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A STUDY OF THE EXTRUSION OF PARTICULATE CELLULOSIC COMPOSITE MATERIALS

by

J C WAHLERS

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THIS THESIS IS DEDICATED TO THE MEMORY OF THE LATE

PROFESSOR BRIAN YATES

Brunel University
Department of Materials Technology
Uxbridge, Middlesex UB8 3PH
John Colin Wahlers
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ABSTRACT

Traditional routes to the manufacture of timber based composite materials such as particleboard rely on platen pressing a premix of carefully chosen and prepared timber particles and an adhesive to produce flat sheets. Historically such routes have made very inefficient use of forest resources, and because of the planar nature of the composite produced, the finished articles have enjoyed a reputation of being of poor quality and "cheap".

The work documented in this thesis sought to examine alternatives to the traditional manufacturing methods in terms of both raw materials and processing route, and to establish technical, economic, and environmental boundaries for the use of the alternatives.

Having settled upon extrusion as the most likely technique with which to proceed, the investigation was carried forward by the construction of a series of experimental instruments with the joint aims of refining the technique to a realistic working level and investigating the underlying mechanical principles behind the process.

Recourse to the literature of polymer rheology, fluid mechanics, and soil mechanics gave the basis for a crude mechanism hypothesis, and iterative improvements in the instrumental techniques yielded data with which this could be refined. Although some correlation between the extrusion behaviour of this system with other more easily quantifiable systems was found, there is still considerable work required in defining the dynamic changes in the material properties as the raw material is extruded.

Small scale production experiments proved successful, however, and an elementary financial model of a suitable production facility indicated that the techniques developed could be used as the basis of an environmentally acceptable, economically viable, low technology industry.

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DECLARATION

All work documented in this thesis, including the design and construction of experimental apparatus, was carried out by the author, except where due acknowledgement has been made to the contrary.

No part of the work has been published or submitted in support of any other degree or academic qualification.

CHAPTER ONE. INTRODUCTION

This chapter is intended to set the scene for the overall strategy of the research presented in this thesis, and therefore includes an outline of the historical use of timber before moving on to the growth of the particleboard and wood composite industries.

This introduction is drawn to a conclusion with a chapter by chapter summary of the remainder of the thesis in order to give an overview of the work in the context of the research strategy.

1.0 Historical Background

Wood is an important natural resource, and has almost certainly been so since man first trod the earth. Its abundance, renewability, and relative cheapness have been major factors in its popularity not only as a building material but also for use in tools, weapons, furniture and for decoration. In addition to those factors there are further properties which distinguish wood from other materials, for example:

- wood is generally light in weight which makes it easy to handle and transport,
- wood is relatively easy to work with simple tools,

- the porous, cellular nature of wood makes it easy to fabricate using fasteners such as nails and is also responsible for wood's ability to hold decorative and protective finishes,
- the high strength to weight ratio of most woods means that they compare favourably with other structural materials such as steels,
- wood is a good insulator for heat, sound and electricity, dimensionally wood is very stable with respect to temperature (at constant moisture levels)
- the environmental stability of wood is relatively high, offering good resistance to acids, weathering, and in many cases attack by insect and animal life,
- generally wood structures can be disassembled with ease, and the salvage value is high in relation to initial cost,
- wood can be decorative and ornamental in itself without the need for embellishment,
- wood retains its properties over a wide temperature range and in general gives plenty of warning of the onset of failure.

In addition to the advantages of wood per se, the origin of the raw material, the forest, also has an important part to play in maintaining the balance of nature. The control of erosion and stream flow, the provision of habitats for wildlife and even the provision of recreation facilities are all parts of the production and use of timber for whatever end purpose.

Despite all these seemingly advantageous features of a thriving and controlled timber industry, it is sad to relate that only in relatively recent years has attention been focussed on the devastation caused by the indiscriminate and unthinking use of timber resources. Recent figures (1), for the UK alone indicate that in the 30 years from 1947 to 1977 the loss of semi natural broad leaved timber was equal to all of the losses of the previous four centuries. Obviously this cannot be blamed totally on forest management techniques since the urbanisation of the countryside must also should some of the responsibility, however the fact that Britain currently imports some £2750 million worth of wood and wood products indicates that better use of our natural resources would benefit not only the environment but also the balance of payments. Britain is of course not alone in having lost much of its natural timber resource since the tropical rain forests of the world are currently being cleared at the rate of 10 to 20 million hectares a year. Brazil is a typical example of an area in which this devastation is occurring, with its tropical rain forest having been reduced from 20.5 million hectares in the year 1500 to a projected 750,000 hectares in the year 2000 (2), a reduction of almost 80%.

Reasons for the continuing deforestation of the world are manifold and it is beyond the scope of this thesis even to list them. It is necessary for the coherent development of the theme of this research to concentrate on one of the major causes however, the inefficient use of felled timber, and examples are plentiful.

The most significant limitations to the usefulness of a tree for man's purposes are geometrical, and this necessarily influences the amount of wood which remains unused. A survey published in 1947 (3), gave indications of the percentage usage of wood from trees felled for different end products. Trees felled for cooperage yielded only 28% useful timber, for building ties 30%, for general lumber 32% and for veneer 34%. On a (US) national basis only 43% of all timber felled was deliberately converted into wood products of any kind, including fuel. A further 22.5% was converted into fuel from waste, and a total of 34.5% was not used at all. Considering that this waste in 1944 amounted to approximately 105 million tonnes of unused, but felled, wood, there was not only great scope for improvement, but also enormous economic incentive to find a profitable use for all this material which was being burnt to make room for other lumber operations.

Significantly it was about this time that the first commercial plants to produce particleboard from sawdust and machining waste come on stream, although there is still argument over whether it was the German Torfitwerke group or the American Dyas Company who were actually first into production. What is clear is that the idea of producing large wood-like articles from smaller wood particles goes back a lot further, at least to 1887 when Ernst Hubbard published "Die Derwertung der Holzabfalle" and possibly as far back as the time of the Pharaohs and the Roman Empire (4).

After the Second World War the expansion of the particleboard industry was dramatic. In the United States of America alone the annual output of particleboard increased from 10,000m^3 in 1950 to 521,000m^3 in 1960 (5). For Europe the corresponding figures are 10,000m^3 and 2.2 million m^3. The average growths of the two industries as reported by the same source for the period 1960-1973 were 21.9 and 18.1 per cent per annum respectively. This source also predicted that the growth of the industry would continue, albeit at a decreasing rate, through the 1980's and into the 1990's, a statement which has so far been shown to be correct (6).

The situation with regard to the maximum utilisation of timber resources has consequently seen a reversal of the 1944 picture outlined earlier, with the demand for raw material outstripping the supply of waste and residual wood. Large quantities of groundwood, mainly in the form of forest thinnings and small diameter trees, are currently used by the particleboard industries both in the UK and abroad. Despite the notionally higher cost of such raw material, the consistency and uniformity of roundwood mean that greater control over wood type and chip size can be exercised, resulting in more consistent, higher quality products. It is therefore not unusual today for manufacturers to use low grade chips made from waste material for the core of a board and high grade chips made from selected roundwood for the outer surfaces where the better properties are required.

1.1 Product Development

Despite the many technological advances which have accompanied the growth of the particleboard industry, almost the entire output still consists of simple rectangular panels. The largest panels currently produced are some 2438 x 12500mm in size although the bulk of production centres around panels of 2438 x 1219mm. Post-press sawing and machining operations are then used to produce a range of stock sizes from these larger panels. The panels are intended for two principal markets, the construction industry (for structural panels such as floors or wall cladding, or for temporary construction aids such as shuttering and closures), and the furniture industry (for the manufacture of low and mid-quality products).

All of the current applications, consequently call only for flat pieces of particleboard and it is not surprising therefore that 95-98% (6) of the board on the market is produced by the simple process of platen pressing. The remaining few per cent is produced using various extrusion processes which, because of chip orientation during the process, result in boards having unfavourable anisotropy and consequent poorer strength and stiffness properties than the platen pressed equivalent. Extruded board is cheaper than pressed board however and consequently finds use in undemanding applications or in situations where the board forms part of a composite product and is therefore reinforced, for example in the cores of laminated doors. These aspects of the technology are dealt with in detail in Chapter 2.

Shaped products such as skirting board, decorative panelling, and banister rails are also manufactured from wood particles by pressing operations using contoured platens. Although there are obvious advantages in producing such articles, and in fact any article whose cross-section is constant, by the use of an extrusion technique, there appear to be no manufacturing facilities at present based on this concept. Patents exist, (7-28), which outline details of machinery for accomplishing the task, but there is no evidence of any of this work being taken further than the drawing board stage.

Based on the predictions cited earlier (5) regarding the progress of the particleboard industry, it is logical to assume that a process for manufacturing articles for which a market has been shown to exist, but which is in some way or ways more advantageous than those processes at present in use, would find commercial acceptability. If at the same time the process utilises raw materials which result in no more, or perhaps less damage to the environment than present processes, then the search for such a process can only be regarded as worthwhile.

The purpose of the research documented in this thesis was to investigate the possibility of producing wooden artefacts by an extrusion route, a technique which historically has produced little in the way of commercial application, despite the obvious financial and technical advantages of doing so.

1.2 The Research Strategy

The starting point for any investigation of this kind must be a definition of the research aim followed by a comprehensive search of the available literature to ascertain whether such a project has been undertaken previously, and if it has not, then why not. If the end result of the research is to have commercial value, then the research itself should be directed towards a specific goal. Target products and/or areas must be identified early in the work so that as little time and effort as possible are wasted in pursuing unproductive avenues of work.

As has been mentioned earlier, the production of items from comminuted wood is a well established field and embraces everything from floors and walls to decorative furniture and fittings. Any new product or process which is to be successful will therefore have to be competitive in terms of either quality or cost, which leads naturally to the conclusion that starting from wood-chips, the product will necessarily possess high added value. With solid timber artefacts a high added value generally implies that much of the original wood from which an article is made has to be removed by way of machining and is therefore lost as a waste product. Examples of such products are window frames, decorative mouldings and picture frames. Although limited quantities of such articles have been produced from comminuted wood feedstock, these have all been manufactured by platen pressing, with consequent high capital plant costs which are then passed on in the product. Items such as those mentioned above, however, do lend themselves to manufacture from particles as they generally require the product to possess only moderate strength, a feature typical of particle based materials.

An obvious conclusion to be drawn from the above arguments then is that an extrusion process, simply by its nature, would be a profitable production route providing that the mechanical drawbacks which have inhibited the use of extruded products could be overcome. Technical and scientific literature in the field of the extrusion of highly wood-filled composites (wood content $>$ 80%) is exceedingly sparse as, not surprisingly, is that covering the rheological aspects of the flow of such systems. This suggests that detailed research and investigation of the kind proposed in this study has not been applied to the area and therefore that any new work stands a good chance of being useful. The main theme of the research presented in this thesis therefore will be the development of as full an understanding as possible of the processes and mechanisms occurring during wood composite extrusion. This will involve using models and theories to describe the process rather than relying solely on the empirical methods which have been used in the past.

Clearly with a starting material having such a distinctive geometric shape and internal anisotropy as wood chips this will involve the coalescence of ideas and theories from a range of subject areas, and the literature of rheology, fluid mechanics, soil mechanics, powder handling, and metal and plastics processing as well as the more scientific treatises from the particleboard literature should all provide relevant information and were investigated.

On the basis of the information gained from the literature, the next stage is to narrow the likely field down and identify the possible manufacturing approaches. This will involve the establishment of feasibility boundaries and the identification of practical limitations to the chosen process(es). The application of the accumulated information from all of the preceding stages to factors such as power requirements, tool design, costs, and of course the nature of the desired product is therefore required.

At this stage the design of an experimental approach to supply basic process information is both necessary and possible. Although it is sometimes possible to adapt existing apparatus to give the results required, in this instance it was thought more likely, in view of the unusual nature of the raw material, that the equipment would have to be specially designed and constructed in order to yield the required information. Initial trials using a very simple piston rheometer type of apparatus fitted to an Instron universal testing machine indicated that in order to yield realistic results a "rheometer" of very generous proportions and capacity would be required. It was also obvious that none of the commercially available instruments would be suitable for investigations using the rather unusual feedstock required for this research. The instrument finally used for this study is described fully in Chapter 8.

On the basis of the theoretical and practical results from the research, the commercial aspects can finally be attended to by an outline design for a pilot scale production plant. This should include not only mechanical and process details but also brief costing information and a limited survey of potential markets and areas of application.

The remainder of this thesis therefore follows the outline structure given throughout this introduction and this is laid out in detail below.

1.3 Summary of Contents

Chapters 2 and 3 of this thesis are concerned mainly with a literature review of the subjects covered, Chapter 2 describing commercial and speculative wood conversion and "improvement" processes, while Chapter 3 covers the more general literature on extrusion and rheology.

The description of the experimental work begins with Chapter 4 and covers, in sequence, the raw material properties (Chapters 4 and 5), the design philosophy and construction of the basic extrusion rig (Chapter 6), and finally the iterative building programme and experimental results are presented in Chapters 7 and 8. The results from the earlier work of Chapter 6 showed some limitations and ambiguities as detailed in Chapter 7 and an extruder with more comprehensive instrumentation and control was designed and constructed, as detailed in Chapter 8, and was used to provide new and more specific data.

Products resulting from the various experimental programmes were examined in a limited product evaluation exercise, and the details of this, together with the results are presented in Chapter 9.

Chapter 9 concludes this work with a summary of the main conclusions and the identification of those areas which require clarification by further work.

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CHAPTER TWO. WOOD CONVERSION PROCESSES

The inherent advantages of wood as a material of construction were outlined in Chapter 1. It must be said, however, that when considered in isolation and not in the light of design which capitalises on its good points, wood does have many disadvantages too.

The most severe disadvantage is the highly anisotropic nature of timber with its concomitant effects on properties [for example, the longitudinal tensile strength is generally of the order of 40 times the transverse value, (1)]. The mechanical properties are not the only ones affected though, and fire, insect, and fungal attack resistance, water absorbency and electrical and thermal conductivities are all variable depending on the orientation of the timber during testing.

Conversion processes such as are discussed later in this chapter are thus man's attempts to ameliorate this situation for his own advantage. The title of this chapter could therefore equally well be Wood Improvement processes, and in order to present a more complete picture of the routes used to reach an improved product, the first part of this chapter gives a review of some of the more common processes used for this purpose. The second, major part of this chapter deals more specifically with the subject of particleboard, and because of the close analogy between particleboard and the product of the research which this thesis documents, this subject is dealt with in more detail than those in the earlier parts of this chapter.

2.0 General Background

Wood conversion processes fall broadly into one of two categories, physical modification or chemical modification, although some processes also rely on combinations of the two. Examples of the former, in which the raw timber is broken into smaller units then re-assembled are laminates, plywood, particleboard, fibreboard, and paper, while the latter covers those processes which involve impregnation of the natural wood structure, densification by the use of heat, pressure or both, or again some combination of the two.

Although the technology of both routes to an improved "wood" has advanced greatly over the last 50 years, neither can be said to be a new concept. Pliny the Elder reports the use of beech veneers and citrus wood in "The Natural History" (2), and Noah was told to "pitch it within and without with pitch" in Genesis 7, 14 when building the ark. It is probably not too much of a generalisation to say that the chemical routes to modification are used with success both for improving the mechanical properties and for the preservation of timber, while the physical routes are first and foremost ways of improving mechanical performance, or incorporating decorative finishes.

2.1 Chemical Treatment Methods

Chemical treatments applied to wood generally confer resistance to one or many of the natural degrading processes which can occur, from insect attack, through moisture absorbtion to fire. Many of the techniques such as creosoting, painting and the tarring with pitch mentioned above have been in existence for such a long time that a detailed discourse is unnecessary in this thesis, however the underlying principles are not as well known and these, together with some of the less well known treatments will be dealt with briefly in the following sections.

2.1.1 Impregnation mechanisms

In order for a substance to impregnate the body of a piece of wood fully, it is necessary for that substance to move through the internal structure of the cellular assembly which makes up the wood. The structure and complex internal nature of woods is too vast a subject to be discussed in depth here, and the reader is referred to one of the standard texts listed in the references (9, 10). A much simplified description of the structure of softwoods will assist in the understanding of the processes involved during impregnation, however, and is a useful point of reference for later chapters.

The structure of a softwood can be likened to a bundle of drinking straws in that it consists of large numbers of parallel hollow cells or tracheids. The cells are of finite length, (hence the drinking straws are sealed at each end), and they do not all begin and end at the same place (there is a variation in both the length and the axial position of the individual straws within the bundle). The length of individual tracheids is generally significantly greater than the diameter (hence the analogy with drinking straws) and membrane covered holes in the cell walls, "pits", allow intercommunication between adjacent cells.

It can be seen from this very simple model that bulk movement through the wood of any substance, be it solid, liquid, or gas will involve movement on a micro scale through the voids at the centres of the tracheids (the lumen), through the cell walls proper, and through the membrane covered pits. This movement can be either a flow mechanism under externally applied pressure or capillary forces, or a diffusion mechanism driven by concentration gradients within the cellular structure.

2.1.1.1 Flow through the wood structure

Referring back to the model above, it is clear that flow through the cellular structure involves flow through the lumen in series with the parallel flow combination of the pits and the cell walls. The volume flow through the lumen therefore is equal to the combined flow through the pits and the cell walls. If all of these paths are assumed to be composed of capillaries then a modified version of Poiseuille's equation (11), can be used to quantify the flow through each of the three sections, thus:

$$V = \frac{\pi r^4 \Delta P}{8 \eta L} \qquad (2.1)$$

where V = rate of flow ($cm^3 \, s^{-1}$)

r = radius of capillary (m)

L = length of capillary (m)

P = pressure drop over length of capillary (Nm^{-2})

η = viscosity of fluid ($Ns \, m^{-2}$)

for a number, n, of parallel capillaries we get

$$V = \frac{n \pi r^4 \Delta P}{8 \eta L} = \frac{q r^2 \Delta P}{8 \eta L} \qquad (2.2)$$

where q is the combined cross section of all capillaries, ($n \pi r^2$).

Now $V_{\text{lumen}(l)} = V_{\text{pit}(p)} + V_{\text{wall}(w)}$ (2.3)

and substituting from (2) gives:

$$\left(q_l r_l^2/L_l\right)\Delta P_l = \left(q_p r_p^2/L_p\right)\Delta P_p + \left(q_w r_w^2/L_w\right)\Delta P_w \qquad (2.4)$$

Since ΔP_p and ΔP_w are equal, then if $\Delta P_c = \Delta P_p$

$$\left(\frac{q_l\ r_l^2}{L_l}\right)\Delta P_l = \left[\left(\frac{q_p\ r_p^2}{L_p}\right) + \left(\frac{q_w\ r_w^2}{L_w}\right)\right]\Delta P_c \qquad (2.5)$$

where ΔP_c = combined pressure across the cell wall.

Stamm (12) substituted measured values for the quantities in equation (2.5) and showed that for longitudinal flow (parallel to the tracheids) the pressure drop across the lumen of the cells was negligible compared with that across the wall, and that the flow through the wall is negligible when compared with that through the pits. The inference in the case of Stamm's figures is that it is the number and size of the cell wall pits which govern the rate of flow of a fluid through a softwood structure. This in turn explains why there is such a wide variation in penetrability from species to species. A similar substitution carried out for transverse flow shows that the pressure drop across the lumen is even lower than the previous value, while flow through the cell walls is still negligible. A survey of the relative penetrability of major North American woods by liquid preservatives was prepared by MacLean (13), in 1935 and Table 2.1 is a summary of some of his findings, as an illustration of the variation which exists within the softwood species alone.

The situation with regard to the flow of gases through wood is somewhat complicated by the fact that gases are compressible and that molecular flow can occur through the pit pores. Comstock (14), however, has shown that the permeability of wood to gases and non-wetting liquids is of a similar order.

It is agreed generally within the literature (15, 16, 17, 18, 19), that the longitudinal flow rate through a wood is at least 10,000 times greater than the transverse or radial flow rates. As a consequence, impregnation treatments rely almost entirely on flow along the grain of the wood to achieve their penetration. This fact was demonstrated clearly in the course of this research during the experiments involving the use of X rays to define particle orientation. Details of this are included in Chapter 8 of this thesis. These results also demonstrate that the process of impregnation is not as straightforward as the preceeding explanation suggests. This may be due to structural differences in the real wood from the over simplified model used,

WOOD TYPE	
Bristlecone Pine, (Pinus aristata)	Heartwood easily penetrated
Pinon, (Pinus eduils)	
Ponderosa Pine (Pinus ponderosa)	
Douglas Fir (coastal) (Pseudotsuga taxifolia)	Heartwood moderately difficult to penetrate
Jack Pine (Pinus banksiana)	
Loblolly Pine (Pinus taeda)	
Longleaf Pine (Pinus palustris)	
Norway Pine (Pinus resinosa)	
Shortleaf pine (Pinus echinata)	
Western Hemlock (Tsuga heterophylla)	
Eastern Hemlock (Tsuga canadensis)	Heartwood difficult to penetrate
Engelmann Spruce (Picea engelmannii)	
Lowland White Fir (Abies grandis)	
Lodgepole Pine (Pinus contorta)	
Noble Fir (Abies nobilis)	
Sitka Spruce (Picea sitchensis)	
Western Larch (Larix occidentalis)	
White Fir (Abies concolor)	
White Spruce (Picea glauca)	
Alpine Fir (Abies lasiocarpa)	Heartwood very difficult to penetrate
Corkbark Fir (Abies arizonica)	
Douglas Fir (mountain), (Pseudotsuga taxifolia)	
Northern White Cedar (Thrya occidentalis)	
Tamarack, (Larix laricina)	
Western Red Ceda (Thuja plicata)	

TABLE 2.1 Relative penetrability of the major North American softwoods

or it may be that the impregnation in this case involved the alternative mechanism mentioned in section 2.1.1, diffusion.

2.1.1.2 Diffusion through the wood structure

Since diffusion is dependent only on the effective cross sectional area of an interface, while flow is dependant on both this and the square of the radius of the pore sizes, it is to be expected that diffusion controlled impregnation will be significantly different from the previously discussed flow controlled regime. The driving force for diffusion is derived either from a concentration gradient or from a vapour pressure gradient, hence those processes which involve diffusion fall into one or other of these categories.

