.0046	584.2	18.176	55.67	5.46
11C	1	40	446	472.3	0.0049	448.8	14.996	55.24	4.56
12C	3	40	446	476.3	0.0046	478.2	14.996		4.29
13C	5	40	446	482.9	0.005	498.4	14.996	56.33	4.69
14C	8	40	446	482.4	0.0047	498.5	14.996	57.57	4.42
15C	10	40	446	476.8	0.0046	453.4	14.996	52.38	4.29
16C	12	40	445	484.2	0.0051	489.4	14.996	56.53	4.85
17C	17	40	446	479.3	0.0049	478.2	14.996	55.24	4.56
18C	15	40	444	471.7	0.0043	469.1	14.996	54.18	4.02
19C	1	40	447	501.3	0.0131	417.3	14.996	48.20	12.32
20C	3	40	446	499.7	0.0131	410.5	14.996	47.41	12.32
21C	5	40	446	516.5	0.0125	480.4	14.996	55.50	11.80
22C	8	40	445	515.7	0.0124	480.4	14.996	55.50	11.66
23C	10	40	446	498.2	0.0128	406.0	14.996	46.89	12.07
24C	12	40	446	495.1	0.0125	408.3	14.996	47.16	11.80
25C	15	40	446	528.9	0.0121	530.0	14.996	61.21	11.39
26C	17	40	445	502.0	0.0128	421.8	14.996	48.72	12.07
27C	1	40	446	499.8	0.0087	466.9	14.996	52.91	8.18
28C	3	40	446	499.3	0.0088	462.4	14.996	53.42	8.31
29C	5	40	445	502.1	0.0086	478.2	14.996	55.24	8.04
30C	8	40	446	491.2	0.0088	428.6	14.996	49.50	8.31
31C	10	40	445	508.6	0.0087	503.0	14.996	58.09	8.18
32C	12	40	446	501.9	0.0086	475.9	14.996	54.98	8.04
33C	15	40	445	490.8	0.0088	428.6	14.996	49.50	8.31
34C	17	40	446	497.0	0.0088	448.9	14.996	51.86	8.31
35C	5	40	447	462.9	0.0040	444.3	14.996	51.32	3.75
36C	5	40	445	483.9	0.0070	455.3	14.996	50.28	6.57
37C	5	80	446	490.8	0.0070	475.9	18.176	45.55	8.35
38C	5	80	446	475.4	0.0037	530.0	18.176	50.51	4.44
39C	5	80	445	525.3	0.0113	541.3	18.176	51.58	13.47
40C	1	80	446	518.0	0.0131	489.4	18.176	46.64	15.69
41C	10	80	446	527.9	0.0125	530.0	18.176	50.51	15.01
42C	8	80	447	546.1	0.0125	609.0	18.176	58.04	14.68
43C	17	80	446	532.4	0.0123	552.6	18.176	52.65	14.68
44C	15	80	447	532.5	0.0120	557.1	18.176	53.09	14.33
45C	12	80	445	520.7	0.0120	512.0	18.176	48.79	14.33
46C	5	80	446	525.3	0.0120	532.3	18.176	50.73	14.33
47C	1	80	446	522.1	0.0094	557.1	18.176	53.09	11.26
48C	5	80	445	499.5	0.0088	473.7	18.176	45.14	10.58
49C	10	80	447	519.8	0.0093	548.1	18.176	52.74	11.09
50C	8	80	446	502.7	0.0091	509.7	18.176	48.57	10.92

Table 10 continued ...

No	$\frac{1}{2}$ Glass	Extrusion Ratio	(°C) Initial Temp.	(°C) Final Temp.	(M/sec) Ram Speed	(MN/m) Final Load	P/K	(MN/m) Final Flow stress	(sec <sup>-1</sup> ) Strain Rate
<b>(HE9 + Glass)</b>									
51C	17	80	445	521.0	0.0094	548.1	18.176	52.24	11.26
52C	20	80	446	507.8	0.0086	509.7	18.176	48.57	10.24
53C	8.8	80	447	505.2	0.0088	496.2	18.176	47.29	10.58
54C	20	80	447	491.5	0.0040	586.4	18.176	55.88	4.78
55C	20	80	446	536.5	0.0120	570.6	18.176	54.37	14.53
56C	8.8	80	446	534.6	0.0128	554.9	18.176	52.88	15.36
57C	8.8	80	446	507.1	0.0047	622.5	18.176	59.32	5.63
58C	5	80	486	498.5	0.0059	548.1	18.176	52.24	4.61
59C	5	80	488	546.1	0.0128	500.7	18.176	47.72	15.36
60C	5	80	488	508.7	0.0050	527.8	18.176	50.30	5.98
61C	5	80	489	525.2	0.0067	531.6	18.176	50.94	8.02
62C	5	80	490	536.1	0.0084	534.6	18.176	50.94	10.07
63C	5	80	488	529.5	0.0081	507.5	18.176	48.36	10.07
64C	5	80	537	556.2	0.0083	507.5	18.176	48.36	9.90
65C	5	80	537	566.9	0.0128	462.4	18.176	44.06	15.36
66C	5	80	535	523.7	0.0040	557.1	18.176	53.09	4.78
67C	5	80	537	533.9	0.0049	543.6	18.176	51.81	5.81
68C	5	80	537	550.8	0.0076	505.2	18.176	48.15	9.04
69C	5	80	536	567.7	0.0083	557.1	18.176	53.09	9.90
70C	5	80	396	492.9	0.0086	568.4	18.176	54.16	10.24
71C	5	80	396	473.4	0.0045	593.2	18.176	56.53	5.12
72C	5	80	397	502.2	0.0135	545.8	18.176	52.01	16.21
73C	5	80	397	471.7	0.0050	557.1	18.176	53.09	5.98
74C	5	80	398	481.7	0.0066	557.1	18.176	50.51	7.85
75C	5	80	396	492.5	0.0114	530.0	18.176	56.95	13.65
76C	5	80	352	483.4	0.0117	597.7	18.176	56.95	14.00
77C	5	80	352	455.6	0.0044	597.7	18.176	56.95	5.29
78C	5	80	351	485.0	0.0134	590.9	18.176	56.31	16.04
79C	5	80	352	461.9	0.0054	597.7	18.176	56.95	6.49
80C	5	80	353	464.1	0.0076	566.1	18.176	53.95	9.04
81C	5	80	352	473.6	0.0091	584.2	18.176	55.67	10.92
82C	8.8	80	447	522.7	0.0097	550.3	18.176	52.45	11.60
83C	8.8	80	447	495.6	0.0044	581.9	18.176	55.46	5.29
84C	8.8	80	446	532.8	0.0133	543.6	18.176	51.81	15.86
85C	8.8	80	447	494.0	0.0051	543.6	18.176	51.81	6.14
86C	5	80	447	509.2	0.0064	566.1	18.176	53.95	7.67
87C	5	80	447	523.0	0.0112	550.0	18.176	50.51	13.31
88C	5	40	446	496.0	0.0113	412.8	14.996	47.68	10.59
89C	5	40	397	497.4	0.0125	525.5	14.996	60.69	11.80
90C	5	40	397	449.0	0.0043	473.7	14.996	54.72	3.89
91C	5	40	396	462.8	0.0047	514.3	14.996	59.41	4.42
92C	5	40	398	469.8	0.0068	489.4	14.996	56.53	6.43
93C	5	40	397	482.3	0.0097	496.2	14.996	57.31	9.12
94C	5	40	397	486.0	0.0117	489.4	14.996	56.53	10.99
95C	5	40	536	569.5	0.0107	496.2	14.996	57.31	10.06
96C	5	40	535	56.13	0.0125	437.6	14.996	50.54	11.80
97C	5	40	536	502.6	0.0043	424.8	14.996	49.07	4.04
98C	5	40	536	532.0	0.0053	499.2	14.996	57.66	4.98
99C	5	40	537	552.0	0.0071	511.4	14.996	59.06	6.72
100C	5	40	535	541.3	0.0091	415.3	14.996	47.96	8.60
101C	5	40	536	542.0	0.0113	378.7	14.996	43.73	10.62
102C	5	40	536	553.9	0.0127	404.4	14.996	46.71	11.96
103C	5	40	514	553.6	0.0093	512.7	14.996	59.22	8.74
104C	5	40	468	502.4	0.0096	411.2	14.996	47.49	9.00
105C	5	40	421	--	--	432.8	14.996	49.99	--
106C	5	40	468	--	--	428.7	14.996	49.52	--
107C	5	40	372	456.0	0.0094	453.2	14.996	52.34	8.87
108C	5	40	351	--	--	491.1	14.996	56.73	--
109C	5	40	351	421.4	0.0059	450.4	14.996	52.05	3.63
110C	5	40	351	438.7	0.0051	483.8	14.996	51.04	4.80
111C	5	40	350	437.4	0.0074	449.1	14.996	51.87	6.99
112C	5	40	351	--	--	480.3	14.996	55.46	--
113C	5	40	352	452.4	0.0139	455.8	14.996	52.56	13.04
114C	5	40	446	505.9	0.0137	428.7	14.996	49.52	12.88
115C	5	5	447	--	0.0125	--	7.083	--	--
116C	5	10	446	--	0.0129	--	9.507	--	--
117C	5	80	446	--	0.0125	--	18.176	--	--
118C	5	180	445	--	0.0123	--	22.304	--	--
119C	5	60	446	--	0.0126	--	16.818	--	--
120C	5	80	468	--	0.0093	--	18.176	--	--
121C	5	80	421	--	0.0097	--	18.176	--	--
122C	5	80	372	--	0.0090	--	18.176	--	--
123C	5	80	514	--	0.0091	--	18.175	--	--
<b>(Al-Mg + Glass)</b>									
1NR	1	80	446	541.2	0.0089	645.1	18.176	61.47	10.58
2NR	3	80	446	553.2	0.0091	687.9	18.176	65.56	10.94
3NR	5	80	445	548.9	0.0086	681.2	18.176	64.92	10.76
4NR	7	80	447	549.2	0.0083	687.9	18.176	65.56	9.92

No	% Glass	Extrusion Ratio	(°C) Initial Temp	(°C) Final Temp	(M/sec) Ram Speed	(MN/m <sup>2</sup> ) Final Load	T/K	(MN/m <sup>2</sup> ) Final Flow stress	(sec <sup>-1</sup> ) Strain Rate
<u>(Al-Ng + Glass)</u>									
5NR	9	80	446	554.5	0.0080	715.0	18.176	68.14	9.57
6NR	11	80	446	549.1	0.0083	685.7	18.176	65.35	9.92
7NR	13	80	447	553.7	0.0080	710.5	18.176	67.71	9.57
8NR	15	80	445	558.0	0.0080	727.4	18.176	69.32	9.57
9NR	5	80	446	570.8	0.0127	701.5	18.176	66.86	15.21
10NR	5	80	446	505.9	0.0040	651.1	18.176	62.34	4.79
11NR	5	80	487	535.3	0.0045	687.9	18.176	65.56	5.13
12NR	5	80	487	563.1	0.0080	658.6	18.176	62.77	9.57
13NR	5	80	488	589.9	0.0130	672.1	18.176	64.05	15.56
<u>(Al + Glass)</u>									
1A1C	5	80	446	524.2	0.0139	507.5	18.176	48.36	16.59
2A1C	5	80	446	520.5	0.0089	559.3	18.176	53.30	10.61
3A1C	5	80	447	517.2	0.0083	557.1	18.176	53.09	8.92
4A1C	5	80	444	500.3	0.0041	624.7	18.176	59.53	4.95
5A1C	5	80	446	492.1	0.0044	568.4	18.176	54.16	3.30
6A1C	5	80	447	493.1	0.0043	579.6	18.176	55.24	5.13
7A1C	5	80	421	482.3	0.0013	581.9	18.176	55.46	5.13
8A1C	5	80	421	522.1	0.0129	570.6	18.176	54.37	15.59
9A1C	5	80	396	517.8	0.0133	611.2	18.176	58.25	15.91
10A1C	1	80	396	467.4	0.0036	597.7	18.176	56.95	4.31
11A1C	3	80	536	580.7	0.0106	557.1	18.176	53.09	12.65
12A1C	5	80	534	585.1	0.0084	624.7	18.176	59.53	10.09
13A1C	7	80	537	575.7	0.00100	548.1	18.176	52.24	11.97
14A1C	9	80	536	584.3	0.0091	602.2	18.176	57.38	10.94
15A1C	11	80	535	578.5	0.0077	618.0	18.176	58.89	9.23
16A1C	15	80	537	569.8	0.0086	557.1	18.176	53.09	10.26
17A1C	5	80	536	571.3	0.0083	570.6	18.176	54.37	9.92
18A1C	5	80	536	556.3	0.0076	530.0	18.176	50.51	9.06
19A1C	5	80	351	523.6	0.0130	746.5	18.176	71.14	15.56
20A1C	5	80	351	476.6	0.0044	694.7	18.176	66.20	5.30
21A1C	5	80	351	500.1	0.0100	683.4	18.176	65.13	11.97
22A1C	5	80	372	517.7	0.0096	710.5	18.176	67.71	11.45
23A1C	5	80	372	536.7	0.0137	739.8	18.176	70.50	16.41
24A1C	5	80	372	487.0	0.0047	687.9	18.176	65.56	5.64
25A1C	5	80	468	519.4	0.0040	672.1	18.176	64.05	4.79
26A1C	5	80	468	570.7	0.0134	658.3	18.176	60.83	16.07
27A1C	5	80	514	588.9	0.0131	599.9	18.176	57.18	15.73
28A1C	5	80	514	531.9	0.0039	647.3	18.176	61.68	4.62
29A1C	5	80	536	581.2	0.0086	604.4	18.176	57.59	10.26
30A1C	5	80	536	606.0	0.0137	604.4	18.176	57.59	16.41
31A1C	5	80	536	588.1	0.0097	604.4	18.176	57.59	11.62
32A1C	5	80	536	554.2	0.0046	654.1	18.176	62.34	5.47

<u>TEMP</u> <u>(°C)</u>	<u>EQUATION</u>	<u>CORRELATION</u> <u>COEFFICIENT</u>	<u>REDUNDANT</u> <u>WORK TERM</u>
250	$p = 179.2 + 111.0 \ln R$	0.992	1.61
350	$p = 122.0 + 96.1 \ln R$	0.990	1.27
450	$p = 92.7 + 89.8 \ln R$	0.986	1.03
500	$p = 61.3 + 91.3 \ln R$	0.993	0.67
540	$p = 71.8 + 82.5 \ln R$	0.988	0.87

TABLE 11

Pressure vs.  $\ln R$  equations for HE9 extrusions  
at different temperatures

<u>300°C</u>		<u>350°C</u>		<u>400°C</u>		<u>450°C</u>		<u>500°C</u>	
<u>Twist Rate</u> (Revs/sec)	<u>Max Torque</u> (NM)	<u>Twist Rate</u>	<u>Max Torque</u>	<u>Twist Rate</u>	<u>Max Torque</u>	<u>Twist Rate</u>	<u>Max Torque</u>	<u>Twist Rate</u>	<u>Max Torque</u>
0.14	10.25	0.16	7.11	0.16	5.81	0.16	4.57	0.16	3.51
0.42	12.02	0.42	7.53	0.45	6.38	0.46	5.15	0.47	4.04
2.99	12.56	2.99	10.16	3.02	8.13	3.17	6.21	3.03	5.38
7.55	13.47	7.66	10.39	6.30	8.63	7.69	7.45	7.77	5.51
15.09	14.03	14.81	11.12	12.50	8.91	15.38	8.38	15.38	6.81
				10.87	8.75				
				15.34	9.07				

TABLE 12

HE9 torsion test results

<u>400°C</u>		<u>450°C</u>		<u>500°C</u>		<u>550°C</u>	
<u>Twist</u> <u>Rate</u> <u>(Revs/</u> <u>sec)</u>	<u>Max</u> <u>Torque</u> <u>(NM)</u>	<u>Twist</u> <u>Rate</u>	<u>Max</u> <u>Torque</u>	<u>Twist</u> <u>Rate</u>	<u>Max</u> <u>Torque</u>	<u>Twist</u> <u>Rate</u>	<u>Max</u> <u>Torque</u>
0.43	8.82	0.16	7.58	0.16	5.48	0.16	3.07
3.03	11.31	0.43	7.58	0.46	5.84		
7.69	12.16	3.18	9.31	3.07	7.84		
14.45	12.63	7.04	10.50	7.81	7.47		
		15.06	10.81	16.00	9.48		

TABLE 13

HE30 torsion test results

Temperature (°C)	Shear Strain rate (sec <sup>-1</sup> )		0.5	2.0	10	25	50
	m	n values					
300	0.006	-0.148	---	-0.139	-0.227	-0.146	
350	0.100	-0.124	-0.073	-0.135	-0.210	-0.273	
400	0.136	-0.001	-0.170	-0.096	-0.147	-0.300	
450	0.165	0.060	0.001	-0.217	-0.265	-0.252	
500	0.070	0.183	0.060	-0.255	-0.233	-0.333	

TABLE 14 Backofen analysis exponents for HE9

Temperature (°C)	Shear Strain rate (sec <sup>-1</sup> )		0.5	2.0	10	25	50
	m	n values					
400	0.124	-0.088	-0.119	-0.094	-0.048	-0.08	
450	0.107	0.159	-0.016	---	-0.115	-0.123	
500	0.146	0.103	-0.231	0.038	-0.116	-0.022	
550	---	0.211	---	---	---	---	

TABLE 15 Backofen analysis exponents for HE30



<u>300°C</u>		<u>350°C</u>		<u>400°C</u>		<u>450°C</u>		<u>500°C</u>	
<u>Shear</u> <u>Flow</u> <u>Stress</u> <u>(MN/m<sup>2</sup>)</u>	<u>Shear</u> <u>Strain</u> <u>Rate</u> <u>(sec<sup>-1</sup>)</u>	<u>Shear</u> <u>Flow</u> <u>Stress</u>	<u>Shear</u> <u>Strain</u> <u>Rate</u>	<u>Shear</u> <u>Flow</u> <u>Stress</u>	<u>Shear</u> <u>Strain</u> <u>Rate</u>	<u>Shear</u> <u>Flow</u> <u>Stress</u>	<u>Shear</u> <u>Strain</u> <u>Rate</u>	<u>Shear</u> <u>Flow</u> <u>Stress</u>	<u>Shear</u> <u>Strain</u> <u>Rate</u>
37.3	0.49	26.94	0.49	21.19	0.49	17.49	0.49	13.46	0.49
---	---	26.25	1.32	24.09	1.40	20.76	1.43	14.95	1.46
45.9	9.39	38.35	9.38	31.47	9.78	23.35	9.96	17.38	9.52
47.7	23.71	38.23	24.07	---	---	27.51	24.17	20.12	24.40
51.1	47.42	40.02	46.54	32.75	48.18	31.08	48.18	23.73	48.38

TABLE 16

Flow stress vs. strain rate data from HE9 torsion tests

<u>400°C</u>		<u>450°C</u>		<u>500°C</u>		<u>550°C</u>	
<u>Shear</u> <u>Flow</u> <u>Stress</u> <u>(MN/m<sup>2</sup>)</u>	<u>Shear</u> <u>Strain</u> <u>Rate</u> <u>(sec<sup>-1</sup>)</u>	<u>Shear</u> <u>Flow</u> <u>Stress</u>	<u>Shear</u> <u>Strain</u> <u>Rate</u>	<u>Shear</u> <u>Flow</u> <u>Stress</u>	<u>Shear</u> <u>Strain</u> <u>Rate</u>	<u>Shear</u> <u>Flow</u> <u>Stress</u>	<u>Shear</u> <u>Strain</u> <u>Rate</u>
28.79	0.49	26.28	0.49	18.86	0.49	10.39	0.49
30.38	1.36	26.05	1.36	17.89	1.45		
43.13	9.51	39.09	22.12	28.62	9.64		
45.55	24.17	35.87	47.31	27.97	24.54		
47.25	45.40			33.53	50.27		

TABLE 17

Flow stress vs. strain data from HE30 torsion tests

<u>Temperature</u> (°C)	<u><math>\beta</math></u>	<u><math>n_2</math></u>	<u><math>\alpha</math></u>
300	0.212	14.311	0.015
350	0.210	10.602	0.020
400	0.185	8.126	0.023
450	0.237	8.512	0.028
500	0.276	6.400	0.043

TABLE 18

Hot working theory "constants" for HE9  
at different temperatures

<u>Temperature</u> (°C)	<u><math>\beta</math></u>	<u><math>n_2</math></u>	<u><math>\alpha</math></u>
400	0.082	4.016	0.020
450	0.175	9.693	0.018
500	0.157	6.600	0.024

TABLE 19

Hot working theory "constants" for HE30  
at different temperatures

	<u>HE9</u>	<u>HE30</u>
$A_1$ ( $\text{sec}^{-1}$ )	$0.898 \times 10^{11}$	$0.271 \times 10^{11}$
$\alpha$ (M /MN)	0.038	0.040
n	5.16	3.87
$\Delta H$ (J/mole)	167224	170881

TABLE 20

Hot working theory constants and activation energies produced by optimization procedure

		HE9 (with pads)				
Ram Speed (M/sec)	Temp (°C)	250	350	450	500	540
0.003		C	A	B	A	A
0.008		C	C	C	A	A
0.013		A	A	A	A	A

		HE9 + 5 wt.% Glass (with pads)						
Ram Speed (M/s)	Temp (°C)	250	300	350	400	450	500	550
0.003		--	--	C	B	A	A	A
0.008		--	--	C	A	A	A	A
0.013		--	--	C	A	A	A	A

		(with pads) HE9 + x% Glass, ram speed = 0.004 m/sec.					
Temp (°C)	wt.% Glass	1	5	8	12	15	17
350		--	B	--	--	--	--
400		--	C	--	--	--	--
450		A	A	C	F	F	G
500		--	A	--	--	--	--
550		--	A	--	--	--	--

(with pads) HE9 + x% Glass, Ram Speed = 0.008 m/sec

Temp (°C)	wt.% Glass	1	5	8	12	15	17
350		--	B	--	--	--	--
400		--	B	--	--	--	--
450		B	B	F	G	G	H
500		--	A	--	--	--	--
550		--	A	--	--	--	--

(with pads) HE9 + x% Glass, Ram Speed = 0.013 m/sec

Temp (°C)	wt.% Glass	1	5	8	12	15	17
350		--	B	--	--	--	--
400		--	A	--	--	--	--
450		A	A	F	G	G	H
500		--	A	--	--	--	--
550		--	A	--	--	--	--

(with pads) HE30

Ram Speed (M/sec)	Temp (°C)	250	350	450	500	540
0.003		B	B	B	B	B
0.008		--	A	A	A	A
0.013		--	A	B	A	A

Al (no pads)

Ram Speed	Temp (°C)	250	300	350	400	450	500	550	600
0.003		--	D	D	D	D	D	D	D
0.008		--	--	--	--	D	D	--	--
0.013		--	D	D	D	D	D	D	A

Al + 5wt.% Glass (with pads)

Ram Speed m/s	Temp (°C)	350	375	400	425	450	475	525	550
0.003		--	D	D	D	D	D	D	D
0.008		--	D	--	D	D	--	--	C
0.013		C	C	F	--	F	C	C	C

Al-Mg (no pads)

Ram Speed	Temp (°C)	400	450	500	550
0.003		--	A	A	A
0.008		B	A	A	A
0.013		--	B	--	B

Al-Mg (with pads)

Ram Speed	Temp (°C)	450	500
0.003		D	A
0.008		B	B
0.013		A	B

TABLE 21

Effect of temperature, strain rate and glass content variations on extrude surface finish

Extrusion No.	Room Temp.		Room Temp. % Elongation	400°C		400°C % Elongation
	0.2% P.S. (MN/m <sup>2</sup> )	U.T.S. (MN/m <sup>2</sup> )		0.2% P.S. (MN/m <sup>2</sup> )	U.T.S. (MN/m <sup>2</sup> )	
(HF9)						
1	--	--	--	16.6	26.4	--
2	73.5	142.0	--	16.0	26.6	--
3	--	--	--	15.2	25.2	--
4	72.8	129.0	--	16.6	25.4	--
5	77.8	128.7	--	18.5	25.2	--
6	--	--	--	12.5	25.1	--
7	88.1	132.9	25.8	--	23.6	--
8	88.8	151.5	25.8	16.4	26.2	56.6
9	86.0	130.3	25.4	17.2	24.5	31.6
10	83.6	129.5	27.8	13.6	25.3	38.7
11	85.9	129.9	27.0	15.1	24.9	38.0
12	90.1	129.6	28.2	13.0	25.7	37.7
13	88.4	151.9	29.2	15.2	25.5	43.5
14	82.1	134.6	26.5	14.8	25.2	59.4
15	88.1	126.8	28.1	13.9	24.2	37.6
16	86.3	124.7	27.9	11.8	23.9	40.9
17	86.1	124.0	27.8	13.2	23.2	40.8
18	104.7	150.4	23.9	16.2	24.8	42.3
19	69.1	130.9	31.5	15.0	24.5	27.9
20	113.4	159.7	22.2	17.8	29.2	33.3
21	--	--	--	--	--	--
22	104.9	153.1	24.0	18.2	25.9	44.9
23	101.0	143.3	27.0	14.5	26.5	42.5
24	77.3	136.8	26.0	15.6	22.9	38.6
25	91.7	137.2	26.9	--	25.1	--
26	95.5	155.4	27.1	18.6	24.6	40.6
27	81.8	143.2	26.1	17.8	24.1	43.1
28	71.7	148.3	--	14.3	--	--
29	69.0	146.9	--	16.1	23.8	--
30	87.5	148.4	--	17.0	24.0	--
31	--	--	--	--	--	--
32	80.3	144.8	21.6	16.0	23.6	43.1
33	99.2	141.1	--	18.7	24.2	--
34	79.2	135.2	26.1	--	--	--
35	81.2	145.8	27.6	15.8	24.4	39.7
36	92.2	150.1	27.3	11.8	24.2	42.3
37	84.5	144.0	29.9	--	--	--
38	61.2	142.4	28.6	18.1	23.7	--
39	85.3	132.5	--	16.9	24.5	--
40	80.6	136.4	22.3	11.4	24.6	41.9
41	77.4	138.8	23.0	16.9	22.9	40
42	79.9	139.5	--	18.2	22.6	--
43	93.3	143.5	--	15.9	23.3	--
44	91.2	147.7	26.4	14.7	24.3	40.8
45	89.7	141.7	28.4	18.7	22.6	--
46	97.4	141.6	--	--	23.9	--
47	72.5	129.4	28.9	15.8	22.6	43.1
48	--	--	--	--	--	--
49	71.7	150.5	--	15.7	23.4	--
50	84.2	142.4	--	17.6	25.4	--
51	65.2	151.1	--	14.5	22.6	--
52	85.5	148.5	--	14.6	21.8	--
62	--	136.0	27.8	--	24.1	--
63	77.0	132.8	21.9	14.4	22.4	39.0
64	92.2	146.4	26.5	14.7	23.7	13.3
65	88.9	132.0	--	17.3	25.0	--
66	77.9	129.8	27.3	--	26.1	--
67	81.1	129.8	27.4	17.1	24.6	41.7

Extrusion No.	Room Temp.		Room Temp. % Elongation	100°C		400°C	
	0.2% P.S. (MN/m <sup>2</sup> )	U.T.S. (MN/m <sup>2</sup> )		0.2% P.S. (MN/m <sup>2</sup> )	U.T.S. (MN/m <sup>2</sup> )	% Elongation	
<b>(HE9)</b>							
68	109.5	147.5	27.5	18.7	26.7	45.4	
69	122.6	159.1	22.7	21.5	32.8	32.8	
70	104.3	152.4	23.4	18.0	30.1	41.6	
71	112.9	152.8	28.0	--	--	--	
72	--	--	--	--	--	--	
73	--	--	--	--	--	--	
74	107.2	149.6	--	16.0	31.4	--	
75	--	--	--	--	--	--	
76	112.5	152.8	--	--	--	--	
77	105.9	155.5	24.2	13.5	28.6	47.1	
78	79.6	123.1	30.7	14.6	25.7	46.2	
79	83.6	130.2	--	14.2	24.4	--	
80	87.3	122.8	--	17.5	24.7	--	
81	80.2	132.3	--	18.2	24.2	--	
82	87.7	143.9	30.1	13.4	24.8	42.7	
<b>(HE50)</b>							
1A	141.5	210.8	37	25.5	47.7	53	
2A	124.4	181.4	--	20.0	41.5	--	
3A	120.0	189.6	40.7	22.5	35.3	52.9	
4A	116.7	181.7	40.2	--	47.9	--	
5A	129.9	185.1	39.2	26.9	41.5	54.3	
6A	--	--	--	--	--	--	
7A	--	--	--	--	--	--	
8A	108.3	170.3	--	24.2	39.1	--	
9A	104.2	174.8	--	25.4	37.7	--	
10A	116.0	175.2	46.0	--	--	--	
11A	114.7	172.5	34.6	28.2	39.8	60.8	
12A	123.8	177.6	41.4	--	45.6	--	
13A	139.8	198.6	52.4	26.1	45.8	46.2	
14A	142.0	198.1	33.5	24.2	47.7	58.7	
15A	145.3	190.2	--	42.9	48.7	--	
16A	140.8	192.2	51.8	27.6	46.9	53.7	
17A	142.1	189.8	37.8	26.7	49.2	52.7	
18A	158.3	212.6	39.6	26.2	48.4	60.1	
19A	--	--	--	--	--	--	
20A	--	--	--	--	--	--	
21A	--	--	--	--	--	--	
22A	--	--	--	--	--	--	
23A	--	--	--	--	--	--	
24A	149.8	200.5	38.2	20.0	51.0	57.0	
25A	157.8	212.6	36.1	28.6	48.0	60.5	
26A	141.1	191.8	--	26.1	46.7	--	
27A	126.7	181.5	--	24.2	47.9	--	
28A	144.5	208.9	36.8	25.5	46.4	56.0	
29A	117.0	183.9	40.1	26.7	38.4	53.9	
30A	126.7	178.7	40.1	30.6	43.4	55.2	
31A	145.1	202.2	--	30.4	48.3	--	
32A	--	--	--	--	--	--	
33A	--	--	--	--	--	--	
34A	144.0	191.2	--	27.5	46.5	--	
35A	131.0	190.3	39.6	29.3	49.6	55.6	
36A	127.5	179.0	--	31	43.8	--	
37A	101.4	173.7	44.0	--	--	--	
38A	115.6	185.8	40.1	22.0	41.4	57.6	
39A	131.1	187.2	--	20.9	47.7	50.3	

Table 22 continued ...