Concentration gradient controlled diffusion is important where a carrier solvent is used to introduce a solute into the wood structure. In the flow example it was stated that the cell walls played only a very small part in the movement of the fluid through the wood structure. In contrast to this, provided that the molecules of the impregnant are sufficiently small to pass through the cell wall, [Tarkow et al (20), found that polyethylene glycol molecules up to molecular weight 3500 could diffuse through the cell walls of water swollen wood], the thickness of the cell walls is small enough (7) to make cell wall diffusion a significant contribution to the overall diffusion rate. Again the total system consists of the diffusion through the lumen in series with that through the parallel combination of the pits and the cell walls. The diffusion rates in the parallel combination are added directly, and then reciprocally summed with the diffusion rate of the other series element. Treatment processes involving solute diffusion have the advantage that the active component of the mixture actually penetrates into the solid parts of the wood structure where it is generally required, and can be left there when the carrier solvent is removed either for re-use or for cosmetic purposes.

Vapour phase diffusion is far less specific in terms of mechanism since processes in which a vapour is impregnated generally develop sufficient pressure to cause flow through the wood as well as the diffusion attributable to the pressure gradients.

In both types of diffusion mentioned above, the driving force, be it concentration gradient or vapour pressure gradient, is continuously changing. The diffusion is therefore dynamic, and is described by Fick's second law of diffusion (21).

$$\frac{dc}{dt} = D\left(\frac{d^2c}{dx^2}\right) \qquad (2.6)$$

This states that the rate of change of concentration with time, $\frac{dc}{dt}$, at a distance x from the interfacial boundary, is proportional to the rate at which the rate of variation of concentration with distance changes, i.e. the second derivative of concentration with respect to distance, $\frac{d^2c}{dx^2}$. Unfortunately there is no single general solution to the equation in an integrated form, however by specifying certain conditions, simplified solutions have been obtained (22), which enable use to be made of the relationship in practical applications.

As was the case with the flow regime, diffusion in the fibre direction is a faster process than that in the transverse direction. The difference is much smaller in the case of diffusion though, involving a factor of 10 to 15 (23), as against the 10,000+ factor found in flow.

For impregnating the entire structure of timber, diffusion treatment is considered to be too slow a process to be widely commercial, however it does find use in surface treatments and remedial "bandage" treatments for large sections, and for the treatment of freshly cut veneers prior to seasoning.

2.1.2 Treatment processes

Whether a wood treatment is required to give resistance to insect attack, fungal attack, fire, or air and structure borne moisture, or whether it is designed to improve the mechanical properties of the wood, or even any combination of these, the aim of the process is always to introduce an agent of some kind into the structure of the timber in order to confer the desired properties.

In the first three areas, the results are achieved by chemical means, that is the treatment introduces a substance which is toxic in the case of fungus or insects, or which promotes charring rather than flaming in the case of fire (although surface resident oxygen barriers can be just as effective for this purpose providing they are thermally stable).

Moisture resistance is a more complicated subject altogether and sufficient could be written about this aspect of wood treatment to fill an entire thesis. Resistance to moisture is not generally an end in itself, since so many other properties of wood are dependant upon the moisture content. For example, if the moisture content of timber is maintained below about 20%, the timber will generally be resistant to fungal attack without further treatment. Maintaining a constant moisture content is also almost totally effective in affording dimensional stability to timber.

Unlike the treatments in the case of fungus, fire and insects, the conferring of moisture resistance is generally a physical rather than a chemical action, and can therefore involve mechanical as well as chemical treatment. The most simple of these treatments is painting or otherwise coating the surface with an impermeable layer. Internal coating by means of water repellants such as waxes and polyethylene glycols is less effective but does confer a certain amount of protection against the moisture of humid atmospheres. Internal coating taken one step further, that is to completely fill the cell walls of a timber with a substance, is called bulking. This term is also applied to salt treatments used to saturate the cell walls then leave solid salts behind after evaporation of the solvent. These salts then inhibit the movement of the cell walls by their physical presence and thereby confer dimensional stability. Completely filling the cellular structure is obviously another way of precluding moisture. Treatments which involve this technique, either throughout the timber strucrture or just in the outer exposed regions to form a barrier, include the use of curable phenolic resins and radiation and free radical cross linked polymer blends.

The final reason for wood treatment, to improve physical and mechanical properties, also relies very heavily on filling the structure of the wood with chemicals which increase the stability of the natural structure. This too is an area in which the use of monomer impregnation followed by in-situ polymerisation has increased greatly in recent times.

No matter what chemicals are involved, however, the impregnation processes fall into three main groups; sap replacement, capillary absorption and diffusion, and vacuum/pressure methods. For background information, some typical examples from each of the three areas are included in the following sub-section.

2.1.2.1 Sap displacement processes

This is the simplest form of deep impregnation for timber and the methods are consequently widely used in countries possessed of poor resources, or in those places where distance makes access to pressure plant inconvenient and uneconomical.

2.1.2.1.1 The Boucherie process

This process, invented in 1838 (24), uses hydrostatic pressure to force a water borne salt solution through a newly felled log. End caps are fitted to each end of the log, one as an inlet and one an outlet on which a low vacuum can be drawn to assist the flow of fluid. Copper sulphate or copper chrome arsenate are the usual salts employed, and flow rates of up to 15 $lm^{-2}hr^{-1}$ can be achieved with a 10m head.

2.1.2.1.2 Modified Boucherie process

Hudson (25), reported the development of a new design of end seal which could withstand pressures of 1.4 MPa enabling flow rates of 40 $lm^{-2}hr^{-1}$ to be achieved, thus making the process considerably more efficient. A further modification reported by the same author (26), involves the use of a vertical steel cylinder in which as many as 20 logs can be treated simultaneously. The logs stand on a thick cotton mat supported on a perforated plate. Fluid containing sand particles is then pumped into the container around the logs and the sand settles and compacts to form a seal around the base of the logs. Pure fluid is then pumped into the area around the logs and is forced into the free end of the logs by the pressure. The fluid then flows through the log in conventional Boucherie manner and drains from the bottom of the tube. This system is said to be more efficient still in terms of treatment time with no disadvantages apart from the slight increase in capital cost of equipment.

2.1.2.2 Capillary absorption and diffusion processes

This section covers the more commonly used impregnation processes of brushing, spraying and dipping, and some of the less well known processes such as the "bandage" treatment mentioned earlier.

2.1.2.2.1 Brush and spray coating

Both of these methods are so well known that no specific process details need be given here. Brushing, although less efficient, is generally considered to be more effective than spraying since the latter can have a tendency not to wet the surface unless a great deal of attention is paid to the details of the process. Both treatments are generally only considered as short term protection. Specially thickened fluids can be employed to good effect and the use of starch as a thickening agent to produce brush-on paste treatments has been reported (27). These are left in place for periods varying from 4 hours to several weeks during which time the protective compounds diffuse into the wood interior.

2.1.2.2.2 Dipping

Some processes which involve the dipping of timber also involve the use of externally applied pressure and vacuum and these are therefore mentioned in the appropriate section. Simple immersion of wood to introduce preservative into its structure is second only to painting as the most widely used wood treatment process. The time required in the solution varies greatly according to the type of timber (28), the chemical nature of the treatment (24) and the period of protection the treatment is required to give. Examples of extremes are the "Timborising" process (29), which involves dipping in a solution of a soluble borate for two to ten minutes depending on the timber type and thickness (30), and the "double diffusion" treatment (31), which involves successive soaking in each of two solutions for a period of 3 days. The principle behind this second process is that the first solution (usually copper or nickel sulphate or zinc chloride), fully impregnates the wood with a very soluble salt. The second solution (sodium arsenate or disodium phosphate following copper or nickel sulphate, or sodium chromate folowing the zinc chloride) then causes precipitation of a sparingly soluble salt within the structure of the wood. Following both the Timborising and double diffusion soakings, timber is stored under conditions which prevent the wood from drying out in order to allow further deep diffusion of the salts to take place.

A variation on conventional cold dipping is the hot and cold open tank technique (32). The timber to be treated is immersed in cold preservative which is then heated slowly to between 65°C and 90°C depending on the timber type. This temperature is maintained for between ½ and 4 hours, again depending on the timber type, during which time entrained air in the wood structure expands and is lost. On subsequent cooling, atmospheric pressure forces the preservative into the voids previously occupied by the lost air, where it is then bound. With a knowledge of the timber characteristics it is possible to use the peak temperature and time at temperature to control the depth of preservative penetration and consequently the quantity of preservative used.

2.1.2.2.3 Remedial treatment

The "bandage" treatment, (9,p309), falls into this category and, as its name suggests, involves wrapping a bandage saturated with preservative around the timber to be treated to boost or restore a level of protection. The process is almost always carried out in situ and may even be used below ground level.

2.1.2.3 Vacuum and pressure methods

Methods using vacuum or pressure and combinations of both are without doubt the most efficient ways of introducing modifying agents into the wood structure.

Both organic and water borne preservatives can be added in this way, and increasingly such processes are also being used in the manufacture of wood-plastic composites for the impregnation of the plastic monomer mixture.

There are numerous patented and proprietary processes for carrying out vacuum and pressure impregnation, the differences all pertaining to the particular combination of process stages (pressure, vacuum or both, and impregnant introduction) and the order in which they are carried out. It is beyond the scope of this thesis to deal with all of the processes in detail, and Table 2.2 summarises the most prominent applications of such treatment. The reader is directed to the standard texts, (9, 10), for more specific information on the widespread applications within the preservation field.

2.1.3 Mechanical and thermal methods

The methods which fall into this category of treatment all rely on compressing the wood (and therefore increasing its density), without destroying the natural structure.

PROCESS	SUMMARY
ACETYLATION	Hydroxyl groups in wood react with acetic anhydride over pyridine catalyst to form esters. Lumen remain empty. Anti-shrink efficiency (ASE) 70%
AMMONIA	Evacuated wood exposed to ammonia vapour at 1MPa. Plasticises wood temporarily (90° bends in 13mm stock).
ETHYLENE OXIDE	High pressure gas process over amine catalyst. ASE 65%
OZONE	Gas phase treatment degrades cellulose and lignin. Sometimes used prior to pulping.
POLYMERS	Variety of monomers introduced into wood structure then polymerised using gamma rays or heat. ASE up to 65%.

TABLE 2.2 Processes using Vacuum or Pressure Impregnation Techniques.

The simplest processes use only heat to plasticise the timber, and as long ago as 1936, Kollmann (33), reported the widespread use of the technique in Germany. Goring (34), described the mechanisms involved during the process in detail and the full description will not be included here. The results of the work can however be summarised as indicating that the polymer molecules within the structure reach a thermally induced level of vibration at which inter-molecular bonds break. Large scale relative displacement of molecular chains can then take place, with rebonding in the new positional arrangement taking place on cooling of the timber. Although at the moderate temperatures usually employed for plasticisation (100°-125°C) the deformation introduced is not permanent and can be lost if the timber is subjected to high moisture levels (35), timber treated at higher temperatures (175°-260°C) undergoes permanent deformation to levels of up to 90% (36). A commercial product of this type, "Staypak" (35), has been used successfully for exterior cladding boards in the United States.

At the expense of a degree of toughness, moisture and environmental stability can be conferred on compresssed timber by impregnating the structure with a heat curing polymer prior to compression (36). Compreg (37), is an example of such a composite, although this is usually found in the form of a laminated structure rather than solid timber.

Thermal stabilisation of timber without any mechanical treatment is also possible, as reported by Stamm (38), with the efficacy of the treatment being dependant on the temperature, the moisture content of the timber, and the duration of the treatment.

2.2 Physical Treatment Methods

The disparity between the longitudinal and transverse properties of raw timber was mentioned in the introduction to this chapter, and its effect on the methods of chemical treatment of timber was dealt with in the preceeding section. With the exception of the decorative applications of veneer, the physical treatment methods discussed in this section are aimed exclusively at the improvement of mechanical properties by the elimination of the deleterious effects of this natural anisotropy which exists within the timber structure.

The principal products which fall into this category are veneer, plywood (including laminates), fibreboard, particleboard, and paper. Since the applications of paper cannot in general be described as structural, and the subject is diverse and well covered in more specific texts, it proposed to include only the first four of these applications in this chapter.

2.2.1 Veneer and plywood

Until the late 1920's, the term "veneer" covered not only those products known by the term today, but also those products known now under the description "plywood"(39). Since plywood is manufactured by the assembly of individual veneers this is totally logical, and the technologies of the two fields therefore overlap and can be dealt with contiguously.

2.2.1.1 The manufacture of veneer

The oldest surviving portrayal of the art of decorative veneering is thought to be in the mural "Sculpture of Thebes" of about 1450BC, (39), while the oldest surviving example is said to be the throne of the young Egyptian king Tut ankh Amun from about 1350 BC, (40). The veneers used in both instances were likely to have been prepared by sawing from solid timber then grinding or sanding to produce the characteristic fine surface texture. This is obviously a wasteful process since much useful wood is lost as sawdust (as much as 50% by weight for 1.5mm veneer), and today it is only employed for very high quality veneer produced from the more exotic timbers of the world, when cost is secondary to aesthetic considerations.

The more economical alternative to sawing veneers is to slice them; the action is similar to that of a jack plane but on a much larger scale. The losses in this process are minimal since no sawdust is produced, and the only waste is that trimmed from the sheets when sizing or shaping them. An even more economical process, particularly for larger sheets, is to slice continuously or peel the veneer circumferentially from the parent log which is mounted in a lathe.

The earliest records of the slicing of veneer are contained in the French Patents granted to Charles Picot in 1834 (41), in which a veneer slicing machine is described. Dresser followed this in 1840 with a design for a rotary log peeler (42), capable of peeling logs up to 2m in length at a linear speed of between 4 and 5 m min^{-1}.

The increasing reliability and cost effectiveness of plywood led to significant increases in its popularity as a material of construction towards the end of the nineteenth century (43), and the twentieth century has consequently seen significant progress in the techniques for the manufacture of veneers. Modern veneer peelers capable of peeling 300mm diameter logs in less than 20 seconds (44), are now commonplace and the recent dramatic progress in control and automation technologies has enabled increasingly smaller diameter logs to be peeled economically. This is said to have been a major contributory factor in the continued success of the Finnish timber industry where technology to peel logs from 20 cm diameter down to 60mm cores has been perfected (45).

With very few exceptions, picea sitchensis being one of them, logs as felled are neither soft nor wet enough to be fed directly to a peeler or slicer. The first operation caried out on timber destined for veneer manufacture is therefore a plasticising process, usualy accomplished by "boiling", (at temperatures significantly less than 100°C), or steaming the as-felled log. Within these broad definitions there are numerous proprietary variations in techniques and for this reason the reader is referred to more specific texts on the subject rather than details being given here, (9, 10).

2.2.1.2 The manufacture of plywood

By cross-banding veneers together with a suitable adhesive it is possible to reduce the grain induced anisotropy of natural timber significantly. For example by orientating alternate plies at right angles, boards with equal longitudinal and lateral properties can be produced. This includes not only the expected improvements in tensile strength but also in shear and bending strengths, in stiffness and in dimensional stability. Decorative aspects of the timber are generally maintained by the use of an odd number of plies so that the visible grain in the outer or "skin" veneers runs in the same direction.

There is broad international agreement with regard to the classification of timber plywoods (46), which divides the commodity into two broad groups; veneer plywood which is defined as "plywood in which all the plies are made of veneers up to 7mm thick orientated with their plane parallel to the surface of the panel", and core plywood, defined simply as "plywood having a core", (47). The first definition is self explanatory, while the second is further sub-divided as follows:

1) Wood core plywood: "plywood having a core of solid wood or veneers".

a) Battenboard: "plywood, the core of which is made of strips of solid wood more than 30mm wide, which may, or may not be glued together".
b) Blockboard: "plywood, the core of which is made of strips of solid wood more than 7mm wide, but not wider than 30mm, which may or may not be glued together".
c) Laminboard: "plywood, the core of which is made of strips of solid wood or veneer not wider than 7mm placed on edge and glued together".

2. Cellular plywood: "plywood, the core of which consists of a cellular construction. There shall be at least two cross-banded plies on both sides of the core".

3. Composite plywood: "plywood, the core (or certain layers) of which are made of materials other than solid wood or veneers. Composite plywood with a core shall have at least two cross-banded plies on each side of the core".

For further clarification of the above the reader is directed to reference (47). Whichever form the final product is to take, the production route once the raw material has been prepared by peeling or sawing is substantially the same.

1) The prepared raw material is coated with a suitable adhesive. The nature of the adhesive will be dependant on the proposed end use of the product, for example boards intended for interior decorative purposes do not demand the use of weather and micro-organism resistant adhesives. For more information on the types of adhesive available the reader is directed to references (48) and (49), since the subject is too vast to be included in this brief overview of plywood manufacture.

2) The material from 1) above is assembled into the form it will take in the finished product.

3) The pre-assembled structure from 2) is transferred to a press for consolidation and adhesive curing. In general the presses for the production of plywood have between 10 and 30 spaces or "daylights" between the platens and so can produce a number of boards simultaneously. Although modern technology has added refinements to the equipment, the original patent for a hot platen plywood press was apparently granted to Luther in 1896 (50). The temperature of the platens and consequently that to which the plywood is raised is a careful balance between production rate and board properties, and is determined largely by the nature of the adhesive in use and the type of timber from which the board is manufactured. Similarly the specific pressure exerted on the board is also material dependant but is generally in the range 1.179 to 1.965 MPa. For more information on the effects of temperature and pressure on the production and properties of plywood, the reader is again directed to one of the standard texts on the subject, already quoted (9, 19, 50).

4) The board is machined to size and allowed to equilibrate in the atmosphere. Sizing of boards is generally accomplished by the use of automatic saws to remove the areas of the board near the edges where defects are most likely to be found. Thickness variations from board to board are eliminated by sanding or scraping the cut board to standard thicknesses.

5) Post manufacture treatment. Again depending on the final use to which a board is to be put, one or several finishing treatments may be included in the production cycle. Metal foil, plastic, phenolic film or even hardboard may be laminated to the surfaces of the board and painting, texturing and printing are all common forms of post manufacturing treatment. Impregnation with fire retardants or preservatives may also be carried out at this stage (51), although ideally this should be carried out during the veneer preparation.

2.2.1.3 Properties of plywood

The tremendous increase in the popularity of plywood for use in furniture and in construction has prompted a great deal of research into the properties of plywood, both for specific timbers and for the composite product in general. There are therefore many excellent reference works on the subject from as early as 1943 (52), including more advanced treatises based on tensor notation, (see 53, 54) and the reader is referred to these and to standard texts quoted earlier (9, 10), for detailed information on the subject. There are also British and American Standards which define the properties of plywood and methods by which they can be assessed, (55, 56).

This section is therefore limited to those properties in which plywood displays an advantage over raw timber, and typical values are quoted where appropriate. The properties in question are; moisture resistance (and dimensional stability), isotropy and homogeneity, thermal conductivity, and mechanical properties.

a) Although the equilibrium moisture contents of plywood and seasoned solid wood are quite similar at between 10 and 12%, (50, 57), those plywoods bonded with moisture resistant adhesives exhibit better dimensional stability in response to changes in the environmental humidity than do solid timbers. This is particularly true in the sheet direction due to the stabilising effect of the cross-laminated structure.

b) The elimination of the natural anisotropy of solid timber has already been quoted as one of the major advantages of cross bonded plywood.

The nature of the veneer assembly clearly also effects the homogeneity of the product, eliminating potential weaknesses such as through knots, and making machining and fabrication of the material more straightforward.

c) Poor thermal conductivity is advantageous for applications such as building construction, where heat loss needs to be minimised. Although the thermal conductivity of solid wood and plywood of an equal density manufactured from the same timber are very similar (0.12 $WK^{-1}\ m^{-2}$ for softwoods, 0.15 $WK^{-1}\ m^{-2}$ for hardwoods), the advantage of plywood lies in the ability to be able to tailor the product density and therefore thermal conductivity, to the intended application, within the constraints imposed by other properties. The possibility of combining different timbers to optimise product properties is also advantageous. Since the sound transmission loss is directly proportional to the logarithm of the weight of a wall per unit area, the ability to alter the density of plywood within limits also offers advantages in respect of sound insulation applications.

d) As was mentioned in the opening paragraph of this section, a great deal of research has been carried out on the mechanical properties of plywood, and it is beyond the scope of this thesis to deal with the subject in any detail. Since plywood is a composite material, mechanical properties are affected not only by the type of timber in use, but also by the nature and characteristics of the glue lines. The comments made above regarding the tailoring of properties to suit the applications are also relevant here, and tensile, compressive and bending strengths, moduli of rigidity and elasticity, and hardness are all properties open to manipulation.

Other properties of plywood tend to reflect the properties of the timber from which it is made, and treatments applied to solid timber, for example to impart insect or fungal attack resistance, can also be used on plywood with the same effect. The only specific property of plywood which is always poorer than solid timber is the nail and screw retention in the edge direction. This is a predictable failing owing to the wedging effect of such fasteners forcing the plies apart, and is generally overcome by joint design rather than by manipulation of mechanical properties.

2.2.2 Fibreboard

Fibreboard is produced by the assembly of ligno-cellulosic fibres, usually originating from wood or woody type materials such as bagasse. Most of the strength of such boards comes from the interlacing or "felting" together of the fibres, and their own intrinsic adhesive properties, but additional adhesive is sometimes used to confer additional properties such as moisture resistance.

Fibreboard is unique in that it is the only wood-based sheet material which is reconstituted rather than re-assembled from discrete timber particles. Attributes which this method of manufacture confers on the product include;

a) total absence of surface defects. This makes the product ideal for painting and surface finishing.

b) since much of the sugar and starch content of the timber is removed during the fibre preparation, the product is much more stable with respect to insect and fungal attack.

c) the end product has an inherently lower equilibrium moisture content than solid timber.

Broad characterisation of fibreboard is carried out on the basis of product density, with international agreement as to coarse specifications, however ISO standards are much more rigorous and grade the board according to:

1) type of raw material
2) method of sheet formation
3) density of product
4) type and place of application.

All boards still fall under two broad categories, low density softboards, or insulating boards, and higher density hardboards. It is the degree of compaction or compression during manufacture which determines the ultimate board density, and therefore the properties of the final product.

2.2.2.1 The manufacture of fibreboard

Credit for the conception of the principle of fibreboard manufacture is given to the ancient Japanese (circa 6th Century BC), but the first European patent was granted to British inventor Clay in 1772 (58), for the application of "papier mache". During the hundred years between 1858 and 1958 over 800 patents relating to fibreboard were granted and for an informed bibliography the reader is directed to references 59, 60 and 10.

There are two principal methods of converting the raw timber into fibreboard, the wet process which is used for the whole range of fibre based products, and the dry process which is used only for the medium and high density materials. Both processes require fibrous starting material however, and there are two routes to the production of the fibres, both equally suitable to either of the finishing processes.

2.2.2.1.1 Mechanical defibration

This is the technique most often favoured by large plants with consistent raw material supplies since the process is continuous. Wood particles, in the form of large chips from a chipper or hammer mill, or less commonly sawdust, are pressure fed by an Archimedian screw into the narrow gap between two segmented grinding discs. One, or both of the discs are rotated at high speed (1500 rpm is common) to produce a field of high shear between them which causes mechanical disintegration of the wood particles into their constituent fibres. Various modifications are sometimes employed to increase the productivity of the machine or improve the consistency of the product, for example high pressure steam may also be injected with the chips, but for brevity the reader is directed to reference 39 for more information on this subject rather than the details being included here.

2.2.2.1.2 Explosive defibration

This process was developed by Mason from an original idea by Lyman in 1858, and has been the subject of over 70 patents since 1924 (61). The process, known as the Masonite process, involves the use of cylindrical pressure vessels or "guns" about 2m long and 500mm in diameter. A charge of wood chips is introduced into the gun and steam at 3.93 MPa is injected for a period of about 60 seconds. At the end of this time the wood particles are fully saturated with steam and the pressure is rapidly increased to 7.9 MPa for about 5 seconds, which raises the temperature to about 300°C. This period is followed by explosive decompression of the chamber down to atmosphere, when the wood particles also explode with the release of the entrapped steam to form the fibres required for subsequent processing. The product from this process is significantly different from that produced by mechanical defibration, the lignin content of boards manufactured from it being about 38%, compared with 18-22% for boards from mechanical fibre and 26% for an average softwood.

For the wet process the fibres from the primary fibre production are taken up into a slurry to give a fibre content of approximately 5%. This is continuously agitated to prevent settling and is metered onto a moving wire mesh belt in the same manner as in the Foudrinier paper making process (for further details see reference 62). As the slurry, termed the "wet lap", moves with the belt, so some moisture drains away due to gravity, more is removed by vacuum pumps, and a further amount by passing the material through pinch rollers to thickness size the material. This partially dried material can now support a structure and is cut into board sizes. For insulating or softboard this material is then transferred to an oven and simply dried. For the production of medium and high density hardboards the sheets are transferred to a press and subjected to heat and pressure to form the desired final product.

The dry process for the manufacture of hardboard was developed by the American Plywood Research Foundation about 1945. The major incentive for the work was the elimination of the vast quantities of water used in the wet process, which caused problems both in the supply of high quality feed-water, and in the disposal of the waste water by-product (63). The process utilises air as the suspension medium for the wood fibres, and as might be expected, the interlacing or felting of the fibres is less efficient and isotropic than in the wet process.

This is particularly so in view of the degree of dryness of the fibres required to enable air suspension to be used. As a consequence, a binder or adhesive medium has to be incorporated into the boards to give them a useable level of strength, and this negates some of the economic advantage of the water elimination. The process once the binder has been introduced and the fibres have been layed into the mat is similar to the wet process in that a combination of heat and pressure is used to tailor the board properties and produce the final product.

2.2.2.2 Properties of fibreboard

The products resulting from both manufacturing routes compete in the same sectors of the market and are widely used in the building and furniture industries. The range of products and product properties available by the manipulation of the various process variables is so large that a detailed breakdown of properties is impossible within the constraints of this thesis. The reader is therefore referred to the many excellent texts already quoted in this section, and to British Standard BS 1142, "Specification for Fibre Building Boards" (1971 - 1972), which gives very detailed and specific property and testing information.

2.2.3 Particleboard

A brief history of the birth and development of the particleboard industry was given in the introductory chapter of this thesis and there is nothing to be gained from repeating the information here. The most important conclusion which can be drawn from a historical overview of the industry, however, is that there is a trend away from waste wood and towards specifically prepared timber as its raw material, as part of a search for higher quality, more consistent products. The subject matter of this section will therefore be restricted to those aspects of raw materials and process variables which influence the properties of the finished products. This will provide a source of reference for later chapters dealing with the preparation and characterisation of the feedstock material used during this research programme.

Some idea of the complexity of the interactions between the numerous variables involved in the production of particleboard can be obtained from the information presented in Figure 2.1, (64,39). It is clear from this that an investigation of individual variables in isolation is unrealistic and most of the published work has been carried out by varying groups of factors where the inter-relationships between group members are much more clearly defined. The remainder of this section will therefore be presented in this way, with only the macro effects of the factors involved being detailed.

Within the range of influences illustrated in Figure 2.1, there is a natural division between the influences dependant on the materials properties and those dependant on the pre-treatment of those materials.

2.2.3.1 The influence of material properties.

Particleboard is made up from three major components; particles, an adhesive, and additives to confer special properties. The amount of a fourth component which is always present in particleboard, water, is made up from the contribution to the total of each of the other three. Although the total amount of water present in a board is of very great significance at all stages of board production, it is generally agreed (64,39,10), that the source of the water, be it the particles, the binder, or the additives, is largely unimportant.

Dealing with each of these major components in turn then:-

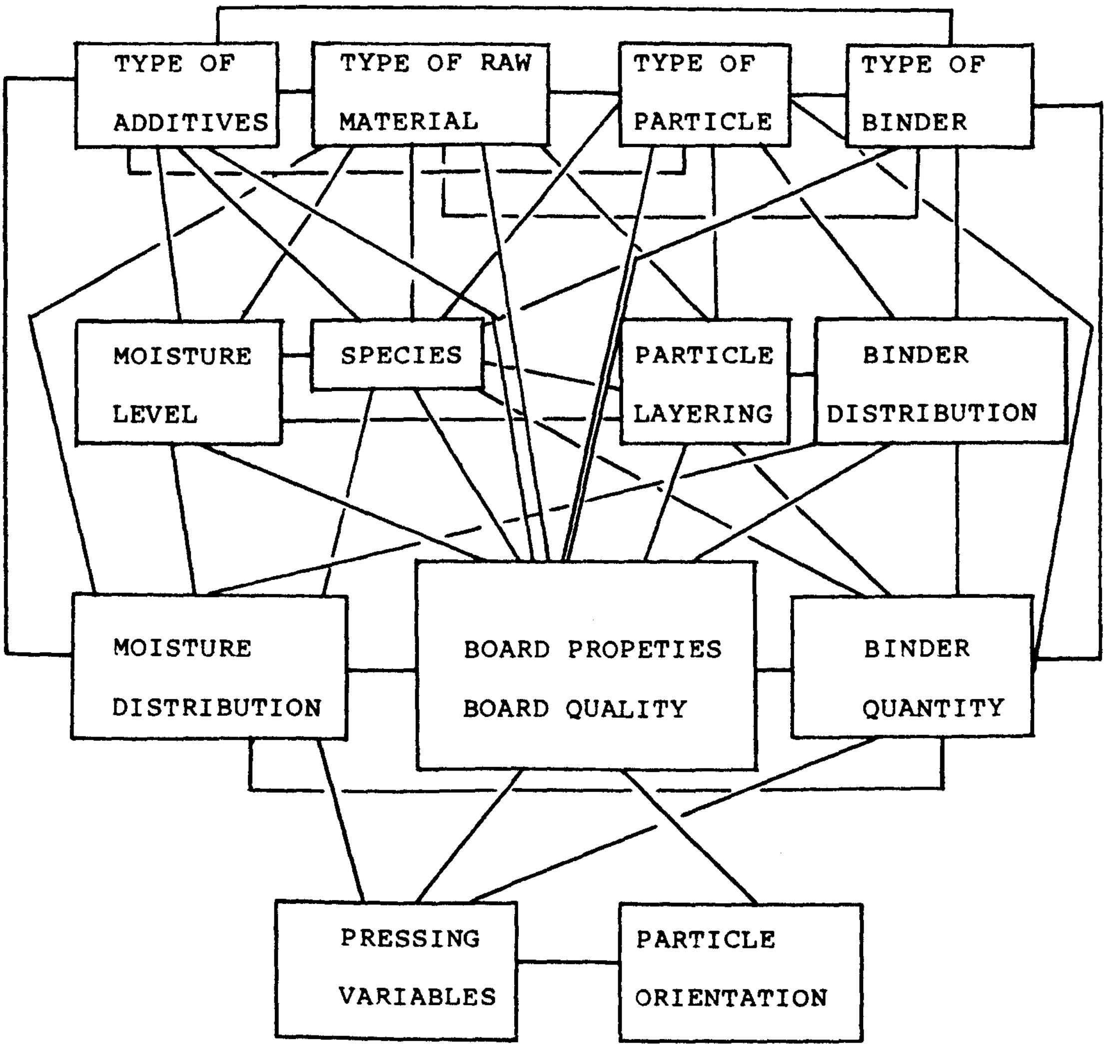

FIGURE 2.1. The possible interactions which influence the properties of particleboard.

2.2.3.1.1 The particles

Consideration is given in Chapter 4 to materials other than wood as the source of the particulate raw material. Since the vast majority of particleboard is produced from wood, however, this section will be restricted to the influences of wood particles on the properties of wood based particleboard.

Clearly the species of timber used as the raw material will have the greatest effect on the finished product, since wood will account for about 90% of the total weight. Commercial plants must therefore be based on a sound knowledge of the properties of their raw material species. This is particularly true where the major source of raw material is timber waste, which is likely to be made up from at least several timber species. The properties of the various timbers need to be known in detail so that suitable particle blends can be produced, and the properties of the other components tailored to give optimum properties in the final product using this blend. It can be seen from Figure 2.1, that all of the other variables influencing board properties are linked to the timber species and are therefore influenced by it. This is not the major complication it appears to be however, since properties within a single species are generally consistent, thus if the effect of a single property is known, then the effect of a particular species in relation to that property can be calculated. The effects of specific properties and not the effects of specific species forms the next section of this Chapter.

a) Density

This is generally considered to be the most important variable which affects final board properties, and its influence is twofold:-

1) in general the denser the wood, the smaller the area of wood for a given weight of chips and therefore the less adhesive is used to coat them - conversely the number of chips for a given weight will also be smaller, hence the inter-chip contact area will be smaller and the board proportionately weaker. The quantity of adhesive used in boards produced from high density raw material therefore tends to be higher than in low density equivalents in order to compensate for this fact.

2) The denser the wood, the less compressible the chips tend to be, and the higher the press pressure required to deform the chips to produce adequate chip to chip contact becomes. This not only results in higher capital machinery costs, but also in very high density boards which are difficult to handle and machine.

As a consequence the majority of the particleboard currently produced is manufactured from low density species which give a medium density product with a strength level which is adequate for most purposes. Because of the increased cost of boards produced from high density species, their use is limited to applications where the increased strength and hardness are specificaly advantageous.

b) Acidity

Provided a single species is used as raw material, and that the adhesive is tailored to suit the pH of that species, pH in itself has no influence on the final properties of the board. It is in the effect that the wood pH has on the curing properties of the binder that the influence of this property lies. Adhesive manufacturers have made life simple for single species particleboard mills by producing ranges of adhesives whose properties are tailored to a specifc timber species.

The rate of cure of the most common binder in use, urea formaldehyde resin, is increased by increasing temperature and decreasing pH and is consistent and therefore predictable over a range of combinations of the two. Table 2.3. shows typical pH values for a range of common hardwoods and softwoods (65) and illustrates the problems which could face a manufacturer using mixed species feedstock for this

HARDWOODS	pH	SOFTWOODS	pH
BROWN OAK (fungal attack)	2.8	YELLOW PINE (Sapwood)	2.7
AMERICAN WHITE OAK	3.9	SITKA SPRUCE	3.35
BEECH	5.0-6.0	JAPANESE LARCH	4.2
ENGLISH ELM	7.15	REDWOOD	5.15
		WHITEWOOD	6.0
		PARANA PINE	8.8

TABLE 2.3. Typical pH Values for a Range of Common Timbers (65).

process. The most desirable rate of adhesive cure is always a compromise between minimising the length of time the board must be held at termperature to effect resin cure, and ensuring that the resin does not begin to precure before the mat is consolidated and thus decrease the strength of the finished product. The pH of the furnish is therefore critical in determining the quantity of additional acid catalyst which must be added to the binder to achieve optimum production conditions. Buffering capacity at a given pH also varies from species to species and is another factor which must be included in the overall equation describing adhesive cure.

c) Permeability

Table 2.1 illustrates the differences between the permeabilities of some of the common timber species and a factor of 10,000 difference between longitudinal and transverse values was mentioned in Section 2.1.1.1. Clearly the resin efficiency of a furnish, i.e. the quantity of adhesive required to give adequate bonding at the chip/chip interfaces, will be very dependant on the permeability of the chips and therefore on the timber species present. Any resin absorbed into the chips is not contributing to the bonding process and is in effect wasted. Not only the inherent permeability of the timber is important in this respect, but the ratio of exposed end grain to normal surface is also very significant.

This aspect will be dealt with more fully later in this Chapter.

d) Moisture content

During curing, because wood is an inherently bad conductor of heat, most of the heat transmission through the board, from the faces adjacent to the hot platens to the central core of the board, occurs by the generation of steam. This forms in the surface of the mat and permeates into the centre where it sustains the resin cure. Clearly the final moisture content of the furnish is going to be critical in ensuring that the most efficient bond is created in the shortest possible press time. Too little steam generation means longer press times and the possibility of charring on the board surface. Too much water on the other hand can lead to steam pockets remaining in the board until press opening, causing the board to delaminate explosively or "blow" as the platens open. Rayner (66), covers the subject in great detail.

Once the optimum mat moisture content has been established, chip moisture content, water in the resin, and water used in the application of any additives can be controlled to achieve the optimum value. In this sense the moisture content of the feedstock also influences the economics of the process since the chips will have to be dried down to a level suitable for use. Moisture content of the raw material also exerts an influence on the chip preparation economics, since very wet timber tends to tear rather than chip and can end up with a "fuzzy" fibrous surface which is difficult to glue. A very wet feedstock does not generate much dust or fine particles during chip preparation however, and the proportion of particles of useable geometry produced is correspondingly high. Fibre breakage from wet chipping is also low, and the chips produced are consequently stronger. Chips produced from wet feedstock do have a correspondingly high moisture content, however and the energy requirement for drying the chips down to a useful moisture content is greater than for dry feedstock. The production of chips from dry feedstock involves the opposite of almost every characteristic of the wet process noted above, but does involve lower energy costs at the drying stage. Clearly if there is any choice in the type of feedstock for use in a particular plant, the decision is going to be based on achieving the optimum balance of all the factors mentioned above. Mills utilising mixed or unknown feedstock are once more faced with the need to cater for the worst possible case and adjust their processing conditions to meet the immediate requirements of the situation in hand. In extreme cases this can involve the use of large soaking pits in which to condition the logs prior to the chipping process.

e) Extractives content

The extractives contained in timber are comprised chiefly of tannins, polyphenolics, and essential oils. More specific details are given in Chapter 4, Tables 4.1 and 4.5, and as intimated in that section, most of the extractives content of a timber is contained in the bark or in the sapwood just below the bark.

Extractives can interfere with resin cure and with bond integrity, but the problems can be overcome by tailoring the adhesive system to the species in use. Mixed species feedstock is clearly a problem in this respect, and although some work has been carried out on the subject, certain species, for example Western Red Cedar (Thuja Plicata), Hemlock (Tsuga Heterophylla) and White Fir (Alies Concolor), are still largely unusable for economic production because of the problems they cause.

2.2.3.1.2 The binder

A detailed discourse on the properties of the various binder systems available is included in Chapter 4, Section 4.1 of this thesis. This present section will therefore deal only with the broad implications of binder specification with respect to board properties.

The properties of the binder will always be reflected in the properties of the board, for example if a non-water resistant binder such as casein is used, then the board will not exhibit moisture resistance either. This being the case the binder is always chosen with the desired properties of the finished board in mind.

The resin level used in the manufacture of a board exerts the most influence on the mechanical properties of the finished product, and a great deal of work has been carried out in this area (67-70). At the levels of addition generally encountered in commercial board production (6-10% by weight on dry wood) there can be no possibility of a continuous binder phase and the adhesion occurs through a series of "spot welds" between adjacent chips. The strength of the bond is therefore dependant on the number and size of adhesive droplets present on the chip surface (this will be dealt with in more detail later in this Chapter), and the adhesives used are always formulated to have greater strength than the timber which they are being used to join. Bond failure resulting in board failure almost always occurs in the wood or at the wood/binder interface as a consequence, and there is therefore an optimum level of binder addition, beyond which the return on further additions becomes less and less cost effective.

2.2.3.1.3 Additives

Since additives are always incorporated with the intention of improving or modifying the performance of the board in some way, they must by definition affect the board properties. The most commonly used additive is wax, and there are many proprietary variations on the market all tailored to produce specific end effects, or be effective with certain species of timber. The primary function of a wax addition is to confer a measure of moisture resistance to the finished board, and they are particularly effective against infrequent heavy wetting. They are frequently employed for shuttering boards for example or for boards whose end use is designed to be interior but subject to occasional flooding, such as in kitchen or bathroom flooring.

Less frequently used additives confer fire retardant or insect resistant properties on the board. The increasing use of particleboard as a major building material is likely to result in increased restrictions on the use of boards without fire retardant characteristics, and additives are therefore likely to come into much greater prominence in the near future, despite adverse effects on other board properties (71).

2.2.3.2 The influence of material treatment

Although the effects of material properties influence board performance significantly, the way in which the raw material is subsequently prepared and incorporated into the pre-pressing mat has as much if not more influence on the properties of the final product. Again this section can be divided conveniently into three parts dealing with the wood, the binder and the additives, although the actual final pressing also has considerable influence on the board properties and will also be covered in this section.

2.2.3.2.1 The particles

Although the term particle is generally used to indicate that product of the preparation operations which will be used for the board manufacture, ASTM D 1554 defines a particle as "the aggregate component of a particleboard manufactured by mechanical means from wood or other lignocellulosic material including all small subdivisions of the wood". In the context of this section however the former definition will be deemed to apply, although the term particle must not be inferred to imply any particular shape characteristic. The size, shape, and geometry of the wood particles all influence the ultimate board properties, and the preparation of the particles is thus a critical stage in particleboard manufacture.

The two alternative routes to a particle from raw timber are breaking or crushing, and cutting, and both produce quite distinct particle characteristics. For high quality board, "engineered" chips are a pre-requisite and not a luxury, random chips being far from ideal for particleboard production. Such is the influence of this preparation stage of the process that a considerable number of patents were taken out during the early years of particleboard development which dealt with this aspect alone (72,73,74). Research and industrial interests are still focussed on the area as can be judged from more modern publications on the subject (75,76).

The first exhaustive study on the subject was carried out by Turner (77), in which the general principles underlying the relationships between particle geometry and board properties were established. Heebink and Hann (78), followed up this work and Table 2.4. shows a summary of the results of their tests on oak particleboard.

In general, short, stocky chips tend not to become interwoven in the mat and therefore the board produced from them will have lower mechanical properties. Such chips also have a large end grain to total area ratio and tend to absorb more binder than longer chips might. Long slender chips, on the other hand confer the mechanical strength of the

PARTICLE TYPE	STATIC BENDING LOADING MODULUS OF RUPTURE (MPa)	MODULUS OF ELASTICITY (MPa)	PROPORTIONAL LIMIT (MPa)	TENSION PERPENDICULAR TO FACE (MPa)
Sawdust	11.1	1.72	6.3	2.12
Fines	16.6	1.88	7.5	2.38
Slivers	18.2	2.43	9.9	2.47
Slivers + 1% wax	17.3	2.54	9.9	2.35
Planer shavings	19.9	2.86	11.7	1.97
Planer shavings + 1% wax	20.0	3.21	12.3	1.80
12.7 mm flake	23.6	3.33	13.7	2.60
12.7 mm flake + 1% wax	23.6	3.45	13.4	2.49
25.4 mm flake	43.6	5.13	26.9	2.92
25.4 mm flake + 1% wax	43.5	4.76	23.4	3.01
Red Oak plywood				
Parallel to face grain	79.2	9.95	35.1	2.20
Across face grain	50.7	4.99	35.1	-
Oak timber	109.0	9.93	71.8	-

TABLE 2.4. THE EFFECT OF PARTICLE GEOMETRY ON MECHANICAL PROPERTIES OF PARTICLEBOARD (78).

interwoven structure and expose less end grain and are therefore more resin efficient. The surface of boards produced using long slender chip does not tend to be very smooth, however, since the interweaving of the chips leaves voids in the struture. A combination board comprised of an internal structure of long slender chips to confer strength with an exterior of smaller, squatter chips to give a good surface finish, therefore has many advantages. This was the basis of the patent granted to Fahrni (73), for his Novopan process. Since that time many variations on the same idea have been put forward, including graded density board, in which the particle size and shape changes gradually from large coarse particles at the centre of the board to fine, almost dust like particles at the surface. Difficulties in ensuring adequate control over the quality of graded density boards has hampered their progress and three layer board with 2mm thick fine surface layers forms the bulk of particleboard produced commercially at present.

There is clearly a need for the discrete production of both long slender particles and short stocky particles in any commercial particleboard operation. The two routes to particle production mentioned earlier owe their joint existence in large part to this requirement. Particles produced by crushing or hammering tend to have much smaller "slenderness ratios" than those produced by cutting, while the latter process also causes less fibre damage thus preserving the natural strength of the wood structure in those particles which most influence the strength of the finished board.

Although it is impossible to generalise on the effect of particle geometry on board properties because of the almost infinite number of other variables which exist, Maloney (39) summarised the effects of flake geometry on flakeboard properties as follows:

> Increasing flake thickness causes an increase in bending strength and stiffness in medium medium density boards up to the practical thickness limit. In high density boards, however there appears to be a peak in board strength below maximum flake thickness even if all other factors are held constant.
>
> The internal bond in flakeboard tends to decrease with increasing flake length within practical limits.
>
> With medium and high density boards, thickness swelling increases with increasing flake thickness. Low density boards exhibit a maximum at an intermediate value of flake thickness.

For a given flake geometry, the method of production of the flake influences the property of the board, even when other factors remain unchanged (79).

Boards manufactured from flakes with mixed geometrical characteristics tend to exhibit properties which are the "average" of the properties of board produced from the individual component particles.

Particles having a length to thickness ratio of about 200:1 appear to confer optimum strength and stiffness properties to the boards (80).

5 to 20% of fines incorporated in a board tend to lower strength and stiffness properties, but do confer a degree of short term moisture resistance.