Extrusion No.	Room Temp.	Room Temp.	Room Temp.	400°C	400°C	400°C
	0.2% P.S. (MN/m <sup>2</sup> )	U.T.S. (MN/m <sup>2</sup> )	% Elongation	0.2% P.S. (MN/m <sup>2</sup> )	U.T.S. (MN/m <sup>2</sup> )	% Elongation
<u>(Al-Ni)</u>						
4N	176.2	305	22.1	--	--	--
5N	163.2	298.4	21.7	--	--	--
6N	155.1	285.7	21.7	--	--	--
7N	147.0	278.7	21.1	--	--	--
8N	152.0	283.2	22.1	--	--	--
9N	159.2	290.8	21.4	--	--	--
10N	167.5	299.0	21.6	--	--	--
11N	168.6	302.5	21.7	--	--	--
13N	170.5	301.5	20.3	--	--	--
14N	161.9	289.9	18.4	--	--	--
15N	155.4	282.8	19.5	--	--	--
21N	146.8	265.3	17.7	--	--	--
<u>(Al)</u>						
15A1	66.3	85.4	2.6	--	--	--
18A1	57.4	78.6	2.9	--	--	--
19A1	53.3	73.5	3.0	--	--	--
22A1	65.1	120.7	9.5	--	--	--
23A1	77.2	162.6	10.5	--	--	--
<u>(HE9 + GLASS)</u>						
39C	93.3	207.4	18.5	--	--	--
40C	89.9	149.3	27.9	--	--	--
41C	82.0	243.7	14.1	--	--	--
42C	81.0	221.9	15.7	--	--	--
45C	--	--	--	--	--	--
46C	79.0	198.0	22.3	--	--	--
48C	120.0	189.4	19.0	--	--	--
58C	82.2	223.6	21.0	--	--	--
59C	79.6	195.9	25.2	--	--	--
60C	112.9	206.4	22.1	--	--	--
61C	76.7	216.0	23.9	--	--	--
62C	71.2	212.0	23.6	--	--	--
63C	87.4	215.3	22.8	--	--	--
69C	76.5	218.9	28.7	--	--	--
70C	83.6	189.7	21.2	--	--	--
120C	85.3	204.7	22.7	--	--	--
121C	78.2	197.0	23.3	--	--	--
123C	59.9	210.8	26.0	--	--	--
125C	81.8	167.6	16.4	--	--	--
126C	75.9	180.0	13.5	--	--	--
127C	75.9	193.6	13.7	--	--	--
128C	75.4	197.6	19.9	--	--	--
129C	73.8	166.2	23.2	--	--	--
130C	78.3	204.3	27.3	--	--	--
<u>(Al-Mg + GLASS)</u>						
1NR	158.8	297.1	20.3	--	--	--
2NR	147.1	305.2	16.8	--	--	--
3NR	137.4	289.1	9.9	--	--	--
4NR	131.1	215.1	6.3	--	--	--
<u>(Al + GLASS)</u>						
11A1C	58.3	96.7	3.8	--	--	--
12A1C	59.4	91.2	3.1	--	--	--
13A1C	58.5	101.0	4.0	--	--	--
14A1C	62.6	101.4	4.1	--	--	--
15A1C	73.5	139.6	4.4	--	--	--



HE9		HE9 + GLASS		HE30		Al-Mg		Al-Mg + GLASS		Al		Al + GLASS	
<u>Extrude</u> <u>No.</u>	<u>HV</u>	<u>Extrude</u> <u>No.</u>	<u>HV</u>	<u>Extrude</u> <u>No.</u>	<u>HV</u>	<u>Extrude</u> <u>No.</u>	<u>HV</u>	<u>Extrude</u> <u>No.</u>	<u>HV</u>	<u>Extrude</u> <u>No.</u>	<u>HV</u>	<u>Extrude</u> <u>No.</u>	<u>HV</u>
1	36.5	11c	40.0	4A	54.1	7N	77.9	4NR	38.9	2A1	43.1	5A1c	41.3
2	41.3	12c	38.6	5A	54.1	18N	77.9	9NR	63.6	4A1	39.4	9A1c	28.8
3	37.5	13c	32.8	12A	55.4	19N	78.4	10NR	78.0	10A1	39.7	19A1c	27.0
4	37.3	14c	24.0	13A	60.0	20N	75.4	11NR	77.0	17A1	30.3	21A1c	29.9
6	35.2	15c	40.6	14A	58.8			13NR	61.3	20A1	39.3	25A1c	46.6
7	35.4	16c	18.2	25A	60.7							32A1c	49.7
8	36.2	17c	--										
9	38.2	18c	--										
10	40.4	22c	37.3										
11	34.2	31c	26.6										
12	36.8	72c	32.8										
13	39.7	79c	40.0										
14	43.1	87c	23.5										
17	42.5	90c	32.4										
18	41.4	95c	34.8										
65	42.8	113c	34.8										
75	41.3	114c	32.5										
78	42.2	130c	46.6										
103	41.0												
106	41.0												
107	46.0												

TABLE 23

## Hardness tests results

<u>HE9</u>	
<u>Extrude No.</u>	<u>Energy Absorbed (J)</u>
10	49.7
11	41.2
12	45.9
13	44.8
14	39.9
15	41.5
16	44.8
17	44.6
18	42.4
19	47.9
20	42.0
22	49.5
25	36.8
26	52.8
33	38.1
35	39.7
36	35.0
37	38.3
42	39.6
43	41.4
45	42.0
64	28.8
65	39.1
81	46.6
82	43.7

TABLE 24

HE9 fracture toughness  
results

<u>HE30</u>	
<u>Extrude No.</u>	<u>Energy Absorbed (J)</u>
1A	34.9
2A	42.0
3A	30.3
17A	38.8
18A	44.0
24A	42.8
25A	43.3
28A	38.1
29A	34.0
31A	38.0
38A	39.1
39A	38.8

TABLE 25

HE30 fracture toughness results

<u>Soaking Temperature</u> (°C)	500	520	560	580
<u>0.2% P.S.</u> (MN/m <sup>2</sup> )	124.9	140	144.8	142.4
<u>U.T.S.</u> (MN/m <sup>2</sup> )	165.7	177.7	178.6	179.5
<u>% Elongation</u>	19.2	18.8	13.7	16.1

(specimens soaked for 5 hours, water quenched, aged for 6 hours at 180°C)

TABLE 26  
Soaking temperature variation results (HE9)

<u>Ageing Temperature</u> (°C)	140	160	180	200	220
<u>0.2% P.S.</u> (MN/m <sup>2</sup> )	96.3	151.1	144.8	124.5	101.6
<u>U.T.S.</u> (MN/m <sup>2</sup> )	166.6	195.3	177.5	159.5	139.7
<u>% Elongation</u>	19.3	13.5	13.7	15.7	21.3

(specimens soaked for 4 hours at 560°C, water quenched, aged for 6 hours)

TABLE 27  
Ageing temperature variation results (HE9)

<u>Ageing Time (hours)</u>	1	2	4	6	20
<u>0.2% P.S.</u> (MN/m <sup>2</sup> )	87.7	96.6	111.3	144.8	153.1
<u>U.T.S.</u> (MN/m <sup>2</sup> )	147.1	146.6	155.5	178.5	185
<u>% Elongation</u>	20.8	19.0	17.9	13.7	10.9

(specimens soaked for 4 hours at 560°C, water quenched, aged at 180°C)

TABLE 28

Ageing time variation results (HE9)

<u>Ageing Temperature (°C)</u>	170	180	190	200	220	240
<u>0.2% P.S.</u> (MN/m <sup>2</sup> )	231	244.5	233.4	191.1	162	120
<u>U.T.S.</u> (MN/m <sup>2</sup> )	273.4	276.8	267.2	232	205.3	171.8
<u>% Elongation</u>	21.2	18.2	20.1	17.3	21.7	21.1

(specimens soaked for 5½ hours at 550°C, water quenched, aged for 6 hours)

TABLE 29

Ageing temperature variation results (HE30)

<u>Ageing Time</u> (hours)	1	2	7	16	24
<u>0.2% P.S.</u> (MN/m )	169.6	216.1	235.4	235.3	226.4
<u>U.T.S.</u> (MN/m )	236.6	257.5	264.2	267.1	258.6
<u>% Elongation</u>	22.2	21.4	18.9	17.1	16.6

(Specimens soaked for 5½ hours at 550°C, water quenched, aged at 180°C)

TABLE 30

Ageing time variation results (HE30)

<u>0.2% P.S.</u> (MN/m <sup>2</sup> )	88.0	78.5	76.1	76.3	76.2	72.5
<u>U.T.S.</u> (MN/m <sup>2</sup> )	143.9	125.9	126.4	126.6	126.3	124.8
<u>Ageing Time</u> (hours)	0	2	3	3.75	5	6

(Specimens extruded, air (press) quenched, aged at 180°C)

TABLE 31

Results of press quenching HE9 extrudes

<u>Condition</u>	<u>0.2% P.S.</u> (MN/m <sup>2</sup> )	<u>U.T.S.</u> (MN/m <sup>2</sup> )	<u>% Elongation</u>
TB	70	120	13
TE	110	150	7-8
TF	130-160	150-185	6-8

TABLE 32

Properties of heat treated cast and wrought alloys

<u>Extrude</u>	<u>Soak</u>	<u>Quench</u>	<u>Age</u>	<u>Cool</u>	<u>U.T.S.</u> (MN/m <sup>2</sup> )	<u>0.2% P.S.</u> (MN/m <sup>2</sup> )
HE9+10% glass	--	--	--	--	241	87
"	--	--	6 hr @ 180°C	air	239	91
"	--	--	6 hr @ 180°C	water	229	93
"	5½ hr @ 500°C	water	6 hr @ 180°C	air	236	88
"	5½ hr @ 500°C	air	6 hr @ 180°C	air	232	91

TABLE 33

Summary of results of heat treatments carried out on HE9 + 10 wt.% glass extrude

Material	Room Temperature Test	400°C Test
HE9	Cup + cone (Ductile) Transparticle	Cup + cone (Ductile) Transparticle
HE9 + 5wt.% glass	• •	• •
HE30	• •	• •
Al	Brittle Interparticle	————
Al + 5wt.% glass	• •	————
Al-3%Mg	45° shear (Ductile, some necking) Transparticle	Cup + cone (Ductile) Transparticle
Al-3%Mg + 5wt.% glass	• •	————

TABLE 34

Type of fracture experienced by various extruded materials

## CHAPTER 6

## CONCLUSIONS

1. Aluminium, Al-Mg and Al-Mg-Si alloy powders produced by water and air atomization techniques possess very fine structures which are dendritic in nature.
2. Al-Mg-Si powder compacts formed by cold pressing, obey Konopicky's relationship giving an equation :-

$$\ln \left( \frac{1}{1-D} \right) = 0.293 \times 10^{-2} P + 1.349$$

D = relative density

P = compaction pressure

The strength of such compacts increases rapidly with sintering temperature if the cold pressed compacts are sintered. The powder particle size also affects compact strength markedly, the strength decreasing with increasing powder particle size.

3. The load-ram displacement curves obtained during powder compact extrusion differ significantly in form from typical solid billet extrusion curves, particularly in the initial, upsetting phase.
4. Powder compacts obey a law of the form:-

$P/\text{constant}' = \text{constant}'' + \ln R$  during extrusion at one temperature.

P = extrusion pressure

R = extrusion ratio

The equation becomes (For Al-Mg-Si at 540°C):



$$\frac{P}{\text{constant}} = 0.87 + \ln R$$

Additions of 5wt.% glass have a negligible effect on this behaviour. The value of the constant on the right hand side indicates the amount of redundant work involved in extrusion. This value is much higher for powder compact extrusion than for solid billet extrusion. This redundant work is probably the energy source involved in "building" the extrude as the powder traverses the deformation zone.

5. Powder compact extrusion load-ram displacement *curves* exhibit initial peaks, the magnitude of the peak increasing with strain rate and with decreasing temperature. This indicates that the phenomenon is connected with a dynamic process involving dislocation multiplication and annihilation.
6. As the redundant work term and the extrusion temperature obey an Arrhenius type of relation it is fairly clear that powder consolidation during extrusion is a thermally activated process.
7. During extrusion of Aluminium, Al-Mg-Si and Al-Mg powder compacts the peak extrusion pressure is linearly related to the extrusion temperature.
8. A minimized plane strain upper bound solution may be used to predict powder compact extrusion pressures if a knowledge of the strain and strain rate dependence of the flow stress exists. Torsion and tensile tests predict a relation of the form:-

$$K_{ij} = \frac{40}{\sqrt{3}} \frac{\dot{\epsilon}}{\epsilon}^{0.1} \frac{1}{\epsilon}^{0.21}$$

For HE9 at 400°C.

The predicted loads are, however, very inaccurate and the method is unacceptable. Attempts to modify the solution for axi-symmetric extrusion produce answers which are commonly 50% less than the experimentally measured values.

9. The use of a minimized axi-symmetric upper bound solution enables much more accurate predictions of powder compact loads to be made. Typically, for HE9 and HE30 the predicted values are never greater than 10% in error over the extrusion ratio range 5:1 to 180:1.
10. This latter theory also predicts that the strain required for complete consolidation ( $\sqrt{2.3}$ ) is not reached until a late stage in the deformation zone. eg. 70 % of the deformation zone experiences a strain of less than 2.3 even at an extrusion ratio of 80:1. This agrees with the long held belief that, during powder compact extrusion, complete consolidation and therefore final extrude properties are not attained until the compact has almost traversed the deformation zone.
11. Extrusion strain rates predicted by this theory agree reasonably well with those predicted by Feltham's equation at high extrusion ratios.
12. Graphs of maximum torque V. ln. twist rate produced from torsion data for both HE9 and HE30 are linear. Both materials also obey equations of the form:-

$$\text{Flow stress} = \text{const. } \dot{\epsilon}^n$$

The strain rate exponent,  $n$ , increases with temperature.

13. The torsion test data yields activation energies of 167,224 J/mole for HE9 and 170,881 J/mole for HE30. These values are somewhat higher than the value of 156,000 J/mole usually quoted for aluminium. This discrepancy is probably due to the oxide content of the powder extrudes.
14. The Zener-Holloman constants  $\alpha$  and  $n$  have values of 0.038 and 5.16 respectively for HE9 and 0.04 and 3.87 for HE30. These values agree quite well with those obtained by other workers for solid aluminium extrusion.
15. Generally, the surface finish of the extrudes is very dependent upon the extrusion temperature and strain rate. For HE9, HE30 and aluminium powder compact extrudes, the surface finish improves with increasing ram speed and temperature. However, the surface finish of Al-Mg extrudes improves with decreasing ram speed. Additions of up to 5 wt.% glass have little effect on the surface finish. Additions of glass to aluminium and Al-Mg extrudes have a deleterious effect on the strength and elongation properties.
16. Generally the strength and elongation properties of as-extruded HE9, HE30 and Al-Mg powders are very much superior to those of their "solid" counterparts (Often by as much as 50%). The strength of the extruded aluminium powder is also much greater than that of commercially extruded solid aluminium although the elongation value is considerably less due to the high oxide content.
17. The Al-Mg powder extrudes exhibit serrated yielding during room temperature tensile tests.

18. Additions of glass to aluminium and Al-Mg powder extrudes have a deleterious effect on the strength and elongation properties.
19. Additions of glass to HE9 powder extrudes improve the as-extruded U.T.S., value by 40% (for 8 wt.% glass). Generally, the proof stress and elongation values are not greatly affected.
20. The elastic modulus of HE9 is reduced by approximately 10-12% by the addition of 5 wt.% glass.
21. The toughness of reinforced HE9 decreases rapidly with increasing glass content.
22. The mechanical properties of the powder extrudes are very dependent upon the extrusion temperature. Generally the strength of the unreinforced extrudes decreases with increasing temperature (except for aluminium powder) and the elongation improves. Reinforced HE9 exhibits an increase in strength and elongation with increasing temperature.
23. The decrease in strength, with increasing test temperature, shown by HE9 powder extrude is in agreement with the predictions made by Seeger's analysis.
24. Tensile tests performed at different temperatures show that the strain rate sensitivity of HE9 and HE30 powder extrudes increases with temperature. (At room temperature both sensitivities are slightly negative).
25. The tensile strength of powder extrudes decreases rapidly with increasing initial powder particle size.

26. The strength of HE9 and Al-Mg powder extrudes increases with glass content to a maximum and then falls off rapidly. The deterioration is caused by matrix cracking due to elastic expansion of the glass. The optimum content for HE9 is 8 wt.% for Al-Mg, approximately 2 wt.%.
27. The strength and elongation properties of reinforced HE9 decrease rapidly with increasing glass particle size.
28. Generally the hardness of reinforced powder extrudes decreases with increasing glass content.
29. Both HE9 and HE30 powder extrudes respond well to a TF type, three stage heat treatment. Reinforced HE9 extrudes are not responsive to such treatment. Press quenching is also completely ineffective.
30. HE9, HE30, Al and Al-Mg powder extrudes reveal fibrous, cold worked microstructures under the optical microscope. The structure is elongated in the extrusion direction and "equi-axed" in the transverse direction.
31. Optical microscopy reveals the imperfection of the fibres. A variation in fibre morphology across the extrude section manifests the variation in strain encountered.
32. Electron microscopy of HE9, HE30 and Al-Mg powder extrudes reveals a total lack of recrystallisation under any of the extrusion conditions investigated. This is due to the presence of the oxide particles. In all of the extrudes subgrains elongated in the extrusion direction and produced by dynamic recovery,

are present. Increasing the extrusion temperature produces subgrains which are more "equi-axed" in nature. The subgrain size also increases markedly with extrusion temperature.

33. Unreinforced HE9 and HE30 extrudes exhibit cup and cone ductile fracture during tensile tests. Unreinforced aluminium extrudes exhibit brittle interparticle fracture. Additions of up to 5 wt.% glass cause embrittlement.
  
34. During extrusion the reinforcing glass chemically reacts with the metal matrix. A spinel,  $Mg Al_2 O_4$ , is produced at the interface (in the case of HE9 and HE30). This reaction of the matrix magnesium in the formation of this spinel is one of the main reasons for the lack of response shown by reinforced extrudes to heat treatments.

```

00100 PROGRAM UPPERE(INPUT,OUTPUT)
00200 1 PRINT 4
00300 4 FORMAT(*READ IN RADIUS D,FR,OM,PHI*)
00400 READ*.D,FR,OM,PHI
00500 A=C-(D/FR)
00600 R=D-A
00700 N=1
00800C CONVERT OMEGA AND PHI TO RADIAN
00900C
01000 R1OM=OM/57.29578
01100 R1PHI=PHI/57.29578
01200C
01300C TO CALCULATE THE DEPTH OF WORKING
01400 100 A1=(A**2)/((1.+(A/R))*(SIN(R1OM)**2))
01500 B1=(A**2)/((1.+(A/R))*(SIN(R1PHI)**2))
01600 X=SQRT(A1+B1)
01700 N=N+1
01800C
01900C TO CALCULATE AND WRITE OUT NEW ANGLES
02000 FOM=(4.*A)/(3.*X)
02100 FPHI=(4.*R)/(3.*X)
02200 R2OM=ATAN(FOM)
02300 R2PHI=ATAN(FPHI)
02400 DIF1=SQRT((57.29578*(R1OM-R2OM))**2)
02500 DIF2=SQRT((57.29578*(R1PHI-R2PHI))**2)
02600 N=N+1
02700 IF (DIF1.LT.0.01.AND.DIF2.LT.0.01)GO TO 9
02800 IF (N.EQ.50)GO TO 101
02900 R1OM=R2OM
03000 R1PHI=R2PHI
03100 GO TO 100
03200C CALCULATE LOAD USING VALUES OF X,OM,PHI
03300 C1=(2.*(A**2))/(X*(SIN(R2OM))**2)
03400 C2=(2.*A**2)/(X*(SIN(R2PHI))**2)
03500 C3=(3.*A)/TAN(R2OM)
03600 C4=(3.*A)/TAN(R2PHI)
03700 PRINT 15,C1,C2,C3,C4
03800 15 FORMAT(3HC1=,F10.4,2X,3HC2=,F10.4,2X,3HC3=,F10.4,2X,3HC4=,F10.4)
03900 ALOAD=(2.*X)+(2.*X**2)/8+C1+C2-C3-C4
04000 PRESS=ALOAD/D
04100 OM=R2OM*57.29578
04200 PHI=R2PHI*57.29578
04300 PRINT 10,A,B,D,FR,X,OM,PHI,ALOAD,PRESS
04400 10 FORMAT(2HA=,F10.4,2X,2HE=,F10.4,2X,2HD=,F10.4//
04500+16HEXTRUSION RATIO=,F10.4/2HX=,F10.4,2X,3HOM=,F10.4,
04600+2X,4HPHI=,F10.4//10HALOAD/2K=,F10.4//13HPRESSURE/K=,F10.4)
04700C
04800C
04900 PRINT*,'ANY MORE INPUT'
05000 READ 106,NSWER
05100 IF (NSWER.EQ.1)GO TO 1
05200 106 FORMAT(A1)
05300 STOP 1
05400 101 PRINT 102
05500 102 FORMAT(*N=50*)
05600 105 STOP
05700 END