Although the summary above deals largely with flake and flakeboard and is therefore not totally relevant to particleboard and the type of particles used for this research programme, it serves to illustrate the very empirical nature of the knowledge which exists regarding the overall technology of "reassembled" wood products. The quantity and quality of information regarding the influences of particles more closely resembling those used for this study are even lower and the successful operation of commercial ventures in this field relies largely upon lessons learned from experience, and on the manipulation of other more manageable properties to control board quality.

2.2.3.2.2 The binder

Binder preparation for particleboard production very much depends on the form in which the binder arrives at the plant, and the form in which it is used. In plants where the resin is sprayed onto the chips in a liquid state, delivery may take the form of a dry powder or a concentrated aqueous syrup. Both of these are then mixed with water to produce the liquid to be sprayed onto the particles. It is the properties of this final syrup which have most effect on the properties of the finished board. In general the catalyst to assist resin cure is mixed into the resin at this stage and the mixture sprayed directly onto the particles. Clearly the level of hardener needs to be such that optimum curing of the boards in the press is achieved without the resin curing before the furnish mat reaches the press, thus lowering bond strength. This level will be calculated with a knowledge of the press characteristics, but also with a knowledge of the timber characteristics, as mentioned earlier in this section.

The room temperature viscosity of the blended resin is also important as it influences the spraying characteristics of the syrup. Lehmann (81), found that resin efficiency is highly influenced by the degree of atomisation of the resin, which in turn is affected by the resin viscosity, other spraying parameters being constant. The significance of these findings is emphasised by the fact that between 19.6 and 27.9% less resin was required to produce board of a given strength with a fine spray than with a coarse spray. The explanation for this result is probably the increased number of "spot weld" bonds which are statistically more likely with a large number of small resin droplets rather than with a smaller number of large droplets (82).

Resin viscosity at temperatures at or about the pressing temperature is also of some importance. From Poisieuille's equation (Section 2.1.1.1) it can be seen that as the fluid viscosity decreases, so the rate at which it is absorbed into the wood increases. Thus if the resin viscosity is too low at pressing temperatures, the amount which is absorbed into the wood and therefore wasted will become significant and the bond efficiency will be impaired. If the resin viscosity is too high at pressing temperature, however, spreading of the resin due to the compression of the particles will not occur or will be limited, the area of the individual bonds will be decreased and the overall bond strength will be reduced.

In those plants where resin is delivered and used as a powder, the resin application is generally carried out by tumbling the particles and the resin together. Although this technique does have the advantage that no water is added to the furnish and the final moisture content can therefore be closely controlled, the increase in surface moisture required to help the resin to adhere to the wood can cause blowing problems during and after pressing. There are other disadvantages of dry resin application, for example segregation of resin in the mat and unpredictable dusting losses during particle transport, however the process does have an advantage in its use of higher moisture content furnish with consequent lower drying energy costs and is used in some commercial operations. High performance phenolic resins are almost always applied in the dry powder state.

2.2.3.2.3 The additives

The most important additive to be added to the furnish for particleboard is wax. There are many proprietary grades of wax, all with basically the same purpose, to confer moisture resistance to the finished board. The balance between adequate moisture resistance and impaired board strength depends largely on the quantity of wax added, and this is both binder and timber species dependant. Generally the wax manufacturers advise on rate of addition and compatibility with binder and species, and a modern commercial mill relies on the data provided by them.

The method of introducing the wax also affects the properties of the board since uniformity of cover is vital for optimum moisture resistance. Subject to compatibility, the wax can be emulsified with the resin and the resulting mixture sprayed onto the chips in the normal way. This is the most widely used method since it gives optimum moisture resistance, however if the resin and wax cannot form a stable emulsion together, then the wax can be applied by spraying it on in the molten state as a separate operation. There are proponents of both systems, but the advantages and disadvantages tend to be linked to the mechanical aspects of furnish preparation and the relative costs of the alternative systems, and both work equally well (when properly carried out) in terms of board properties.

Other additives are constrained by the same factors as is wax, efficiency of action and detrimental side effects. Most of the additives are water soluble however, and can therefore be sprayed on with the binder and thus confer maximum benefit for minimum addition.

2.2.3.2.4 Treatment of the mixed furnish

The treatment of the furnish to produce a uniform pre-pressing mat is as critical as any of the other processes mentioned previously. Uniformity at this stage will be dependant on the preceding operations as well, however, since it is impossible to obtain a consistently uniform mat if there is variability in the prepared particle feed material.

The only material variable that it is possible to adjust at this stage is the moisture content, since, as stated earlier, the location of the water in the furnish is unimportant as long as it is uniform (64,39,10). With on line monitoring of furnish moisture content, water can be sprayed onto the prepared furnish to adjust the level to that which is known to give optimum processing.

The moisture content of the furnish does not only influence board properties through curing rate, however. Hot water and steam have been used for many years as plasticisers to enable wood to be bent and manipulated without breaking (83). It follows then that if the moisture content, and therefore the quantity of steam generated, varies through the thickness of a board, then the amount of plastic deformation of the wood particles will also vary. This can be used to advantage by ensuring a higher moisture content in the surface layers of the board, when they deform and compact more on pressing. This produces a board with a high density "skin" and a lower density core, and such a combination gives a board with good stiffness and bending strength without resorting to very high density. This technique can be, and is used within the limits of board "blowing" mentioned previously to tailor the properties of the finished board.

Particle size gradation was mentioned in Section 2.2.3.2.1 as a way of influencing board properties, and this manipulation of the particle feed is always carried out at the mat preparation stage. There are many ways of achieving this effect, (most of them patented), from sifting the particles into coarse and fine fractions and then introducing them into the furnish by separate routes, to ingenious mechanical and pneumatic devices which carry out sifting and matlaying simultaneously "on-line". Since the specific techniques are not relevant to the study documented in this thesis the reader is directed to reference 39 for a particularly detailed coverage of the subject, rather than attempt to summarise the information here. Clearly particle size gradation will also affect the steam plasticising effects mentioned above, and can be used as an alternative route to the "duplex" product described there.

Provided that the particles to be converted into board are not spherical, or to some extent cubic, then there is also the possibility of particle orientation within the mat. Although often a great deal of effort is put into ensuring that the particle orientation is random, this can clearly only be the case in the plane perpendicular to that of the board, since the mat forming and pressing processing themselves will orientate the particles in the direction parallel to the plane of the board. This accounts for the differences in the directional properties of the board, tensile strength and internal bond being much greater parallel to the board face than perpendicular to it. The general exception to this state of affairs is in the case of extruded chipboard, mentioned in Section 1.1, in which orientation normal to both the board length and to a lesser extent the board thickness produces significant anisotropy in terms of board properties, but in the opposite sense to the platen pressed equivalents. So called "random" board is to be preferred therefore for general use.

Advantages in a board which had some particle alignment parallel to the board length as well as thickness could be envisaged for applications requiring good longitudinal bending and stiffness, for example for flooring panels. Several techniques for introducing this kind of anistropy have been developed, including mechanical combing devices, electrostatic charge devices (84) and combinations of the two. The effects of particle orientation on the properties of board made from hammer mill particles were investigated by Talbot (85), who showed that the properties of suitably aligned board were equivalent to those of high quality sawn timber. Not only has this made the use of particleboard acceptable in limited structural applications, but it has also enabled timber species which produce low strength "random" boards to find application in more demanding areas.

Press closing time, defined as the time taken to close the platens to final board thickness once they have come into contact with the mat, was recognised as having a significant effect on board properties as long ago as 1959 (86). As well as the influence on surface precuring mentioned earlier, press closing time can also influence the board density profile independant of the moisture content. The faster the press closes, the more compaction of the surfaces of the mat takes place before the onset of steam generation and the thicker the high density face layers tend to be. Clearly since the wood is not plasticised during compaction, an increase in the pressure required to produce a given thickness of board also accompanies the reduction of press closing time. Conversely, slower press closing times permit more extensive cure of the binder before compaction is complete and can result in lower density board surfaces. If this causes problems then boards can be pressed over thickness and the outer low density layers removed by sanding. The temperature of the platens is also linked to board properties through its effect on density. In the same way as variations in moisture content affect rate of resin cure through the board, so changes of platen temperature will have an equivalent effect on furnish of uniform moisture content.

By manipulating all, or any of the variables mentioned in the previous two sections of this Chapter, the particleboard manufacturer has the ability to tailor the properties of his boards to suit the end user's needs, the only limit being the extent of his knowledge regarding the complex actions and interactions of all the variables involved.

Although there is a considerable amount of information in the literature regarding the finishing of boards subsequent to pressing, it is felt again that as this has little or no relevance to the study documented in this thesis, reference to the standard texts quoted in this Chapter is more efficient than including this information here.

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CHAPTER THREE. RHEOLOGY, FLOW, EXTRUSION AND TRIBOLOGY

In any description of an extrusion process there are three clear areas which must be covered; the properties of the material to be extruded, the formal mechanical description of the extrusion process, and the prediction of the response of the material to that process. A further complication in the system under examination for this study is the thermosetting process required to provide ultimate product strength and rigidity. This clearly affects all three of the areas above since the curing is dynamic and the material is constantly changing in nature during the process.

Bearing in mind the oddities of the overall material system under investigation, and the almost unlimited range of variables open to manipulation, it was considered extremely unlikely that any one branch of science would be able to furnish all of the background information required to describe the system in the terms outlined above. For this reason, and because of the technological orientation of the work in terms of process development, background information was gleaned from review articles of the contributing fields whenever possible, and original papers were consulted only in the key areas identified during these preliminary overviews.

It was considered reasonable to assume that the behaviour of the system was likely to have more in common with polymer systems and soil mechanics than with ideal Newtonian fluids and dilute dispersion rheology, and the emphasis throughout this chapter therefore reflects this view.

3.0 Universal Definitions

Although the material for this chapter has been drawn from a range of disciplines and sub-disciplines, there are certain terms and definitions, (though not always symbols) which are common to most of them. This section is therefore included as a source of reference to those terms which occur frequently throughout the chapter, repeated definitions of which would not only be inconvenient but also be tedious to the reader.

SHEAR STRENGTH - In soils, defined as that property which enables a material to remain in equilibrium when its surface is not level. Generally defined as the resistance of a material to a shear stress of critical value immediately prior to yield occurring.

SHEAR FORCE - The resolved force normal to the direction of the principal applied force on a body.

VISCOSITY (η) - A measure of the internal friction in a fluid defined by the ratio of shear stress to the corresponding rate of shear.

$$\text{Viscosity} = \frac{\text{shear stress}}{\text{rate of shear strain}} = \frac{T}{\dot{\gamma}}$$

SHEAR STRESS (T) - Defined as the applied shear force divided by the area over which it acts, the shear face.

SHEAR STRAIN (γ) - Defined as the amount of shear displacement divided by the distance between the shearing surfaces.

PRINCIPAL PLANE - A plane that is acted upon by a normal stress only. There is no tangential or shear stress present on a principal plane.

PRINCIPAL STRESS-The normal stress acting on a principal plane. At any point in a body the applied stress system that exists can be resolved into three principal stresses which are mutually orthogonal.

3.1 Rheological Background

Rheology owes its name to a statement, reputedly made by Heraklitus about 495 BC, (1), "panta rhei" or "everything flows". It was formally defined as "the science of the deformation and flow of matter" at the first Conference of Rheologists, the inaugural meeting of the American Society of Rheology in December 1929 in Washington DC, USA (2). By definition, therefore, the science does not confine itself to the study of fluids but also deals with the flow of less mobile matter such as glass and even rock. Although the rheological system under investigation in this thesis falls nearer the latter category than the former, an overview of the rheological principles involved in describing more free flowing systems and "ideal fluids" is necessary for completeness.

3.1.1 Ideal or Newtonian Fluids

The simplest case of fluid behaviour, that of a so-called "ideal fluid", was described by Newton (3). Viscosity is defined as the ratio of the shear stress to the rate of shear strain in a system, and the ideal Newtonian fluid is defined as one in which this relationship is constant. This relationship between a stimulus and a response is clearly a function of the "constitution" of the system and equations of motion describing the relationship are therefore referred to as "constitutive equations". The very simple constitutive equation of a Newtonian fluid is therefore

$$\eta = \tau/\dot{\gamma} \qquad 3.1.-1$$

where η = viscosity in Pascal seconds (Pa.s), τ = shear stress in Pascals (Pa), and $\dot{\gamma}$ = shear rate in reciprocal seconds (s^{-1}). In a Newtonian fluid therefore, the plot of shear stress against strain rate at constant temperature and pressure would be represented by the solid line OA in Figure 3.1.

The many explanations for the phenomenon of viscosity centre on the molecular interactions within fluids causing varying degrees of "slipperiness", and, although the ideal Newtonian fluid is defined as ;being incompressible, those external influences which affect the kinetics of molecular interactions are clearly going to affect viscosity. Such is the case with both temperature and pressure, and although exact predictions of their effects have not yet been formulated, the dependance is described approximately by the following equation:

$$\eta(T,P) = \eta_o \exp \frac{\Delta E}{R} \left[\frac{T_o - T}{T_o T} \right] \exp \beta(P-P_o)\ldots \qquad 3.1.-2$$

where η_o is the viscosity at T_o, P_o (reference temperature and pressure), ΔE is the activation energy for flow, R is the universal gas constant, and β is a material specific property (m^2/N).

All gases and most homogenous liquids behave as ideal Newtonian fluids.

3.1.2 Non-Newtonian Fluids

Non-Newtonian behaviour is exhibited by solutions, suspensions, molten polymers and most fluids which are non-homogenous, although many such media will behave in a Newtonian manner over part of the stress/ strain curve. When the stress/strain curve is non-linear, as for example line OB in Figure 3.1, the viscosity of the fluid can be defined in two ways for any given rate of shear.

The apparent viscosity, η_a, is given by the slope of the secant OC from the nominated point on the curve to the origin O, and is represented by

$$\eta_a = \tau / \dot{\gamma} \qquad 3.1\text{-}3$$

The consistency of the fluid, η_c, at the same shear rate corresponding to point C is given by the slope of the stress/strain curve at that point. It is therefore an instantaneous value given by the formula

$$\eta_c = d\tau / d\dot{\gamma} \qquad 3.1\text{-}4$$

and clearly is always less than or equal to the apparent viscosity.

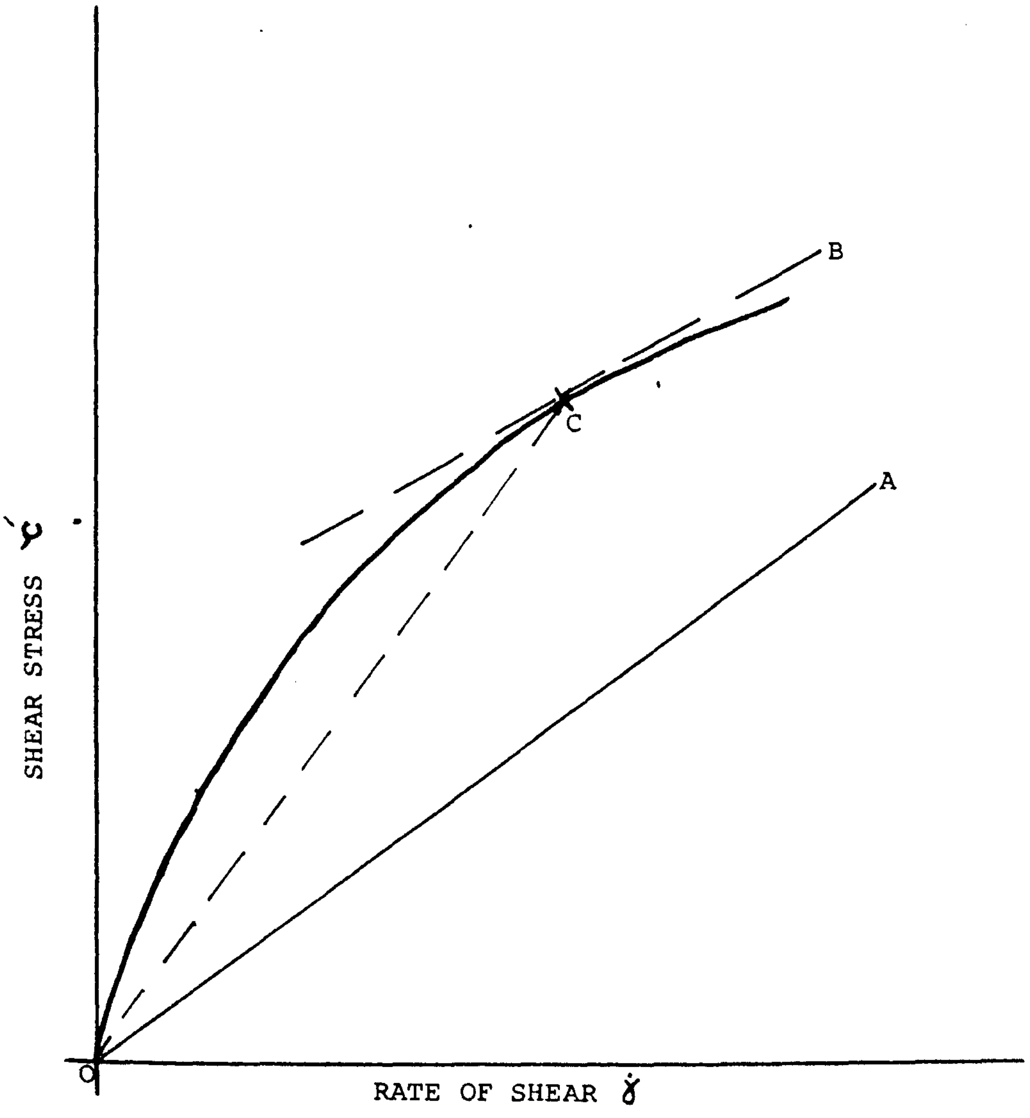


FIGURE 3.1 Shear stress vs strain rate curves for typical Newtonian (OA), and non-Newtonian (OB) fluids.

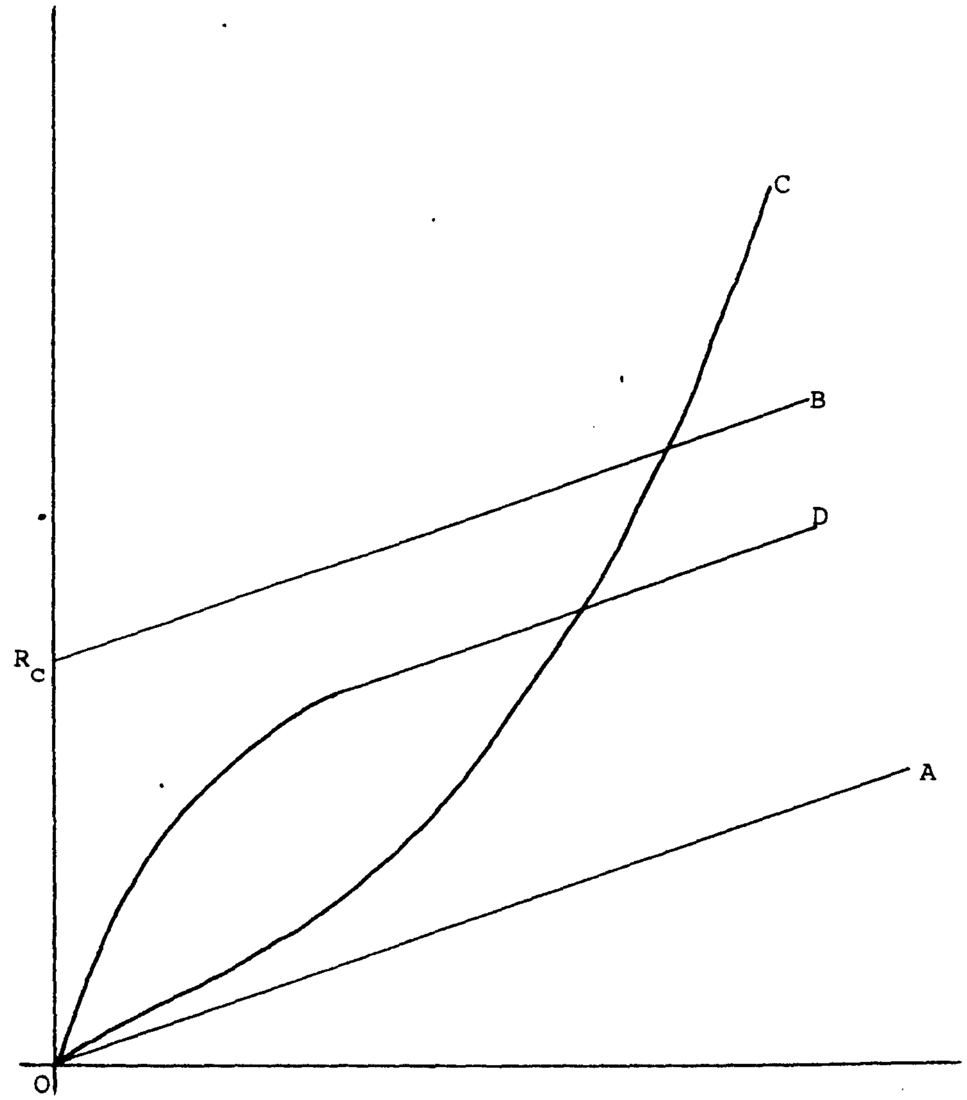


FIGURE 3.2 Shear stress vs shear strain curves for four different systems. OA=Newtonian Fluid, RB=ideal plastic "Bingham Body",

Curve OD represents a less severe form of the behaviour illustrated by curve OB in Figure 3.1 and is characteristic of a range of materials described as "pseudoplastics". Such materials exhibit their maximum apparent viscosity at zero shear rate, hence the alternative term of reference for such systems is "shear thinning". A suspension of polystyrene in toluene is an example of such a system.

The behaviour of both the dilatant and the pseudoplastic systems can be described approximately by a suitable power law equation which holds good over several decades of change in shear rate. The two basic equations are:

$$\tau = K\dot{\gamma}^{n} \qquad 3.1\text{-}6$$

and

$$\eta_a = K\dot{\gamma}^{n-1} \qquad 3.1\text{-}7$$

where K and n are constants (5, 6). For Newtonian behaviour $n = 1$ and $K = \eta_a$, with the difference between n and unity indicating the degree of departure from Newtonian behaviour. Although n normally takes values of less than one (pseudoplastic behaviour, for example polymer melts), values of $n > 1$ do exist and indicate dilatant behaviour.

Although there is some agreement that in the systems which most commonly display pseudoplastic power law behaviour, polymer melts and dispersions, the role of polymer chain interaction has the overriding effect on the shear rate dependance of viscosity there is less agreement about the form which these interactions take. Büche (7), Graessley (8) and Williams (9, 10) have formulated complex theories to explain the phenomenon. Cross (11), on the other hand developed a general empirical equation which enabled curve fitting to be carried out on curves of the typical sigmoidal shape. The formula is as follows:

$$\eta_a = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + \Omega \dot{\gamma}^m} \qquad (3.1\text{-}8)$$

where η_0 and η_∞ are the limiting Newtonian viscosities at very low and very high shear rates respectively, and Ω and m are material dependant constants. In polymer systems m is generally taken to be $(\bar{M}_n/\bar{M}_w)^{0.2}$, where $\bar{M}_n$ is the number average molecular weight and $\bar{M}_w$ is the weight average molecular weight and is therefore generally within the range 0.66 to 1.0. Ω frequently assumes a value of unity.

The complex theories of Bueche, Graessley and Williams do approximate that the power law equation at high shear rates. For monodisperse polymers, Bueche predicts a value of $n = 0.5$ for the power law equation, Graessley a value of $(n-1) = -9/11$, and Williams $n = 0$, showing the wide disparity between the theoretical predictions, however published experimental data still shows enough variation for all the values to be valid.

All of the preceding discussion has been based upon viscosity measurements under shearing conditions. A further property, which is particularly important in polymer studies and polymer processing is the tensile or extensional viscosity, measured, as the name suggests, while the system is under tensile loading. An applied tensile stress, σ, is calculated from the formula

$$\sigma = \frac{\text{Force to cause elongational flow}}{\text{Area normal to the flow}} \qquad 3.1\text{-}9$$

and the corresponding strain, ϵ, from

$$\epsilon = \frac{L_t - L_o}{L_o} \equiv \ln (L_t/L_o) \qquad 3.1\text{-}10$$

where L_t = instantaneous length at time t and L_o = original length. The tensile viscosity, η_t, is calculated from the formula

$$\eta_t = \frac{\sigma}{d\epsilon/dt} \qquad 3.1\text{-}11$$

For Newtonian liquids it has been shown (12), that the tensile viscosity is three times the shear viscosity, the Trouton relationship, however for polymeric liquids the value maybe many times more. In polymer processing, particularly fibre spinning, extrusion and injection moulding where polymer is flowing through tubes or channels in which the cross-section is decreasing, tensile or extensional viscosity is of extreme practical importance.

The final common form of viscosity evaluation is carried out using shearing plates whose relative velocity varies in sinusoidal manner. Viscosity measured in this way is termed complex viscosity, η^*, and angular frequency becomes analogous to the rate of shear term which appears in simple shear type rheological measurements. The complex viscosity is defined by the relationship

$$\eta^* = \eta' - i\eta'' \qquad 3.1\text{-}12$$

where η' is the dynamic viscosity and η'' is the imaginary viscosity of the system. i represents the $\sqrt{-1}$, and has no significance except that by using the complex plane, the representation of orthogonal vectors is made relatively simple.

If γ_o and τ_o are the maximum values of the shear strain and shear stress respectively, then at any time t, the instantaneous values are,

$$\gamma = \gamma_o \sin wt \qquad 3.1\text{-}13$$

and $$\tau = \tau_o \sin (wt + \delta) \qquad 3.1\text{-}14$$

where w is the sinusoidal frequency and δ is the phase angle between the stress and the strain.

δ has its origins in the nature of the material. In a perfectly elastic material, stress and strain are completely in phase, while in a perfectly viscous material the strain lags the stress by 90°, i.e. the strain is at a maximum when the stress is passing through the axis of the sine wave. In a perfectly viscous material then, δ would be 90°. It is the imperfect viscous nature of a fluid which causes that fluid to behave in a non-Newtonian manner, however, hence in a real system, δ will have a value of between 0° and 90° depending on the combination of properties of that system. It is clear that if the maximum phase difference is 90°, then it is relatively easy to resolve both the stress vector and the strain vector along two axes 90° apart and thus obtain values for those components of stress and strain which are in phase and those which are totally out of phase with each other. It is these values which are then used for the determination of the component parts of the complex viscosity, with the in-phase components resulting in the dynamic viscosity and the out-of-phase components resulting in the imaginary viscosity.

In the same way that in conventional mechanics, Youngs Modulus (E) can be calculated from values of stress (σ) and strain (e) thus

$$E = \frac{\sigma}{e} \qquad 3.1\text{-}15$$

so an absolute shear modulus, $|G|$, can be defined as the magnitude of the stress vector divided by the magnitude of the strain vector,

$$|G| = \frac{|\tau|}{|\gamma|} \qquad 3.1\text{-}16$$

It is conventional in the case of complex viscosity to break this relationship down into the in-phase and out-of-phase components of the modulus so that G' represents the in-phase shear modulus and G" the out-of-phase shear modulus, a single prime indicating what is termed a storage function and a double prime a loss function. The development of these relationships in terms of the complex viscosity described by equation (3.1-12) defines further characterisation of a non-Newtonian fluid system.

It is assumed that part of the energy imparted to a system to cause deformation or flow will be converted into kinetic energy, (movement and heat), and will therefore be lost from the system and be non-recoverable. This part of the energy will

therefore be lost from the system and be non-recoverable. This part of the energy will

therefore be related to the dynamic viscosity and, by analogy, to the steady state viscosity of a Newtonian fluid. The close correlation of steady state viscosity, (η), and the dynamic viscosity, (η') at low frequencies or rates of shear was confirmed experimentally by Schroff (13), and the out-of-phase shear modulus (G"), calculated using η' in the formula

$$G'' = \omega\eta' \qquad 3.1\text{-}17$$

is consequently also known as the "Loss Modulus" of a system.

The dynamic shear modulus (G'), comprises of the in-phase components of the complex shear modulus (G*, defined as G' + iG"), and therefore represents the work expended in causing elastic deformation of the material, which is therefore recoverable. It is defined by the relationship

$$G' = \omega\eta'' \qquad 3.1\text{-}18$$

where the imaginary viscosity η'' represents a measure of the elasticity or stored energy of a system. At low frequencies when η' is independant of ω(viz $\eta' = \eta$ as stated previously) the value of G' and hence the elasticity of a system is very small, and at high frequencies it tends towards a maximum value of about 10^5 or 10^6 Nm^{-2}, a value which is comparable to that of an elastic band. In some systems this value may exceed the value of G" which has also been found to pass through a broad maximum at high frequencies (14). [Similar examples of non-Newtonian flow have also been observed in low frequency, large amplitude oscillatory tests (15), and Simmons (16), showed that the superimposition of steady state flow on oscillatory motion in a dynamic test caused an upward shift in the frequency at the onset of non-Newtonian behaviour. This latter effect is thought to be related to description of entanglements within the polymer structure by the steady state flow.] The ratio G":G' can be shown to be equal to the tangent of the phase angle,

$$\tan \delta = \frac{G''}{G'} \qquad 3.1\text{-}19$$

This value is known as the loss tangent or dissipation factor and is the ratio of the mechanical energy dissipated to that stored, per cycle.

Over the range of frequencies or strain rates for which values have been obtained, it has also been shown that η^* has a close correlation with η_a (the apparent viscosity, discussed earlier), a relationship which was subsequently verified for a range of polymer systems, (16,17,18).

It will be clear by now that the trend throughout this section has been away from simple fluid systems which exhibit very simple stress/strain relationship and towards complex systems which are even yet not fully described and which exhibit properties more usually associated with solid rather than liquid systems. The final section touched on the topic of visco-elasticity, which is a whole subject area in its own right, and an in-depth treatment is therefore beyond the scope of this thesis. Where specific areas of the topic are relevant or are required for discussion or clarification of detail they will be included as when necessary, but the reader is referred to the excellent work by Aklonis and MacKnight for more detailed study of the subject should this be required (19).

3.2 Flow

Flow, whether it is flow of solids, liquids, gases or some substance which is a mixture, or has the properties of, any or all of these three, is vitally important to both chemical and physical processing. The subject is therefore well researched and formulae developed in the nineteenth century are still in common use today. As a consequence there is a great deal of information on the subject available in the reference books for most branches of science and technology, and therefore for brevity only work of particular relevance or historical interest will be included in this section.

3.2.1 Fluid flow

Whether a fluid is flowing over a surface or through an enclosed pipe, the pattern of the flow is related to the velocity and physical properties of the fluid, and to the geometry of the surface. Reynolds (20), examined the problem in some detail in 1883, and the results of his work still form the basis of many chemical engineering solutions to flow problems to this day. In his work he identified two easily discernible types of flow:

> that in which the flow appears to exist as parallel streams which do not interfere with each other and in which there is therefore no bulk movement of fluid at right angles to the flow direction. This he termed laminar or streamlined flow.

and

> that in which lateral oscillations in the fluid break up to form eddies and in which there is therefore dispersion of the fluid across the tube section. This he termed turbulent flow.

The experiments also indicated that for a given fluid in a pipe of fixed cross section there was a critical velocity above which laminar flow broke up into turbulent flow. By using different diameters of pipe and by changing the temperature of the experiments to change the viscosity of the fluid, Reynolds was able to develop an empirical criterion for flow in enclosed pipes based upon the velocity of the fluid, the diameter of the tube, and the viscosity and density of the fluid. The resulting criterion, aptly called the Reynolds Number, (R_e), is a dimensionless value obtained from the formula

$$R_e = \frac{d.u.\rho}{\mu} \qquad 3.2\text{-}1$$

where d is the pipe diameter, u is the linear flow velocity, ρ is the density of the fluid and μ its viscosity.

It is generally accepted that for conditions resulting in a value of R_e less than 2000 the flow is usually laminar, while for values greater than 4000 it is usually turbulent. The transition between the two conditions occurs gradually between the two values when the flow is unstable and oscillating between laminar and turbulent, with the proportion of turbulent flow increasing as R_e approaches 4000. Turbulent flow is clearly unstable by its nature and if conditions alter such that R_e becomes less than 2000, the flow will become streamline some distance from the transition point. Streamline flow, however, once established, has been shown to be stable in the absence of any disturbance. If the pipe diameter is increased gradually, established streamline flow can persist at R_e values of 40,000, when they are reached in this way.

Although in most chemical engineering applications, turbulent flow is desirable because of the high. rates of heat and mass transfer which accompany the rapid mixing of the fluid elements, the pressure drop along a pipe is greater for turbulent flow than for laminar flow. This, coupled with the increased velocities required, generally means that the energy required for pumping purposes is also greater. Again Reynolds demonstrated empirically that below the critical velocity the pressure gradient is directly proportional to the fluid velocity, whilst above the critical velocity, the proportionality is the velocity raised to the power of 1.8.

Following up the work by Reynolds, Stanton and Parnell (21), investigated the effect of surface roughness on the pressure drop along a cylindrical pipe. Again a dimensionless group was used to describe the experimental conditions, this time

$$\frac{R}{\rho u^2} \qquad 3.2\text{-}2$$

where p and u are as before, and R is the resistance to flow per unit surface area of pipe. By plotting this function against Reynolds number they discovered that for a given surface condition, a single curve could represent the results for all fluids, pipe diameters and velocities. In common with Reynolds' work, three distinct regimes were shown to exist.

For Reynolds' numbers of less than 2000, $R/_{pu}{}^2$ was independant of the surface condition of the pipe.

For Reynolds' numbers of greater than 2500, $R/_{pu}{}^2$ varies with the surface roughness.

Depending on the fluid, at very high values of R_e (20,000 to 100,000), $R/_{pu}{}^2$ becomes independent of the Reynolds number and varies only with the surface roughness of the pipe.

Several workers have investigated these phenomena (22,23,24,25), and there is agreement that the effects can be explained by the presence of a so-called "boundary layer" within the flowing medium. In a flowing system, the fluid in contact with the stationary walls must also be stationary, since otherwise the velocity gradient and shear stress at the interface would be infinite. A velocity gradient must therefore exist through the fluid from the wall to a point where the velocity of the flow front becomes essentially constant (clearly if the flow in the bulk of the fluid is turbulent, then within each eddy the instantaneous linear velocity of a given point must be continuously varying, but the average velocity can still be constant). The viscous forces within the fluid will determine the efficiency with which the wall induced drag is transmitted through the fluid and will therefore also determine the thickness of the boundary layer. Because of the velocity gradient within the boundary layer, the Reynolds number will also vary with distance from the wall, hence the boundary layer can be further subdivided into laminar and turbulent sub-layers.

For low Reynolds numbers, when the velocity is low or the fluid very viscous, the boundary layer can be totally streamlined and the velocity at any distance from the surface is a function only of that distance. In the extreme this represents totally streamlined flow when the thickness of the boundary layer is equal to the radius of the pipe.

Under more typical conditions, however, there exists a certain critical thickness beyond which the streamlined flow becomes unstable, and a transition from laminar to turbulent flow begins. The thin streamlined layer is termed the laminar sub-layer, and the zone in which the transition from this state to the fully developed turbulent boundary layer occurs is known as the buffer layer. Even under apparently fully turbulent flow, a laminar sub-layer of finite thickness has been shown to exist, and its thickness can be calculated from the formula (26):

$$\frac{d_b}{d} = 62\ R_e^{-7/8} \qquad 3.2\text{-}3$$

where d_b is the thickness of the laminar sub-layer, d is the diameter of the pipe, and R_e is the Reynolds number. It is clear from the equation that the laminar sub-layer is very thin under most practical circumstances, however its presence does go some way to explaining the results obtained by Stanton and Parnell. At low Reynolds numbers, surface asperities can all fall within the laminar sub-layer, and under these conditions all surface finishes will appear to the bulk flow of fluid as a hydrodynamically smooth pipe. As the Reynolds number increases, so larger asperities will exceed the thickness of the laminar layer and will influence the flow. This explains the dependance of flow resistance on surface roughness and hence on the size and number of asperities, above the critical value of Reynolds number. In the previous two instances, the resistance to flow is due largely to the frictional drag at the fluid surface, so called skin friction. Under more turbulent conditions still, when the surface is very rough or the Reynolds number is so high that it appears very rough, skin friction becomes less important, and the drage due to the formation of eddy currents as the fluid impacts upon the asperities becomes the dominant resistance to flow. This is termed form drag, and since it involves dissipation of the kinetic energy of the fluid, the losses are proportional to the square of the fluid velocity. The Reynolds number at which the onset of this behaviour occurs is dependant only on the surface finish of the pipe wall.

All the preceding descriptions are based on non-compressible ideal fluids, although the solutions given do apply very closely to most liquid fluid flow. If the fluid is compressible, however, the above simplifications cannot generally be applied since thermodynamic effects must also be taken into account. The "fluid" under consideration in this investigation does not fall neatly into either of these descriptions, however, and the following section on granular flow is therefore necessary in order to complete the range of flow systems which may exist within the process.

3.2.2 Granular flow

Although the flow of powders and granular masses is frequently encountered in industrial applications, for example the flow of polymer granules in the hopper of an injection moulder at one extreme and the flow of powdered coal in the fuel hopper of a power station boiler at the other, the science of the mechanisms involved is sparsely investigated. Much of the pioneering work on granular masses dates back to the Nineteenth Century (27), and as was the case with Reynolds and fluid flow, many of the empirically developed laws and theorems are still in wide usage today.

Most powders or granular materials can be characterised as either cohesive or non-cohesive systems. Cohesive systems, as the name implies, are those in which adjacent particles adhere to each other, or exhibit some sort of particle interaction. This interaction may be due to one or several of a variety of factors, for example:

1) Rough particle surfaces or convoluted shapes may give rise to mechanical interlocking of particles.
2) The surfaces of the particles may be coated with an adhesive substance.
3) There may be interparticle forces present, for example magnetism in ferromagnetic materials or electrostatic changes on electrically insulating materials.
4) The presence of a fluid at points of interparticle contact may exert surface tension forces.

In non-cohesive systems, on the other hand, particle/particle interaction is limited to physical contact, and individual particles otherwise obey Newtons laws of motion.

A major difference between the flow behaviour of granular material (both cohesive and non-cohesive) and ideal Newtonian fluids is in their response to hydrostatic pressure. While as mentioned earlier in this chapter, the viscosity of Newtonian fluids is largely independant of hydrostatic pressure, the equivalent property of a granular mass is extremely dependant upon the pressure exerted on its surface. For this reason, instruments used in the evaluation of the properties of granular masses generally incorporate some mechanism by which a normal force can be applied to the material surface.

An instrument having its origins in the familiar rotational viscometer is frequently used, and is shown schematically in Figure 3.3. The means of applying the normal loading is obvious, and the roughened surface on the rotor enables the transmission of forces into the material under test which clearly cannot "wet" the rotor as a fluid would. A further complication to such testing is the property, quantified by Rankine (27), that powders and granular masses cannot transmit hydrostatic pressure in the way that fluids do, and as a consequence the normal force will vary with distance from the material surface. The horizontal pressure at any distance from the surface of a powder bed can be calculated approximately by the use of Rankine's formula:

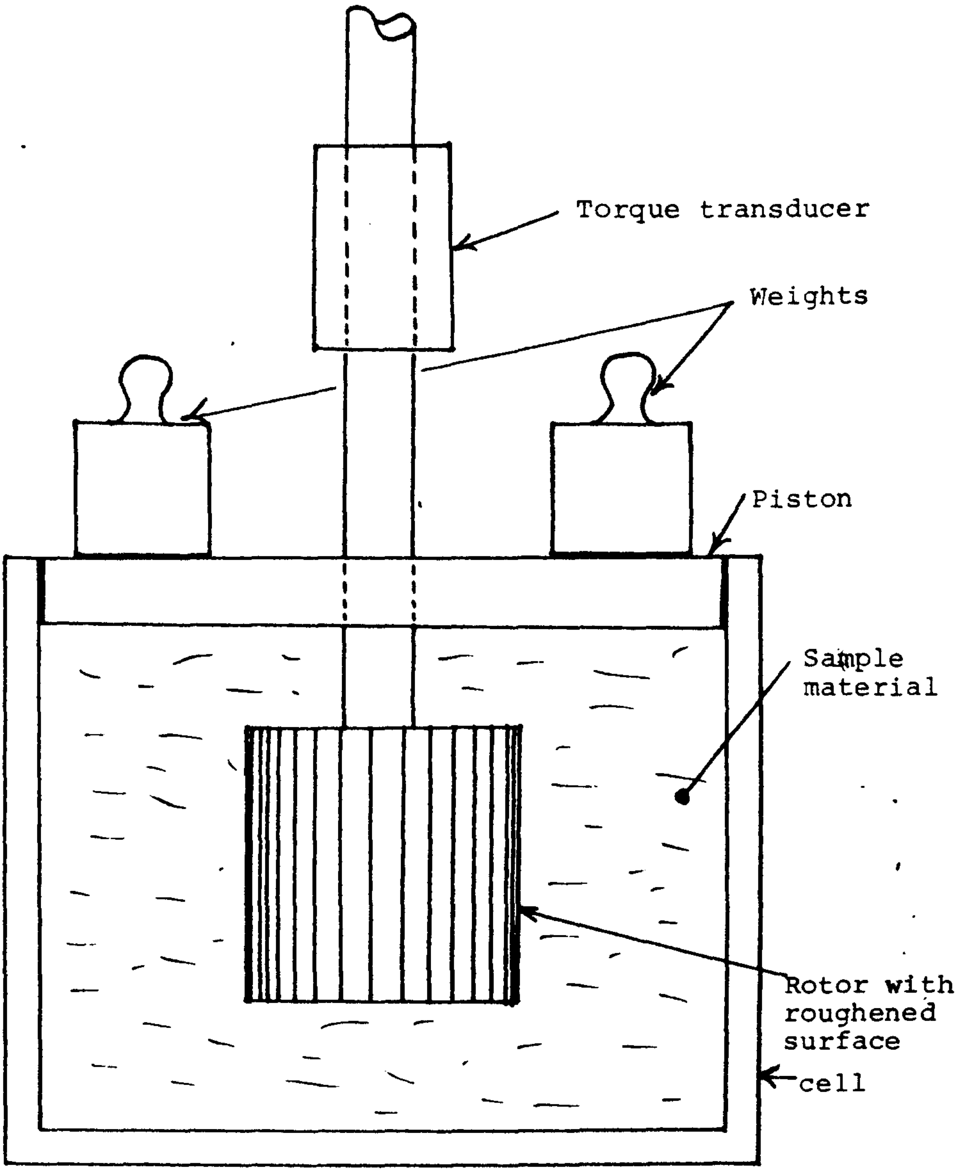


FIGURE 3.3. Schematic diagram of rotary viscometer for granular materials.

$$P_{horizontal} = P_{vertical} \times \frac{1 - \sin \phi}{1 + \sin \phi} \qquad 3.2\text{-}4$$

where ϕ is termed the internal angle of friction of the material in question.

A second type of powder rheometer is illustrated in Figure 3.4. This is a modification by Jenike (28), of the apparatus commonly found in soil testing laboratories for the determination of shear strength of soils, and is generally termed a shear cell apparatus. In this instrument the force required to cause the mass to shear for a given value of normal loading is measured.

More basic methods of characterisation have also been employed from time to time, such as the measurement of the time for the flow of a given amount of material through an orifice of known dimensions (which clearly could not be used for cohesive materials which will not flow), and the measurement of the angle of repose of a mound of the material formed by dropping it from a known height. Neither of these two methods is universally applicable, unlike the first two mentioned, hence their use is very limited, particularly for scientific work.