```

## APPENDIX 2

```

00100 PROGRAM UPBOUND(INPUT,OUTPUT,TAPE 5=INPUT,TAPE 6=OUTPUT)
00200 COMMON/1/ FB,ALP,BET,PI,FM,SUM,RE,RE,V1,V3
00300 COMMON/2/ X(2),ED12,ED23,ED24,ED2,AA,EB,FPSEB,THET
00400C *****VERSION 1*****
00500C RB=BILLET RADIUS
00600C RE=EXTRUDE RADIUS
00700C ER=EXTRUSION RATIO
00800C ALP=ANGLE ALPHA=ANGLE BETWEEN BILLET WALL AND DEAD METAL ZONE
00900C BET=ANGLE BETA=ANGLE BETWEEN V23 AND VERTICAL
01000C FM=ANGLE M=ANGLE BETWEEN V12 AND VERTICAL
01100C V1=RAM VELOCITY
01200C V3=EXTRUDE VELOCITY
01300C ED23=ENERGY DISSIPATED ALONG E23/K
01400C ED24=ENERGY DISSIPATED ALONG E24/K
01500C ED2=ENERGY DISSIPATED WITHIN TRIANGLE 2/S,PT3 K
01600C THIS PROGRAM CALCULATES VALUES OF P/K USING A SINGLE TRIANGLE UPPER
01700C BOUND SOLUTION, INVOLVING INTEGRATION BY SIMPSONS RULE
01800C IT CALLS UPON THE LIBRARY ROUTINE FC4CBF TO MINIMISE THE VALUE OF
01900C P/K BY VARYING THE ANGLES ALPHA AND BETA. IT ASSUMES THAT THE SHEAR
02000C STRESS, K, TAKES A CONSTANT VALUE THROUGHOUT THE DEFORMATION ZONE
02100 PRINT 199
02200 199 FORMAT(*XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX*,/
02300+26X,*X ALPHA *X,./
02400+27X,*X X,./
02500+28X,*X X,./
02600+29X,*X X,./
02700+30X,*X X,./
02800+10X,*BILLET*,15X,*X X,./
02900+32X,*X X,./
03000+33X,*X X,./
03100+34X,*X X,./
03200+35X,*XXXXXXXXXXXXX*,/
03300+34X,*X,./
03400+33X,*X---BETA*,/
03500+32X,*X,./
03600+31X,*X EXTRUDE*,/
03700+30X,*X,./
03800+29X,*X,./
03900+-----*,////
04000+9X,*POSITIONS OF ANGLES ALPHA AND BETA*)
04100C
04200C
04300 CALL TYPTN
04400 CALL UNITS
04500 X(1)=ALP
04600 Y(2)=BET
04700 PI=3.14159
04800 V3=V1*FB*RE/(RE*RE)
04900C
05000 CALL MINIMIZ
05100 STOP
05200 END
05300C
05400 SUBROUTINE TYPTN
05500 COMMON/1/ FB,ALP,BET,PI,FM,SUM,RE,RE,V1,V3
05600 303 PRINT 301
05700 301 FORMAT(*PROGRAM REQUIRES EXTRUSION RATIO OR EXTRUDE RADIUS. *,/
05800+*IF ENTERING EXTRUSION RATIO TYPE 'A', IF ENTERING EXTRUDE RADIUS *,/
05900+*TYPE 'R'.*)
06000 READ 302,NSWER
06100 302 FORMAT(A1)
06200 IF(NSWER.EQ.1HA)GO TO 304
06300 IF(NSWER.EQ.1HR)GO TO 306
06400 PRINT*, ' IDIOT, CANT YOU READ? '
06500 GO TO 303
06600 304 PRINT 305
06700 305 FORMAT(*ENTER BILLET RADIUS IN MMS, EXTRUSION RATIO, AN ESTIMATE*
06800+*OF ANGLES ALPHA AND BETA IN DEGREES AND RAM VELOCITY IN MM/S-C *)
06900 READ(5,*)RB,RE,ALP,BET,V1
07000 RE=RE/SQRT(ER)
07100 GO TO 308
07200 306 PRINT 307
07300 307 FORMAT(*ENTER BILLET RADIUS, EXTRUDE RADIUS IN MMS, AN ESTIMATE
07400+*OF ANGLES ALPHA AND BETA IN DEGREES AND RAM VELOCITY IN MM/SEC.*)
07500 READ(5,*)RB,RE,ALP,BET,V1
07600 ER=RE*RE/(RE*RE)
07700 308 RETURN
07800 END
07900C
08000C
08100 SUBROUTINE UOB(N,X,ALOAD)
08200 COMMON/1/ FB,ALP,BET,PI,FM,SUM,RE,RE,V1,V3
08300C THE FOLLOWING RELATES ALPHA AND BETA TO M
08400C
08500C DIMENSION X(2)
08600 ALP = X(1)

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08700 BET = X(2)
08800 CALL GEOM(ALP,PI)
08900 CALL GEOM(BET,PI)
09000 TEM=( (RB-RE)/TAN(ALP)-RE*TAN(BET) )/RB
09100 EM=ATAN(TEM)
092000
093000 PRINT 10,ALP,BET
094000 C10 FORMAT(*UPB.,ALP,BET= *,2E12.5)
09500 CALL SUMEM
096000
09700 ALOAD=SUM/(PI*RB*RE*V1)
098000
099000 ALOAD=P/K
100000
101000
10200 RETURN
10300 END
104000
10500 SUBROUTINE GEOM(ANGLE,PI)
106000 THIS ROUTINE LIMITS THE VALUES OF ALPHA AND BETA TO LESS THAN 90
107000 AND GREATER THAN 0 DEGREES.
108000 IF (ANGLE)30,20,10
109000 10 IF (ANGLE.LT.PI/2.) RETURN
110000 ANGLE=ANGLE-PI/2
111000 GO TO 10
112000 20 PRINT*, 'THERE IS AN ERROR ALPHA OR BETA = 0 DEGREES.'
113000 30 IF (ANGLE.GT.0.0) RETURN
114000 ANGLE=ANGLE+PI/2.
115000 GO TO 30
116000 END
117000 SUBROUTINE UNITS
118000 COMMON/1/ ER,ALP,BET,PI,FM,SUM,RE,VE,V1,V3
119000 COMMON/2/ X(2),ED12,ED23,ED24,ED2,AA,BE,EP,PSBR,THET
120000
121000 THIS ROUTINE PUTS RE AND VE IN METRES. ANGLES IN RADIANS AND V1
122000 IN METRES PER SECOND
123000
124000 RE=RE/1000.
125000 VE=VE/1000.
126000 ALP=ALP/57.2958
127000 BET=BET/57.2958
128000 V1=V1/1000.
129000 RETURN
130000 END
131000
132000 SUBROUTINE SUMEM
133000 COMMON/1/ ER,ALP,BET,PI,FM,SUM,RE,VE,V1,V3
134000 COMMON/2/ X(2),ED12,ED23,ED24,ED2,AA,BE,EP,PSBR,THET
135000
136000 THIS ROUTINE CALLS TWO OTHER ROUTINES THAT PRODUCE THE VALUES OF
137000 ENERGY DISSIPATION AT VELOCITY DISCONTINUITIES AND WITHIN THE SINGLE
138000 TRIANGLE, AND SUMS THEM.
139000
140000 CALL E1E2E3
141000
142000 CALL F2CALC
143000
144000 SUM = ED12+ED23+ED24+ED2
145000 RETURN
146000 END
147000
148000
149000 SUBROUTINE F1E2E3
150000 COMMON/1/ ER,ALP,BET,PI,FM,SUM,RE,VE,V1,V3
151000 COMMON/2/ X(2),ED12,ED23,ED24,ED2,AA,BE,EP,PSBR,THET
152000
153000 THIS ROUTINE PRODUCES THE THREE VALUES OF ENERGY
154000 DISSIPATION AT VELOCITY DISCONTINUITIES V12,V24,V23
155000
156000 RB2=RB*RB
157000 A=V1*SIN(ALP)/COS(ALP+EM)
158000 B=PI*RB2/COS(EM)
159000 ED12=A*B
160000
161000 RE2=RE*RE
162000 C=V3*SIN(ALP)/COS(ALP-BET)
163000 D=PI*RE2/COS(BET)
164000 ED23=C*D
165000
166000 E=2.*PI*RB2*V1/COS(ALP)
167000 F=TAN(PI/4.+BET/2.-ALP/2.)
168000 G=TAN(PI/4.-BET/2.-ALP/2.)
1690000 PRINT 101,E,F,G
170000 C101 FORMAT(*E,F,G= *,3E12.5)
171000 ED24=F*ALOG(F/G)
172000 RETURN

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```

17300 END
17400 SUBROUTINE F2CALC
17500C
17600 COMMON/1/ FF,ALP,BET,PI,EM,SUM,PS,RE,V1,V3
17700 COMMON/2/ Y(2),F012,F023,F024,F02,AA,BE,PSBP,THET
17800C THIS ROUTINE CALCULATES THE ENERGY DISSIPATION WITHIN TRIANGLE 2
17900C AND CALLS UPON A SIMPSONS RULE ROUTINE
18000C
18100C DIMENSION F(200)
18200C
18300C NSTEP = 100
18400C THETMIN=PI/2.0-EM
18500C THETMAX=PI/2.0+BET
18600C THETSTP=(THETMAX-THETMIN)/100.
18700C THET=(THETMIN-THETSTP)
18800C
18900C DO 200 I=1,NSTEP+1
19000C THET=THET+THETSTP
19100C
19200C CALL F0THET
19300C
19400C F(I)=(PSBP*SIN(THET))/(3B*BB)
19500C 200 F(I)=F(I)*B*BB/AA
19600C
19700C CALL SIMPV(F,NSTEP,THETSTP,SINT)
19800C
19900C F02=PI*SQRT(2.)*SINT
20000C RETURN
20100C END
20200C
20300C
20400C SUBROUTINE F0THET
20500C COMMON/1/ FF,ALP,BET,PI,EM,SUM,PS,RE,V1,V3
20600C COMMON/2/ X(2),F012,F023,F024,F02,AA,BE,PSBP,THET
20700C
20800C THIS ROUTINE CALCULATES STRAIN RATES IN POLAR COORDINATES
20900C
21000C AA=COS(EM)*COS(EM)/(COS(EM+ALP)*COS(EM+ALP))
21100C BB=SIN(THET-ALP)
21200C CC=COS(THET-ALP)
21300C DD=V1*AA*BB/SIN(THET)
21400C EE=1.+(COS(THET)+COS(THET))/(SIN(THET)*SIN(THET))
21500C FF=EE*V1*AA*SIN(ALP)*DD
21600C FF=DD*2.*CC
21700C
21800C EPSL1=FF+EE
21900C
22000C GG=BB/TAN(THET)
22100C
22200C EPSL2=DD*(CC+GG)
22300C
22400C HH=-DD*BB*2.
22500C II=V1*AA*SIN(ALP)
22600C QQ=CC*PS*DE
22700C
22800C FPSL3=HH+QQ
22900C
23000C FPSL1S=FPSL1*FPSL1
23100C FPSL2S=FPSL2*FPSL2
23200C FPSL3S=FPSL3*FPSL3
23300C
23400C FPSBRI=SQRT((FPSL1S+FPSL2S+FPSL3S/2.)*2./3.)
23500C
23600C FPSBP=FPSBRI/SQRT(2./3.)
23700C RETURN
23800C END
23900C
24000C SUBROUTINE SIMPV(F,NSTEP,H,SINT)
24100C
24200C THIS ROUTINE PERFORMS AN INTEGRATION BY SIMPSONS RULE
24300C THE FIRST PART CHECKS THAT NSTEP IS AN EVEN NUMBER
24400C
24500C DIMENSION F(200),X(2)
24600C IF (MOD(NSTEP,2).EQ.0) GO TO 1
24700C WRITE (6,61)NSTEP
24800C 61 FORMAT ('EXECUTION TERMINATED IN SIMPV BY ODD NSTEP = ',I6)
24900C STOP
25000C 1 SINT=0
25100C DO 2 J=2,NSTEP,2
25200C 2 SINT=SINT+F(J-1)+4.*F(J)+F(J+1)
25300C SINT=SINT*H/3.
25400C RETURN
25500C END
25600C SUBROUTINE MINIMIZ
25700C COMMON/1/ FF,ALP,BET,PI,EM,SUM,PS,RE,V1,V3
25800C COMMON/2/ X(2),F012,F023,F024,F02,AA,BE,PSBP,THET

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25900 EXTERNAL UPR,WRITOUT
26000 DIMENSION EPS(2),GRAD(2),DFIRST(2),W(17)
26100 N=2
26200 IW=7*N+N*(N+1)/2
26300 ITERAT=200
26400 DO 1 I=1,N
26500 EPS(I)=1.0E-4
26600 1 DFIRST(I)=0.001
26700 ACC=1.0E-10
26800 ESTIM=7.0
26900 EF=1.0E-9
27000 IERR=0
27100
27200 CALL F040BF(N,X,ALOAD,GRAD,EPS,ESTIM,ACC,W,IW,DFIRST,EF,UPR,WRITOUT
27300 +,ITERAT,IERR)
27400 IF (IERR.NE.0) GO TO 10
27500 ALP=ALP*57.2958
27600 BET=BET*57.2958
27700 RB=RB*1000.
27800 PRINT* 'XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX'
27900 PRINT 16,EP,RE
28000 16 FORMAT(/////,7X,*EXTRUSION RATIO = *,F8.4,3X,*BILLET RADIUS = *,F8.4)
28100 WRITE(6,15)ALP,BET,ALOAD
28200 150 FORMAT(* MINIMUM IS AT: ALPHA = *,F10.6,* DEGREES */
28300 +17X,* BETA = *,F10.6,* DEGREES */18X,* P/K = *,F10.6,* METRES SQUARED*)
28400 PRINT*, ' THIS IS A SINGLE TRIANGLE SOLUTION. '
28500 PRINT 17
28600 17 FORMAT(/////,10X,*XXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXXX*,15X)
28700 STOP
28800 10 WRITE(6,120)IERR,X,ALOAD
28900 120 FORMAT(* FAIL*,12/* AT */2F12.8/* P/K = *,E16.8)
29000 STOP
29100 END
29200
29300 SUBROUTINE WRITOUT (N,X,ALOAD,GRAD,ITERAT)
29400 COMMON/1/ ALP,BET,PI,EM,SUM,FB,RE,V1,V3
29500 DIMENSION X(2),GRAD(2)
29600 WRITE(6,1)ITERAT
29700 1 FORMAT(* ITERATION = *,I4)
29800 RETURN
29900 END

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08700C
08800C
08900C PRINT*, 'ENTER THE FREQUENCY WITH WHICH THE MONITORING SUBROUTINE IS TO
09000C BE CALLED. ZERO WILL GIVE NO CALLS WHILE 1 WILL CALL IT EVERY CYCLE.
09100C IF SHOULD BE NOTED THAT THIS PROGRAM EMPLOYS AN ITERATIVE METHOD FOR
09200C SOLUTION AND MAY THEREFORE TAKE SOME TIME BEFORE GIVING AN ANSWER.'
09300C READ(5,*) IPRIN
09400C
09500C
09600C IT=0
09700C
09800C
09900C IXIYP(1)=90
10000C IXIYP(2)=90
10100C IGTYP(1)=2
10200C IGTYP(2)=2
10300C IGTYP(3)=2
10400C IGTYP(M)=1
10500C
10600C LINE(1)=.TRUE.
10700C LINE(2)=.TRUE.
10800C LINE(3)=.TRUE.
10900C LINE(M)=.FALSE.
11000C
11100C M2N=M+2*N
11200C IW=5*N+4*M+M*N+2*N*N
11300C
11400C
11500C SUBROUTINE ECUHAF IS THE LIBRARY MINIMISING ROUTINE
11600C
11700C CALL ECUHAF(N,M,X,EST,MAX,AXX,IST,INF,MET,LOGW,M2N,W,IW,IXIYP,IGTYP
11800C ,LINE,GR10,DGR10,DDGR10,MUN,MUNE,IPRIN,MAXJ,IFAIL)
11900C
12000C V1=V1*1000.
12100C
12200C WRITE(6,951)
12300C WRITE(6,951)IFAIL
12400C WRITE(6,952)EST, (X(1),I=1,N)
12500C WRITE(6,953)C,V1,C.CONSTAN,C0
12600C WRITE(6,951) IPRIN,MAXJ
12700C
12800C STOP
12900C
13000C 951 FORMAT(75(1HX),///)
13100C 901 FORMAT(/,17X,I3,* ITERATIONS *,I5,* PENALTY EVALUATIONS*,//75(1HX))
13200C 902 FORMAT(20X,*PRESSURE IS *,E12.5,4X,*AT */17X,*ALPHA = *,F10.6,7X
13300C ,*PHI = *,F10.6)
13400C 961 FORMAT(32X,*IFAIL =*,I2,///)
13500C 966 FORMAT(4X,*EXTRUSION RATIO IS *,F6.2,* : 1 AT A RAY SPEED OF *,
13600C ,F10.4,*INS/SEC.*/,1X,*2 = *,F6.4,9X,*CONSTANT = *,F6.4,4X,
13700C ,*STRAIN HARDENING EXPONENT = *,F5.4)
13800C
13900C
14000C
14100C END
14200C
14300C
14400C SUBROUTINE MONE(N,M,URMIN,XMIN,GMIN,UMIN,IORD)
14500C
14600C
14700C THIS ROUTINE GIVES A MONITOR OF THE DUAL AND EXTRAPOLATED SOLUTIONS
14800C AT THE END OF EACH CYCLE.
14900C
15000C DIMENSION URMIN(5),XMIN(2),GMIN(5),UMIN(5)
15100C
15200C COMMON/HAF/ MET,IPRIN,IT
15300C
15400C WRITE(6,1007)
15500C WRITE(6,1005) (I,URMIN(I),I=1,M)
15600C WRITE(6,1006) IORD
15700C WRITE(6,1001)
15800C WRITE(6,1002) (I,XMIN(I),I=1,N)
15900C WRITE(6,1003)
16000C WRITE(6,1004) (I,GMIN(I),I=1,M)
16100C WRITE(6,1005)
16200C WRITE(6,1006) (I,UMIN(I),I=1,M)
16300C
16400C
16500C 1000 FORMAT(/,*EXTRAPOLATION SOLUTION ORDER= *,I5)
16600C 1001 FORMAT(/,*SOLUTION VECTOR*)
16700C 1002 FORMAT(10(4(1X,14Y,I3,=12.5)/))
16800C 1003 FORMAT(*CONSTRAINTS AND OBJECTIVE FNS*)
16900C 1004 FORMAT(10(4(2X,3HCON,13,E12.5)/))
17000C 1005 FORMAT(*DUAL SOLUTION OF EXTRAPOLATED SOLUTIONS*)
17100C 1006 FORMAT(10(4(1X,=HQUAL,13,=12.5)/))
17200C 1007 FORMAT(//*DUAL SOLUTION AT END POINT OF CYCLE*)

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173000
174000
175000 RETURN
176000 END
177000
178000
179000 SUBROUTINE MON(N,M,XR,PRXR,PG,PUPONK,R,ITCH,NEWCYC,NP,IG,NH,RRF)
180000
181000
182000 THIS ROUTINE MONITORS THE CHANGE OF ANGLES ALPHA AND PHI AND THE
183000 RESULTING CHANGE IN THE PRESSURE.
184000
185000 LOGICAL NEWCYC
186000 COMMON/HAF/ MET,IPRIN,IT
187000 DIMENSION XR(2),PG(2),PUPONK(5)
188000
189000 IT=IT+IPRIN
190000 IF(.NOT.NEWCYC) GO TO 101
191000 IT=C
192000
193000 WRITE(6,1000) R
194000
195000 101 WRITE(6,1001) IT,PRXR
196000
197000
198000 IF(ITCH.LE.0) WRITE(6,1006)
199000 IF(ITCH.EQ.3.OR.ITCH.EQ.-3) WRITE(6,1007)
200000 IF(ITCH.EQ.2.OR.ITCH.EQ.-2) WRITE(6,1008)
201000
202000
203000 WRITE(6,1009)
204000 WRITE(6,1010) (J,XR(J),J=1,N)
205000
206000 IF(MET.LT.1) GO TO 106
207000
208000 WRITE(6,1002)
209000 WRITE(6,1003) (I,PG(I),I=1,N)
210000
211000 106 WRITE(6,1011)
212000 WRITE(6,1012) (I,PUPONK(I),I=1,M)
213000
214000
215000 1000 FORMAT(/*BEGINING OF CYCLE FOR R= *,E15.6)
216000 1001 FORMAT(/*ITERATION NO *,I3,* PENALTY FUNCTION VALUE *,E15.6)
217000 1002 FORMAT(*PENALTY GRADIENT*)
218000 1003 FORMAT(10(4(3X,1HG,I3,E12.5)/))
219000 1006 FORMAT(/*LINE SEARCH TROUBLES*)
220000 1007 FORMAT(* H NOT FAC*)
221000 1008 FORMAT(* H INDEF*)
222000 1009 FORMAT(/*SOLUTION VECTOR*)
223000 1010 FORMAT(10(4(3X,1HX,I3,E12.5)/))
224000 1011 FORMAT(*CONSTRAINTS AND OBJECTIVE FNS*)
225000 1012 FORMAT(10(4(2X,3HCON,I3,E12.5)/))
226000
227000
228000 RETURN
229000 END
230000
231000
232000
233000 SUBROUTINE GRID(N,X,I,PUPONK)
234000
235000
236000 THIS IS THE MAIN CALCULATION ROUTINE CALLED BY THE MINIMISATION
237000 ROUTINE AND ITSELF CALLING THE REMAINING ROUTINES TO CALCILATE THE
238000 PRESSURE.
239000
240000
241000 COMMON/1/ R1,Y,Z,DELTA,ALAMDAT,ALAMDAC,RR,RRC
242000 COMMON/2/ RE,RE,V1,V3,PI
243000 COMMON/3/ SINALPHA,SNBETA,SNBETA,CSGPA,CSALPHA,CSBETA,SNDB2,CSTB2,
244000 +Q,CG,CONSTAN,SNBND,CSAMP,SNP,CSP,SNTHETA,CSTHETA,CSGAN
245000 COMMON/ANGLE/ ALPHA,GAMMA,PHI,THETA,UTHEA,BETA
246000 COMMON/ENER/ E2,E24,E25,E12,E12T,E25T,E2T,E2T
247000 COMMON/STN/ TEMP4,STRAIN2(5),STRN(10,5),ST(10),STRAIN(5)
248000
249000
250000 DIMENSION X(2)
251000 ALPHA=X(1)/57.2958
252000 PHI=X(2)/57.2958
253000
254000
255000 IF(I.EQ.1) GO TO 889
256000 IF(I.EQ.2) GO TO 999
257000
258000 TGAMMA=ABS((((RB-RE)/TAN(ALPHA))-(RE*TAN(PHI))))/RB)

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25900 GAMMA=ATAN(TGAMMA)
26000 IF (I.EQ.3) GO TO 699
26100 A1=RE*TAN(PHI)
26200 A2=(RS-RE)/TAN(ALPHA)
26300 C
26400 E24T=0
26500 E12T=0
26600 E23T=0
26700 E2T=C
26800 C
26900 C
27000 JJ=10
27100 K=5
27200 C
27300 IF (A1.GT.A2) GO TO 799
27400 DTHETA=(GAMMA+PHI)/10.
27500 THETA=((PI/2.)-GAMMA)-(DTHETA/2.)
27600 GO TO 599
27700 799 DTHETA=(PHI-GAMMA)/10.
27800 THETA=((PI/2.)+GAMMA)-(DTHETA/2.)
27900 599 Z=1
28000 C
28100 C THE FOLLOWING DO-LOOPS DO THE CALCULATIONS DOWN EACH SET OF ELEMENTS
28200 C AND AROUND THE ELEMENTS RADially.
28300 C
28400 DO 100 L=1, JJ, 1
28500 C
28600 THETA=THETA+DTHETA
28700 Y=1
28800 C
28900 DO 200 MM=1, K, 1
29000 C
29100 BETA=THETA-ALPHA
29200 SNALPHA=SIN(ALPHA)
29300 SNBETA=SIN(THETA-ALPHA)
29400 SNGAM=SIN(GAMMA)
29500 CSGAM=COS(GAMMA)
29600 C
29700 C
29800 IF (A1.GT.A2) GO TO 4001
29900 C
30000 C
30100 CSGPA=COS(GAMMA+ALPHA)
30200 C
30300 C
30400 GO TO 4002
30500 4001 CSGPA=COS(ALPHA-GAMMA)
30600 4002 CSALPHA=COS(ALPHA)
30700 C
30800 C
30900 CSBETA=COS(THETA-ALPHA)
31000 SMDT2=SIN(DTHETA/2.)
31100 CSDT2=COS(DTHETA/2.)
31200 SNBMD=SIN(BETA-(DTHETA/2.))
31300 CSAMP=COS(ALPHA-PHI)
31400 SNF=SIN(PHI)
31500 CSP=COS(PHI)
31600 SNTHTA=SIN(THETA)
31700 CSTHTA=COS(THETA)
31800 C
31900 C
32000 R1=RS/CSGAM
32100 C
32200 C
32300 C
32400 CALL CENTRE C(NM, L)
32500 IF (L.EQ.1) CALL E12CALC(MM, L)
32600 IF (MM.EQ.1) CALL E24CALC(MM, L)
32700 IF (MM.EQ.5) CALL TRIANGL(MM, L)
32800 IF (MM.EQ.5) GO TO 300
32900 CALL ENERGYC(MM, L)
33000 300 IF (L.EQ.10) CALL E23 CALC(MM, L)
33100 C
33200 C
33300 C
33400 C
33500 E2T=E2T+E2
33600 C
33700 C
33800 Y=Y+2.
33900 C
34000 C
34100 200 CONTINUE
34200 C
34300 Z=Z+2.
34400 C

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34500 100 CONTINUE
34600
34700
34800
34900
35000
35100 THE SEPERATE ENERGIES ARE SUMMED
35200
35300 P=E12T+E23T+E24T+E2T
35400
35500
35600 THE TOTAL ENERGY IS THEN EQUATED TO THE PRESSURE*AREA*RAM VELOCITY.
35700
35800 PUPCNK=P/(PI*RR**RB*V1)
35900
36000
36100
36200 THE FOLLOWING ARE THE CONSTRAINTS USED IN THE MINIMISATION. THEY ALL
36300 PASS THROUGH AS THE REQUIRED RESULT. I.E. IN PUPCNK. THE CONSTRAINTS
36400 EMPLOYED ARE THAT: NEITHER ALPHA OR BETA CAN EXCEED 90 DEGREES.
36500 THAT ANGLE ALPHA MUST ALWAYS BE GREATER THAN ANGLE GAMMA.
36600
36700
36800 GO TO 1099
36900 889 PUPCNK=(PI/2.)*ALPHA
37000 GO TO 1099
37100 999 PUPCNK=(PI/2.)*PHI
37200 GO TO 1099
37300 699 PUPCNK=ALPHA-GAMMA
37400 1099 RETURN
37500
37600
37700 END
37800
37900
38000 SUBROUTINE CENTREC(MM,L)
38100
38200
38300 ROUTINE TO CALCULATE THE BASIC GEOMETRICAL TERMS REQUIRED BY EACH
38400 ELEMENT. RR IS THE RADIUS FROM THE CENTRE TO THE EDGE OF THE ELEMENT
38500 RPC IS THE DISTANCE FROM THE CENTRE TO THE CENTRE OF GRAVITY.
38600 DELTAR IS THE ELEMENT HEIGHT. ALAMDAT IS THE TOP EDGE DIAMETER.
38700 ALAMDAC IS THE CENTRE OF GRAVITY DIAMETER.
38800
38900
39000 COMMON/1/ R1,Y,Z,DELTAR,ALAMDAT,ALAMDAC,RR,RPC
39100 COMMON/2/ RB,RE,V1,V3,PI
39200 COMMON/3/ SNALPHA,SNBETA,SNGAM,CSGPA,CSALPHA,CSBETA,SNDB2,CSDB2,
39300 +Q,OC,CNSTAN,SNBMD,CSAMP,SNP,CSP,SNTHETA,CSHETA,CSGAM
39400 COMMON/ANGLE/ ALPHA,GAMMA,PHI,THETA,OTHETA,BETA
39500 COMMON/STN/ TEMP4,STRAIN2(5),STRN(10,5),ST(10),STRAIN(5)
39600
39700
39800 RR=(R1*SIN(BETA-(Z*(OTHETA/2.))))/SNBETA
39900
40000 DELTAR=RR/5.
40100 RPC=RR-Y*(DELTAR/2.)
40200
40300 ALAMDAT=(2.*(RPC+(DELTAR/2.))*SNDB2)/SNBMD
40400 ALAMDAC=(2.*RPC*SNDB2)/SNBMD
40500
40600 RETURN
40700 END
40800
40900
41000 SUBROUTINE E12 CALC(MM,L)
41100
41200
41300 ROUTINE TO CALCULATE THE ENERGY REQUIRED IN CROSSING THE DISCONTINUITY
41400 V12.
41500
41600 COMMON/1/ R1,Y,Z,DELTAR,ALAMDAT,ALAMDAC,RR,RPC
41700 COMMON/2/ RB,RE,V1,V3,PI
41800 COMMON/3/ SNALPHA,SNBETA,SNGAM,CSGPA,CSALPHA,CSBETA,SNDB2,CSDB2,
41900 +Q,OC,CNSTAN,SNBMD,CSAMP,SNP,CSP,SNTHETA,CSHETA,CSGAM
42000 COMMON/ENR/ E2,E4,E23,E12,E12T,E23T,E24T,E2T
42100 COMMON/ANGLE/ ALPHA,GAMMA,PHI,THETA,OTHETA,BETA
42200 COMMON/STN/ TEMP4,STRAIN2(5),STRN(10,5),ST(10),STRAIN(5)
42300
42400
42500 V12=(V1*SNALPHA)/CSGPA
42600 DV12=0.
42700 ELEMLN=(RB/CSGAM)/5.
42800 ELEMLNR=(RB/CSGAM)*((10,-Y)/10.)
42900 AREA12=ELEMLN+ELEMLNR*2.*PI*CSGAM
43000

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43100 CALL SPATE(V12,DV12,AK12,ELEMENR,STRATE)
43200 CALL STRAN12(MM,L,V12,BK12)
433000
43400 E12=V12*AREA12+AK12*BK12
435000
436000
437000 THE ENERGY FOR THE PRESELEMENT IS THEN ADDED TO THAT FOR PREVIOUS
438000 SIMILLAR ELEMENTS.
439000
44000 E12T=E12T+E12
441000
442000
44300 RETURN
44400 END
445000
446000
44700 SUBROUTINE E24 CALC(MM,L)
448000
449000
450000 ROUTINE CALCULATES THE ENERGY REQUIRED TO ACCELERATE THE MATERIAL
451000 DOWN THE D.M.Z BOUNDARY.
452000
45300 COMMON/1/ R1,Y,Z,DELTA,ALAMDAT,ALAMDAC,RR,RR
45400 COMMON/2/ RE,RE,V1,V3,PI
45500 COMMON/3/ SNALPHA,SNBETA,SNBETA,CSGPA,CSALPHA,CSBETA,SNDB2,CSDB2,
45600 Q,QQ,CONSTAN,SNBND,CSAMP,SNF,CSP,SNTHETA,CSTHETA,CSGAM
45700 COMMON/ENER/E2,E24,E23,E12,E12T,E23T,E24T,E2T
45800 COMMON/ANGLE/ALPHA,GAMMA,PHI,THETA,DIHETA,CETA
45900 COMMON/STN/ TEMP4,STRAIN2(5),STRN(10,5),ST(10),STRAIN(5)
46000
461000
462000
46300 V24=(V1*CSGAM*CSGAM*SNBETA)/(SNTHETA*CSGPA*CSGPA)
46400 DV24=((V1*CSGAM*CSGAM)/(CSGPA*CSGPA))*((SNTHETA*COS(ALPHA-
46500 +THE TA)-SNBETA*CSTHETA)/(SNTHETA*SNTHETA))
46600 AREA24=ALAMDAT*2.*PI*RR*COS(GAMMA-(Z*DIHETA/2.))
467000
46800 CALL SPATE(V24,DV24,AK24,STRATE2)
469000 PRINT*,'POS 2', MM,L,RR,DELTA,V24,STRATE2,BK24
47000 CALL STRAN2(MM,L,ALAMDAT,V24,STRATE2,BK24)
471000 PRINT*,'POS 1', MM,L,RR,DELTA,V24,STRATE2,BK24
472000
473000
47400 E24=V24*AREA24*AK24*BK24
475000
476000
47700 E24T=E24T+E24
47800 RETURN
47900 END
480000
481000
48200 SUBROUTINE E23 CALC(MM,L)
483000
484000
485000 ROUTINE CALCULATES THE ENERGY INVOLVED IN CROSSING THE BOUNDARY 23.
486000
48700 COMMON/1/ R1,Y,Z,DELTA,ALAMDAT,ALAMDAC,RR,RR
48800 COMMON/2/ RE,RE,V1,V3,PI
48900 COMMON/3/ SNALPHA,SNBETA,SNBETA,CSGPA,CSALPHA,CSBETA,SNDB2,CSDB2,
49000 Q,QQ,CONSTAN,SNBND,CSAMP,SNF,CSP,SNTHETA,CSTHETA,CSGAM
49100 COMMON/ENER/E2,E24,E23,E12,E12T,E23T,E24T,E2T
49200 COMMON/STN/ TEMP4,STRAIN2(5),STRN(10,5),ST(10),STRAIN(5)
493000
49400 V23=(V3*SNALPHA)/CSAMP
49500 DV23=0.
49600 ELEMENL=(RE/CSP)/5.
49700 ELEMENR=(RE/CSP)*((10.-Y)/10.)
498000
49900 AREA23=ELEMENL+ELEMENR*2.*PI*CSP
500000
50100 CALL SPATE(V23,DV23,AK23,ELEMENR,STRATE)
50200 CALL STRAN23(MM,L,BK23)
503000
50400 E23=AREA23*V23*AK23*BK23
505000
506000
50700 E23T=E23T+E23
50800 RETURN
50900 END
510000
51100 SUBROUTINE ENERGYC(MM,L)
512000
513000
514000 ROUTINE CALCULATES THE ENERGY DISSIPATED WITHIN EACH RECTANGULAR
515000 ELEMENT.
516000

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51700 COMMON/1/ R1,Y,Z,DELTA,ALAMDAT,ALAMDAC,RR,RC
51800 COMMON/2/ RE,RE,V1,V3,PI
51900 COMMON/3/ SINALPHA,SNBETA,SNBETA,CSGPA,CSALPHA,CSBETA,SNDB2,CSDB2,
52000 +,CC,CONSTAN,SNBMD,CSAMP,SNF,CSP,SNTHETA,CSTHETA,CSGAM
52100 COMMON/ANGLE/ E2,E24,E23,E12,E12T,E23T,E24T,E2T
52200 COMMON/ANGLE/ ALPHA,GAMMA,PHI,THETA,DTHEA,BETA
52300 COMMON/STR/ TEMP4,STRAIN2(5),STRN(10,5),ST(10),STRAIN(5)
52400
52500
52600 THIS ROUTINE USES A METHOD INVOLVING STRAIN RATE WHICH IS CALCULATED
52700 USING THE METHOD EMPLOYED BY ADIE AND ALEXANDER IN ADIE'S PH.D THESIS.
52800
52900
53000 ELEMENV=ALAMDAC*DELTA*2.*PI*RR*SNTHETA
53100 VELOC=(V1*CSGAM*CSGAM*SNBETA)/(SNTHETA*CSGPA*CSGPA)
53200
53300 DVELOC=((V1*CSGAM*CSGAM)/(CSGPA*CSGPA))*((SNTHETA*COS(ALPHA-THETA)
53400 +SNBETA*CSTHETA)/(SNTHETA*SNTHETA))
53500
53600 ETHET=((VELOC*CSBETA*2.)+(SNBETA*DVELOC))/RC
53700 EPHI=(VELOC/RR)*(CSBETA+(1./TAN(THETA))*SNBETA)
53800
53900 ERTHET=((-VELOC*SNBETA*2.)+(CSBETA*DVELOC))/RC
54000 EBARDOT=SQRT((2./3.)*((ETHET*ETHET)+(EPHI*EPHI)+(0.5*(ERTHET*ERTHET))))
54100
54200 CALL SRATE(VELOC,DVELOC,AK2,RR,STRATE3)
54250 WRITE(7,1004)MM,L,STRATE3
54260 1004 FORMAT(*ELEMENTS*,/ ,12,2X,12,E12.5)
54300 CALL CNTSTRN(MM,L,ALAMDAC,VELOC,
54400 +STRATE3,BK2)
54500 E2=EBARDOT+ELEMENV*AK2*BK2*SQRT(3.)
54600
54700 RETURN
54800 END
54900
55000
55100 SUBROUTINE TRIANGL(MM,L)
55200
55300
55400 ROUTINE CALCULATES THE ENERGY DISSIPATED WITHIN THE 5 TRIANGULAR
55500 ELEMENTS AT THE CENTRE OF THE MESH.
55600
55700
55800 COMMON/1/ R1,Y,Z,DELTA,ALAMDAT,ALAMDAC,RR,RC
55900 COMMON/2/ RE,RE,V1,V3,PI
56000 COMMON/3/ SINALPHA,SNBETA,SNBETA,CSGPA,CSALPHA,CSBETA,SNDB2,CSDB2,
56100 +,CC,CONSTAN,SNBMD,CSAMP,SNF,CSP,SNTHETA,CSTHETA,CSGAM
56200 COMMON/ANGLE/ ALPHA,GAMMA,PHI,THETA,DTHEA,BETA
56300 COMMON/ANGLE/ E2,E24,E23,E12,E12T,E23T,E24T,E2T
56400 COMMON/STR/ TEMP4,STRAIN2(5),STRN(10,5),ST(10),STRAIN(5)
56500
56600
56700 XR1=((ALAMDAC/2.)*SNBETA)/SNDB2
56800 TRIRAD=(2./3.)*(XR/5.)
56900 TRIC=(2.*TRIRAD*SNDB2)/SNBMD
57000 TRIAREA=0.5*ALAMDAC*XR1*SIN(BETA-(DTHEA/2.))
57100 TRIVOL=TRIAREA*2.*PI*TRIRAD*COS(GAMMA-(2*(DTHEA/2.)))
57200 VT=(V1*CSGAM*CSGAM*SNBETA)/(SNTHETA*CSGPA*CSGPA)
57300 DVT=((V1*CSGAM*CSGAM)/(CSGPA*CSGPA))*((SNTHETA*COS(ALPHA-
57400 +THETA)-SNBETA*CSTHETA)/(SNTHETA*SNTHETA))
57500 CALL SRATE(VT,DVT,AKT,TRIRAD,STRATE4)
57550 WRITE(7,1005)MM,L,STRATE4
57560 1005 FORMAT(*TRIANGLES*,/ ,12,2X,12,E12.5)
57600 CALL CNTSTRN(MM,L,TRIC,VT,
57700 +STRATE4,BKT)
57800 E2=TRIVOL*VT*AKT*BKT*SQRT(3.)
57900
58000
58100 RETURN
58200 END
58300
58400
58500 SUBROUTINE DGRID(N,X,I,G)
58600
58700
58800 ROUTINE INCLUDED SINCE A DECLARATION IS REQUIRED BY THE LIBRARY ROUTINE.
58900 IT HAS NO USE IN THE CALCULATION.
59000
59100 DIMENSION G(4),X(2)
59200 RETURN
59300 END
59400
59500
59600 SUBROUTINE JGRID(N,X,I,DG)
59700

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599000
599001 C ROUTINE INCLUDED FOR THE SAME REASON AS DGRID
600000 C
601000 DIMENSION DG(4,4),X(2)
602000 RETURN
603000 END
604000 C
605000 SUBROUTINE SSTATE(V,DV,AK,RADIUS,STRATE)
607000 C
608000 C THIS ROUTINE CALCULATES THE VALUE OF THE STRAIN RATE FOR EACH ELEMENT
610000 C AND RAISES IT TO THE POWER Q DETERMINED ON ENTRY BY THE USER.
611000 C
612000 C COMMON/3/ SNALPHA,SNBETA,SNGAM,CSGPA,CSALPHA,CSBETA,SNDB2,
613000 +CSDB2,Q,Q0,CONSTAN,SNBND,CSAMP,SNP,CSP,SNTHETA,CSTHETA,CSGAM
614000 C COMMON/ANGLE/ ALPHA,GAMMA,PHI,THETA,DTHETA,BETA
615000 C
616000 C
617000 STRAIN=(V*CSBETA*2.+(SNBETA*DV))/RADIUS
618000 STRAINP=(V/RADIUS)*(CSBETA+(1./TAN(THETA))*SNBETA)
619000 STRAINR=((-V*SNBETA*2.)+(CSBETA*DV))/RADIUS
620000 STRATE=SQRT((2./E.)*((STRAIN*STRAIN)+(STRAINP*STRAINP)
621000 +((0.5*(STRAINR*STRAINR))))
622000 C
623000 C
624000 AK=CONSTAN*(STRATE**Q)
625000 C
626000 RETURN
627000 END
628000 C
629000 C
630000 SUBROUTINE STRAN12(MM,L,V12,BK12)
631000 C
632000 C THIS ROUTINE CALCULATES THE STRAIN AT A DISCONTINUITY 12 AND RAISES
634000 C TO THE POWER QQ AS DETERMINED BY THE USER.
635000 C
636000 C COMMON/2/ RE,RE,V1,V3,PI
637000 C COMMON/3/ SNALPHA,SNBETA,SNGAM,CSGPA,CSALPHA,CSBETA,SNDB2,CSDB2,
638000 +Q,Q0,CONSTAN,SNBND,CSAMP,SNP,CSP,SNTHETA,CSTHETA,CSGAM
639000 C COMMON/STN/ TEMP4,STRAN2(5),STRN(10,5),ST(10),STRAIN(5)
640000 C
641000 C
642000 STRAIN(MM)=V12/(SQRT(3.)*V1*SNTHETA)
643000 IF(STRAIN(MM).LT.2.3) GO TO 1
64330 CALL FIRSTST(BK12)
64350 TEMP1=BK12
64370 RETURN
644000 EXP01=(STRAIN(MM)**(QQ+1.))
645000 C
646000 BK12=(EXP01/STRAIN(MM))/(SQRT(3.)*(QQ+1.))
647000 TEMP1=BK12
648000 GO TO 3002
649000 3001 BK12=TEMP1
650000 C
651000 3002 RETURN
652000 C
653000 END
654000 C
655000 SUBROUTINE CNTSTRN(MM,L,ALENGTH,VEL,STRATE,BK2)
656000 C
657000 C THIS ROUTINE CALCULATES THE STRAIN FO EACH ELEMENT EXCLUDING THE
659000 C BOUNDARY.
660000 C
661000 C COMMON/3/ SNALPHA,SNBETA,SNGAM,CSGPA,CSALPHA,CSBETA,SNDB2,CSDB2,
662000 +Q,Q0,CONSTAN,SNBND,CSAMP,SNP,CSP,SNTHETA,CSTHETA,CSGAM
663000 C COMMON/ANGLE/ ALPHA,GAMMA,PHI,THETA,DTHETA,BETA
664000 C COMMON/STN/ TEMP4,STRAN2(5),STRN(10,5),ST(10),STRAIN(5)
665000 C
666000 C
667000 DT=ALENGTH/VEL
668000 IF(L.GT.1) GO TO 777
669000 STRN(L,MM)=(STRATE*DT)+STRAIN(MM)
670000 IF(STRN(L,MM).LT.2.3) GO TO 1
67030 CALL FIRSTST(BK2)
67050 TEMP2=BK2
67070 RETURN
671000 P1=STRAIN(MM)**(QQ+1.)
672000 P2=STRN(L,MM)**(QQ+1.)
673000 BK2=((P2-P1)/(STRN(L,MM)-STRAIN(MM)))/(SQRT(3.)*(QQ+1.))
674000 TEMP2=BK2
675000 GO TO 778
676000 777 STRN(L,MM)=(STRATE*DT)+STRN(L-1,MM)
676500 C

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67700 IF (STRN(L,MM).GE.2.3) GO TO 4001
67800 POWER2=(ST(L,MM)**(QQ+1.))
67900 POWER1=(STRN(L-1,MM)**(QQ+1.))
680000
68100 BK2=((POWER2-POWER1)/(STRN(L,MM)-STRN(L-1,MM)))/(SQRT(3.)*(QQ+1.))
68200 TEMP2=BK2
68300 GO TO 778
68400 4001 BK2=TEMP2
685000
68600 778 RETURN
68700 END
688000
689000
690000 SUBROUTINE STRAN23(MH,L,BK23)
691000
692000
693000 ROUTINE CALCULATES THE STRAIN AT THE BOUNDARY 23.
694000
695000 COMMON/2/ RB,RE,V1,V3,PI
696000 COMMON/3/ SNALPHA,SNBETA,SNBGM,CSGPA,CSALPHA,CSBETA,SNCTB2,CSCTB2,
697000 +Q,QQ,CONSTAN,SNBND,CSAMP,SNP,CSP,SNTHETA,CSTHETA,CSGAN
698000 COMMON/5/ TEMP4,STRAIN2(5),STRN(10,5),ST(10),STRAIN(5)
699000
700000
70100 V203=V3+CSP/CSAMP
70200 V23=V3+SNALPHA/CSAMP
70300 STRAIN2(MH)=V23/(SQRT(3.)*V203*SNTHETA)+STRN(L,MM)
70330 CALL FIRSTST(BK23)
70350 TEMP3=BK23
70370 RETURN
70400 IF (STRAIN2(MH).LE.2.3) GO TO 1
70500 1 EXPON1=(STRN(L,MM)**(QQ+1.))
70600 EXPON2=(STRAIN2(MH)**(QQ+1.))
707000
70800 BK23=(EXPON2-EXPON1)/(STRAIN2(MH)-STRN(L,MM))/(SQRT(3.)*(QQ+1.))
70900 TEMP3=BK23
71000 GO TO 5002
71100 5001 BK23=TEMP3
712000
71300 5002 RETURN
71400 END
715000
716000
717000 SUBROUTINE STRAN24(MH,L,ALANDAT,VELO,STR,BK24)
718000
719000
720000
721000 THIS ROUTINE CALCULATES THE STRAIN AT THE BOUNDARY 24
722000
723000 COMMON/3/ SNALPHA,SNBETA,SNBGM,CSGPA,CSALPHA,CSBETA,SNCTB2,CSCTB2,
724000 +Q,QQ,CONSTAN,SNBND,CSAMP,SNP,CSP,SNTHETA,CSTHETA,CSGAN
725000 COMMON/ANGLE/ ALPHA,GAMMA,PHI,THETA,ETHETA,BETA
726000 COMMON/5/ TEMP4,STRAIN2(5),STRN(10,5),ST(10),STRAIN(5)
727000
728000
72900 DT=ALANDAT/VELO
73000 IF(L.GT.1) GO TO 7799
73100 ST(L)=(STR*DT)+STRAIN(MH)
732000 PRINT*,'LINE 732000 ST(L) AND STRAIN(MH)',ST(L),STRAIN(MH)
73300 IF (ST(L).LE.2.3) GO TO 1
73400 CALL FIRSTST(BK24)
73500 TEMP4=BK24
736000 PRINT*,'AT LINE 71155',TEMP4
73700 RETURN
73800 1 P3=ST(L)**(QQ+1.)
73900 P4=STRAIN(MH)**(QQ+1.)
740000 PRINT*,'P3 AND P4',P3,P4
74100 BK24=((P3-P4)/(ST(L)-STRAIN(MH)))/(SQRT(3.)*(QQ+1.))
74200 TEMP4=BK24
743000 PRINT*,'AT LINE 71211',TEMP4
744000 PRINT*,'BK24'
74500 GO TO 7798
74600 ST(L)=(STR*DT)+ST(L-1)
74700 IF (ST(L).GE.2.3) GO TO 6001
74800 POW2=(ST(L)**(QQ+1.))
74900 POW1=(ST(L-1)**(QQ+1.))
750000 PRINT*,'POW2 AND POW1',POW2,POW1
75100 BK24=((POW2-POW1)/(ST(L)-ST(L-1)))/(SQRT(3.)*(QQ+1.))
75200 TEMP4=BK24
75300 GO TO 7798
75400 6001 BK24=TEMP4
75500 7798 RETURN
75600 END
757000 SUBROUTINE FIRSTST(BK)
758000 COMMON/3/ SNALPHA,SNBETA,SNBGM,CSGPA,CSALPHA,CSBETA,SNCTB2,CSCTB2,
759000 +Q,QQ,CONSTAN,SNBND,CSAMP,SNP,CSP,SNTHETA,CSTHETA,CSGAN

```

```
76000 BK=((2.3*(00+1.))/(2.3))/(SORT(3.)*(00+1.))
76100 PRINT*, 'IN FIRSTST', BK
76200 RETURN
76300 END
```

## APPENDIX 4

```

00100 PROGRAM EXTRUS (INPUT, OUTPUT, TAP5=INPUT, TAP6=OUTPUT)
00110C
00120C
00130C
00140C
00150C
00160C THE MATHS. IS IN THE S.I. SYSTEM.
00170C
00180C THE FOLLOWING HAVE BEEN ASSUMED TO BE CONSTANT:-
00190C BILLET DIAMETER, DB:
00200C BILLET LENGTH, BL:
00210C CONTAINER TEMPERATURE, TEMPCOM:
00220C ACTIVATION ENERGY, Q:
00230C GAS CONSTANT, R:
00240C LAMP LENGTH, ALL:
00250C DENSITIES OF ALUMINIUM ALLOY AND STEEL, RHOAL, RHOST:
00260C HEAT CAPACITIES OF ALUMINIUM ALLOY AND ST.-L, CAL, CST:
00270C THERMAL CONDUCTIVITIES OF ALUMINIUM ALLOY AND STEEL, AKAL, AKST:
00280C
00290C
00300 DIMENSION V1(50), ALOAD(50), TEMPT(50), TB(50), AK6(50), AK8(50), AK10(50),
00310 TIME(50), CTOT1(50), CTOT2(50), CTOT3(50), DELTAT(50), TEMPD(50), STATE(50),
00320 FLOG7(50), SIGMA(50), ALPHAS(50), SINH(50), BLOG(50), V1(50), ALOAD1(50)
00330 1 PRINT 60
00340 60 FORMAT (1READ IN THE EXTRUSION RATIO, ALPHA, THEN THE NUMBER OF RESULTS*
00350 +/ (UP TO 50) FOLLOWED BY THE FAN SPEED (M/S), THE LOAD (MN/MSQED.)'
00360 +/ AND THE INITIAL BILLET TEMP. (DEG.C.) FOR EACH POINT. ')
00370 READ (.ERR.=1, F.*), FF, ALPHA, N, (V1(I), ALOAD1(I), TB(I), I=1, N)
00380 52 PRINT, 'IS THE INFORMATION CORRECT?'
00390 READ 50, MNSWR
00400 50 FORMAT (A1)
00410 IF (MNSWR.EQ.'1HY') GO TO 51
00420 IF (MNSWR.EQ.'1HN') GO TO 1
00430 GO TO 52
00440C
00450C
00500 51 DB = 0.075
00510 BL = 0.090
00520 TEMPCOM = 573.00
00530 Q = 151000.0
00540 R = 2.317
00550 ALL = 0.00508
00560 RHOAL = 2796.9
00570 RHOST = 7868.0
00580 CAL = 1000.0
00590 CST = 489.752
00600 AKAL = 281.0
00610 AKST = 32.6508
00620C
00630C
00640C VALUES OF THE WORK CONSTANT AND THE ANGLE OF
00650C THE DEFORMATION ZONE.
00660C
00670C
00680 IR = IFIX(FF)
00690 IF (IR.NE.40) GO TO 2
00700 ROM=53.53
00710 C=14.096
00720 GO TO 9
00730 2 IF (IR.NE.80) GO TO 3
00740 ROM=54.91
00750 C=12.176
00760 GO TO 9
00770 3 IF (IR.NE.60) GO TO 93
00780 ROM=54.39
00790 C=16.82
00800 93 CONTINUE
00810C
00820C EVALUATION OF THE CONSTANTS USED IN THE PROGRAM
00830C
00840C
00850 9 ANGLE = 90.0-ROD
00860 RANGLE = ANGLE/57.29573
00870 RADROM = ROM/57.29573
00880 TANANG = SIN(RANGLE)/COS(RANGLE)
00890 TANROM = SIN(RADROM)/COS(RADROM)
00900 DE = SQRT((OS*DE)/FF)
00910 ALD = (DB-DE)/2.0*(1.0-TANANG)
00920 ALT = BL-ALD-((DB-DE)/2.5*TANANG)
00930 ALFAST = AKST/(CST*RHOST)
00940 ALFAAL = AKAL/(CAL*RHOAL)
00950 ALFA = SQRT(ALFAST/ALFAAL)
00960C
00970C
00980C CALCULATION OF CONSTANTS IN THE HEAT BALANCE EQUATION
00990C

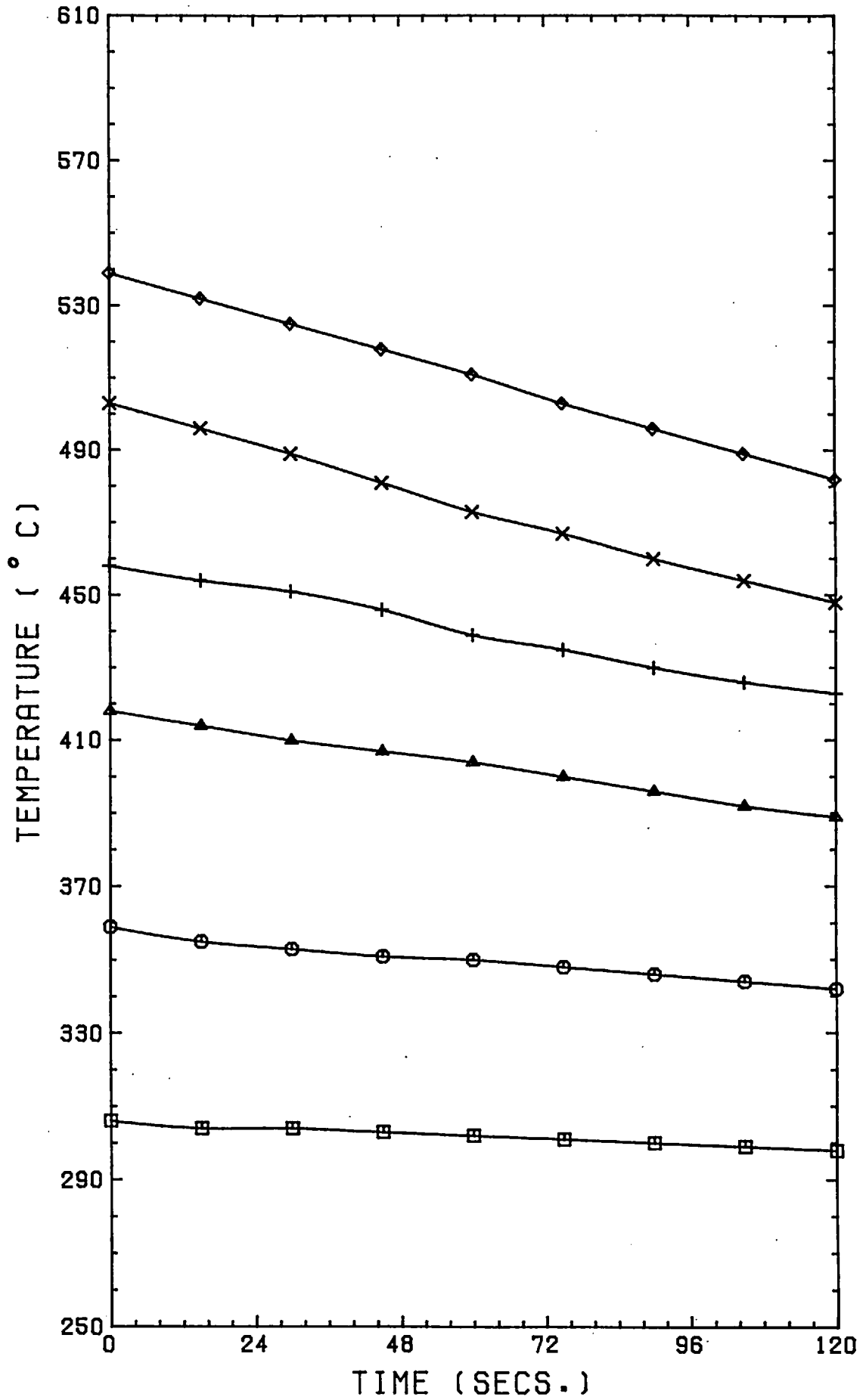
```

```

01000C
01010 AK1 = (3.142*((DB*DB)-(DE*DE))/COS(ANGLE))*SQRT(AKST*CST*RHOST/12.0)
01020 AK2 = 3.142 *ALD/6.0*(18.0*AKST*DB*SQRT(RHOST*CST)**(2.0/3.0))
01030 AK3 = 3.142 *ALD/6.0*DE*(18.0*AKST*DE*((RHOST*CST)**2))**(1.0/3.0)
01040 AK4 = 3.142*ALL/6.0*(18.0*AKST*DE*SQRT(RHOST*CST)**(2.0/3.0))
01050 AK5 = 3.142*ALL/6.0*DE*(18.0*AKST*DE*((RHOST*CST)**2))**(1.0/3.0)
01060 AK7 = 3.142/6.0*ALT*(18.0*AKST*DE*SQRT(CST*RHOST)**(2.0/3.0))
01070 AK9 = 3.142/6.0*(18.0*AKST*(DE**4))*(ALT**3)*(RHOST*CST)**2**(1.0/3.0)
01080 AK11 = 3.142 *DB*DB*SQRT(RHOST*AKST*CST/12.0)
01090C
01100C
01110C      EVALUATION OF THE TEMPERATURE RISE
01120C
01130C
01140 DO 10 I=1,N
01150 ALOAD(I) = ALOAD1(I)*1.00000E0
01160 V(I) = V1(I)/1000.0
01170 AK6(I) = 3.142*DE*DE/4.0*RHOAL*CAL*EP*V(I)
01180 AK8(I) = -AK7*3.0*V(I)/(5.0*ALT)
01190 AK10(I) = -AK9*3.0*V(I)/(4.0*ALT)
01200 TIME(I) = ALT/V(I)
01210 CTOT2(I) = (AK1*SQRT(TIME(I)))+(AK2+AK4)*TIME(I)**(2.0/3.0)+(AK3+
01220 AK5)*TIME(I)**(1.0/3.0)
01230 CTOT1(I) = (AK6(I)*TIME(I))+CTOT2(I)
01240 CTOT3(I) = CTOT2(I)+(AK7*TIME(I)**(2.0/3.0)+(AK8(I)*TIME(I)**(5.0/3.0))
01250 +(AK9*TIME(I)**(1.0/3.0)+(AK10(I)*TIME(I)**(4.0/3.0))
01260 +(AK11*SQRT(TIME(I)))
01265 TB(I)=TR(I)+273.
01267 ALOAD(I)=ALOAD(I)*3.142*0.075*0.075/4.
01270 TEMPI(I) = (TB(I)+(ALFA*TEMPCON))/(1.0+ALFA)
01280 DELTAT(I) = (ALOAD(I)*ALT-(TEMPI(I)-TEMPCON)*CTOT3(I))/CTOT1(I)
01290 TEMPD(I) = TB(I)+DELTAT(I)
01300C
01310 WRITE(6,500) TEMPD(I)
01320 500 FORMAT(F20.6)
01325 10 CONTINUE
01330 STOP
01340 END

```

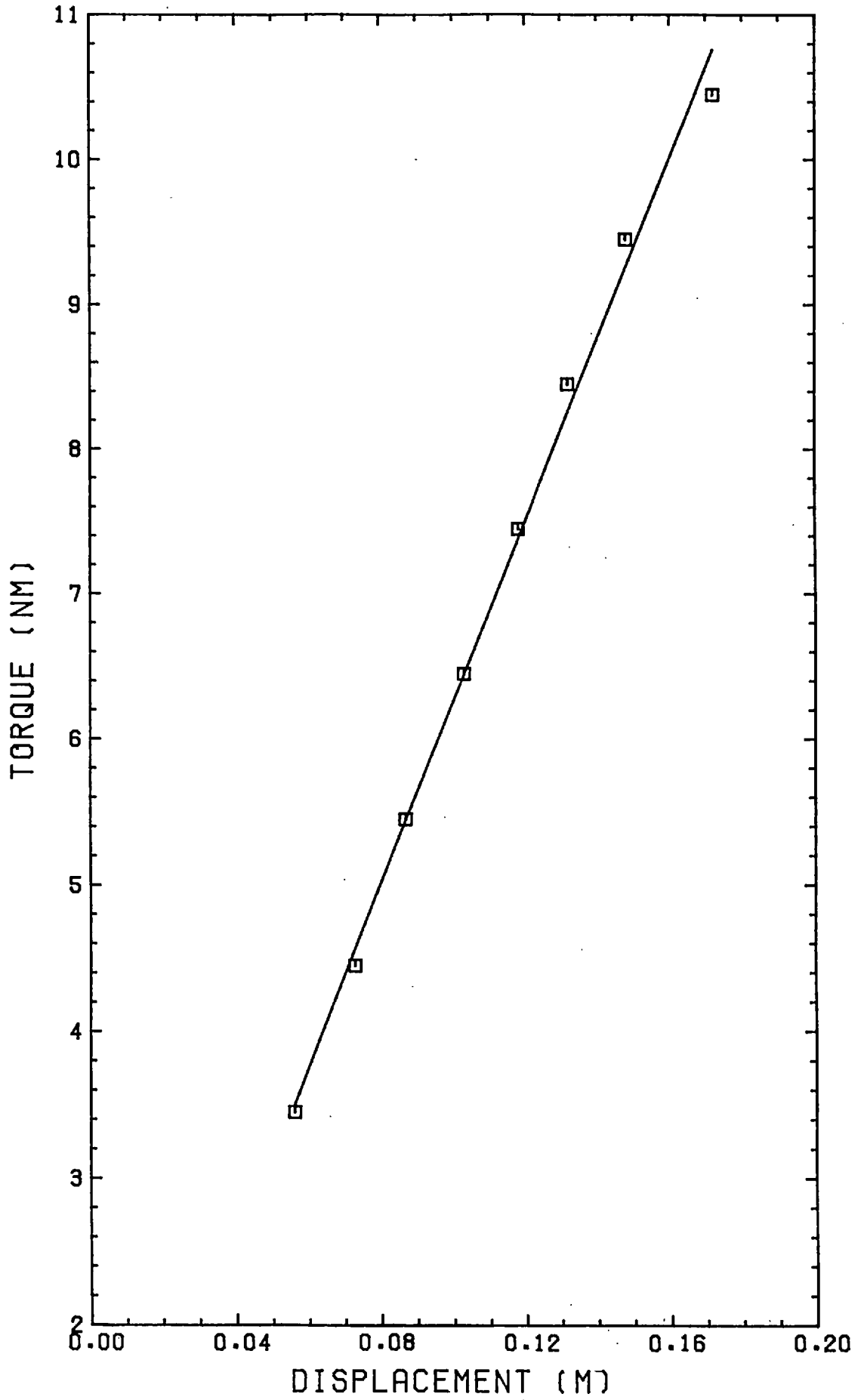
## APPENDIX 5



Billet cooling curves



## APPENDIX 6



Torsion machining calibration curve

## APPENDIX 7

## ERRORS

In the present investigation, during each series of tests, certain runs were duplicated to discover the reproducibility of the results. Listed in the table below are the percentage reproducibility errors for the parameters of major importance. (Each figure is the result of at least five pairs of duplicated runs).

<u>% Reproducibility Error</u>	<u>Parameter</u>
8%	Extrusion load
4%	Extrusion ram speed
4%	Tensile strength (R.T.)
7%	Tensile strength (400°C)
8%	Elongation (R.T.)
10%	Elongation (400°C)

The accuracy of the integral profile technique used to predict the deformation zone temperature during extrusion is rather uncertain. Experimental verification of the predictions presents tremendous difficulties. Extrusion of compacted powder billets containing thermocouples is almost impossible. Raybould and Sheppard who developed the integral profile technique for extrusion, carried out some aluminium extrusions using thermocouples to monitor the die surface and dead metal zone temperatures. They found that the disagreement between experimentally measured temperatures and theoretically predicted temperatures was usually less than 10°C (for R = 30:1 and 50:1 and ram speeds varying from 0.0005 m/s to 0.1 m/s). The maximum variation was 20°C obtained using medium ram speeds at R = 30:1. Raybould and Sheppard also compared experimental results obtained by Singer and

Al-Samarrai for  $R = 12:1$  with integral profile predictions. Once again the error was usually of the order of  $10-15^{\circ}\text{C}$ . It should be remembered that these experiments involved extrusions in which the initial billet temperature and the container temperature were the same. In the present series of experiments this was usually not the case. However, the theory was modified to allow for this initial temperature difference. In general the disagreement between the theoretically predicted temperature and the actual temperature should certainly not exceed  $20^{\circ}\text{C}$  which represents a 5% error in a  $450^{\circ}\text{C}$  extrusion.

## APPENDIX 8

## RECOMMENDATIONS FOR FURTHER WORK

The present investigation has dealt with the extrusion of aluminium, Al-Mg and Al-Mg-Si atomized powders containing various amounts of  $B_2O_3$  - 10%  $SiO_2$  glass. Although these systems have been dealt with in some detail in the present series of experiments several major research topics, touched on by this project, have still to be investigated in a completely satisfactory manner. These include:-

- 1) The temperature rise during extrusion
- 2) The operating mechanism which produces the peak in the extrusion load-ram displacement curve.
- 3) A detailed, scientific investigation of extrusion defects and their causes.
- 4) Other types of glasses which might be used to fibre-reinforce metal matrices.
- 5) The use of other materials, such as metal wires, in the reinforcement of metal matrices.

1) The importance of knowing the deformation temperature accurately is obvious as this parameter controls, to a large extent, the product structure and properties. A more satisfactory model for predicting temperature rise during extrusion might be produced by using a finite element technique or by modifying the minimised axis-symmetric upper bound solution used in this project. These methods are feasible only if excellent computing facilities are available.

2) The magnitude of the peak in the extrusion load curve determines the press capacity required for a particular process. Thus the smaller the peak, the more efficient the process. In order to minimise this peak a thorough

knowledge of the factors believed to cause it (i.e. die-quenching friction effects, dislocation dynamics) must be obtained.

3) In the past, the production of an extrude possessing a good surface finish has usually been more of an art than a science. The exact effect on surface finish of varying parameters such as billet temperature, container temperature, ram speed and lubrication conditions must be investigated. The exact operating mechanisms which produce hot and cold shortness, surface checking etc. must be understood.

4) One of the main reasons for choosing the  $B_2O_3 - 10\% SiO_2$  glass for this project was the low softening temperature of this glass ( $< 300^\circ C$ ). The glass requirements i.e. acceptable chemical and mechanical properties and low softening temperature, limit the choice to some  $B_2O_3$  based glass. If the  $SiO_2$  content were increased the strength of the glass would increase and although the softening temperature would also increase, extrusion in aluminium should still be possible. Therefore further work involving  $B_2O_3-SiO_2$  glasses containing more than 10%  $SiO_2$  might be useful.