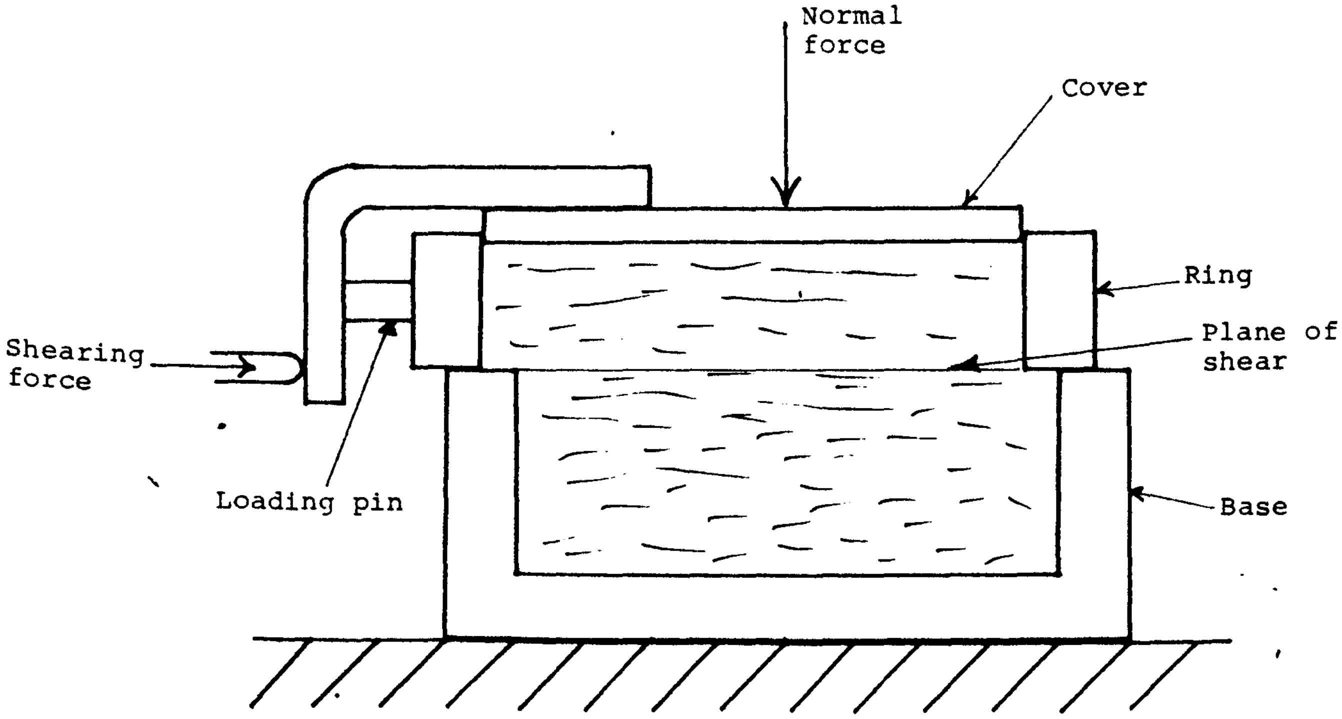


FIGURE 3.4. A Jenike Shear Cell (28)

The differences between the flow of granular materials and that of Newtonian fluids are illustrated very clearly if results from rotational viscometer experiments with both systems are plotted on the same axes.

Figure 3.5 shows the rotor torque as a function of number of rotor revolutions for three systems: a loosely packed powder, the same powder after consolidation, both of these under the same normal load, and a typical Newtonian liquid. The torque for the loosely packed powder increases monotonically as the number of revolutions increases, while that for the consolidated powder rises to a high peak very rapidly then decays to the value of the loose powder. The torque for the Newtonian liquid, on the other hand, remains constant throughout the experiments.

If, instead of number of revolutions, speed of rotation is plotted against rotor torque, the results are as shown in Figure 3.6. In this case the results for two granular materials under the same normal load are shown, illustrating the difference in magnitude but similarity in behaviour between two granular masses. The Newtonian liquid behaves as would be predicted from the information given in section 3.1.1 of this chapter. The behaviour of the two granular samples illustrated is more characteristic of solid state frictional behaviour than of viscosity, since

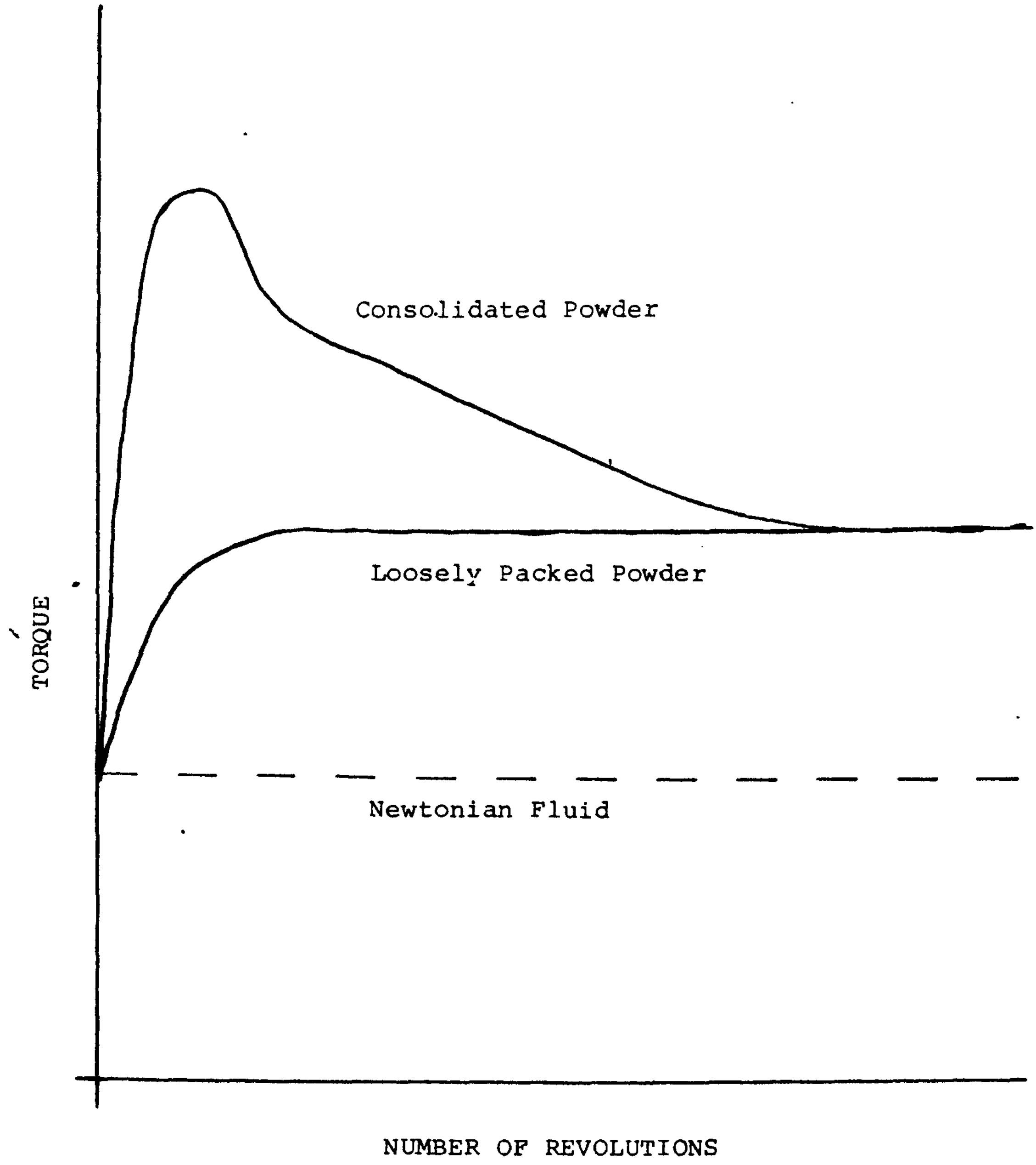


FIGURE 3.5. Effect of duration of rotation on three dissimilar systems under constant normal loading.

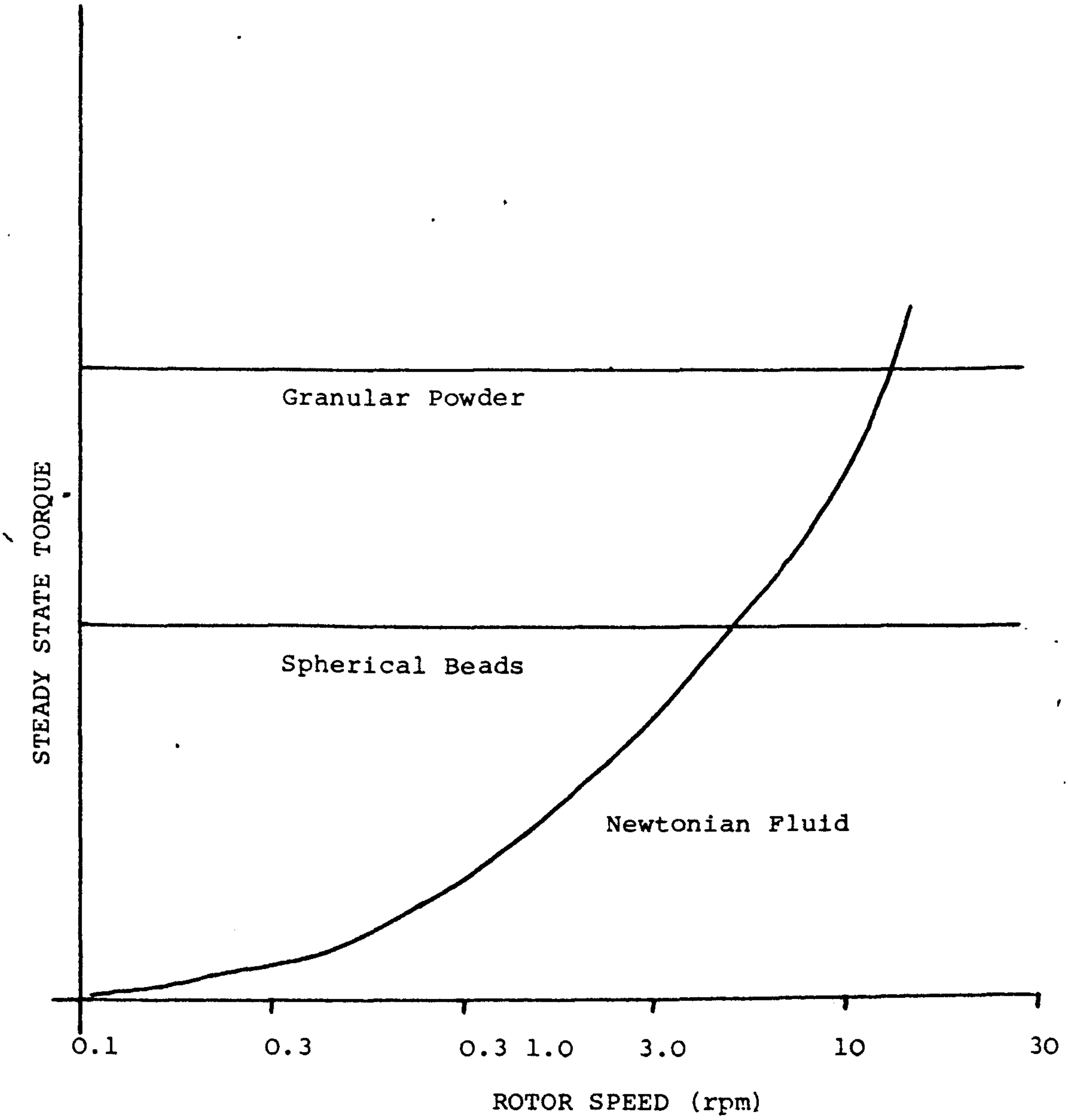


FIGURE 3.6 Difference in the relationship of torque to rotor speed for two different granular solids and a Newtonian Fluid under constant normal load conditions.

the force required to maintain movement against dynamic friction is almost independant of the speed of sliding. The mode of failure in granular shear is across a narrow, well defined slip plane rather than uniformly distributed across the annular gap between the shearing surfaces, and this too is more characteristic of solid state friction than of liquid viscosity.

The difference between cohesive and non-cohesive powders can also be illustrated using rheometer data, as shown in Figure 3.7. It can be seen that for zero normal load, non-cohesive materials exhibit zero shearing force, whilst predictably, cohesive materials exhibit a finite "yield stress" below which no permanent deformation occurs. In both cases, however, the relationship between normal load and torque is virtually constant. The case illustrated in Figure 3.7, where the cohesive material exhibits a smaller coefficient of internal friction than the non-cohesive powder is by no means rare. The implication is that the cohesive powder becomes the easier of the two to stir at high normal loads due to the inability of cohesive materials to pack as densely under normal loading (29).

Material behaviour such as is illustrated in figure 3.7 was first recorded by Coulomb (30), who developed the following simple equation to describe it:

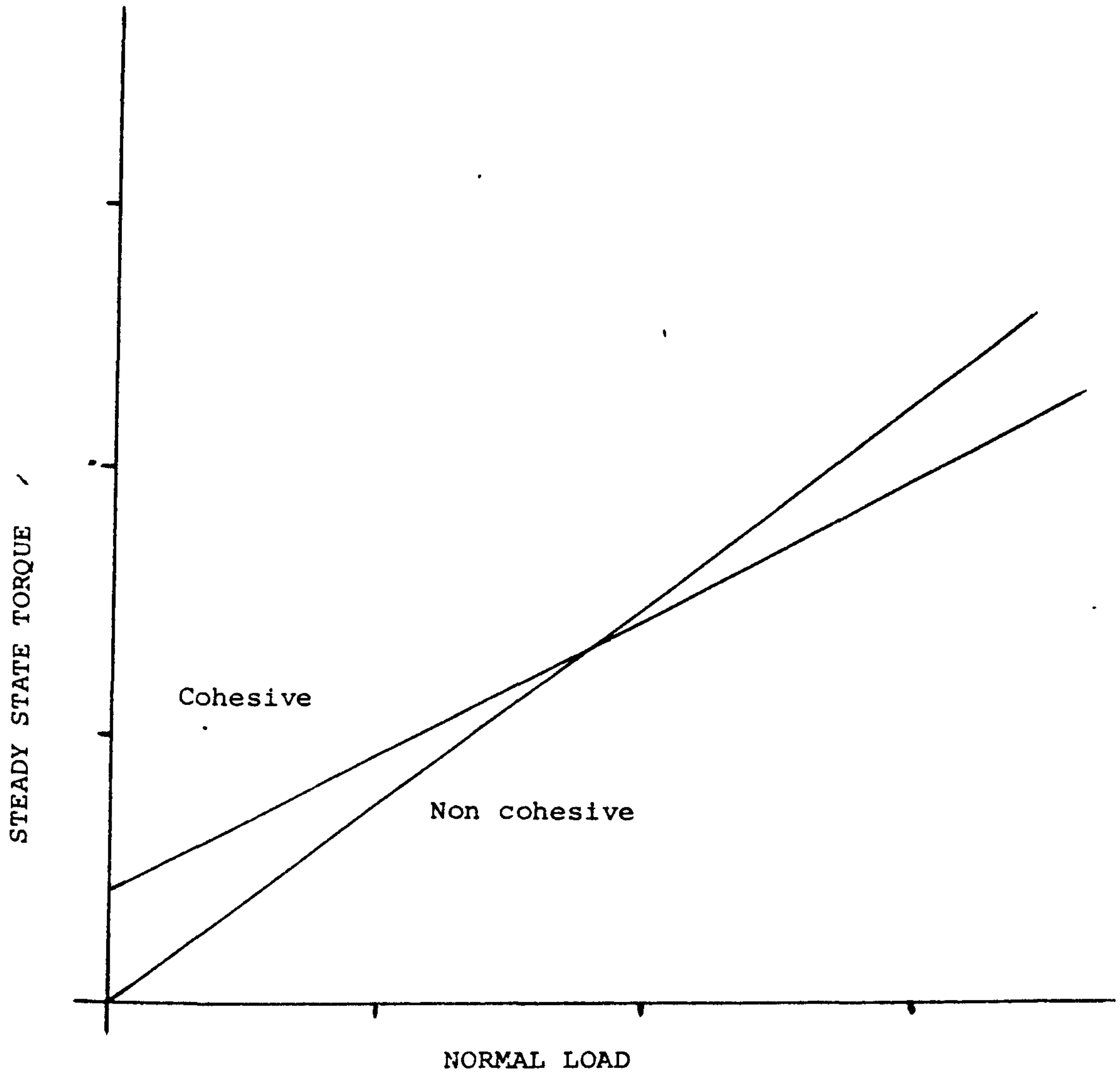


FIGURE 3.7. The behaviour of cohesive and non-cohesive powders under varying loads and at constant rotational speed.

$$\tau = C + \phi\sigma_n \qquad 3.2\text{-}5$$

where τ is the shear stress and σ_n the normal stress on the material. C is termed the coherence of the material, and is the value of the intercept on the torque/shear stress axis while as before, ϕ is the coefficient of internal friction and is the slope of the straight line. This equation is not completely general, however, and those materials which do not conform to it are more accurately described by the following equation due to Ashton et al (31):

$$\left(\frac{\tau}{C}\right)^n = \frac{\sigma_n}{\sigma_b} + 1 \qquad 3.2\text{-}6$$

where σ_b is the tensile strength of the powder and is the intercept of the line for the cohesive powder on the negative normal load axis. n generally varies between 1.0 and 2.0 in practise, with $n = 1$ representing conditions when the Coulomb equation is valid. For non-coherent materials which are perfectly free-flowing, the coherence C, is zero and the Coulomb equation therefore reduces to:

$$\tau = \phi\,\sigma_n \qquad 3.2\text{-}7$$

Although it is convenient to plot the results of rheometer tests on axes such as those illustrated, this is only so because the directions of the stresses involved have been designed by the nature of the instruments to favour this situation. In order to render the results more useful in terms of the interpretation of real systems, it is more normal to plot results in a different manner. By considering the equilibrium of an element within the stressed mass, it can be shown that on any plane inclined at an angle θ to the direction of the principal plane, there is a shear stress, τ, and a normal stress σ_n. With reference to figure 3.8. it can be seen that the magnitudes of these two stresses are:

$$\tau = \frac{\sigma_1 - \sigma_3}{2} \sin 2\theta \qquad 3.2\text{-}8$$

and $$\sigma_n = \sigma_3 + (\sigma_1 - \sigma_3) \cos^2 \theta \qquad 3.2\text{-}9$$

Mohr, (32) showed that providing a consistent nomenclature correcion was used, the locus of all stress conditions describing the situation at any point would fall on a circle, (now generally termed a Mohr's circle) Referring to figure 3.8b, all normal stresses, including principal stresses, are plotted along axis OX and all shear stresses are plotted along axis OY. The convention also assumes that the major principal plane is parallel to OX (i.e. major principal stress is parallel to OY).

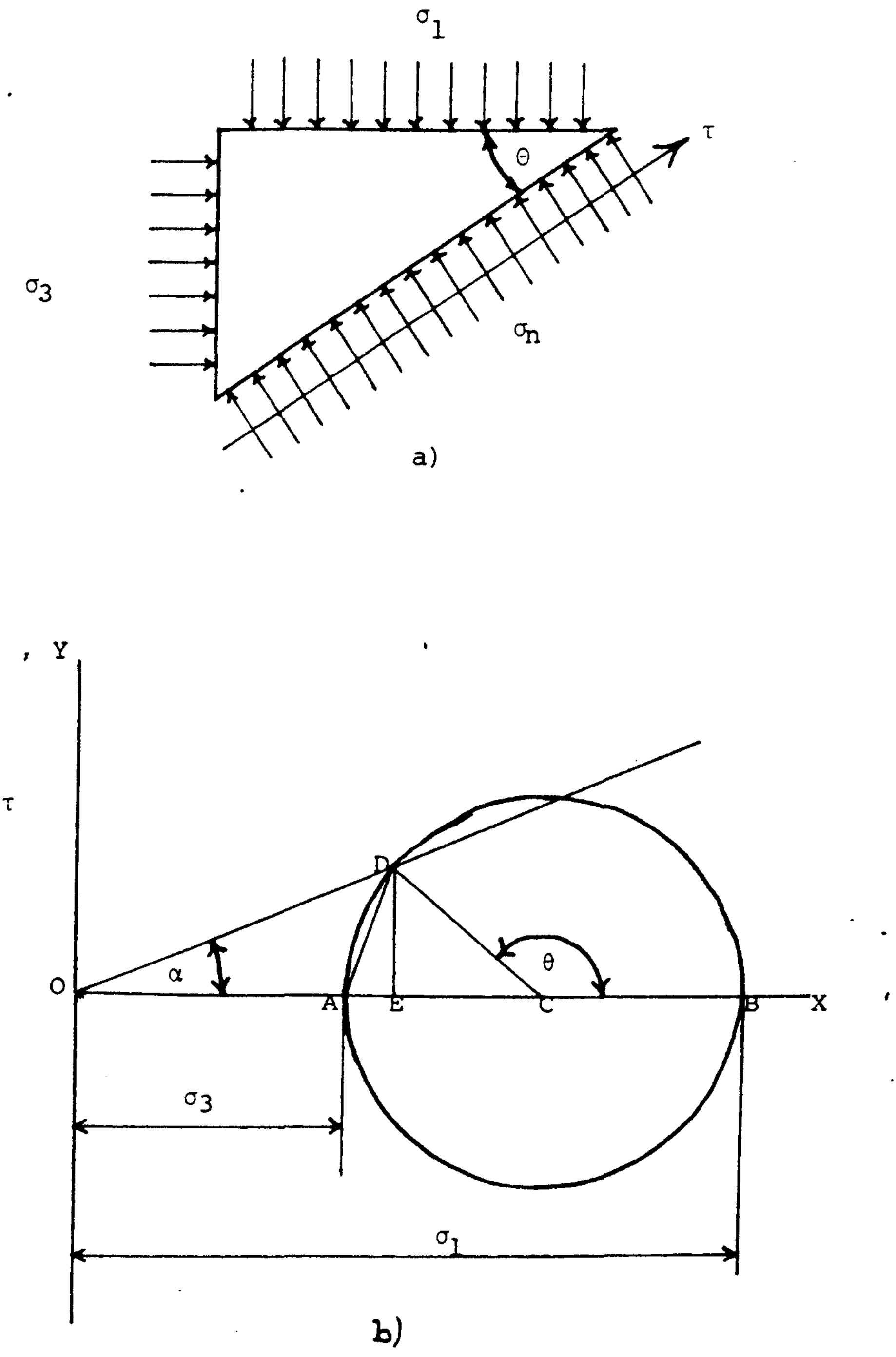


FIGURE 3.8. Graphical derivation of Mohr's Circle of stresses.

Points A and B are located such that OB and OA are the magnitudes of the major and minor principal stresss respectively. The circle of diameter AB passing through both points is then the locus of stress conditions on all planes passing through point A. The stresses on a plane passing through A and inclined at angle θ to the major principal plane are therefore given by the coordinates of the point D. The proof of the technique is simple:

Normal stress σ_n $= OE = OA + AE$

$= \sigma_3 + AD \cos\theta$

$= \sigma_3 + AB \cos^2\theta$

$= \sigma_3 + (\sigma_1 - \sigma_3) \cos^2\theta$

and Shear stress $= DE = DC \sin(180° - 2\theta)$

$= DC \sin 2\theta$

$$= \frac{\sigma_1 - \sigma_3}{2} \sin 2\theta$$

OE and DE represent the components of the complex stress acting on AD, and from the triangle of forces ODE it is clear that OD represents this complex stress on the diagram. Angle DOB therefore represents the angle of obliquity (α) of the resultant stress on plane AD. Clearly if the circle represents the locus of all possible stress states within a system, then failure of the system, i.e. the point at which the mass begins to shear, must occur when the line OD is a tangent to the circle.

(It is interesting to note that this is not at the value of maximum shear stress within the system since the normal force at maximum shear stress is high enough to prevent failure.) According to Coulomb's equation (3.2-5) this critical angle is therefore the angle of internal friction, and the slope of the line, tan ϕ, is the coefficient of internal friction. Although this angle is often taken as the angle of repose of a mount of a material (33), Jenike has refuted this and offers a different explanation of the angle of a mound (28).

If instead of being confined within experimental apparatus the granular material is required to flow in a practical situaiton, for example in a hopper of some sort or within a conveying system, there are two principle ways in which this can occur.

1) Plug flow. This describes flow when the moving part of the body of material moves inside a static channel with walls formed within the solid itself. The solid outside the channel remains at rest, consequently the walls of the containment vessel, and its shape, have no influence on either the shape of the channel or the velocity profile of the material within the channel. Examples of this kind of flow are found in poorly designed hoppers when only a central section of material moves, leaving a static "pipe" of material through which the remainder discharges.

2) Mass flow. This describes the situation when the boundary of the moving mass coincides with the walls of the container. All of the material within the mass is therefore in motion at the same time and this form of flow would be expected in smooth, steep sided hoppers under gravity discharge.

Clearly the forces acting upon and within a material have a large influence on the type of flow which will predominate. On a simplistic level, highly cohesive solids in vessels with low wall friction are more likely to move by mass flow than systems in which the wall friction exceeds the internal friction and cohesive forces within the material. Jenike (28) used these bases to develop sophisticated flow/no flow criteria for bins and hoppers, and more details of this subject will be given in later chapters where they have specific relevance to the work reported.

If, instead of gravity, some other external force causes the mass of particles to move, the simple systems described above no longer apply. One specific case in point, flow caused by the reduction of the volume of the container, describes the process of extrusion, and since this is particularly relevant to the work reported in this thesis the topic will be dealt with in more detail in a later section.

3.3 Tribology

Tribology has been defined as the science of friction, wear, and lubrication (34), and is therefore a topic of extreme relevance to the investigations recorded in this thesis.

3.3.1 Friction

According to Bowden and Tabar (35), the two main factors responsible for dry friction are:-

1) adhesion between surfaces at points of real contact, which must be broken or sheared if sliding is to occur
2) the ploughing or grooving of one surface by the asperities of the other

In static friction, where there is no relative motion between the surfaces, clearly only the first of these factors has any bearing, and even when the second factor is involved, in rolling or sliding friction, its effect is generally much smaller than that of the first. This explanation fits in well with important practical observations, first recorded around 1500 AD by Leonardo da Vinci, namely:

1) The frictional force, F_t, i.e. the force to cause movement of the two surfaces relative to each other and in the plane of their interface, is independant of the nominal contact area.

2) The frictional force is proportional to the normal force, F_n between the two surfaces. The proportionality between the forces for any given pair of surfaces is termed the coefficient of friction, f, i.e.

$$f = \frac{F_t}{F_n} \qquad 3.3\text{-}1$$

The real area of contact was shown by Bowden and Tabar (35), to be of the order of 10^{-4} times the nominal contact area. This implies that even if the normal load is relatively small, the pressure at the points of contact is sufficiently high to reach the yield stress of at least one of the materials in contact, even if both are metallic.

If this is the case then the real area of contact can be calculated very simply from the formula,

$$A_r = \frac{F_n}{\sigma_y} \qquad 3.3\text{-}2$$

where A_r is the real area of contact, and σ_y is the yield strength of the weaker material. Similarly, if the first observation above holds true, then to cause sliding, a force equal to the shear strength of the weaker material, τ_y, multiplied by the area of true contact, must be applied to the system, i.e.

$$F_t = \tau_y A_r \qquad 3.3\text{-}3$$

and by substitution for A_r:

$$F_t = \frac{\tau_y}{\sigma_y} F_n \qquad 3.3\text{-}4$$

The final equation suggests that the static coefficient of friction is therefore a function of the properties of the weaker or softer of the two materials since

$$f = \frac{F_t}{F_n} = \frac{\tau_y}{\sigma_y} \qquad 3.3\text{-}5$$

Equation 3.3-5 is also known as Amonton's Law.

Although this relationship holds true for many metallic systems under certain circumstances, it is only a very approximate generalisation and cannot be applied universally for several reasons.

If two highly polished surfaces of the same material are brought into contact, then interatomic forces of attraction must also influence the characteristics of the system. In the ultimate case, if perfectly smooth, impurity free surfaces of the same material are brought into contact then complete welding will occur since by definition, the atoms at the interface are unable to distinguish which surface they belong to.

Relative motion involving shearing must influence both local temperature and pressure and these in turn will affect the material properties at that point.

Yielding and ploughing will introduce fresh surfaces and alter the topography of the interface.

These arguments have been used to explain the observed differences between static and kinematic friction, and Neilsen (36), used them to explain the so-called "slip-stick" motion which generally occurs during dry sliding. He postulates that during the "stick" stage the real area of contact increases due to the increasing tangential loading. "Slip" occurs when the shearing force becomes equal to the critical yield value and greater than the force required for ploughing. The real area of contact, and hence the friction force then decreases rapidly during the period of slip. Any halting of the motion will allow new adhesive bonds to form and restore the frictional force to its original value.

Other materials, for example polymers, exhibit much more complex frictional behaviour in contact with metals. Considering the differences between the two types of material in terms of hardness, elasticity, and the temperature dependance of the material properties this is to be expected.

The relationship between normal load and frictional force in a typical system is described by the relationship:

$$F_t = CF_n^{\alpha} \qquad 3.3\text{-}6$$

and a load dependant coefficient of friction can therefore be deduced:

$$f = C\ F_n^{\alpha-1} \qquad 3.3\text{-}7$$

where C is a system constant and α is a variable found to be between 0.66 and 1(37). It is suggested that $\alpha = 0.66$ represents the case when all deformation at the yield points is purely elastic, and $\alpha = 1$ the situation in equation 3.3-4, when all deformation is assumed to be plastic. Values between the two extremes would therefore represent systems with visco-elastic properties, and the predictable effects of external influences such as temperature, time of loading, speed of sliding and surface roughness on such a system have been observed experimentally as is detailed below.

With the exception of the case when α =1, the predicted decrease in the coefficient of friction with increasing normal load has been observed experimentally (38,39,40).

The effect of increasing temperature on the coefficient will be twofold, simultaneously causing a decrease in the shear strength of the material and a concomitant increase in surface area. These effects will tend to counteract each other, but since they may change at different rates, a minimum in the plot of f versus temperature might be predicted. Experimental observations have confirmed the presence of such minima in a variety of systems (38,39,40,41,42). At temperatures at or near the glass transition or melting temperature there may be a dramatic increase in coefficient of friction (42), which is indicative of the formation of a thin fluid layer by incipient melting, which deforms by viscous drag flow rather than dry friction. It is worth noting that even before melting occurs, equation 3.3-7 bears the same form as equation 3.1-7, the power law equation describing the non-Newtonian behaviour of dilatant and pseudo-plastic materials.

Since in general the yield strength of a material increases with increasing strain rate, the observed effect of an increase in sliding speed causing an increase in coefficient of friction (38,41), would be predicted from equation 3.3-5. The fact that this effect is less dramatic than might be expected may be due to the opposite effect any friction induced local temperature rises would have.

Because of the much lower yield strength of polymers, it is to be expected that the "ploughing" contribution to kinematic friction would be much less than with metallic systems. Again this has been observed to be the case (41), although the expected trend of increasing friction with increasing roughness has been confirmed. There appears to be an additional temperature dependence of this property, with the largest effects occurring at about 65°C (41) but no satisfactory explanation has been given for this behaviour.

A limited amount of research has also been carried out on organic/metal interfaces, for example with wood (43,44) with results which appear to confirm the general theories of Bowden and Tabor (35), and for more in-depth information on the subject matter of this section the reader is directed to the references and to the many technical journals which have proliferated in the area.

3.3.2 Wear

Defined as "the removal of material from solid surfaces as a result of mechanical action" (45), wear, together with breakage and absolescence, is one of the main reasons for the cessation of usefulness in inanimate objects.

There are four principal types of wear process which can occur individually or concurrently:

1) Adhesive wear. This occurs due to the high forces present at real contact points between two materials, as outlined in the previous section. Fragments of one surface are pulled off during sliding and may adhere to the other surface or may appear in the form of loose wear debris at the interface. Clearly the amount of wear will be influenced by the coefficient of kinematic friction and the real contact area, and Rabinowicz and Shooter (46), obtained data with which they quantified wear rates. Thus if two solids have a ratio of hardness R, then their adhesive wear rates will vary in the ratio $1/R^2$. Although this is intuitively correct, in other observations they have shown that even low adhesion soft polymers can remove measureable quantities of material from relatively much harder surfaces such as mild steel.

2) Abrasive wear. This is the type of wear associated with the second of the friction modes of Bowden and Tabor and occurs when a softer surface is gauged and ploughed by the passage of a harder surface across it. A similar effect occurs when loose hard particles become trapped between two softer surfaces. Abrasive wear almost always results in the formation of loose wear debris. Although abrasive wear can be eliminated by having smooth surfaces free of hard particles, the adhesive wear which may then result can produce wear debris which may then trigger abrasive wear action.

3) Corrosive wear. This type of wear results when the sliding occurs in a corrosive environment. The corrodant need not be anything more than air, which can cause oxidation of newly formed surfaces, changing their characteristics. Whatever the corrosive its action is to eat away at the newly formed surfaces etching deeper into the material surface as it does so. Although in general polymers are non-corrosive and may reduce corrosive wear by coating the other surface thus preventing the ingress of the corrodant, certain polymers, notably PVC, can liberate corrosives under certain temperature conditions and thus exascerbate the problem instead.

4) Surface Fatigue Wear. This type of wear occurs after repeated loading and unloading of a surface, as occurs for example in ball or roller bearings or in the presence of a fluctuating normal load. Although in a perfectly elastic material with no surface defects, no such wear should ever happen, small defects will occur, perhaps as a result of one of the other forms of wear, which can then initiate failure of the surface in this mode. Many polymers will crack and fail under this type of loading.

It is clear from the above that any mechanism which reduces or prevents the physical contact of the two surfaces will affect the friction and wear properties of the combination, and this is the role of the third facet of tribology, lubrication.

3.3.3 Lubrication

At its simplest, lubrication can be divided into three principal categories; fluid or hydrodynamic lubrication, boundary lubrication, and solid film lubrication.

The theoretical foundations for hydrodynamic lubrication were first elucidated by Reynolds (47), in 1886 and have been the subject of considerable research ever since (48). The basic principle is that the two surfaces are completely separated by a continuous, relatively thick (0.01mm to 0.5mm) layer of fluid. The pressure developed in the fluid due to the relative motion of the surfaces may be of the order of several Mega Pascals, and it is this pressure which keeps the surfaces apart and eliminates dry friction. It is not surprising in view of the pressures generated that any eccentricity in the rotation of a journal revolving in a stationary bearing will cause the axis of the journal itself to move. Lubrication of this type is typically found in journal bearings, but is also important in polymer extrusion using an Archimedean screw, when the molten polymer itself acts as the lubricant.

Boundary lubrication, as its name suggests, is characterised by the presence of a very thin film of lubricant, often only an atomic monolayer, which covers the entire real area of contact. Surface to surface contact is thus not eliminated completely, but is reduced to a value which is characteristic of the system. The boundary layer is often chemically bonded to the surfaces and may be formed in situ by reaction of the lubricant with the material surface, particularly as clean bare surfaces form by the action of ploughing and gauging. The boundary layer may also be attracted to the surfaces by physical forces and boundary layer lubricants are frequently formulated to contain polar molecules or dipoles and have flexible chains. Fatty acids and alcohols are examples of such lubricants, and lubricant systems based on hydrodynamic lubrication frequently contain boundary lubricants to afford protection for the surfaces in the event of pressurisation loss or for the period of start-up before full hydrodynamic flow develops.

Solid film lubrication is closely related to boundary layer lubrication in that the film may only cover the real area of contact of two surfaces. The film in this case is generally much thicker, however and frequently covers the entire material surface thus eliminating dry contact completely. Again chemical or physical bonds may be formed between the lubricant and the surfaces, but clearly the film can only remain solid at temperature below its melting point. A variation of dry film lubrication is the use of metallic lubricants in the extrusion industry, where the metal to be extruded is encased within an envelope of a second metal whose coefficient of friction with the walls of the extrusion container is much lower than that of the first metal. Metal stearates are a major family of solid film lubricants in wide use throughout industry.

Lubrication, for the elimination and reduction of wear, and to lower power requirements by decreasing frictional losses, is an area of considerable importance to every branch of industry. As such, there has been a great deal of research carried out since Reynold's original work, and it is impossible to cover the subject in detail in this thesis. The reader is therefore directed to the texts identified in the references (35,39) for a more complete coverage of the subject.

3.4 Extrusion

The process of extrusion involves the propulsion of a body of material through a die of a desired shape to form a product of fixed cross-section in a continuous, or semi-continuous length.

The means by which the material is propelled through the die depends chiefly on the state of the material immediately prior to the die orifice. In general the material will either be a melt, as in the case of polymer extrusion, or a solid as with metal extrusion, however it will be clear from the earlier sections of this chapter that the distinction between the two is rarely clearly defined.

The two major mechanisms employed are the Archimedean screw and the reciprocating ram, the former being limited to melt extrusion whilst the latter is widely employed for both starting states. There are numerous other less widely used mechanisms, for example gear pumps (49) and normal stress pumps, (50) but none are suitable for the study undertaken and the reader is therefore directed to the references for further information.

The advantages and disadvantages of the screw and ram types of extruder are dealt with fully in Chapter 6, with particular reference to their suitability of the work of this project. These will both be expanded upon further in this chapter.

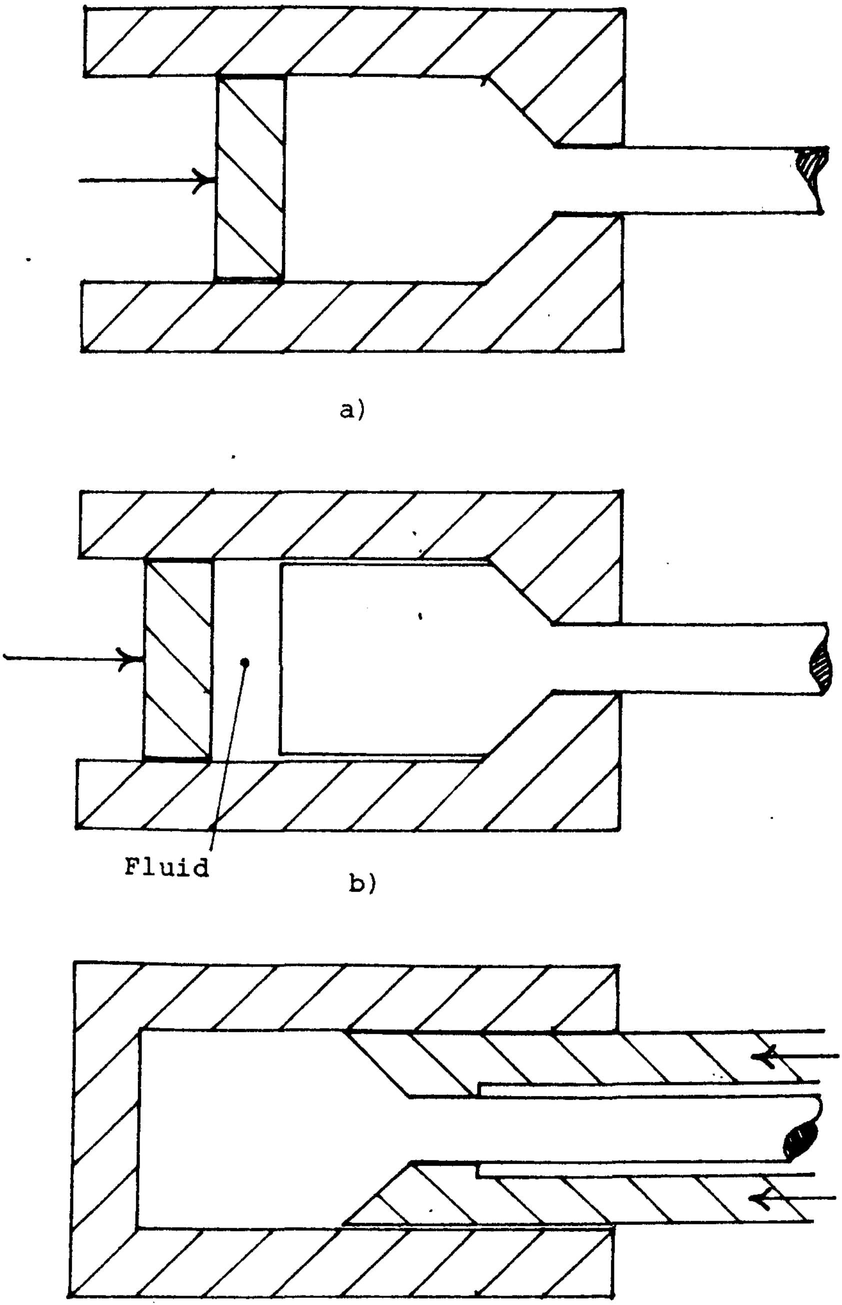
The theories behind the operation of the various forms of screw extruder are complex and although interesting in themselves do not contribute to the understanding of the processes occurring in the present investigation. No further details will therefore be given in this chapter and the reader is directed to suitable references for in-depth discussions of the topic (51, 52, 53, 54).

Ram extrusion, on the other hand, matches closely the process under investigation and will be given a fuller treatment in the following sections.

3.4.1 Ram Extrusion

Although principally used for the processing of metallic materials where the disadvantages of discontinuous operation are outweighed by the high pressure capability and ruggedness of the machinery, ram extrusion has been used to a limited extent in polymer processing, particularly of thermo-setting materials and in wipe covering operations (55). The underlying mechanical principles are the same irrespective of the feedstock, providing it is incompressible, (which in the case of this investigation it clearly is not, and this point will be dealt with in detail in a later chapter), and can be dealt with in a generalised manner.

The most basic form of extrusion, direct extrusion, is illustrated in Figure 3.9a. The material to be extruded, in the form of a billet is enclosed within a container with an orifice of the size and cross-section of the product required, and the material is pressurised and forced through the orifice by movement of the ram. It is clear that as extrusion proceeds the material within the container will move relative to the walls of the container, and the resulting frictional force increases the pressure required on the ram considerably. By eliminating or reducing these frictional forces, the process can be made more energy efficient, and the two most common



a)

b)

c)

FIGURE 3.9. The three most common forms of extrusion, a) direct extrusion, b) hydrostatic extrusion, c) indirect extrusion.

ways of achieving this are illustrated in Figures 3.9b and 3.9c.

Figure 3.9b represents the process of hydrostatic or isotatic extrusion, where the two surfaces are separated by a fluid through which the pressure is transmitted to the bilet. As well as eliminating the wall friction component, this technique has the advantage when processing brittle materials that the isostatic pressure reduces significantly the tendency of the material to crack.

Figure 3.9c illustrates the process of indirect extrusion. In this case the relative motion between the billet and the container is eliminated completely and the only frictional forces acting are those between the die and the deforming billet.

Of course in addition to the alterations to the basic process outlined above, it is possible to use lubricants of various forms to reduce the coefficient of friction between sliding surfaces. Molybdenum disulphide, powdered glass, and oils containing graphite, talc, mica and complex phosphates are examples of typical lubricants. The process of 'canning' also falls into this category, where metals which have a severe tendency to adhere to the equipment walls are enclosed inside a container of a less difficult metal which is then extruded with the principal material. Such a process has added advantage when the principal metal is toxic or reactive and has also been employed as a means of extruding powders.

There is one further source of energy consumption which does not yield useful work in the extrusion process, particularly in the case of the extrusion of metals, and that is the so called "redundant work". This is work performed on the material which produces non-uniform deformation, and is frequently related to friction effects since a common manifestation is the increase in shear strain from the centre out to the skin of an extruded product. This can result in residual tensile stresses being left in the skin of an extruded product, and is also responsible for a common fault in directly extruded sections known as extrusion defect. This occurs because the centre of the billet travels towards the die at a greater speed than the billet surface which is retarded through contact with the walls. As the volume of material in the container decreases below a critical value, so material adjacent to the walls flows across the face of the piston and is then accelerated into the centre of the extrudate. Since this material is frequently oxidised and contains contaminants, these become entrained in the centre of the extrudate causing inhomogeneity and weakness. Solutions to this problem range from discarding the last part of the billet without extruding it to inert atmosphere processing which prevents the initial formation of the oxides.

A quantity of major interest in the extrusion process is clearly the pressure required on the ram to cause the material to flow.