It might also prove possible to incorporate other strong, chemically stable glasses with higher softening temperatures in metals with higher melting points than aluminium. For example silica softens in the temperature range 1500-1600  $^\circ C$ . Additions of boric acid would reduce this softening point to a temperature suitable for the extrusion of iron powder. The properties of silica reinforced iron might prove very interesting.

5) In the past a considerable amount of research involving the use of metal wires to reinforce metal matrices has been carried out. Very often this work has involved steel wires in aluminium. The wires have

usually been aligned individually and then incorporated in a metal powder compact or alternatively vacuum infiltrated with molten matrix metal. Extrusion at fairly low ratios has sometimes been used to consolidate the product and improve the fibre alignment. However, the present research project has demonstrated that powder extrusion at high ratios in fact serves three purposes, namely:-

- 1) Consolidation of product
- 2) Fibre formation
- 3) Fibre alignment

This powder extrusion process also has the advantages of speed and simplicity. Therefore, it might be worth re-examining some metal-metal systems composites of which were originally produced by the tedious methods mentioned in the previous paragraph. Ideally, a strong, stiff metal in powder form would be mixed with a weaker ductile, (hopefully cheaper) metal powder, the two metals having a similar range of hot working temperatures. The table below shows the relevant properties of some of the more likely metals.

Four systems which look promising in view of the tabulated data are:

- 1) Copper reinforced with Beryllium to increase the tensile strength and "stiffness" and to decrease the specific gravity.
- 2) Copper reinforced with Nickel to increase the tensile strength and "stiffness".
- 3) Stainless steel reinforced with Beryllium, once again to increase the tensile strength and stiffness and to decrease the specific gravity.

- 4) Titanium reinforced with Nickel solely to increase the "stiffness".

Material	U.T.S. (MN/M <sup>2</sup> ) (x10 <sup>6</sup> )	E (MN/M <sup>2</sup> ) (10 <sup>6</sup> )	Melting Point (°C)	Specific Gravity
Austenitic Stainless steel (22%Cr, 22%Ni)	0.725	0.207	(1147)	7.8
β-Titanium	2.21	0.117	1677	4.6
Nickel	0.60	0.207	1453	8.9
Aluminium (99.9% pure)	0.06	0.069	660	2.8
Copper	0.41	0.124	1083	8.9
Molybendum	2.07	0.365	2610	10.3
Tungsten	2.90	0.345	3380	19.3
Beryllium	1.10	0.310	1284	1.8

## NOMENCLATURE

A	Area Constant in flow stress equation Extrusion constant
$A_B$	Cross sectional area of ram
$A_1, A_2, A_3$	Constants in hot working theory
a	Radius of billet - radius of extrude Compaction constant Notch depth
B	Extrusion constant
$B_i$	Mobility of species i
b	Burgers vector
C	Compaction constant Heat capacity
$C_s, C_{AL}$	Heat capacities of steel and aluminium
$C^*$	Degree of volume reduction
$dC_i/dx$	Concentration gradient
D	Deformation zone diameter Distance apart of obstacles Compact relative density Grain diameter
$D_E$	Extrude diameter
$D_B$	Billet diameter
d	Radius of extrude Width of obstacle
$E_F$	Elastic modulus of fibre
$E_M$	Elastic modulus of matrix
$E_C$	Elastic modulus of composite
$\dot{E}$	Rate of energy dissipation
G	Shear modulus
$\Delta H$	Activation energy
$J_i$	Rate of self diffusion of species i
K	Shear flow stress Boltzman's constant



$K_s$	Shear flow stress
$K_A$	Athermal component of shear flow stress
$K'$	Compaction constant
$K_{ij}$	Shear flow stress at discontinuity $ij$
$L$	Extrusion load
$L_{crit}$	Critical fibre length
$l_D$	Height of control volume 1
$l_T$	Height of control volume 2
$l_1$	Length of die land
$l$	Dislocation length
$M$	Torque
$m$	Strain exponent
$n$	Strain rate exponent Constant in hot working theory
$n_2$	Constant in hot working theory
$P$	Extrusion pressure Compaction pressure Tensile load on fibre
$Q$	Heat flow
$R$	Extrusion ratio Universal gas constant Freezing rate
$R_B$	Billet radius
$r(\theta)$	Radial distance from $O_2$
$r$	Fibre radius
$S$	Area of velocity discontinuity Distance from heat source
$T$	Temperature
$T_M$	Melting point temperature
$T_B$	Billet temperature
$T_I$	Billet-container interface temperature
$T_s$	Container temperature

$\Delta T$	Temperature rise in deformation zone
$\Delta T_D$	$T_I - T_S$
$T'$	Line tension
$t$	Time
$U, U_0$	Energy barriers (To dislocation motion)
$u$	Velocity
$\overline{u}_{ij}$	Velocity perpendicular to discontinuity $ij$
$V$	Ram speed Actual compact volume under pressure Activation volume
$V_0$	Initial apparent compact volume
$V_F$	Volume fraction of fibres
$v$	Velocity
$\overline{v}_{ij}$	Velocity discontinuity $ij$
$v_M$	Volume fraction of matrix
$v_W$	Volume fraction of whiskers
$dw/dt$	Rate of working
$x$	Depth of deformation zone
$\alpha$	Angle in axisymmetric velocity field Constant in hot working theory
$\beta$	Angle between stream-line and radial line from $O_2$ Constant in hot working theory
$\gamma$	Angle in axisymmetric velocity field Surface energy per unit area
$\dot{\gamma}$	Shear strain rate
$\delta$	Angle in axisymmetric velocity field
$\epsilon$	Natural strain
$\epsilon_{ij}$	Strain due to discontinuity $ij$
$\dot{\epsilon}$	Natural strain rate
$\overline{\dot{\epsilon}}$	Mean strain rate
$\dot{\epsilon}_x$	Natural strain rate in $x$ direction

$\phi$	Angle in plane strain velocity field Inclination of fibres to stress axis
$\rho$	Density Radius of curvature of notch
$\theta^*$	Averaging term
$\theta$	Angle in axisymmetric velocity field Torsional twist
$\lambda$	Interfibre distance Interparticle spacing
$\nu_0$	Frequency of jumps
$\sigma$	Flow stress
$\sigma_c$	U.T.S. of composite
$\sigma_u$	U.T.S. of matrix
$\sigma_w$	U.T.S. of whiskers
$\sigma_F$	U.T.S. of fibres
$\sigma'_m$	Tensile strength of matrix at fibre fracture strain
$\sigma_A$	Bulk applied stress
$\sigma_Y$	Local tensile stress perpendicular to notch
$\tau_s$	Shear yield stress
$\tau_m$	Shear stress in matrix
$\tau_i$	Lattice friction stress
$\omega$	Deformation zone semi-angle

*N.B. on graphs only*

C	Correlation coefficient
S	Standard error of estimate

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probably the inaccuracies arise because of insufficient experimental data in determining the stress/strain and stress/strain-rate relationships. The tests were all conducted at 400°C and other temperatures should be investigated using compression as well as torsion and tensile data to determine the relevant flow and shear stresses. Nevertheless the upper-bound based upon an actual deformation (or physical field) shows very good agreement with experimental observation.

### CONCLUSIONS

1. The plane-strain analogue approach is not suitable for predicting powder compact pressure.
2. The minimized axisymmetric upper-bound theory predicts pressures with acceptable accuracy, provided the extrusion ratio is < 100.
3. The upper-bound solution based upon an observed physical field gives very good agreement with observed pressures.
4. The variation between theoretical and experimental curve slopes requires further experimental investigation.

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# Production of an aluminium-rich magnesium silicide alloy from atomized powder

T. Sheppard and H. McShane

The production of an aluminium-rich magnesium silicide alloy by extrusion from the powder phase is described. The pressure requirements of such a process are investigated and it is shown that the operation may be described as thermally activated. The strain-rate sensitivity is shown to be less than the equivalent cast and extruded material. The mechanical properties of the as-extruded product are far superior to those of the conventional alloy and the powder material is heat-treatable.

Serial No. 426. Manuscript received 15 April 1976. Contribution to a Symposium on 'Hot Processing in Powder Metallurgy' to be held in Eastbourne on 11-13 October 1976. T. Sheppard, MSc, BSc(Eng), MIM, CEng, MIMechE, and H. McShane, BSc(Eng), ARSM, are in the John Percy Research Group, Department of Metallurgy and Materials Science, Imperial College of Science and Technology, London.

Until the advent of modern atomizing techniques the production of components from aluminium parts was hindered to a large extent by the cost of powders. Atomization has overcome this main obstacle, although in general the dispersion-hardening effect is not so pronounced because the  $Al_2O_3$  content is reduced compared with the original SAP-type alloys. An additional obstacle to widespread use of these light alloys is that the oxide dispersion acts as a vacancy barrier during the sintering process, prolonging that operation. A convenient process which ensures that the powder compact forms a homogeneous 100% dense product is extrusion, which can be carried out at high or low temperature with large strains. It has been shown<sup>1,2</sup> that such a process eliminates the sintering operation and it is suggested that energy which is termed 'redundant' in the extrusion of cast ingots is utilized to assemble the powders into solid form. Powder compacts also have an advantage over the normal ingot because less pressure is required<sup>3</sup> to effect extrusion. This is due to the differing rheological nature of the quasi-static deformation zone.

The atomizing process has other advantages in addition to the dispersion-hardening effect of the oxide film which almost invariably develops as an envelope surrounding each particle. The particles are usually cooled at a rate far in excess of those found in the normal continuous casting of aluminium alloys. This rapid cooling gives a fine eutectic, due to nucleation at lower temperatures, and the size and nature of precipitates can be expected to differ. In a heat-treatable alloy the oxide film and the differing nature of the

precipitates may together be expected to alter the heat-treatment reaction. There appears to be no reported literature on this subject.

The Al-Mg-Si system, when aluminium-rich, is among the most widely used aluminium alloys, having architectural and structural applications. The object of the present work was to examine the rate-controlling deformation mechanism, the nature of product densification, the alloy structure, and the effect of oxide dispersions on the ageing process.

## EXPERIMENTAL

### Material

The air-atomized Al-Mg-Si (HE9) alloy powder, supplied by Borax Consolidated Limited, nominally contained 0.5 wt.-%Mg, 0.5 wt.-%Si, balance Al. Initially the powder particle size distribution was obtained using standard 200 mm BS sieves stacked on an Endrock mechanical shaker to sieve a standard weight of powder (175 g) for a specified time (1 h). The results are shown in Table I.

Samples of the powder were analysed by the Imperial College Analytical Services Laboratory. The chemical composition is given in Table II.

### Preparation for extrusion

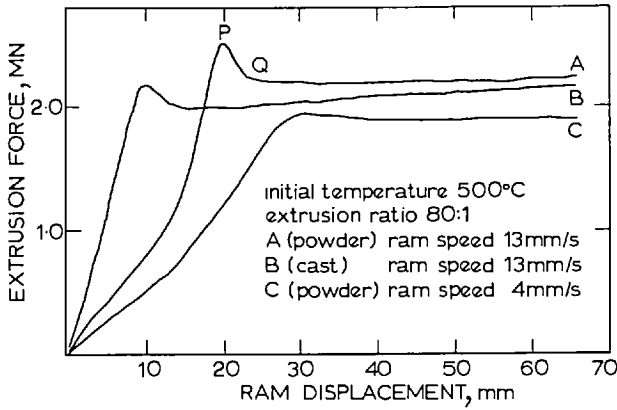
To facilitate handling at the press, the powder was compacted into billets using a 150 ton Tangye hydraulic press. To ensure easy removal of the billets from the die the tool surfaces were cleaned and painted with a stearic acid-chloroform lubricant before each compaction.

Table I Size distribution of Al-Mg-Si alloy powder

Particle size, $\mu\text{m}$	Weight percent
+355	2.14
-355+180	30.77
-180+106	25.73
-106+90	5.39
-90+75	5.20
-75+63	5.65
-63+53	5.15
-53	19.72

Table II Chemical analysis of powder (remainder Al) (wt.-%)

O	Mg	Si	Cr	Cu	Fe	Mn	Ti	Zn	Ni
0.062	0.33	0.12	0.01	0.014	0.15	0.10	0.014	0.025	0.004



1 Load displacement diagrams.

**Extrusion**

Extrusion was performed on an Enefco 5 MN hydraulic extrusion press having a container bore of 76 mm nom. dia. The container was maintained at 300°C for all extrusions. Details of the equipment and instrumentation have been previously reported.<sup>1</sup> Square-edged dies with reduction ratios of 5, 10, 30, 40, 60, 80, and 180 were used. To ensure an adequate surface finish a 10 mm thick pad of solid HE9 material was placed in front of each billet before extrusion. The billets were heated in an air circulating furnace situated adjacent to the extrusion press. The preheat temperature varied between 250 and 540°C. The billet transfer time between furnace and container varied from 10 to 15 s. This necessitated a correction of the temperature at the beginning of extrusion from predetermined cooling curves.

**Preparation of specimens for mechanical testing and microscopy**

The specimens were cut from the same region in each extrusion to allow for the variation in structure and properties along the extrusion length.

The electron microscope foils were prepared by grinding on water-lubricated silicon carbide paper to a thickness of ~0.15 mm, followed by thinning using a standard window technique. The electropolishing was carried out in a solution of 30% nitric acid in methanol under a potential difference of 15 V and at a temperature of -30°C. The transmission microscopy was carried out on an AEI EM6G 100 kV electron microscope.

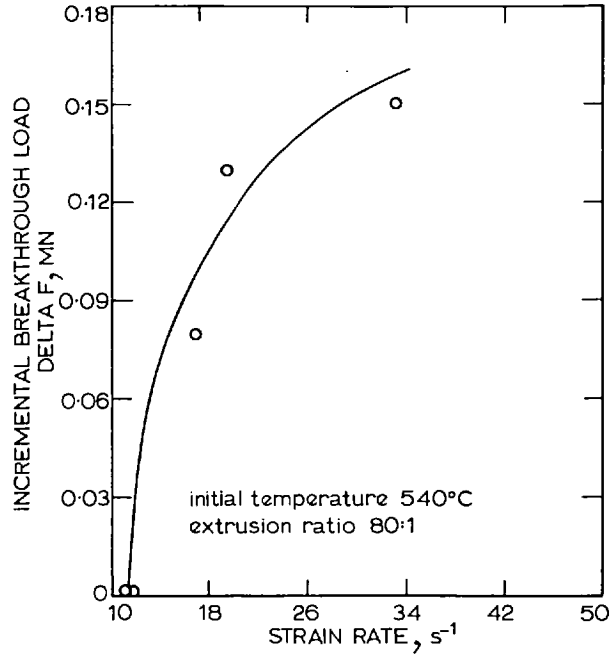
Standard Hounsfield No. 12 specimens were machined for tensile tests performed at room temperature and at various temperatures up to 400°C. The tensile tests were performed on an Instron tensile testing machine fitted with a split furnace for the higher temperature tests.

Impact tests on notched specimens were carried out using a Hounsfield balanced impact machine. The notch cross-section, method of supporting the specimen, and direction of delivery of the blow were the same as in the standard Izod impact test, but specimen size and shape were different (8 mm dia. bar 44.5 mm long, compared with 10 mm square bar 75 mm long for an Izod specimen). The energy reading given by this machine was multiplied by a factor of 2.4 in order to obtain the corresponding Izod value.

**RESULTS AND DISCUSSION**

**Extrusion**

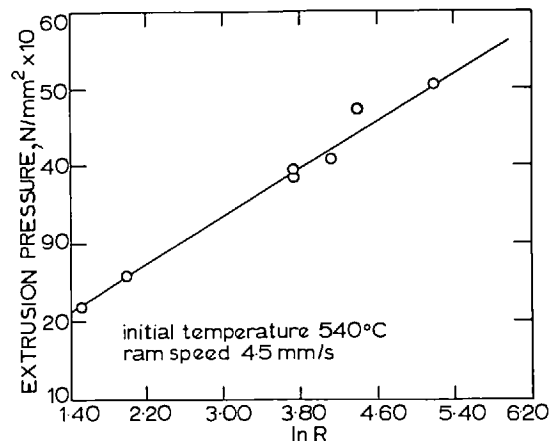
The load-displacement curves for two powder billets and a solid billet of the same alloy composition (excluding Al<sub>2</sub>O<sub>3</sub>) are shown in Fig. 1. The curves differ significantly in the



2 Incremental breakthrough load as function of strain rate.

initial stages of the process (i.e., while the quasi-static deformation zone is being established). The cast billet depicts the normal linear upset forging zone before the peak load is reached, whereas the powder billet shows a non-linear compaction zone which has been shown<sup>1</sup> to correspond to the compact obtaining 100% density. It should be clearly understood that the attainment of theoretical density does not coincide with attainment of optimum properties. In fact, at this stage, the billet is brittle and has practically no resistance to shear. The massive literature on the forging of metal powders<sup>4</sup> supports the thesis<sup>1</sup> that, since deviatoric deformation is a prerequisite for the achievement of acceptable properties, the extruding compact cannot obtain optimum properties until it has completely traversed the deformation zone.

The existence of a peak (denoted by PQ in Fig. 1) in one of the powder billets is contrary to evidence reported on the extrusion of nickel-rich alloys.<sup>5</sup> This breakthrough pressure



3 Extrusion pressure as function of extrusion ratio.

## Nomenclature

$A$	hot working constant
$A_1, A_2$	constants in extrusion equation
$A_3, A_4$	constants in stress/strain-rate equations
$C$	load constant for cast billet
$C_3$	load constant for powder billet
$C_1(t), C_2(t)$	time-dependent constants in temperature calculation
$D_B$	diameter of billet
$D_E$	diameter of extrudate
$G$	gas constant
$\Delta H$	activation energy for deformation process
$L_E$	load
$n, n_1$	stress/strain-rate exponents
$p$	extrusion pressure
$R$	extrusion ratio
$T$	temperature
$t$	time
$\Delta T_D$	temperature differential between product and tools
$U_0$	energy barrier
$V$	ram speed
$V_1$	activation volume
$Z$	Zener-Holloman parameter
$\alpha, \beta$	constants in stress/strain relationship
$\dot{\epsilon}$	strain rate
$\dot{\epsilon}_0$	constant in Seeger analysis
$\sigma_A$	thermal component of flow stress
$\sigma$	flow stress
$\omega$	90° dead metal zone angle

can clearly be seen to be more than that necessary to overcome friction between the compact and the extrusion container and has been attributed to temperature quenching at the die/billet interface.<sup>6</sup> In the present series of experiments billets extruded at the same temperature as the tools exhibited such peaks, so that this explanation is also quite clearly in error. Castle and Sheppard<sup>7</sup> have presented an explanation based on dislocation dynamics but for cast billets their experimental evidence is that the strain-rate has no effect. Fig. 1 suggests that this is not so for the compacts extruded, since the billet extruded at a lower strain-rate shows no peak to occur. However, the maximum load is also lower in this experiment and existing evidence does suggest that there is a threshold load value below which these pressure peaks either do not occur or cannot be detected.

The evidence, then, would support the view that this phenomenon is connected with a dynamic process. At high strain rates an excess of dislocations is generated immediately after the compaction phase and before dynamic softening processes (e.g. recovery) commence to operate; while at lower strain rates the rate of dislocation production is never large enough to overcome the dynamic softening process. The same argument could be applied to those extrusions in which the maximum pressure is low. Fig. 2 shows supporting evidence that the difference between this peak pressure and the steady-state pressure increases with increasing strain rate (calculated using the Feltham equation as shown below). It should be recalled, however, that increasing the strain rate will increase the temperature rise obtaining as well as increasing the flow stress in the absence of temperature rise. Hence the pressure required may be greater or lower depending upon the balance of these variables.

Fig. 3 shows that the usual relationship is found between the steady-state pressure required for extrusion and the logarithm of the extrusion ratio. For this alloy at 540°C the relationship may be written:

$$p = 103.4 + 76.2 \ln R \dots \dots \dots (1)$$

whereas for cast billets the usual form of this equation<sup>8</sup> is

$$\frac{p}{\sigma} = 0.52 + 1.32 \ln R \dots \dots \dots (2)$$

For comparative purposes it is convenient to write eqns. (1) and (2) in the form:

$$\frac{p}{A_1 \sigma} = 1.357 + \ln R \quad \text{for a powder compact} \dots (3)$$

and

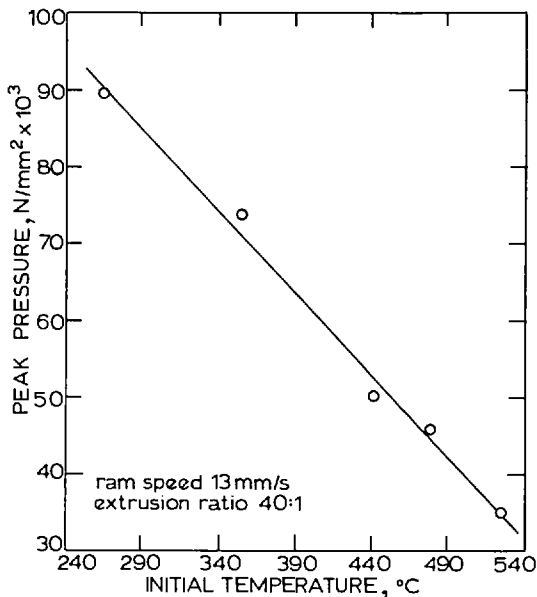
$$\frac{p}{A_2 \sigma} = 0.393 + \ln R \quad \text{for a cast billet} \dots \dots (4)$$

The significant feature here is the relative magnitude of the constant on the right-hand side of these equations since this is generally recognized to be a measure of the so-called 'redundant' work (i.e., work not directly contributing to a shape change). It has been shown<sup>9</sup> that theoretically the energy required for homogeneous deformation will be lower for a powder compact and these equations tend to substantiate this. They also indicate that the redundant portion of the total energy is very large, being 27% at an extrusion ratio of 40:1 compared with only 10% for a cast billet. This is again consistent with the concept of 'building' the alloy as it traverses the deformation zone; weld necks being continuously broken and rewelded until the compact as a whole can withstand the applied deviatoric stresses. Thus the actual construction of the alloy (normally achieved by sintering) is performed by the portion of the extrusion pressure normally termed redundant—clearly a misnomer for powder extrusion processes.

The steady-state extrusion pressure is shown plotted against temperature in Fig. 4. The graph, for an extrusion ratio of 40:1, is linear and suggests that the effective flow stress is directly related to the extrusion temperature. Calculation of the extrusion pressure and comparison of experimental and theoretical results are dealt with in a companion paper.<sup>9</sup>

### Mechanical properties of the extrudate

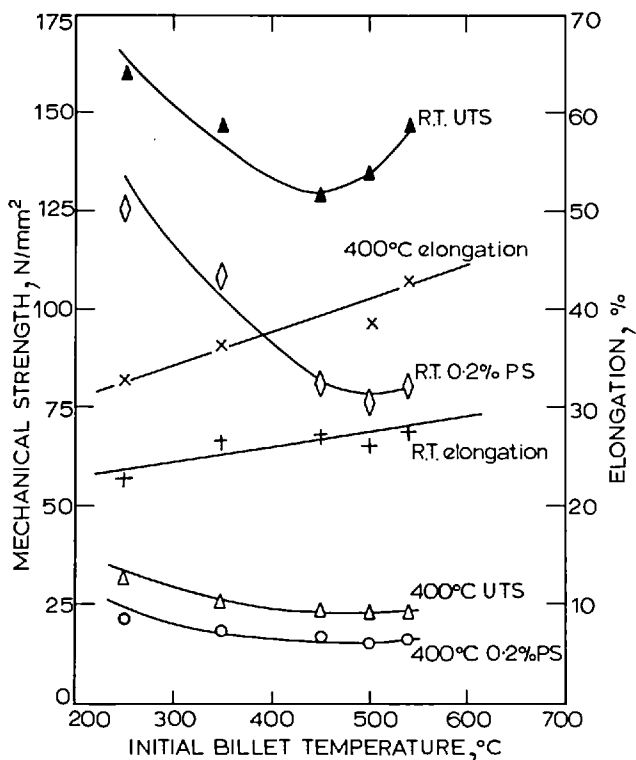
The variation of mechanical properties with initial billet temperature is shown in Fig. 5. The room-temperature proof and ultimate stresses show the expected decrease in value with increasing initial billet temperature (in the range 250–480°C) which is clearly due to softening, probably by dynamic recovery. However, from 480 to 540°C the trend is reversed: the material becomes stronger and elongations continue to improve. This is probably because, at the higher temperatures, a limited amount of magnesium silicide (Mg<sub>2</sub>Si) will be taken into solution and on subsequent air cooling after extrusion a supersaturated solution with a non-equilibrium vacancy concentration will result. It could also be caused by a natural ageing process which occurs at room temperature<sup>10</sup> over a period of weeks and in the cast alloy is due to a fine precipitate of Mg<sub>2</sub>Si. Further study on the transmission electron microscope would be required to verify this point. The room-temperature proof stress varies from 125–75 N/mm<sup>2</sup> over the temperature range investigated, which compares with an as-extruded strength of 50–55 N/mm<sup>2</sup> for the normal cast alloy. In fact this strength



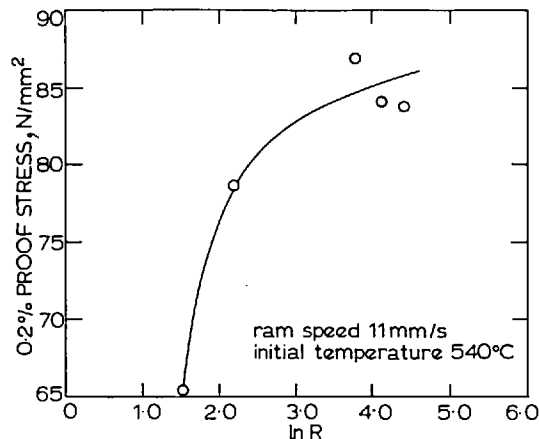
4 Extrusion pressure as function of initial billet temperature.

compares well with the strength of the heat-treated alloy, quoted as 130–160 N/mm<sup>2</sup>. The elongation varies from 20 to 25%, which is remarkable since the normal as-extruded product is assigned a value of 12% for this property as-extruded and only 7% in the heat-treated condition.

The 400°C properties, determined primarily to estimate the product flow stress for insertion into the load model, show a small decrease with increasing billet temperature.



5 Variation of mechanical properties with initial billet temperature.



6 Variation of proof stress with extrusion ratio.

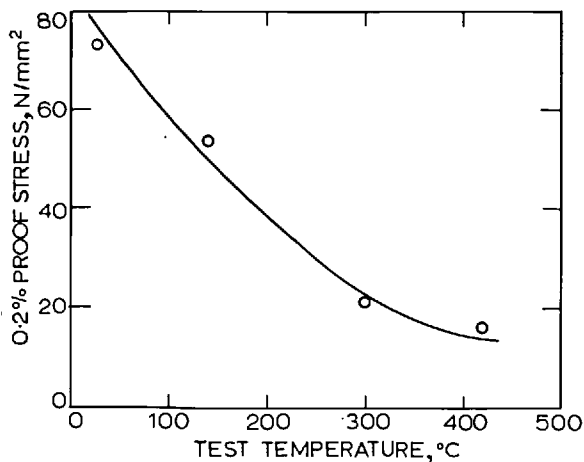
This indicates that at the higher test temperature the form and distribution of precipitate have little effect on resistance to deformation. The alloy is clearly not suitable for high-temperature application, its properties being greatly inferior to those reported previously for commercially pure atomized powders.<sup>1</sup>

Fig. 6 shows the relationship between mechanical properties and extrusion ratio at an initial extrusion temperature of 540°C. This figure does not show optimum values since these are obtained according to Fig. 5 at ~250°C. Nevertheless, the extrusion ratio does affect mechanical properties and the maximum proof stress (86 N/mm<sup>2</sup>) is considerably higher than that for the normal alloy.

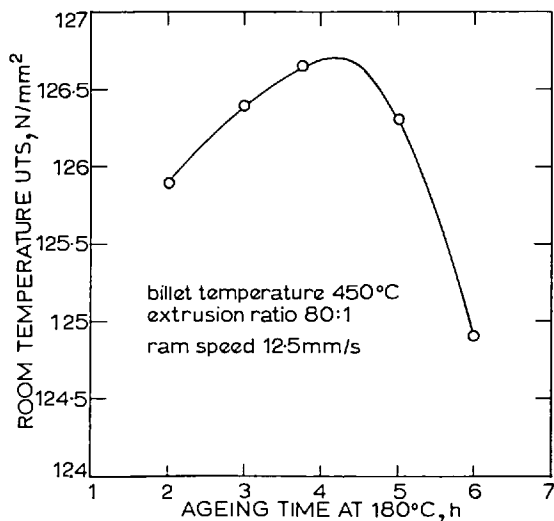
The significant effect of the temperature on the proof stress values is shown in Fig. 7 which is of the same form as that predicted by Seeger's<sup>11</sup> analysis. Thus the governing equation may be written:

$$\sigma = \sigma_A + \frac{[U_0 - GT \ln(\dot{\epsilon}_0/\dot{\epsilon})]}{V_1} \dots \dots \dots (5)$$

which can be regarded as consisting of: a thermally activated component (the second term) due to short-range obstacles such as forest dislocations, solute atoms, and lattice friction effects; and an athermal component,  $\sigma_A$ , due to long-range



7 Flow stress as function of temperature.



**8** Effect of ageing time on strength of press-quenched extrudate.

obstacles such as large precipitates, oxide dispersions, etc. Thus for powder material we would expect  $\sigma_A$  to be larger, and Fig. 7 gives a value of 15 compared with 7–8 N/mm<sup>2</sup> for the normal alloy.

The material exhibited no correlation between extrusion process parameters and notch ductility (Izod) values. However, the results from some 20 tests showed little scatter and gave an impact value of 42.17 N m (31.13 lbf ft) which is almost identical to that reported for cast and wrought material (31 lbf ft nominal).

#### Heat-treatment studies

The alloy investigated is the most widely used precipitation-hardening alloy and is usually subjected to one of three differing heat-treatments designated—

TB: solution heat-treated only, involving soaking at 500–560°C, and quenched.

TE: solution heat-treated, quenched, cold-worked, and naturally aged.

TF: solution heat-treated, quenched, and subsequently precipitation-treated for several hours (depending on geometry) at ~180–200°C.

A transformation of the type: *needle-shaped zones* → *rods* → *platelets of equilibrium  $Mg_2Si$*  occurs with increasing ageing time.

Typical properties for the cast and wrought alloy for the differing heat treatments are given in Table III.

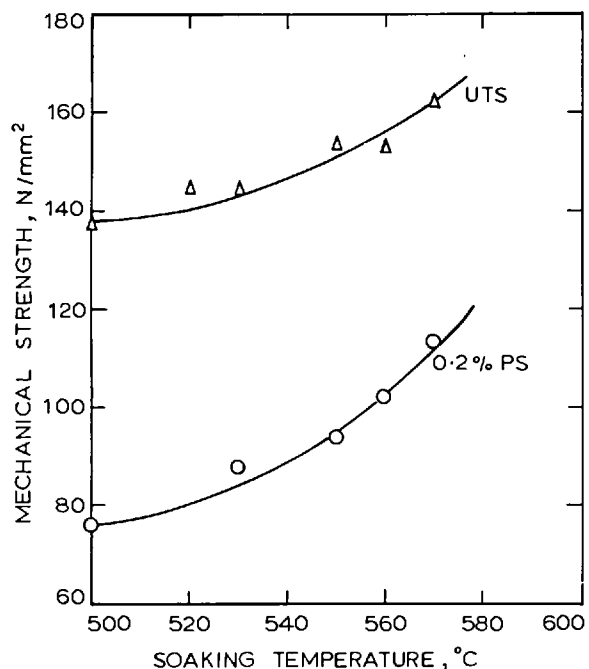
The most common treatment in industrial usage is in fact a compromise. The product is press-quenched (i.e., the heating and extrusion of the billet replaces the solution treatment) and subsequently aged. A homogenization treatment before extrusion serves to organize the precipitate

**Table III** Properties of Alloy 50S

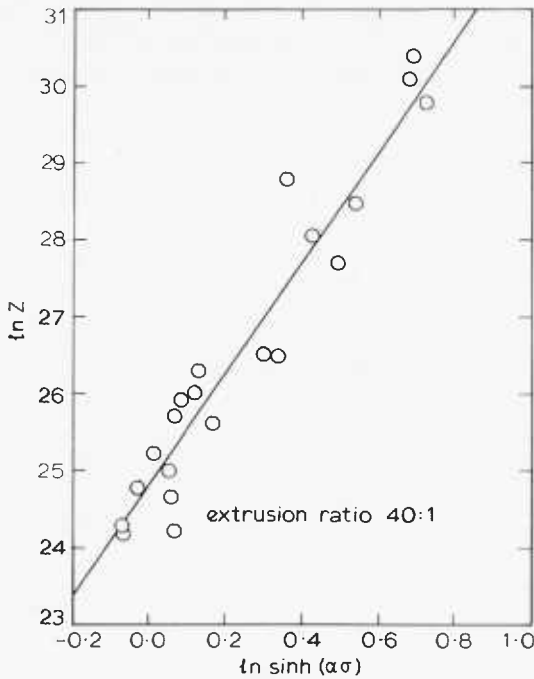
Condition	Proof stress, N/mm <sup>2</sup>	Ultimate stress, N/mm <sup>2</sup>	Elongation, %
TB	70	120	13
TE	110	150	7–8
TF	130–160	150–185	6–8

so that it is taken into solution during the extrusion cycle and this also ensures that minimum die pick-up occurs during the process. When this procedure was applied to the extruded powder material a weakening of the product occurred (see Fig. 8). This is almost certainly due to the omission of the usual homogenization treatment before extrusion, preventing dissolution of the silicide precipitate. The  $Al_2O_3$  layer surrounding each particle will of course also hinder this dissolution. Thus only a small volume fraction of  $Mg_2Si$  precipitate is available for strengthening and Fig. 8 indicates that this grows to optimum size during the initial four hours of treatment. Longer treatment involves further precipitate growth and the total variation in strength is an indication of the low level of activity during the process. The weakening effect occurs because the overall precipitate strengthening is less than the softening effect due to static recovery. It may be possible to overcome this effect by compacting to higher densities (thus, it is hoped, breaking up the  $Al_2O_3$  layer) and carrying out a pre-extrusion solution soak, but it is doubtful whether sufficient disintegration of the oxide layer would occur. Even if such action succeeded it would still be difficult to prevent reoxidation during heat-treatment. Nevertheless this line of approach is at present being pursued.

The product was also subjected to a TF-type heat-treatment involving solution soak, quench, and age. Fig. 9 indicates that substantial strengthening occurs, the properties roughly corresponding to the normal alloy TE treatment but with much better elongation properties (from 15 to 20%). Clearly the oxide barriers have been completely broken by the massive shearing in the deformation zone during extrusion and dissolution of the silicide can now occur during the solution treatment. On subsequent ageing there is sufficient volume of precipitate to cause a substantial increase in strength. The magnitude of the elongation figures suggests that optimum properties have not yet been attained and work continues on varying soaking and ageing times.



**9** Effect of soaking temperature on strength of 3-stage heat-treated extrudate.



10 Variation of ln Z with ln sinh (ασ).

**Thermal activation**

It has been shown previously<sup>12</sup> that the extrusion of light-alloy metal powders is a thermally activated process governed by the general Zener–Holloman equation:

$$Z = \dot{\epsilon} \exp \frac{\Delta H}{GT} = A (\sinh \alpha \sigma)^n \dots \dots \dots (6)$$

It is generally recognized that the applicability of this equation signifies the mechanism by which metals deform and thus it was of interest to examine the behaviour of the alloy according to this thesis.

Since flow stress, strain rate, and temperature are interconnected in a complex manner it is difficult to evaluate the parameters in the above Zener–Holloman equation. The Z value obviously is not constant in the deformation zone since the strain rate, temperature rise, and flow stress all vary incrementally. Since it is difficult to simplify the problem it is general practice to insert average values into the equation. It is remarkable that excellent results can usually be achieved in this way. Thus the strain rate was calculated using a modified version of Feltham’s formula:

$$\dot{\epsilon} = \frac{6CV(\tan \omega)D_B^2}{D_B^3 - D_E^3} \dots \dots \dots (7)$$

and the temperature at the end of extrusion by the integral profile method<sup>8</sup> as modified by Sheppard and Wood,<sup>13</sup> giving:

$$\Delta T = \frac{0.9 L_E V t - T_D C_1(t)}{C_2(t)} \dots \dots \dots (8)$$

The average flow stress was calculated using the minimized upper bound method described in the companion paper<sup>9</sup> but assuming a linear flow-stress variation throughout the quasi-static deformation zone such that it is possible to write:

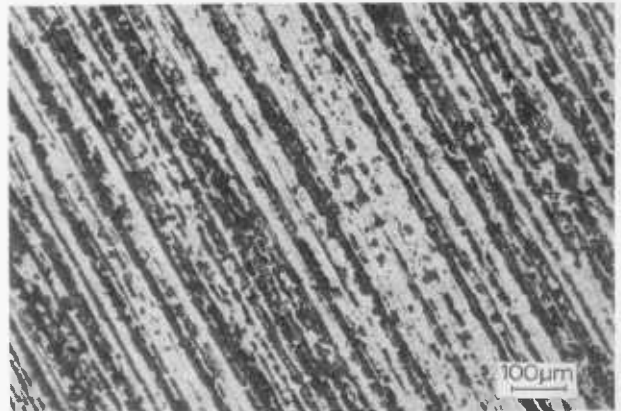
$$p = C_3 \sigma_{\text{exit}} \dots \dots \dots (9)$$

The flow stress, σ, may then be calculated by noting the relationship between C and C<sub>3</sub>.

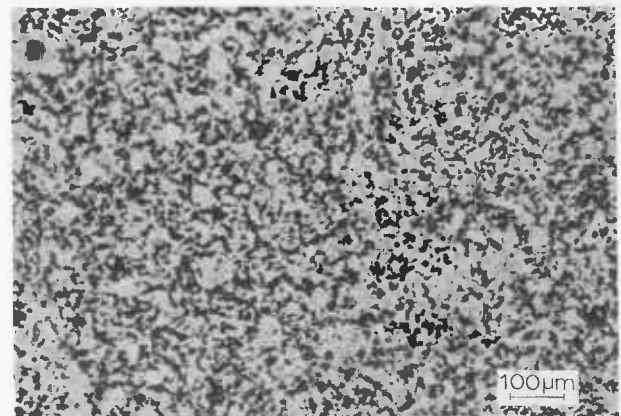
The constant, α, then becomes the remaining unknown. This may be found approximately by noting that at constant temperature and low stresses the relationship between strain rate and stress may be written  $\dot{\epsilon} = A_3 \sigma^{n_1}$  and at high stress  $\dot{\epsilon} = A_4 \exp \beta \sigma$  in which A<sub>3</sub> and A<sub>4</sub> are temperature-dependent. The value of α is then given by the magnitude of β/n<sub>1</sub>. From data plotted in such a manner the value of α was found to be ~0.02. Regression analysis to give a best fit when investigating α between 0.015 and 0.025 gave an α value of 0.022. The value of ΔH is extremely difficult to calculate since it requires extrusion data at constant strain-rate and constant temperature. The interdependence of these two variables has already been noted. However, previous work has indicated a value of 150 000 J/mol which is in agreement with most published literature. This value was adopted since if it were radically in error good correlation with eqn. (6) could not be expected. Fig. 10 shows the result of this exercise giving a slope n=7.2849 and constant A=5.988 × 10<sup>10</sup> and hence eqn. (6) becomes

$$Z = 5.988 \times 10^{10} (\sinh 0.022 \alpha \sigma)^{7.2849} \dots \dots \dots (10)$$

The nominal stress-exponent value found for cast and extruded material varies between 4 and 5 and hence it is clear that the powder material is much less strain-rate sensitive. This observation may be explained by the model proposed<sup>1</sup> for the deformation mode during extrusion, in



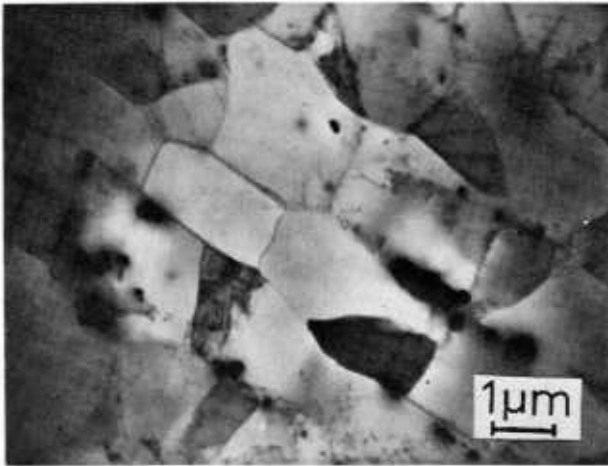
11(a)



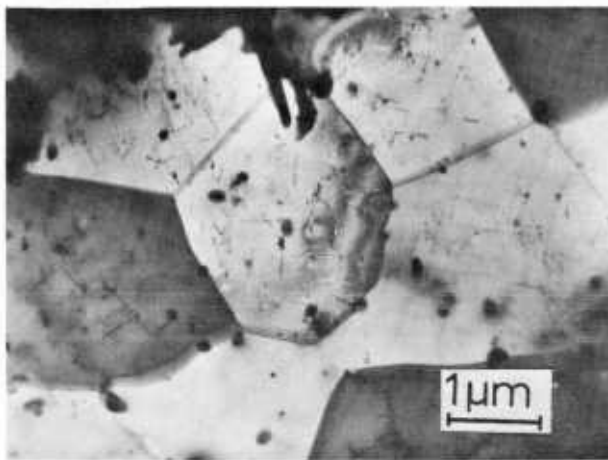
11(b)

Extrusion ratio=80:1 Initial temperature=450°C Ram speed=3.5 mm/s.

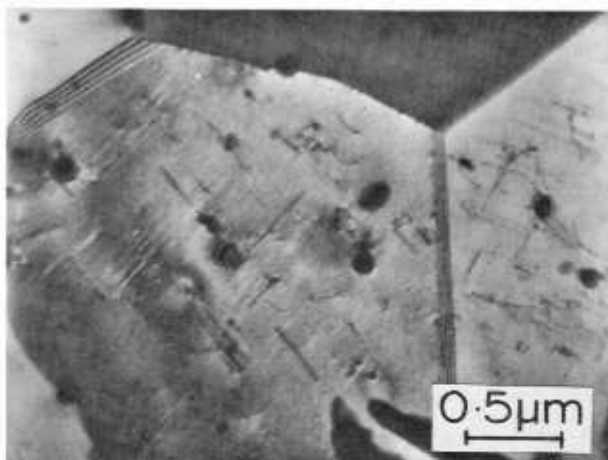
11 Optical micrographs of extruded product: (a) longitudinal; (b) transverse.



12(a)



12(b)



12(c)

Extrusion ratio = 40:1 Initial temperature = 250°C Ram  
speed = 4.5 mm/s.

12 Transmission electron micrographs of extruded product:  
(a) longitudinal; (b) and (c) transverse.

which the material is gradually built by a welding and shearing mechanism, achieving final properties as it passes the final exit discontinuity. It also implies that, provided satisfactory surface finishes can be achieved, it should be possible to obtain higher productivity using powder material than when utilizing conventionally cast billets. The strain-rate sensitivity is, however, higher than that reported previously for powder material and this would almost certainly be due to the more refined temperature calculations used in this work. The value of  $A$ , which is termed an alloy factor, is almost identical to that reported<sup>13</sup> for super-purity aluminium ( $5.62 \times 10^{10}$ ). This is somewhat surprising because evidence<sup>7</sup> suggests that alloy additions should decrease the value of  $A$ . Thus the oxide content would be expected to reduce considerably the value of this constant. Nevertheless the order of magnitude is quite clearly correct.

#### Extrudate structure

Examined under the optical microscope, the product exhibited a fibrous cold-worked structure as shown in Fig. 11(a). The original powder particles can be seen to have been elongated in the direction of extrusion and measurements revealed that the elongation was approximately proportional to the extrusion ratio. This cold-worked microstructure was produced in extrusion performed above the recrystallization temperature of the alloy and the transverse microstructure (Fig. 11(b)) confirms the equiaxed nature of the structure. It was not possible to detect any deviation from this type of structure over the whole range of extrusions investigated, confirming that a substructure must be present and supporting the thermal activation evidence that dynamic recovery is the prevailing deformation mechanism.

Fig. 12 shows typical microstructures observed under the transmission electron microscope. The discontinuous nature of the original oxide film is evident in Fig. 12(a) and original particle boundaries may still be detected and identified by the drawn-out debris of this original oxide. The apparent presence of oxide in the centre of grains is attributed to the orientation selected during the thinning operation, such oxide being the remains of that surrounding a neighbouring grain. The sub-grains are quite clearly elongated, indicating their formation in the deformation zone before exit from the die. This is conclusive proof of dynamic recovery. The sub-grain (or cell) walls are almost perfect although one or two pinned dislocations can be identified. Figs. 12(b) and (c) are transverse sections clearly illustrating the equiaxed sub-grains. Dislocation pinning at precipitate and oxide particles is more easily distinguishable in these sections. Since the precipitates are apparently aligned in the extrusion direction this is direct evidence that pinning occurred during extrusion rather than on subsequent air cooling. It was noticeable that under the electron microscope high-angle boundaries were all sharply defined and even at the highest temperatures there was no evidence of any incipient melting. This clearly indicates that local temperatures due to pressure welding are not excessive and true 'cold' welding must be the dominant cohesion mechanism.

#### CONCLUSIONS

1. Additional 'breakthrough' pressure is required for the extrusion of this heat-treatable alloy. This pressure appears to be strain-rate sensitive for powder materials.
2. The greater useful-work/redundant-work ratio is consistent with the premise that 'deformation energy' is a substitute for sintering energy.
3. As-extruded properties are far superior to those of a conventionally produced alloy.

4. The powder material is heat-treatable. More work is required to optimize this process.

5. The powder process is a thermally activated process, the dominant mechanism being dynamic recovery.

#### ACKNOWLEDGEMENT

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# Hot re-pressing of powder made from mild steel machining swarf

B. Lengyel, C. L. Tan, and G. G. Teh

Since economic studies indicate that powder produced from mild steel machining swarf in a cryogenic hammer mill is significantly cheaper than that made by any other method, it was decided to investigate whether powder-metallurgy products of acceptable properties could be obtained from such powder by a number of alternative methods. The powder was first isostatically compacted, either with or without prior heat-treatment, at room temperature at pressures of up to  $1.38 \text{ GN/m}^2$  ( $200\,000 \text{ lbf/in}^2$ ). The cylindrical preforms were then used to produce specimens by the alternative routes of sintering, or hot forging, or sintering followed by hot forging at small strains. The density and tensile properties of the swarf powder metal were determined as a function of the process variables. By using certain processing routes the density closely approached that of the parent metal while elongation and ultimate tensile strength remained relatively lower, partly because the limited capacity of the forging machine restricted the plastic strain and hence bonding of the particles, partly owing to contamination of the swarf powder. The presence of non-metallic inclusions caused considerable scatter in the results.

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The reclamation of machining swarf by comminution and subsequent powder compaction is attracting considerable industrial interest. While very few details of such work have so far been published, it is known that both steel and cast iron swarf powder metals are used in industry and the utilization of non-ferrous metal swarf via a similar route is being considered.<sup>1,2</sup>

A recent economic study showed that low-cost swarf powder can be obtained by pulverizing mild steel swarf in a cryogenic hammer mill.<sup>3</sup> More recently this method was compared with the water atomization of steel swarf,<sup>4</sup> by considering the main processing stages shown in Chart I.

A modified present-worth method of calculation was used, similar to that developed earlier,<sup>5</sup> with costs estimated from data obtained from industrial sources. Assuming 30% return on investment, the calculations give the following costs per tonne of mild steel swarf powder:

Cryogenic pulverization	
without reclamation of cutting oil	£73.42
Water atomization	
without reclamation of cutting oil	£112.72
Cryogenic pulverization	
with reclamation of cutting oil	£53.00
Water atomization	
with reclamation of cutting oil	£92.20

It is obvious that cryogenic pulverization is the cheaper of the two methods. However, mild steel swarf powder produced by cryogenic comminution is rather coarse (medium particle size  $\sim 450 \mu\text{m}$ ) compared with the medium particle size of iron powder of  $\sim 70 \mu\text{m}$  produced by water atomization. The cost difference is due to the higher production costs and relatively large capital cost of the water atomization plant. The energy cost of atomization is particularly high, which is not surprising since the process of melting metals is energy-intensive. Indeed, more direct methods of swarf recycling are sought, mainly because the present practice of remelting it as light scrap in steel making consumes a great deal of energy, so it would be difficult to justify the replacement of one energy-intensive process with another.

The relative importance of the various cost factors is shown in Fig. 1. The slope (sensitivity) of the initial capital cost line of the water atomization process is much steeper than that for cryogenic pulverization. This is also true of the annual production cost lines. The cost of swarf affects both processes equally, as indicated by the identical slope of the swarf cost lines.

Thus the results of two economic studies prove conclusively that the relatively coarse mild steel swarf powder produced in a cryogenic hammer mill is a cheap basic material, and therefore this route of recycling would be economically attractive if simple and reliable ways could be found to convert it into a swarf powder metal of acceptable quality. To find such process routes was the aim of the present investigations.<sup>4,6</sup>

## EXPERIMENTAL METHOD

The swarf powder, either with or without prior heat-treatment, was isostatically compacted at room temperature into cylindrical billets of  $\sim 18 \text{ mm dia.} \times 60 \text{ mm long}$ . The heat-treatment was carried out in a vacuum furnace using oxygen-free nitrogen atmosphere at  $850^\circ\text{C}$  for 60 min. The preforms so obtained were then forged at elevated temperatures either with or without prior sintering. Sintering was at  $1150^\circ\text{C}$  for 30 min *in vacuo*.

Two forging temperatures were used. Each preform was coated with Dag glass lubricant and heated for 15 min to  $1000$  or  $1150^\circ\text{C}$  in an oxygen-free nitrogen atmosphere before being transferred from the furnace to the forging machine. About 30 s elapsed as the preform was taken out from the furnace until forging began. This time interval was

# Analyses of pressure requirements for powder compact extrusions

T. Sheppard and H. McShane

Three possible theories predicting pressures for powder extrusion are developed, based upon upper-bound analysis. It is shown that a plane-strain analogue does not predict acceptable pressures, but theories based upon an observed physical field or alternatively a minimized axisymmetric approach agree with pressures observed during extrusion. The more complex observed field gives the most accurate assessment. All theories predict curves of differing slope to that observed and it is suggested that the stress/strain/strain-rate relationships require more careful experimental appraisal.

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The production of 100% dense material by extrusion of atomized powders has been described<sup>1-5</sup> for copper, nickel, and aluminium alloys. Sheppard and Chare<sup>1-3</sup> have shown qualitatively that the pressure requirements for such a process should be lower than those for a similar process utilizing cast billets, and Sheppard<sup>4,5</sup> has shown experimentally that for Ni and Al alloys this thesis may be proven. However, it should be noted<sup>6</sup> that for low stacking fault energy alloys in which dynamic recovery is unlikely to occur the pressure reduction may not be very great.

There is no reported literature analysing the process, although techniques exist which can be applied to the problem. The difficulty arises because of the variation in physical properties throughout the quasi-static deformation zone. Thus upper-bound techniques, which assume that all deformation is effected at velocity discontinuities throughout the zone, offers an attractive method of dealing with the analysis. This technique was first proposed by Johnson<sup>7</sup> who assumed a homogeneous material and developed the theory for low extrusion ratios (i.e. 4). Later Sheppard and Raybould<sup>8</sup> extended the analysis to higher ratios and presented minimized solutions showing good agreement with experimental work.

The work reported here is part of a programme investigating composite materials and the experimental work is reported in a companion paper.<sup>9</sup> The matrix material selected for primary investigation was an aluminium-rich magnesium silicide alloy. Three differing methods of calculation are presented (each based upon an upper-bound analysis) and are compared with experimental results.

## THEORY

### Extrusion pressures

The most widely used formulae to predict extrusion loads are those developed by Johnson<sup>7</sup> and are of the form

$$\frac{p}{\sigma} = a + b \ln R \dots \dots \dots (1)$$

It has been shown<sup>2</sup> that this form of equation may also be applied to powder extrusion but the magnitude of the flow stress,  $\sigma$ , is difficult to assess and the constants  $a$  and  $b$  are dissimilar to those observed for as-cast billet extrusion. There is a very clear assumption that the extrusion pressure is dependent only upon reduction and does not vary with differing flow patterns and die shapes associated with various types of material.

An alternative approach based on the flow of metal through the die is possible using slip-line field or load-bounding techniques which are strictly applicable only to processes in which plane-strain conditions obtain. It is possible, however, to apply these methods to the axisymmetric case<sup>8</sup> and the upper-bound approach is more flexible because there is not the requirement of stress equilibrium. The solution must commence with a proposed deformation field which may be derived from practical observations or perhaps suggested by a slip-line field solution. Subsequently the problem becomes one of deriving a rigid triangle hodograph such that the boundary-velocity conditions are satisfied. Since the load calculated will then be higher than that observed it is necessary either to investigate other flow fields, and hence select a minimum load, or to minimize the load by presenting the result in a manner which will enable mathematical techniques to be utilized.

Fig. 1 shows a simple deformation field completely defined by the angles  $\omega$  and  $\phi$  and by the depth of the deformation zone  $x$ . Also shown is the hodograph (or velocity field) which must satisfy the deformation conditions. We may assume that the ram is advancing with unit velocity and that material to the rear of ABC has been compacted to 100% density but has received no shear deformation. Thus material to the rear of ABC will have little or zero shear strength. On encountering the velocity discontinuity AB which, from the hodograph, is of magnitude  $\bar{2}3$ , the powder material is subjected to substantial shear deformation and is confined to flow parallel to AO with velocity  $\bar{1}3$ . Thus all metal in triangle ABO is assumed to attain this velocity and direction. This shear deformation is theoretically assumed to occur instantaneously in zero distance which clearly is impossible. It is assumed that the effective shear strength of the material may be related to the deformation before AB (i.e. = 0) and that after AB. The material next encounters discontinuity BO of magnitude  $\bar{3}4$ , effecting a velocity in direction  $\bar{1}4$ , and finally velocity discontinuity OC ensures that metal leaves the die such that  $\bar{1}5$  is equal to the plane-strain extrusion ratio. It is important to note that axisymmetric calculations

**Nomenclature**

$A_{ij}$	area of discontinuity $ij$
$a$	constant in typical extrusion-pressure formulae
$AB$ etc.	length of discontinuity boundary
$\overline{23}$ etc.	corresponding value of velocity discontinuity (or velocity 'jump')
$k$	shear yield stress
$k_{ij}$	shear yield stress at discontinuity $ij$
$p$	extrusion pressure
$R$	extrusion ratio
$s$	length of velocity discontinuity boundary
$U_{ij}$	velocity perpendicular to velocity discontinuity $ij$
$V_{ij}$	magnitude of velocity discontinuity $ij$
$dw/dt$	rate of energy dissipation
$x$	parameter specifying physical field
$\dot{\epsilon}$	strain rate
$\epsilon_{ij}$	strain at discontinuity $ij$
$\bar{\epsilon}_{ij}$	total equivalent strain at discontinuity $ij$
$\gamma_{ij}$	shear strain at discontinuity $ij$
$\phi, \omega$	angles specifying physical field
$\sigma$	flow stress
$\sigma_o$	strain-rate-dependent stress

Differentiation of eqn. (3) is complicated for powder materials by the inclusion of shear-stress values varying at each discontinuity. It may, however, be assumed that the values of  $\omega, \phi,$  and  $x$  giving minimum pressure for constant shear-stress conditions will also yield the minimum pressure for powder compact extrusion. Thus eqn. (3) could be written

$$\text{Load} = Ak \left[ 2x + \frac{2ax}{d} + \frac{2a^2}{x \sin^2 \omega} + \frac{2ad}{x \sin^2 \phi} - 3a \cot \omega - 3a \cot \phi \right]$$

Differentiation with respect to  $x$  and equating to zero yields

$$x^2 = \frac{a^2}{(1+a/d) \sin^2 \omega} + \frac{ad}{(1+a/d) \sin^2 \phi}$$

Differentiation with respect to  $\omega$  yields

$$\cot \omega = 3x/4a$$

and with respect to  $\phi$  yields

$$\cot \phi = \frac{3x}{4d}$$

Obviously  $\omega$  and  $\phi$  are not independent of  $x$  in this way and iteration is required to ensure accurate minimization of these parameters.

Analyses such as that presented above may be used to obtain the pressure requirements for axisymmetric extrusion because Thomsen<sup>10</sup> has shown that (a) the material flow is similar for identical geometrical configurations, and (b) the magnitude of the velocity during axisymmetric extrusion is the square of the velocity during plane-strain extrusion. There is much experimental evidence to support this second conclusion and it quite clearly satisfies the velocity boundary conditions.

Hence for axisymmetric extrusion the surface area of the discontinuities must be considered, and the magnitude of the velocities must be squared. Hence a revised eqn. (3) will be

$$p \times \pi(a+d)^2 \times 1 = [k_{AB}A_{AB}(\overline{23})^2 + k_{BC}A_{BC}(\overline{24})^2 + k_{BO}A_{BO}(\overline{34})^2 + k_{OC}A_{OC}(\overline{45})^2 + k_{AO}A_{AO}(\overline{13})^2] \dots \dots \dots (4)$$

are based upon the similarity between the material flow obtaining in that and plane-strain processes. Hence the ratio  $(a+d)/d$  must be made equal to  $\sqrt{R}$ .

The rate of energy dissipation at each discontinuity is given by the product of the length (or area) of the discontinuity, the magnitude of the discontinuity, and the shear yield strength of the metal. Thus for linear discontinuities we may write

$$\frac{dw}{dt} = \Sigma k \cdot v \cdot s \dots \dots \dots (2)$$

The plane-strain solution may then be written (considering unit depth) pressure  $\times$  area  $\times$  velocity =  $\Sigma k \cdot v \cdot s$ .

$$p \times (a+d)(1) \times 1 = \Sigma k \cdot v \cdot s$$

$$p(a+d) = [k_{AB}AB \overline{23} + k_{BC}BC \overline{24} + k_{BO}BO \overline{34} + k_{OC}OC \overline{45} + k_{AO}AO \overline{13}] \dots \dots \dots (3)$$

The lengths and velocities may be specified in terms of  $\omega, x,$  and  $\phi$ :

$$AO = \frac{a}{\sin \omega}, \quad \overline{13} = \frac{a}{x \sin \omega}$$

$$OC = \frac{d}{\sin \phi}, \quad \overline{45} = \frac{a}{x \sin \phi}$$

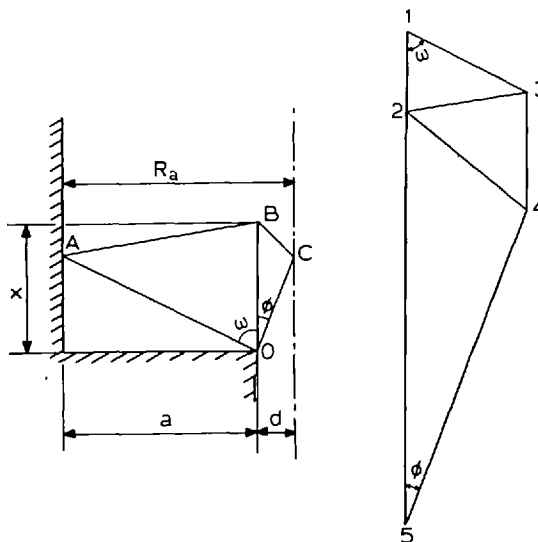
$$BO = x, \quad \overline{34} = \left[ 1 + \frac{a}{d} - \frac{a}{x} \cot \omega - \frac{a}{x} \cot \phi \right]$$

$$AB = \left[ \frac{a^2}{\sin^2 \omega} + x^2 - 2ax \cot \omega \right]^{\frac{1}{2}},$$

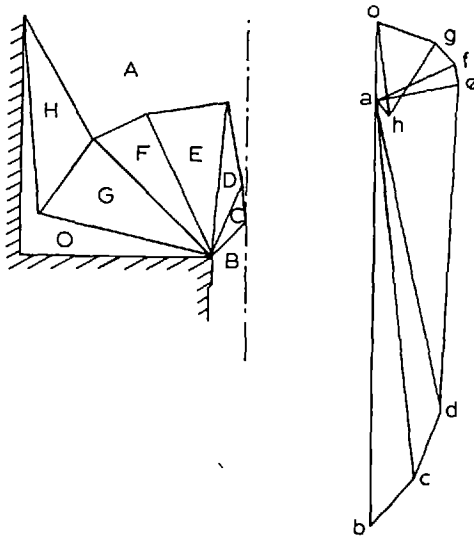
$$\overline{23} = \frac{1}{x} \left[ \frac{a^2}{\sin^2 \omega} + x^2 - 2ax \cot \omega \right]^{\frac{1}{2}}$$

$$BC = \left[ \frac{a^2}{\sin^2 \phi} + x^2 - 2dx \cot \phi \right]^{\frac{1}{2}},$$

$$\overline{24} = \frac{a}{xd} \left[ \frac{d^2}{\sin^2 \phi} + x^2 - 2dx \cot \phi \right]^{\frac{1}{2}}$$



1 Upper-bound solution based upon simple physical field. Extrusion ratio 40:1.



2 Upper-bound solution based upon observed physical field. Extrusion ratio 40:1.

The velocities  $\bar{13}$  etc are identical to the plane-strain analysis and the surface areas by simple geometry become

$$A_{OC} = \frac{\pi d^2}{\sin \phi}, \quad AC = \frac{\pi a(a+2d)}{\sin \omega}, \quad BO = 2\pi xd$$

$$AB = \pi(a+2d) \left[ \frac{a^2}{\sin^2 \omega} + x^2 - 2dx \cot \omega \right]^{\frac{1}{2}}$$

$$BC = \pi d \left[ \frac{d^2}{\sin^2 \phi} + x^2 - 2dx \cot \phi \right]^{\frac{1}{2}}$$

Since metal flows for plane-strain and axisymmetric geometries are similar, the minimized values of the geometric variables will also be identical. Hence the values of  $x$ ,  $\omega$ , and  $\phi$  obtained in the plane-strain analysis may be used in the axisymmetric case.

Both of the methods developed above suffer from the drawback of a limited discontinuity pattern. Thus deformation occurs in three large steps. An alternative approach is to use an observed flow pattern and to build coherent discontinuities about this pattern. This will have the advantage of many discontinuities which for powder material, where the shear yield stress is strongly dependent upon deformation, may be important. Such a physical field with its accompanying hodograph is shown for a reduction ratio of 40:1 in Fig. 2. The extrusion pressure is then given by

$$p \times \frac{\pi(a+d)^2}{4} \times 1 = [k_{AH} A_{AH}(ah)^2 + \dots + k_{BC} A_{BC}(bc)^2] \dots \dots \dots (5)$$

**Shear yield stress**

Equations (3), (4), and (5) each require that the shear stress of the compact be predicted within each rigid zone and thus at each discontinuity. The relative shear deformation at each jump is given by the magnitude of the velocity 'jump' divided by the velocity perpendicular to that jump. The material is also elongated in the direction of its deflected flow and in the three-dimensional case there will be deformations of small magnitude in directions at right-angles to this.

Fig. 3 is identical to Fig. 1 but depicts the relevant parameters. Thus for example the shear deformation at AB

is of magnitude  $\ln(V_{AB}/U_{AB})^2 = \ln(\bar{23}/12')^2 = \gamma_{AB}$  and the deformation on the flow direction  $= \ln(\bar{13})^2 = \epsilon_{AB}$ . At BC the deformations are  $\ln(\bar{24}/14')^2 = \gamma_{BC}$  and  $\ln(\bar{14})^2 = \epsilon_{BC}$ .

The strains in directions perpendicular to these will be an order of magnitude less so that the total equivalent strain may be written

$$\bar{\epsilon}_{AB} = \sqrt{\frac{2}{3}}((\epsilon_{AB})^2 + \frac{1}{2}(\gamma_{AB})^2)^{\frac{1}{2}} \text{ etc.}$$

It should be noted that the use of the velocity-squared relationship is also necessary to determine the shear yield stress to be used in the plane-strain analogue; otherwise the total strain will not correlate with that actually obtaining in the extrusion process. Since the measurement is expressed in terms of natural strain it is also necessary to take the natural logarithm of change in zero time and hence the material must have properties related to those at each side of the discontinuity. This value may best be estimated by taking the mean equivalent strain such that the deformation at each discontinuity is given by

$$\frac{1}{2} \bar{\epsilon}_{AB} \text{ at AB}$$

$$\frac{1}{2} \bar{\epsilon}_{BC} \text{ at BC}$$

$$\bar{\epsilon}_{AB} \text{ at AO}$$

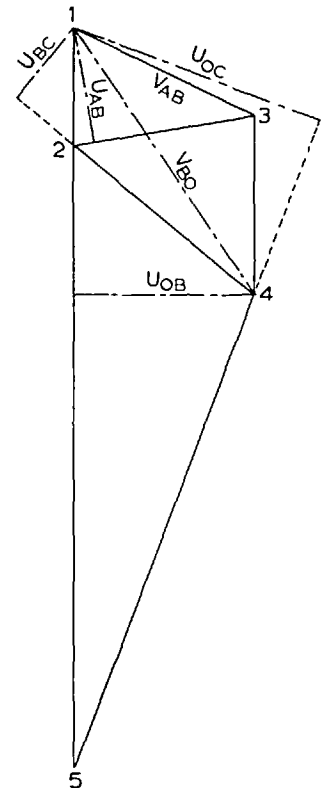
$$\bar{\epsilon}_{AB} + \frac{1}{2} [\bar{\epsilon}_{AB} + \bar{\epsilon}_{BO}] \text{ at BO}$$

$$\bar{\epsilon}_{AB} + \bar{\epsilon}_{BO} + \frac{1}{2} [\bar{\epsilon}_{BO} + \bar{\epsilon}_{OC}] \text{ at OC}$$

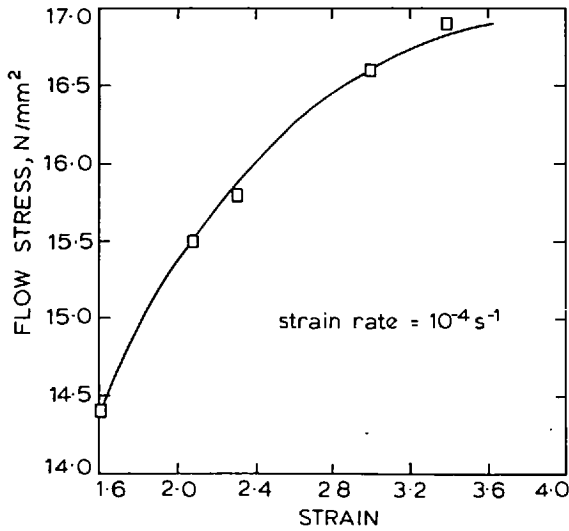
The results of tensile tests, shown for a strain-rate of  $10^{-4} \text{ s}^{-1}$  in Fig. 4, indicate a stress/strain relationship for magnesium silicide powder compacts:

$$\sigma = \sigma_0 \epsilon^{0.21}$$

where  $\sigma_0$  is a strain-rate-sensitive constant.



3 Hodograph of simple field showing strain parameters.



4 Stress/strain relationship at 400°C.

A combination of tensile and torsion tests suggested a relationship between stress and strain rate:

$$\sigma_o = 75 \epsilon^{0.19}$$

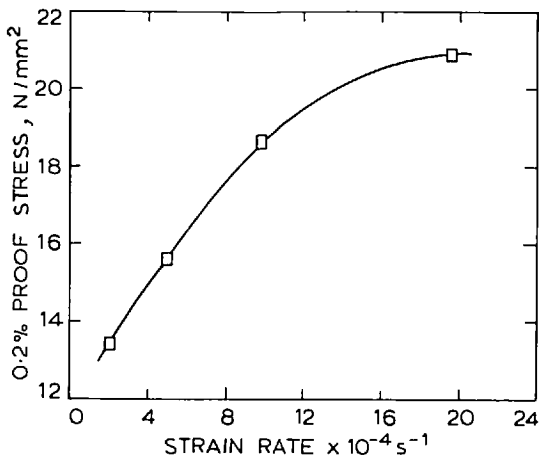
The results of the strain-rate tests giving this best-fit curve are shown in Fig. 5.

Using the Von Mises yield criterion, the shear yield stress at a discontinuity is thus given by

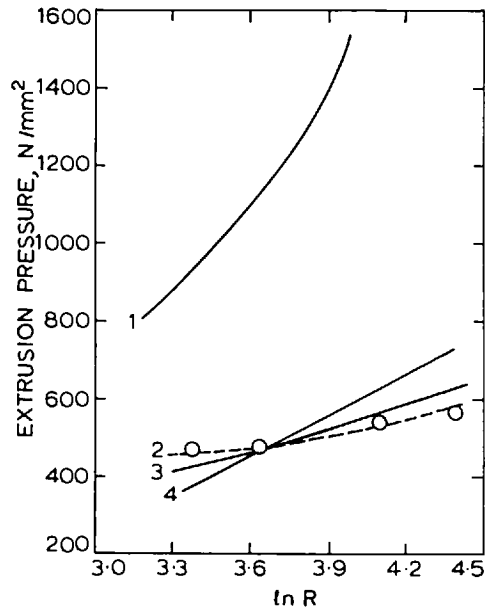
$$k_{ij} = \frac{75}{\sqrt{3}} \epsilon^{0.19} \bar{\epsilon}_{ij}^{0.21}$$

**COMPARISON OF THEORIES WITH EXPERIMENTAL RESULTS**

The plane-strain and axisymmetric extrusion pressure calculated according to eqns. (3) and (4) were programmed for computer and the results are shown in Fig. 6. A program allowing the hodograph shown in Fig. 2 to be drawn automatically is being developed, but for this work the physical fields and hodograph were drawn manually. A sample calculation for an extrusion ratio of 40:1 is given in Table I and the relevant curve has been constructed in Fig. 6.



5 Stress/strain-rate relationship at 400°C.



6 Comparison of experimental and theoretical results: 1=minimized plane strain; 2=experimental; 3=observed physical field; 4=minimized axisymmetric.

The most significant feature in Fig. 6 is the enormous discrepancy between the pressures predicted by the plane-strain analogue approach and those observed in practice. When extruding cast billets this method usually predicts pressures which are ~20% above those actually obtaining. Clearly, for powder compact extrusions the method is not sufficiently accurate.

The minimized axisymmetric solution gives a reasonable correlation with experiment but the predicted slope of this curve is manifestly incorrect and for high extrusion ratios (i.e. > 100) the theory would again be unacceptable.

The best curve is obtained using the approach based upon an observed physical field, although this theory also predicts a curve differing in slope to that observed. It is not clear why this should be so. Certainly for cast materials all three theories would give curves having similar slopes. Most

Table I Specimen calculation using observed physical field at extrusion ratio 40:1

Boundary	$A_{ij}$ , mm <sup>2</sup>	$V_{ij}$ , mm/s	$\bar{\epsilon}_{ij}$	$k_{ij}$ , N/mm <sup>2</sup>	$k_{ij}A_{ij}(V_{ij})^2$
OH	7355	1.22	0.297	56	502 641
OG	6848	0.539	0.359	58.19	214 768
AH	4548	0.033	0.148	48.73	7 314
AF	1156	1.02	0.076	42.64	50 286
AE	376	0.986	0.063	41.08	15 231
AD	238	14.17	1.330	75.60	254 972
AC	18	20.23	1.442	76.83	27 977
HG	2291	1.17	0.508	62.36	167 170
GF	2082	0.118	0.437	60.52	14 868
FE	1291	0.042	0.292	55.45	3 039
ED	736	13.89	1.380	76.16	778 615
DC	2716	0.514	2.776	87.58	122 272
CB	131	0.649	2.964	88.74	7 545

$$k_{ij}A_{ij}(V_{ij})^2 = 2 166 700$$

$$\text{Extrusion pressure} = \frac{2 166 700 \times 4}{\pi \times 75^2} = 490 \text{ N/mm}^2.$$

probably the inaccuracies arise because of insufficient experimental data in determining the stress/strain and stress/strain-rate relationships. The tests were all conducted at 400°C and other temperatures should be investigated using compression as well as torsion and tensile data to determine the relevant flow and shear stresses. Nevertheless the upper-bound based upon an actual deformation (or physical field) shows very good agreement with experimental observation.

### CONCLUSIONS

1. The plane-strain analogue approach is not suitable for predicting powder compact pressure.
2. The minimized axisymmetric upper-bound theory predicts pressures with acceptable accuracy, provided the extrusion ratio is < 100.
3. The upper-bound solution based upon an observed physical field gives very good agreement with observed pressures.
4. The variation between theoretical and experimental curve slopes requires further experimental investigation.

### ACKNOWLEDGEMENT

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# Production of an aluminium-rich magnesium silicide alloy from atomized powder

T. Sheppard and H. McShane

The production of an aluminium-rich magnesium silicide alloy by extrusion from the powder phase is described. The pressure requirements of such a process are investigated and it is shown that the operation may be described as thermally activated. The strain-rate sensitivity is shown to be less than the equivalent cast and extruded material. The mechanical properties of the as-extruded product are far superior to those of the conventional alloy and the powder material is heat-treatable.

Serial No. 426. Manuscript received 15 April 1976. Contribution to a Symposium on 'Hot Processing in Powder Metallurgy' to be held in Eastbourne on 11-13 October 1976. T. Sheppard, MSc, BSc(Eng), MIM, CEng, MIMechE, and H. McShane, BSc(Eng), ARSM, are in the John Percy Research Group, Department of Metallurgy and Materials Science, Imperial College of Science and Technology, London.

Until the advent of modern atomizing techniques the production of components from aluminium parts was hindered to a large extent by the cost of powders. Atomization has overcome this main obstacle, although in general the dispersion-hardening effect is not so pronounced because the  $Al_2O_3$  content is reduced compared with the original SAP-type alloys. An additional obstacle to widespread use of these light alloys is that the oxide dispersion acts as a vacancy barrier during the sintering process, prolonging that operation. A convenient process which ensures that the powder compact forms a homogeneous 100% dense product is extrusion, which can be carried out at high or low temperature with large strains. It has been shown<sup>1,2</sup> that such a process eliminates the sintering operation and it is suggested that energy which is termed 'redundant' in the extrusion of cast ingots is utilized to assemble the powders into solid form. Powder compacts also have an advantage over the normal ingot because less pressure is required<sup>3</sup> to effect extrusion. This is due to the differing rheological nature of the quasi-static deformation zone.

The atomizing process has other advantages in addition to the dispersion-hardening effect of the oxide film which almost invariably develops as an envelope surrounding each particle. The particles are usually cooled at a rate far in excess of those found in the normal continuous casting of aluminium alloys. This rapid cooling gives a fine eutectic, due to nucleation at lower temperatures, and the size and nature of precipitates can be expected to differ. In a heat-treatable alloy the oxide film and the differing nature of the

precipitates may together be expected to alter the heat-treatment reaction. There appears to be no reported literature on this subject.

The Al-Mg-Si system, when aluminium-rich, is among the most widely used aluminium alloys, having architectural and structural applications. The object of the present work was to examine the rate-controlling deformation mechanism, the nature of product densification, the alloy structure, and the effect of oxide dispersions on the ageing process.

## EXPERIMENTAL

### Material

The air-atomized Al-Mg-Si (HE9) alloy powder, supplied by Borax Consolidated Limited, nominally contained 0.5 wt.-%Mg, 0.5 wt.-%Si, balance Al. Initially the powder particle size distribution was obtained using standard 200 mm BS sieves stacked on an Endrock mechanical shaker to sieve a standard weight of powder (175 g) for a specified time (1 h). The results are shown in Table I.

Samples of the powder were analysed by the Imperial College Analytical Services Laboratory. The chemical composition is given in Table II.

### Preparation for extrusion

To facilitate handling at the press, the powder was compacted into billets using a 150 ton Tangye hydraulic press. To ensure easy removal of the billets from the die the tool surfaces were cleaned and painted with a stearic acid-chloroform lubricant before each compaction.

Table I Size distribution of Al-Mg-Si alloy powder

Particle size, $\mu\text{m}$	Weight percent
+355	2.14
-355 + 180	30.77
-180 + 106	25.73
-106 + 90	5.39
-90 + 75	5.20
-75 + 63	5.65
-63 + 53	5.15
-53	19.72

Table II Chemical analysis of powder (remainder Al) (wt.-%)

O	Mg	Si	Cr	Cu	Fe	Mn	Ti	Zn	Ni
0.062	0.33	0.12	0.01	0.014	0.15	0.10	0.014	0.025	0.004

# THE PRODUCTION OF FIBRE REINFORCED ALUMINIUM BY A POWDER EXTRUSION TECHNIQUE

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John Percy Research Group, Department of Metallurgy,  
Imperial College of Science and Technology, London

## SUMMARY

The paper reports results of experiments designed to produce a fibre reinforced alloy using an aluminium matrix and a  $B_2O_3$ ,  $SiO_2$  fibre.

It is shown that during extrusion the glass powder elongates into fibres having a favourable aspect ratio. The relationships between mechanical properties and process variables are reported and include the results of 120 experimental runs. The structure, substructure and bonding are investigated in some detail. It is shown that tensile properties are improved, elongation is acceptable and impact strength only slightly deteriorates when compared with the matrix material.

## INTRODUCTION

Probably the single most important factor precluding the widespread use of fibre re-inforced material is the cost of the production. The most common method of manufacture of composites involves coating of the individual fibres by the matrix material, aligning the coated fibres in a suitable direction and finally bonding by hot or cold pressing. Sheppard and Chare [1] have shown that when extruding aluminium and aluminium alloy powders the particulate nature of the cold compacted billet is retained well into the deformation zone; assuming solid properties only near the exit of this zone. Thus, there is much interparticle movement, cohesion and segregation of particles within this zone. Moreover, individual particles are elongated within the zone and are processed such that their aspect ratio on completion of deformation is related to the extrusion ratio. Thus, if a reinforcing material could be identified which was essentially viscous at extrusion temperature and considerably stronger at room temperature it should be possible to produce a composite by a random mixing process prior to extrusion; the reinforcing fibres being formed as part of the extrusion process.

It was, therefore, decided to investigate a range of glasses as the reinforcing medium. The most suitable appeared to be the boron based group and from these a  $B_2O_3 - 10\% SiO_2$  was selected because:

- (i)  $B_2O_3$  has a low softening point which is much less than the temperatures obtained when extruding aluminium or aluminium alloys. It is thus viscous at processing temperatures.
- (ii)  $B_2O_3$  forms a good bond with aluminium during extrusion. An essential feature which is necessary to

ensure that the fibres do not 'pull-out' of the matrix on application of a load

- (iii)  $B_2O_3$  with 10%  $SiO_2$  is a glass with good mechanical properties and hence a strong composite would be expected.

The later consideration is based quite simply on consideration of the rule of mixing:-

$$\sigma_{COMP} = (1 - x) \sigma_m + x \sigma_G$$

It should be noted, however, that a critical fibre length exists which must be exceeded if the stress on the fibres is to approach their ultimate tensile stress. Shorter fibres produce a stress concentration effect which produces composite failure by decohesion of the fibres with the matrix and hence the potential load bearing capabilities of the fibre are not utilized. Appendix I shows that the critical length for the system reported in this communication cannot be achieved at extrusion ratios less than 10. The main weakness of the glass selected is that its modulus is approximately equal to that of the matrix so that transfer of load within the composite cannot be expected to be 100% efficient.

The experiments reported here, therefore used  $B_2O_3-10\% SiO_2$  in an aluminium rich magnesium silicide matrix. The matrix material was selected because of its widespread commercial use as a medium strength, cheap, extruded aluminium alloy for architectural and other purposes.

This communication compares the properties and structure of the reinforced extrude with those of an undoped product produced from the matrix material. The effect of the process parameters upon the product are reported. Thus, the interrelation of structure and properties with



extrusion temperature, ram speed, extrusion ratio, wt-% glass, tensile test temperature and strain rate are examined

## EXPERIMENTAL WORK

### Materials

The glass of composition 90% B<sub>2</sub>O<sub>3</sub> - SiO<sub>2</sub> was prepared by roller mixing carefully weighed amounts of the constituent oxides which were fused in fireclay crucibles and poured into steel moulds. The product was then crushed, ground and sieved to produce a glass powder. The size distribution of the glass was found by sieving a 200g sample for one hour in a standard sieve shaker.

The air atomized Al-Mg-Si powder was also supplied by Borax Consolidated Ltd., and the particle size distribution was obtained using standard 200mm BS sieves stacked on an Endrock mechanical shaker to sieve a standard weight of powder (175g) for a specified time (one hour). The results of the powder analysis are shown in Table I.

Table I - Particle Size Distributions

B <sub>2</sub> O <sub>2</sub>		SiO <sub>2</sub>		Al-Mg-Si	
Size $\mu$	wt%	Size $\mu$	wt%	Size $\mu$	wt%
+250	9.42	+355	2.14		
+180	20.03	+180	32.91		
+125	33.33	+106	58.64		
+ 90	46.71	+ 90	64.03		
+ 75	52.03	+ 75	69.23		
+ 53	62.86	+ 63	74.92		
+ 45	69.50	+ 53	80.07		
- 45	28.15	- 53	18.72		

Samples of the metal matrix powder were analysed by the Imperial College Analytical Services Laboratory and the chemical composition is given in Table II.

Table II - Chemical Analysis of Powder

(Remainder Al) wt %

O	Mg	Si	Cr	Cu	Fe
0.062	0.33	0.12	0.01	0.014	0.15
Mn	Ti	Zn	Ni		
0.10	0.014	0.025	0.004		

### Preparation of Billets

The glass and metal powders were roller mixed in pre-determined proportions for two hours. The billets were produced by compacting this powder in a 75mm diameter

boronised tool steel ram and die set. Pressures were exerted by a 1.5MN Tangye hydraulic press giving a maximum compacting pressure of 346MN/m<sup>2</sup>. The tool surfaces were lubricated with a mixture of chloroform and stearic acid. After preparation it was necessary to store the billets in plastic containers with silica gel in order to prevent disintegration due to adsorption of water from the atmosphere.

### Extrusion

The extrusions were performed on a 5MN ENEFCO extrusion press after preheating the billets in an air circulating furnace. A disc of solid Al-Mg-Si material was placed in front of the billet as has been reported in previous work<sup>2</sup>. This was found to be more critical than in the extrusion of purely metallic components.

### Examination of Product Properties and

#### Structure

Specimens were prepared from the same location in each extrusion to ensure that structure and property variation along the extrusion did not influence the results. The electron microscope foils were prepared by grinding on water-lubricated silicon carbide paper to a thickness of ~15mm followed by thinning using a standard window technique. The electropolishing was carried out in a solution of 30% Nitric Acid in methanol under a potential difference of 15V and at a temperature of -30°C. The transmission microscopy was carried out on an AEI EM6 100kV electron microscope.

Standard Hounsfield Number 12 specimens were machined for tensile tests performed at room temperature and temperatures up to 400°C. The tensile tests were performed on an Instron Tensile Testing machine fitted with a split furnace for the high temperature tests.

Impact tests on notched specimens were carried out using a Hounsfield balanced impact machine, the mode of testing being identical to the standard Izod test but with a differing specimen geometry. The energy reading given by this machine was therefore multiplied by a factor of 2.4 in order to obtain the corresponding Izod value.

## RESULTS AND DISCUSSION

### Extrusion Processes

The effect of varying temperature and extrusion ratio on the pressure required for extrusion is shown in Fig. 1. Extrusion ratios up to 180:1 were investigated with temperature varying between 250° and 540°C. It is clear that the normal form of extrusion law is obeyed (i.e.  $p=a+b \ln R$ ) and from Fig. 1 the relationships shown in Table III can be obtained.

The ratio of a/b has traditionally been considered to be a measure of the redundant

work occurring during the extrusion process. Such work which does not contribute to a shape change has been shown to contribute to the welding and re-welding process during powder compact extrusion. It is useful to investigate this ratio since in powder extrusion the flow stress varies with strain and strain-rate and it is not easy to define an average flow stress. The value of  $a/b$  for a cast Al-Mg-Si alloy of similar chemical composition is 0.3 at

Table III Pressure - Extrusion Ratio  
Formulae

Temp.	Law	a/b
250°C	$p=179.2+111.0 \ln R$	1.61
350°C	$p=122.0+ 96.1 \ln R$	1.27
450°C	$p= 92.7+ 89.8 \ln R$	1.03
500°C	$p= 61.3+ 91.3 \ln R$	0.67
540°C	$p= 71.8+ 82.5 \ln R$	0.87

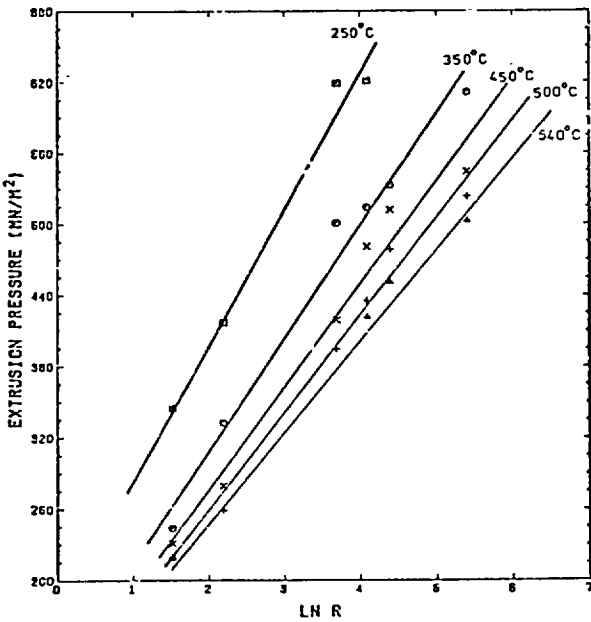


FIG 1 Effect of temperature and extrusion ratio on extrusion pressure

540°C and hence it becomes clear that modes of deformation in cast and powder extrusion are not similar.

The variation of  $a/b$  with temperature is shown in Fig. 2 indicating a linear decrease in proportion of redundant work with increasing temperature. This then would support the thesis that within the redundant work term there is a component which essentially represents the energy required to ensure final cohesion of the powder mass. Clearly local welding will be easier at elevated temperatures and we

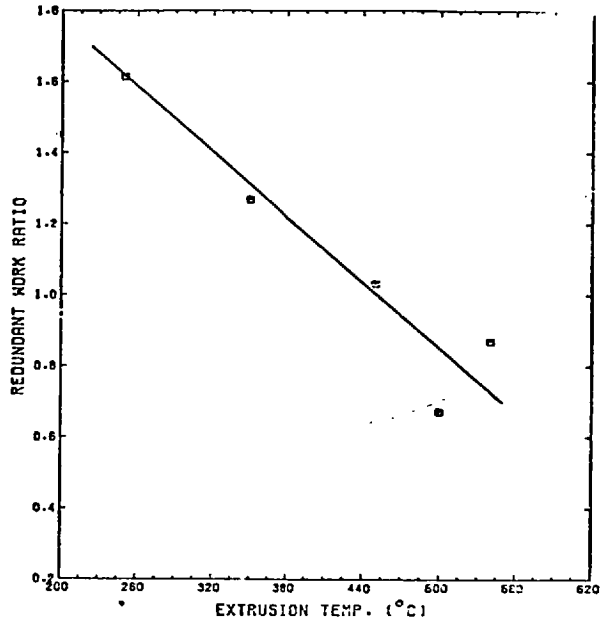


FIG 2 Effect of temperature on redundant work

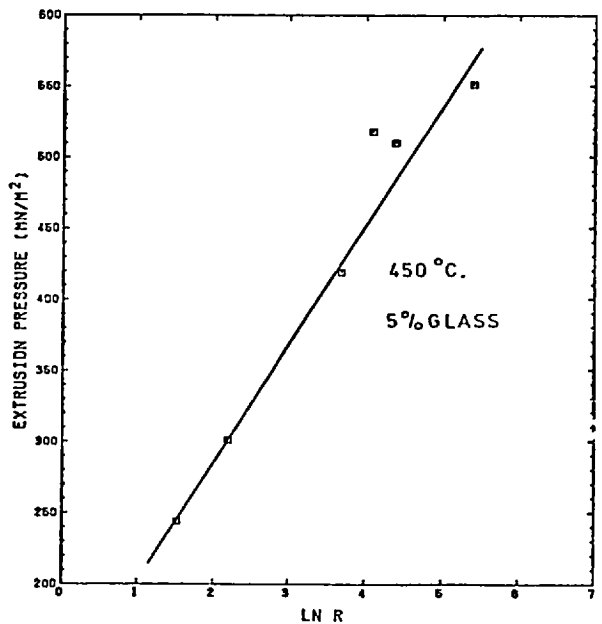


FIG 3 Effect of extrusion ratio on extrusion pressure for a composite billet,

would expect the powder mass to be fully compact at an earlier site in the deformation zone than at lower temperatures. This variation in  $a/b$  ratio with temperature has been observed by Ashcroft and Lawson[3] for cast commercial purity aluminium but they also observed that the ratio remained constant with temperature for an Al-Mg-Zn-Cu alloy. It would appear therefore, that the redundant work is very much a function of alloy micro-behaviour and depends upon the values of  $A$ ,  $n$  and  $\alpha$  in the hyper-

bolitic hot working relationship  $Z=A(\sin h \sigma)^n$  where A,  $\sigma$  and n are constant,  $\sigma$  is the flow stress and Z is the temperature compensated strain rate. It is known that for cast materials the values of A and  $\sigma$  depend upon alloy complexity and it has been shown that these values differ for cast and powder extrusion routes [4]. Fig. 3 shows that the extrusion of composite billets also produces pressure-reduction ratio relationship of the same form giving  $p=48.16+93.10 \ln R$  giving an a/b ratio of 1.05 at 450°C. This would indicate that the addition of 5wt% B<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> has very little effect on the process of extrusion. It is perhaps surprising that the lubricating effect of the viscous glass does not modify the process in some way.

vacancy concentration will result. It is further aided by a natural precipitation hardening process which occurs at room temperature over a period of weeks and is due to a fine precipitate of Mg<sub>2</sub>Si. The room-temperature proof stress varies from 125-75 N/mm<sup>2</sup> over the temperature range investigated which compares with an as-extruded strength of 50-55 N/mm<sup>2</sup> for the normal cast alloy. The elongation varies from 20-25% which is remarkable since the cast as-extruded product is assigned a value of 12% for this property. These high elongation values are compatible with the very fine grain size observed in powder extrusion and which are a direct result of the rapid quench involved in the production of powders. Clearly, dynamic recovery processes have been the dominating deformation mechanism since recrystallisation would have modified the grain size.

The effect of varying the tensile test strain rate is shown in Fig. 5. It is clear that the strain-rate sensitivity of the material is quite different at room

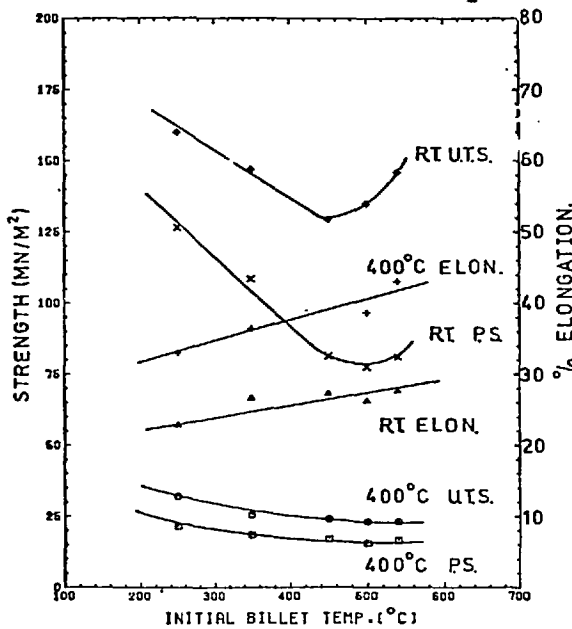


FIG 4 Effect on initial billet temperature on mechanical properties

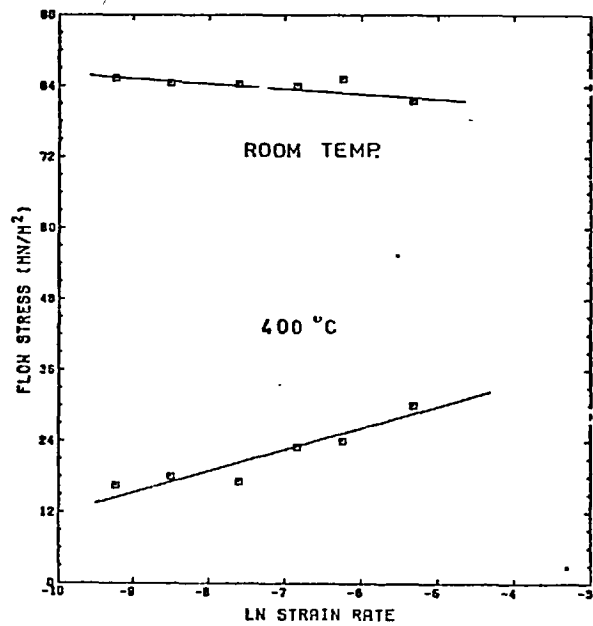


FIG 5 Effect of strain rate on mechanical strength

Mechanical Properties

(i) Matrix Material

The variation in mechanical properties with initial billet temperature is shown in Fig. 4. The room-temperature proof and ultimate stresses show the expected decrease in value with increasing billet temperature (in the range 250-480°C) which is clearly due to softening, probably by dynamic recovery. However, from 480°C to 540°C the trend is reversed; the material becomes stronger and elongations continue to improve. Thus, it is clear that at the higher extrusion temperatures a limited amount of Mg<sub>2</sub>Si is taken into solution and on subsequent air cooling a supersaturated solution with a non-equilibrium

and elevated temperature; the strain rate exponent changing from positive at 400°C to negative at room temperature. This data agrees with that presented by D'antonio et al<sup>3</sup> who noticed a complete change in dependence over the same temperature range and in the same strain-rate range (i.e. < 10<sup>-2</sup> per sec.).

The increase in proof stress with strain rate at 400°C is readily explained by the fact that dislocation generation occurs more easily than does dislocation annihilation by dynamic recovery. The negative exponent observed at room temperature is less easily explained. Lubahn [5]

has reported, also for an Al-Mg-Si alloy, that the strain-rate sensitivity again achieves a minimum at room temperature and observes that it is at this temperature that strain ageing effects are most apparent. Strain ageing effects occur when a material is being plastically deformed and dislocations are pinned by solute atom clusters. The increasing concentration of stress eventually tears the dislocations away from the pinning clusters. Since the stress required to obstruct motion of dislocations is less than that required to free them, the flow is reduced. However, the solute atoms diffuse rapidly and 'repin' dislocations. Thus, the cycle is repeated. If the strain rate is increased, diffusion may not be rapid enough for 'repinning' to occur and the flow stress will be reduced. We should note however, that since serrated yielding was not observed, this explanation may be inadequate.

The effect of powder particle size on extrude mechanical properties was also investigated and it should be noted that no correlation could be found between extrusion pressure and particle size. Fig. 6 shows that decreasing particle size results in increased material properties. This can be related to the existence of a Hall-Petch type relationship in which the particle size becomes the grain size analogy and strength becomes proportional to the reciprocal of the particle dia. to some power  $\chi$  (where  $\chi$  is 1-2). As the particle size decreases the average number of dislocations per unit volume increases because grain boundaries may be formed by dislocations. It is also possible to observe dislocations in as-atomized powders which are presumably due to the high residual stresses due to rapid cooling at the atomizing stage.

(ii) Composite Material

The weight percent fraction of glass in the billet was found to have no significant effect upon the extrusion process variables indicating that attainment of properties occurred at a late stage in the deformation zone.

The significance of glass fraction on mechanical properties is illustrated in Fig. 7. Simple fibre theory and the rule of composites would suggest that strength should vary linearly with the glass content. The ultimate stress appears to agree with such simple theory in the range 0-8 wt% glass. However, above this fraction there is a decrease in strength which occurs because of the disparity in bulk moduli between matrix and glass ( $K_{matrix} = 26.2 \times 10^9 \text{ N/m}^2$ ,  $K_{glass} = 13.8 \times 10^9 \text{ N/m}^2$ ) Thus when the material emerges from the die the glass recovers elastically, significantly more than does the metal matrix and causes premature failure of the mate-

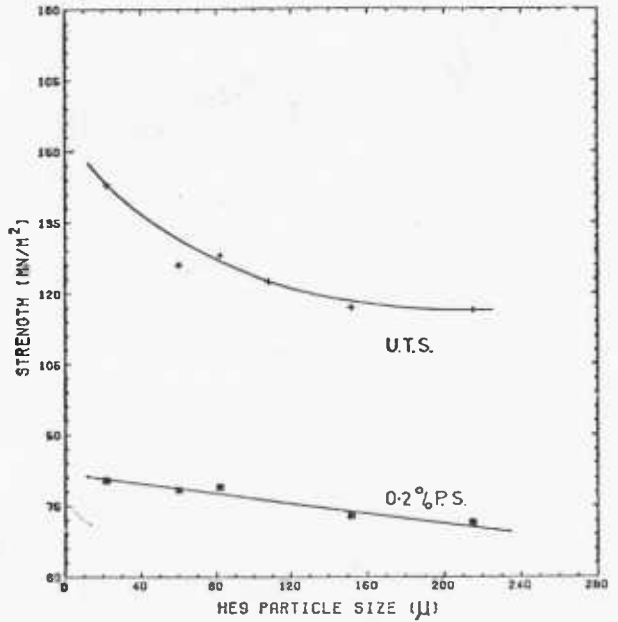


FIG 6 Effect of powder particle size on mechanical properties

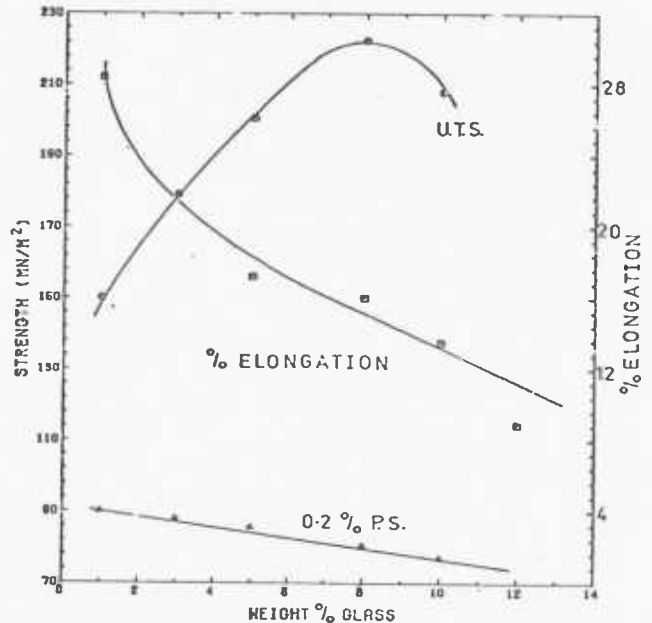
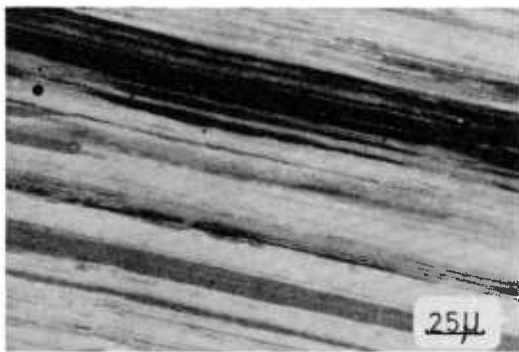


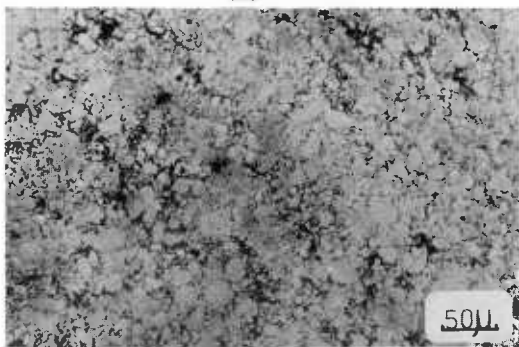
FIG 7 Effect of glass content on mechanical properties

rial. The strengthening would be improved if 'ideal' cylindrical fibres were formed. In fact Fig. 8 shows that formation within the die is far from ideal, the glass being squeezed as a viscous liquid into spaces between adjacent powder particles.

The effect of glass content on the proof stress is as expected, very small. Basic



(a)



(b)

FIG 8

fibre theory pre-supposes that the matrix will yield in order to fully exploit the load transfer. Thus, we might expect a very small decrease in proof stress with increasing glass content.

Fig. 9 illustrates the effect of extrusion temperature upon the room temperature strength of the composite. The ultimate tensile stress shows an almost linear increase with temperature whilst the proof stress deteriorates very slightly. The process parameters affect ultimate stress values in a manner completely opposite to the matrix material. This is because the chemical bonding of matrix and fibre is so much more efficient at high temperatures. This strengthening effect more than compensates for the softening in the initial matrix due to dynamic recovery. The proof stress is seen to decrease rather more rapidly with temperature than is the case for the matrix material. This is again clearly associated with the stress concentration effect as the load is transferred from the matrix to the composite.

Decreasing the glass particle size has a similar effect to decreasing the matrix particle size except that the major change occurs in the levels of ultimate strength obtainable. Fig. 10 shows that the optimum glass particle size is about 40 µ. The explanation must be that as particle size decreases the surface/volume ratio increases exposing a greater area for chemical bonding with the matrix. Under these conditions fibres would then tend to break

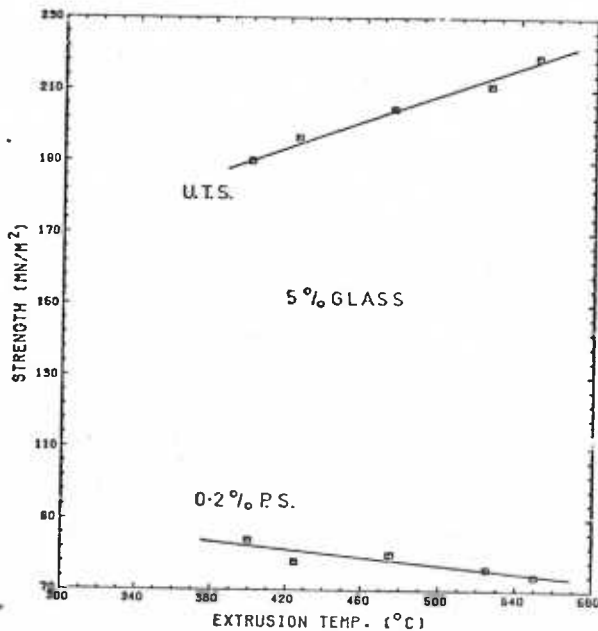


FIG 9 Effect of extrusion temperature on mechanical strength of composite

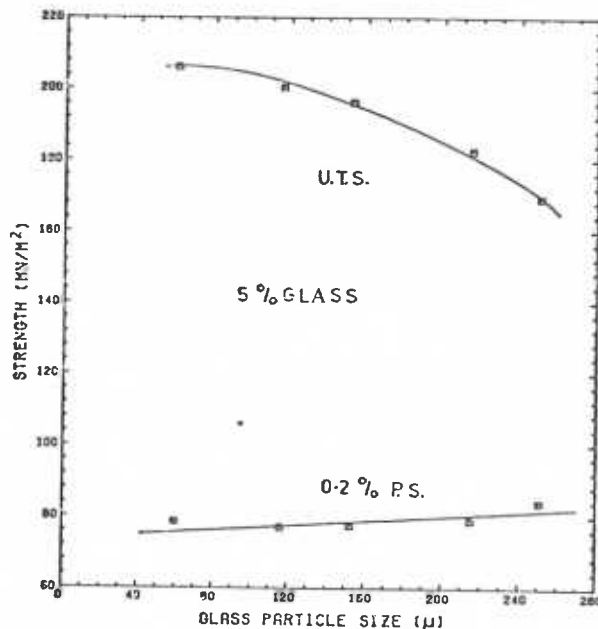


FIG 10 Effect of glass particle size on mechanical properties

rather than 'pull-out' and hence the composite strength increases. There must however, clearly be some stage at which all fibres are breaking and hence no further advantage would accrue from further increases in surface area/volume ratio.

The glass particle size has little effect on the proof stress since at this stress the fibre is not bearing a substantial portion of the load.

One of the primary aims of fibre strengthening is to protect strong but brittle fibres by surrounding with a weak but ductile matrix. Thus, in theory the notch ductility of a composite should be satisfactory if only because if a notch forms in one fibre (perpendicular to the fibre) it will only propagate throughout that fibre before being blunted in the ductile matrix. Thus the notch ductility of the composite is of some interest, indicating whether the composite is behaving correctly and indeed will continue to behave correctly during service. Fig. 11 shows that for glass weight fractions up

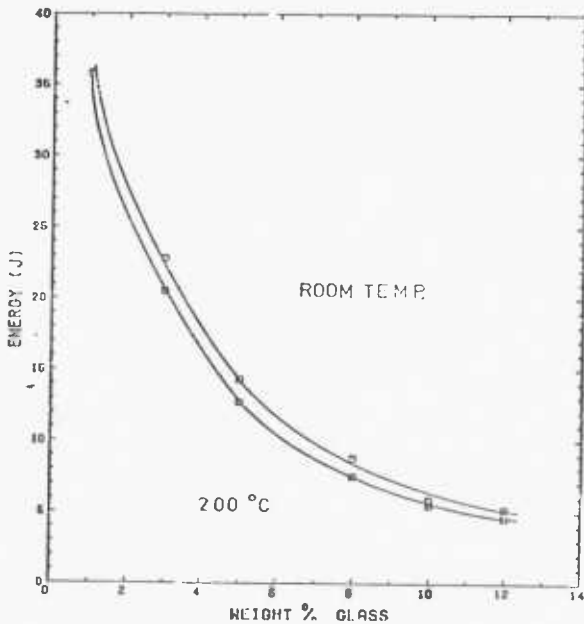


FIG 11 Effect of glass content on notch ductility

to 2% the notch ductility is 28.4 N-m compared to 42.17 N-m for the matrix material. Further additions of glass cause a rapid decrease in notch ductility which appears to be caused by the difference in elastic constants of the fibre and matrix materials. This results in premature cracking of the matrix weakening its resistance to crack propagation. Impact tests were also conducted using specimens heated to 200°C and Fig. 11 indicates that at this temperature there is only a small reduction in toughness.

Table IV shows the comparison between as-extruded composite and matrix material properties together with typical figures for an as-cast and extruded alloy.

Table IV -Optimum Properties of Materials (extruded at 450 C)

	Proof Stress N/mm <sup>2</sup>	Ultimate Stress N/mm <sup>2</sup>	Elongation %	Notch Ductility N-m
'As-cast'	55.0	101	12	43.1
Matrix	81.5	129.5	27	42.2
Composite (5% glass)	87	201	17	14.4

Extrudate Structure

(i) Matrix Material

When examined under the optical microscope the product exhibited a fibrous cold-worked type structure as shown in Fig. 12(a) The original particles have been elongated into the extrusion direction and measurements confirmed that elongation of the particles was approximately proportional to the extrusion ratio. This cold-worked type microstructure was produced in extrusions performed above the recrystallisation temperature of the alloy and the transverse microstructure (Fig. 12(b)) confirms the equiaxial nature of the structure. It was not possible to detect any deviation from this structure over the wide range of extrusions investigated confirming that a substructure must be present and that dynamic recovery was the prevailing deformation mechanism.

The transmission electron microscope reveals a structure consisting of sub-grains which are in general elongated in the extrusion direction (Fig. 13(a)) and are equiaxed in the transverse direction (Fig. 13(b)). This confirms that dynamic

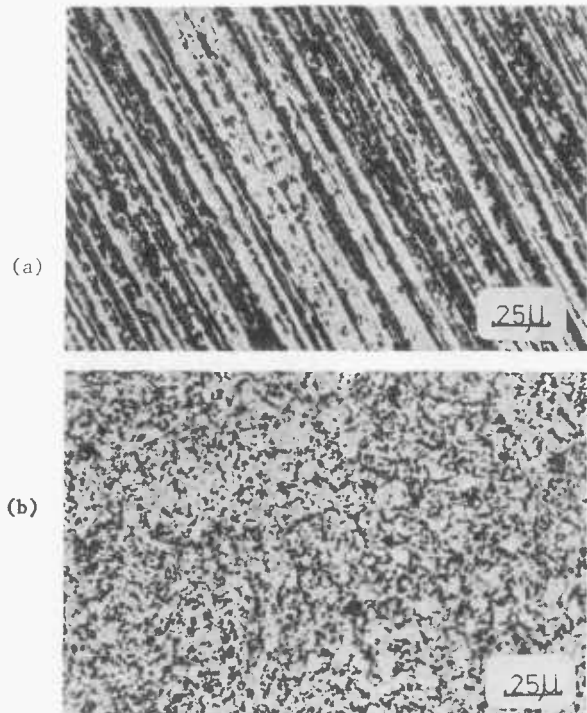


FIG 12

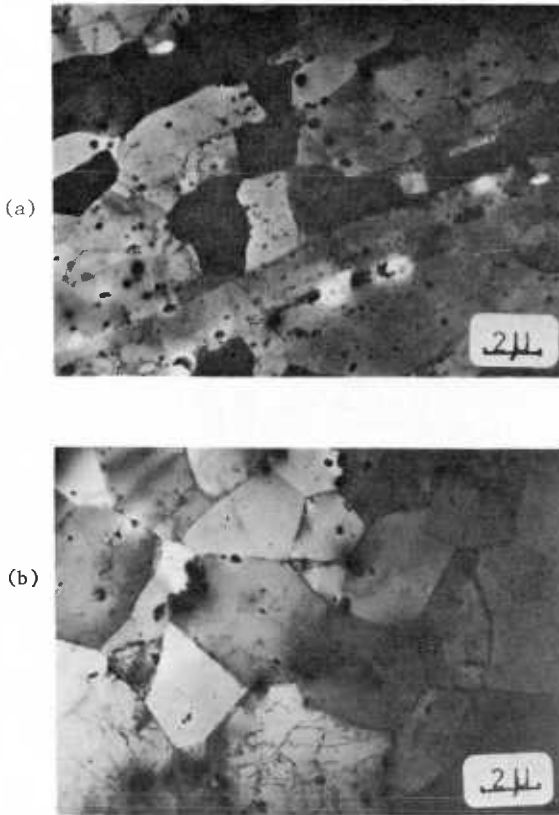


FIG 13

recovery has occurred. The discontinuous nature of the original oxide film is evident and original boundaries may still be detected and identified by the drawn out debris of the original oxide. The apparent presence of oxide in the centre of grains is attributed to the orientation selected during the thinning operation, such oxide being the remains of that surrounding a neighbouring grain. Fig. 14 is an electron micrograph taken from a foil prepared

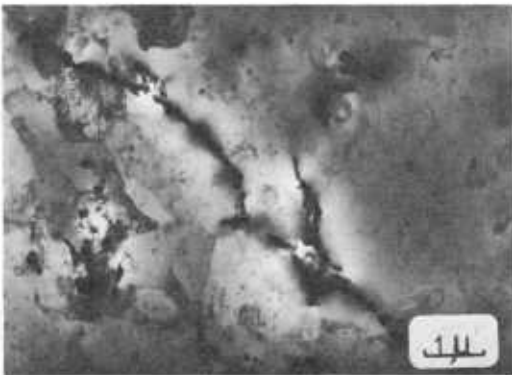


FIG 14

from a region in the deformation zone of a partially extruded billet. It illustrates very clearly how the oxide layer surrounding a particle is broken up and elongated during the extrusion process. This photograph was taken at a location in the middle of the deformation zone and it is evident that the particulate nature of the billet is still preserved at this stage. The oxide has already been elongated and it is also possible to discern that the hard oxide layer penetrates the original particle before assuming its final position in the extrusion direction.

Fig. 15 shows the increase in sub-grain size which occurs with increase in extrusion temperature (i.e. decrease in temperature compensated strain rate). Such a

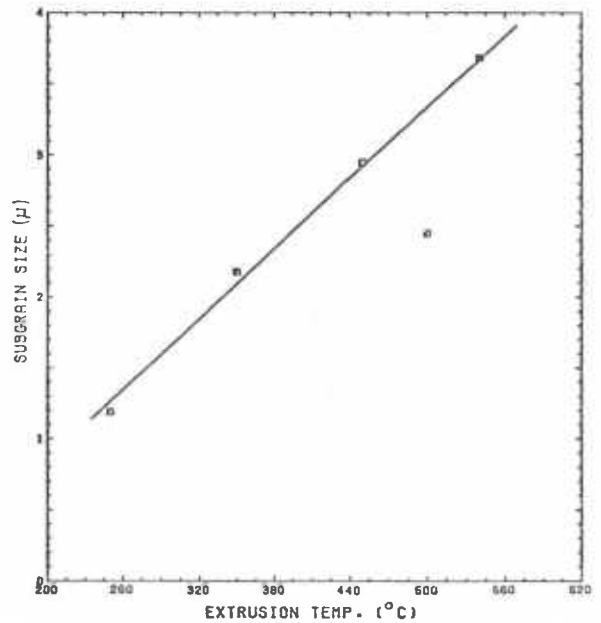


FIG 15 Effect of extrusion temperature on sub-grain size.

relationship has been established for a number of aluminium alloys produced by the conventional method by Sheppard and Raybould [6]. At higher temperatures dislocations have a greater thermal energy and the recovery mechanisms, usually climb and cross slip, operate more readily. Hence, sub-grains are first formed and recovery occurs by tangling and untangling of sub-grain boundaries such that the sub-grain size increases thus lowering the overall dislocation density. This is a direct result of the sub-grain boundaries consisting of tangles of dislocations.

## Extrudate Structure

### (ii) Composite

The optical micrographs Figs 8(a) and (b) illustrate the imperfect nature of the fibres formed by the extrusion process. The transverse section illustrates the wide variety of transverse shapes of the fibres and shows quite clearly that transverse viscous flow of glass has occurred during passage of the composite through the deformation zone. The longitudinal micrograph indicates that fibre cross-section also varies along its length but in general the aspect ratio is satisfactory. The 'cold worked' appearance of the matrix material suggests once more that dynamic recovery remains the deformation mode during composite extrusion. It is just possible to locate very small microcracks in this micrograph which of course, would have a deleterious effect on properties. Fig. 16 is a macrograph taken of an extrusion stopped during the ram stroke and clearly shows the development of structure within the deformation zone. It illustrates the

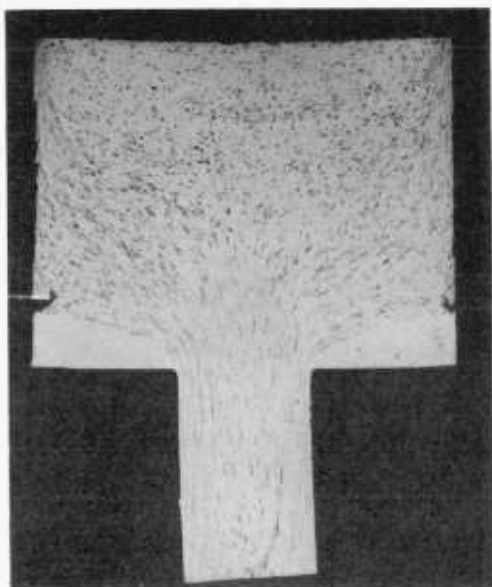


FIG 16

inhomogeneous nature of the extrusion process which causes glass particles located near the surface of the billet to be elongated to a much greater extent than those located on the central axis. Nevertheless all fibres seen to have a suitable aspect ratio although this wide variation in fibre cross-section makes analysis difficult. It would certainly be worthwhile to investigate the effect of indirect extrusion upon the formation of fibres since this is inherently a more homogeneous process.

It was not possible to investigate the composite material under the electron microscope because thinning of foils caused the glass fibres to fall out of the matrix. However, the scanning electron micrograph shown in Fig. 17 is

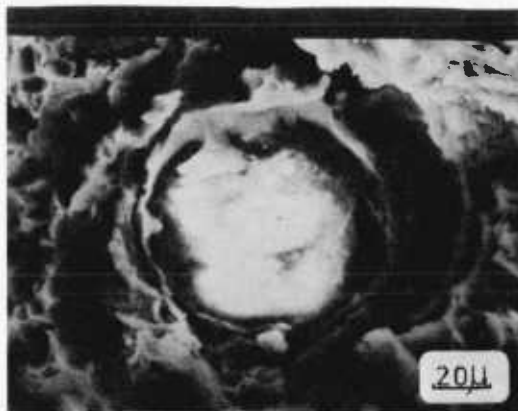


FIG 17

interesting. This shows the crater left by a fibre on the fracture surface (the fibre has disappeared by hydrolysis). Microcracks can be discerned around the fibre-matrix interface and even voids between fibre and matrix. These voids are in fact too large to be due to thermal shrinkage so most probably they have occurred during the polishing operation. The shell around the crater is most distinct and consists of glass-metal interaction products which include the spinel  $Mg_2Al_2O_4$  [7]

## CONCLUSIONS

- 1) The matrix material produces an interesting and useful alloy without the addition of fibre strengthening.
- 2) The extrusion of randomly mixed glass and aluminium alloy particles produces a coherent composite material containing discontinuous glass fibres having a suitable aspect ratio.
- 3) The maximum useful wt % glass is approximately 8. The addition of a greater glass content causes mechanical properties to fall.
- 4) High extrusion temperatures (low temperature compensated strain rates) are desirable for optimum mechanical properties.
- 5) Greatly enhanced tensile properties are produced by incorporating glass fibres in the matrix.

## ACKNOWLEDGEMENTS

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## APPENDIX

The Minimum Extrusion Ratio required for a Coherent Product

Assuming that the glass particles are spherical with radius  $r_1$  and that after extrusion their shape is cylindrical with radius  $r_2$  and length  $\ell$ .

The ratio  $\frac{r_1^2}{r_2^2} = R$  the extrusion ratio.

Continuity requirements of the glass reinforcement yields

$$\frac{4}{3} R^{\frac{3}{2}} r_2 = \ell$$

The critical fibre length  $\ell_c = \frac{r_2 \sigma_f}{\tau}$

where  $\sigma_f$  is the stress in the fibre and  $\tau$  the fibre shear strength.

Hence for fibre reinforcement to be satisfactory

$$\frac{4}{3} R^{\frac{3}{2}} r/2 > \frac{r_2 \sigma_f}{\tau}$$

For this system  $\sigma_f = 2412 \text{ MN/m}^2$

$\tau = 100/\sqrt{3} \text{ MN/m}^2$

Hence  $R = \underline{\underline{9.94}}$

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