In the ideal case of extrusion, when there is zero friction and all work is used to cause uniform deformation, then this can be calculated on the basis of simple strain. If A_o is the original billet cross sectional area and A_p is the final extrudate cross sectional area, then the ratio A_o/A_p is called the extrusion ratio, and the strain is given by:

$$\epsilon = \ln (A_o/A_p) = \ln (L_f/L_o) \qquad 3.4\text{-}1$$

where L_o and L_f are the original and final lengths of the material. The work done per unit volume, u is therefore

$$u = \sigma_o \epsilon = \sigma_o \ln (A_o/A_f) \qquad 3.4\text{-}2$$

Now the volume of the billet is A_oL_o, thus the total work done W, is:-

$$W = A_oL_o \sigma_o \ln (A_o/A_f) \qquad 3.4\text{-}3$$

and since it is also given by:

$$W = p\ A_oL_o \qquad 3.4\text{-}5$$

where p is the pressure on the ram, then the extrusion pressure under ideal conditions is:-

$$p = \sigma_o \ln(A_o/A_f) \qquad 3.4\text{-}6$$

If friction is still neglected, but an adequate allowance is made for redundant work, then values up to 50% greater than the ideal value can be expected. Redundant work might be expected to be related to the die angle of the extruder since this will to some extent govern the shear strains in the outer layers of material. Avitzur (56), postulated an upper bound theory with spherical velocity discontinuities (for the derivation and theoretical assumptions made, the reader is directed to the reference. Only the result is of direct relevance to the current work, and the underlying explanations and assumptions are complex and add little of value to this section of the thesis). This took account of the effects of redundant work and die angles as follows:-

$$\frac{p}{\sigma_o} = 2F(\beta)\,\ln\frac{D_o}{D_1} + \frac{2}{\sqrt{3}}\left[G(\beta) + f\cot\beta\,\ln\frac{D_o}{D_1}\right] \qquad 3.4\text{-}7$$

where β is the die half angle and:

$$F(\beta) = \frac{1}{\sin^2\beta}\left[1 - \cos\beta\sqrt{1 - \frac{11}{12}\sin^2\beta} + \frac{1}{\sqrt{11.12}}\ln\left(\frac{1 + \sqrt{11/12}}{\sqrt{11/12}\cos\beta + \sqrt{1 - 11/12\sin^2\beta}}\right)\right]$$

and $$G(\beta) = \frac{\beta}{\sin^2\beta} - \cot\beta$$

Bay (57), took this derivation one stage further and incorporated an integral term for the axial friction component, which clearly changes as the length of the billet decreases. His analysis resulted in the following pressure equation:

$$\frac{p}{\sigma_o} = 2F(\beta)\,\ln\frac{D_o}{D_1} + \left[\frac{2}{\sqrt{3}}\,G(\beta) + f\cot\beta\,\ln\frac{D_o}{D_1} + \int_{Z_o}^{Z}\frac{2\tau_n}{k}\cdot\frac{dz}{D_o}\right]$$

3.4-8

where Z and Z_o refer to distances as shown in Figure 3.10, τ_n is the friction stress along the cylinder wall and k is the yield stress of the material in pure shear. σ_o in both cases is the equivalent yield

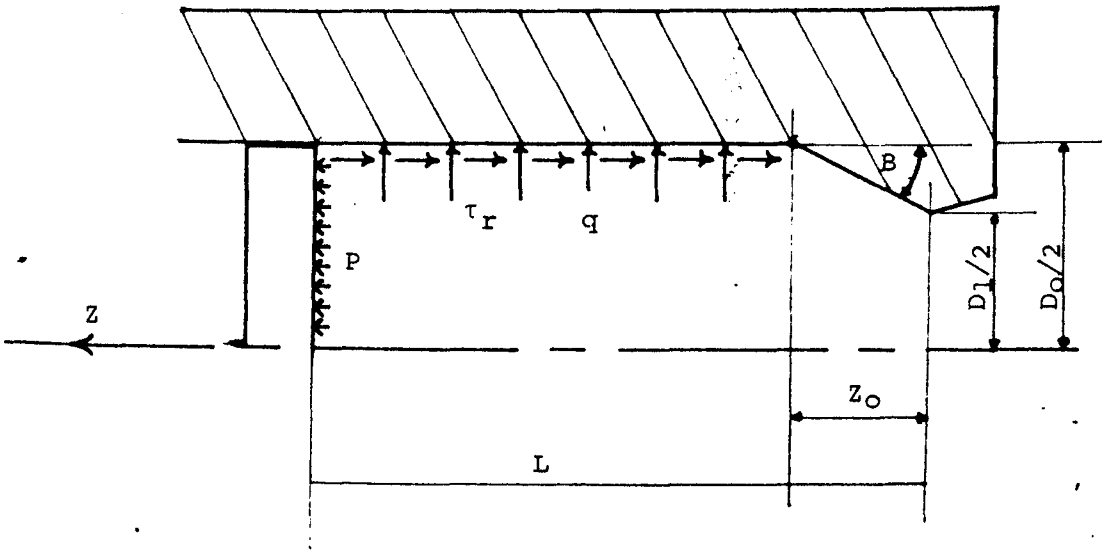


FIGURE 3.10. Notation of forces during direct extrusion.

stress of the material being extruded, and the quantity τ_n/k corresponds to the "friction factor" of Wanheim et al (58) in the case where the real and apparent areas of contact, (section 3.3.1, reference 35) are considered to be equal due to the high normal pressures involved.

There are several assumptions made in the theoretical derivation of this expression, however the major one which causes difficulties in using the expression to describe the system under investigation in this thesis is that the material is totally incompressible. This clearly is not the case with the woodchip starting material, and allowances are made for this in the later chapters concerned with the discussion and interpretation of the results.

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CHAPTER FOUR. CHOICE OF RAW MATERIALS

The work documented in this thesis has two complementary aims:

1) To devise an extrusion procedure, for the continuous manufacture of wood particle composites, which should have potential industrial significance, and which in some way, financial, aesthetic, environmental, or physical, is an improvement over currently available alternatives.

2) To gain an understanding of the mechanical and rheological processes which occur during the extrusion of very high concentration mixtures of spindle shaped particles.

It is clear then that the design of the experimental programme must take cognisance of both the mechanical and financial implications of the raw materials variables as well as those of the process route itself. This chapter therefore deals with the decisions taken regarding the source and type of wood and binder which would form the basic experimental material for the investigation.

In the following sections it should be remembered that the financial implications of the choice of raw materials and in particular timber type will depend almost entirely on the country or area in which the operation is to be carried out. Factors such as relative abundance, location (and therefore transport costs), and the local economy will all need to be taken into consideration. It is beyond the scope of this thesis to include financial breakdowns of all the possible alternatives in each area, and the decisions made on the basis of the facts and figures documented below therefore refer only to the United Kingdom, although the factors considered and the final model are capable of being tailored to suit any individual situation.

Since the pre-extrusion material is an intimate mixture of two components, the timber or cellulosic phase, and the adhesive or binder phase, the following discussion will be divided into two broad areas, one covering each phase.

4.0 The Cellulosic Phase

The title of the subsection deliberately avoids the use of the words timber or wood, since at the very outset of the project the nature of the major phase was only defined as cellulosic. The following paragraphs detail the argument put forward before finally arriving at wood as the basic starting material.

4.0.1 Available cellulosic starting materials.

The analogy of the intended product with particleboard has already been drawn several times during the course of this thesis, and since the physical nature of the two products is very similar, much of the raw material data for particleboard is relevant to the current research.

The two main alternatives to wood as the major component of particleboard are flax shives and bagasse. Other materials such as jute, hemp, maize stalks, cotton stalks, palm fibre and quinine stems are used for particleboard manufacture in various parts of the world, but the output is so low [less than 6% of the total of non-wood particleboard in 1973 (1)] that these were disregarded as possible raw materials for this study.

Despite the fact that non-wood fibrous particleboard amounted to only between 2% and 3% of the total output of particleboard in 1975 (1), the tonnage outputs are significant (bagasse 100,000 tonnes, flax shives 600,000 tonnes) and warrant a more detailed examination of the properties of the two alternatives.

4.0.1.1 Bagasse

Bagasse is the fibrous ligno-cellulosic residue which remains after the extraction of the sugar from sugar cane. Most of the annual world output of 65 million tonnes is still used by the sugar manufacturers as low grade fuel for heating, however as table 4.1, (2), shows, the chemical composition of bagasse is similar to that of wood and it is therefore a possible substitute for wood in particleboard.

The yield of bagasse from harvested sugar cane is about 45% of the dry weight of the cane, and of this about 75% of the residue is usable. The economic advantages of using the bagasse to manufacture a saleable product are therefore obvious, providing that the cost of the conversion is not too high.

The residue from the sugar extraction has a moisture content of about 100%, comparable to that of green timber from forest harvesting, and also contains between 2% and 4% residual sugars. This combination, together with the fact that sugar cane, and therefore bagasse, is a seasonal commodity, present the biggest disadvantages to the further utilisation of bagasse as a raw material. The seasonal nature of the crop means that for a particleboard mill to run the full year round, bagasse raw material must be stored in some way for use during the inter harvest period.

COMPONENT	BAGASSE	BEECH	PINE
	%	%	%
CELLULOSE	46	45	42
LIGNIN	23	23	29
PENTOSANS & HEXOSANS	26	22	22
OTHERS	5	10	7

TABLE 4.1 THE APPROXIMATE CHEMICAL COMPOSITIONS OF BAGASSE, BEECH AND PINE.

The residual sugar and high moisture content of the raw bagasse render it prone to fungal, insect, and animal attack however, and successful storage can generally only be accomplished with the bagasse either totally wet or at a moisture content below the fibre saturation level. Neither of these states is cheap to attain or maintain. The modern solution to the problem is to depith the raw bagasse using hammer mills as it leaves the extraction plant, returning the pith to be burnt as fuel, and baling the bagasse fibres ready for storage. This has a further benefit to the producer since the baled bagasse begins to ferment rapidly on storage, turning the unwanted sugars to alcohol which, together with the moisture still present, evaporates due to the increase in temperature. A product which is stable at 25%-30% moisture content results after 4-6 weeks, and at this level the bagasse is resistant to both bacterial and fungal attack and can be stored economically.

The production of bagasse based particleboard follows the same route as its timber based equivalent, outlined in Chapter 2, the baled product being reduced from the storage moisture level of 30% to a working level of 3% to 5% before refining and grading begins. Slightly longer press times are also required than are common with timber based particleboard due to the much more homogenous and densely packed nature of the bagasse fibres which inhibit the dissipation of vapour curing products from the board.

The strength and other mechanical properties of bagasse based particleboard compare favourably with those of timber based equivalents, as reference to Table 4.2 shows, and in sugar cane growing areas where timber resources are poor or scarce, bagasse is a very economical alternative as the raw material for particleboard production.

4.0.1.2 Flax shives

Unlike bagasse which is a product of tropical countries, flax is a native of temperate zones, and is grown extensively in the Soviet Union, Poland, France, Belgium, and until recently, Holland. As an annual fibre plant it is second only to cotton in terms of economic importance. When the two principal products, flax fibres from the stems and linseed oil from the seeds, have been extracted from the plant, the residue is composed of the woody stem fragments known as flax shives and various organic contaminants such as weeds, rogue seeds and dust.

After separation of the shives from the extraneous matter (usually accomplished by dry processes such as combing, screening and air separation), the by-product consists of elongated granular particles of relatively narrow particle size distribution.

PROPERTY	RAW MATERIAL BAGASSE	FLAX SHIVES	MIXED SPRUCE/ PINE CHIPS	MIXED SP/ PINE 3 LAYER BOARD	BIRCH/OAK 3 LAYER BOARD
DENSITY ($kg\ m^{-3}$)	610	599	652	651	653
BOARD THICKNESS (mm)	19	19	18.1	20	20
RESIN CONTENT (SURFACE)	8%	8%	10%	12%	12%
RESIN CONTENT (CORE)	-	-	-	8%	9%
BENDING STRENGTH ($N\ mm^{-2}$)	21.5	18.7	18.9	16.6	20.4
TENSILE PERPENDICULAR STRENGTH ($N\ mm^{-2}$)	0.6	0.4	0.4	0.5	0.5
SWELLING AFTER 120 mins	6.1%	6.4%	3.7%	9.8%	6.8%
SCREW HOLDING (kg)	61	90	62.6	98	58.5

TABLE 4.2

A COMPARISON OF THE PROPERTIES OF SIMILAR PARTICLEBOARDS MANUFACTURED FROM DIFFERENT RAW MATERIALS.

This granular nature of the raw material means that there is no need for traditional size reduction machinery and the material requires only minimal size classification before it can be used for particleboard production. Another advantage of the as-delivered material is that the fibre-removal process leaves the shives with a moisture content of between 11 and 14% which significantly reduces the size of particle drying and conditioning equipment and the concomitant energy requirements.

As might be expected for a raw material which is indigenous to the temperate zones, these being generally accepted to be the more industrially advanced areas of the world, a considerable amount of work has been carried out on the conversion of flax shives into particleboard. Originally a process developed in England, the Linex Verkor process (3), perfected in Belgium, is now the basis of most flax-shive based particleboard production. This process differs from a conventional platen process in that formers or "trays" are used in the production of the pre-pressing particle mats. These are an economic measure since the nature of the raw material is such that a low angle edge occurs on a free formed pile. This would result in significant material wastage in the form of post pressing trimmings if trays were not used.

As can be seen from Table 4.2, particleboard manufactured from flax shives compares favourably with timber based boards and particularly in the Soviet Union and in Belgium, flax shive based board is produced in greater quantities than its timber based equivalent. In 1973, Belgium produced 930,000m^3 of flax based particleboard, around 60% of the European total (1).

As an alternative to timber for particleboard manufacture therefore, in those areas where the raw material is an industrial by product and is relatively cheap, flax shives offer no particular disadvantages other than the marginally more complex processing plant. On the contrary the advantageous energy savings in the use of flax over timber raw material (1), may be a very significant factor in areas where fuel and energy costs are high.

4.0.2 Additional constraints on the cellulosic raw material

In order to reduce the three alternatives to one, an arbitrary set of constraints was laid down for the raw material. These were:-

1) The material must be readily available. If development to a commercial scale were to be undertaken then an estimated volume of at least 6.5 m^3 of material per hour would be required for economic operation of a medium sized plant.

2) The raw material must be cheap. The envisaged product would compete commercially with sawn, dried and machined timber, and a low initial material cost would give more flexibility in terms of processing whilst still maintaining a competitive price structure.

3) Because some of the envisaged product applications are for structural components, the product must have good mechanical properties, e.g. bending and tensile strength. By analogy with other composite materials, and in particular with particleboard, this infers that the ideal individual particles will have a high aspect ratio (length to width and thickness).

4) For economic mechanical processing the raw material should be as clean as possible, i.e. it should not contain any metallic or silicaceous contamination.

Since the project at this stage was highly speculative, and the location of any final production plant was not decided, the financial aspects could not readily be assessed when defining the raw material. The availability of the material and its potential for producing a high quality board were therefore considered to be the paramount factors to be taken into account. These, together with the need for a consistent and reproducible material to be compatible with the research aspects of the programme, gave rise to the decision to base the work on a timber raw material. It was also felt that if promising results were achieved in this pilot study, the conversion to full scale production could also include a change of raw material should local conditions dictate this.

4.0.3 Timber based alternative materials

It was mentioned in the introduction to this thesis that the major source of wood raw material for the particleboard industry has shifted away from lumber industry waste toward specifically harvested roundwood logs. The most recent comprehensive figures published by the United Nations (1), show that in 1972, 55% of the material used for world particleboard production came from chips and residues with the remainder coming from pulpwood. The same source also emphasises the point made earlier regarding regional variations, stating that in Japan, the particleboard and fibreboard industries derive 80% of their raw material from chips and residues, in the USA the figure is still 90% for the particleboard industry only, while in Europe this figure is as low as 30%.

In the same United Nations survey a significant increase in the use of hardwoods in what began as a traditionally softwood based industry was also noted. In Japan the use of hardwood, both indigenous and imported, grew from 15% in 1956 to 58% in 1970. More significantly, in the traditional coniferous forest areas of Finland, Norway and Sweden the use of indigenous hardwoods leapt from less than 3% of the total pulpwood harvested in 1950 to more than 16% in 1972.

From the above brief introduction to the subject it must now be very clear that the area in which any production facility based on this preliminary work is sited will be the most influential factor in the ultimate choice of cellulosic raw material. For this reason it was decided that projected usage was not a valid consideration in the choice of raw material for this research project.

The model of the case of a plant situated in the United Kingdom was therefore used for the economic base of the decision, and the factors mentioned previously of consistency and reproducibility of raw material supply were considered within that framework.

The following sections deal with the selection of a suitable raw material on the basis outlined above.

4.0.3.1 Standing timber and roundwood in the UK

Standing timber is defined as timber which is actually still growing at the time of pricing, and to which an overall cost for harvesting must be added in order to obtain a realistic price. Because the harvesting is done on an area basis, the size of the timber purchased is not graded and will vary within limits. Roundwood or sawlogs on the other hand are sized and graded and are bought as such, with the price reflecting the extra work involved. The cost of roundwood therefore tends to be higher than that of standing timber.

In the UK most of the timber sold is produced on Forestry Commission Plantations or Concessions and consists almost entirely of coniferous species. Table 4.3 (4), shows the prices of standing timber for a range of various sizes and locations for the two years 1980 and 1981.

		ENGLAND		SCOTLAND		WALES	
	AVERAGE VOLUME PER TREE (m^3)	VOLUME PRODUCED (m^3)	COST (£ m^{-3})	VOLUME PRODUCED (m^3)	COST (£ m^{-3})	VOLUME PRODUCED (m^3)	COST (£ m^{-3})
1980	Up to 0.074	40598	4.37	26201	1.81	27553	3.03
1981		39947	2.79	24435	1.11	13752	1.57
1980	0.074 to 0.224	10763	9.74	6801	9.83	28070	9.82
1981		10023	5.66	34675	5.00	12431	4.31
1980	Over 0.224	25209	23.21	80032	12.83	35456	20.27
1981		34256	12.79	47016	7.76	22784	11.27

TABLE 4.3

COSTS AND VOLUME OUTPUTS OF CONIFEROUS STANDING TIMBER FOR ENGLAND, SCOTLAND AND WALES IN 1980 AND 1981.

This data not only reinforces the point already made several times about the economic effect of location but also illustrates the wide variation in price from year to year, with almost 40% decrease from 1980 to 1981 in all regions. From the same source (4), an average price of £4.61 m^{-3} for standing timber over the period 10/1980-9/1981 was calculated. In January 1982 the price for roundwood was £9-16.8 m^{-3} depending on the size and grade of the timber. To both of these prices an average of £3.50 m^{-3} must be added to cover the cost of transport to the production facility.

4.0.3.2 Wood residues from manufacturing processes in the UK

There are basically three types of wood residues produced in the UK; sawdust, hog-milled chips, and routing flakes.

1) Sawdust

Sawdust is inevitably produced during any woodworking operation and tends to be extremely varied in terms of particle size, moisture content, and species mix, even when obtained from a single supplier. Enquiries to a local supplier (5), indicated that some 4000 tonnes per annum were produced, and this was sold mainly to particleboard manufacturers at £10-12.50 per tonne ex works.

2) Hog-milled chips

So-called because they are produced by a machine called a hog-mill, these chips can vary in size from about 2mm square to flakes of 20mm x 50mm x 0.5mm thick depending upon the end use for which they are intended. Because timber machinists use hog-mills to produce a saleable product from roundings, off-cuts and joinery waste, commercially available hog-mill chips tend to be variable in moisture content and species mix and frequently contain a significant proportion of bark. The source quoted in reference (5) indicated a production rate of between 700-800 tonnes per month, which were again sold mainly to particleboard manufacturers, at a price of £11-13 per tonne ex works.

3) Moulding and routing flakes

This is waste produced during the manufacture of shaped timber articles, e.g. door and window frames, from machined stock. The flakes are generally rectangular in shape 20-30mm long, 10-20mm wide, and 0.5mm thick in the centre tapering to 0.05mm at the ends. Because they are machined from seasoned timber, the moisture content of the flakes is generally between 10% and 30% on dry wood. There is still considerable species mix, however, and softwoods and hardwoods are equally likely.

Because of their tapered profile and because the machining methods used tend to produce curled flakes, moulding and routing flakes have a density about 25% of that of the wood from which they are produced, much less than either of the other types of waste mentioned above. This makes the flakes expensive to transport, although the low price of £8 per tonne is attractive enough to persuade local particleboard manufacturers to utilise them in their products.

4.0.4 The choice of the source of the timber based raw material

From the data presented in the two preceding sections, a comparison of the available sources of raw material can be drawn up on the basis of cost. This is shown in Table 4.4.

A simple comparison of this kind clearly does not tell the full story since timbers from the various sources will require different types and levels of further treatment before they are suitable raw material for the processing studies. It has already

SOURCE	COST (£ per tonne)
Small standing timber	3.67[1]
Large standing timber	22.03[1]
Roundwood	18.50
Hog-milled chips	12.00
Moulder and router flakes	8.00
Sawdust	11.25

TABLE 4.4

COMPARISON OF COST OF WOOD RAW MATERIAL FROM VARIOUS SOURCES.

NOTES

(1) Cost has been calculated assuming $1m^3$ of standing timber weighs 0.7 tonnes.

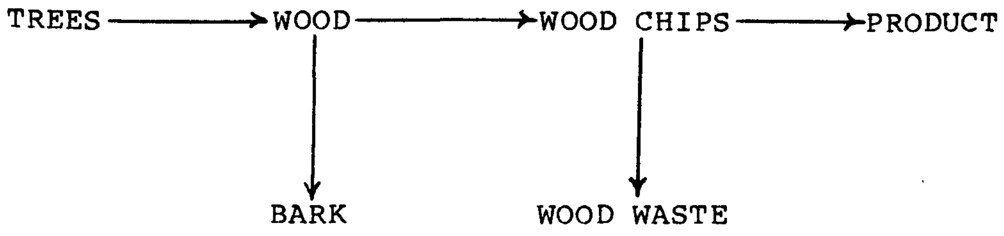
(2) All values have been calculated as an average where a range of costs has been quoted.

been stated that the cost of the raw material for the research work is not the prime consideration, consistency of supply and material quality being of paramount importance, and the use of any of the very variable materials detailed in section 4.0.3.2 can therefore be ruled out. On the basis of the figures presented in Table 4.4 it would appear that the cheapest available raw material would be small standing timber. This, or any of the other sources involving specifically felled timber, would also have the advantage that within limits, the species could be chosen to give optimum processing properties. It must be borne in mind, however, that as the diameter of the timber decreases, so the proportion of useful wood to bark also decreases, and the economics of the process change. This consideration is dealt with in more detail in the following section.

4.0.4.1 The economic production of a feedstock

Still drawing heavily on the analogy between the envisaged commercial product and particleboard, the economics of the processing route from raw green timber were examined assuming that the required end products from this stage (i.e. the starting material for the manufacturing process) are consistently sized, spindle shaped wood chips. The optimum size for the chips and the justifications for its choice are given in detail elsewhere in the thesis (Chapters 2 and 5).

The products and by products at the various stages of the process are as follows:-



It can be seen that there are two principal cellulosic by products formed during the manufacture of the product from the standing timber.

Experiments carried out during other commercially confidential work within Unilever Research indicated that a waste level of at most 50% would be expected, depending upon the timber size. Bark generally accounts for between 10% and 20% of the volume of a tree depending upon size and species, and for the work anticipated in this research, the raw material cost was relatively insensitive to the size of tree chosen. It was therefore clear that unless other factors emerged during the course of the research, the most cost effective starting material for the proposed work was small diameter standing timber.

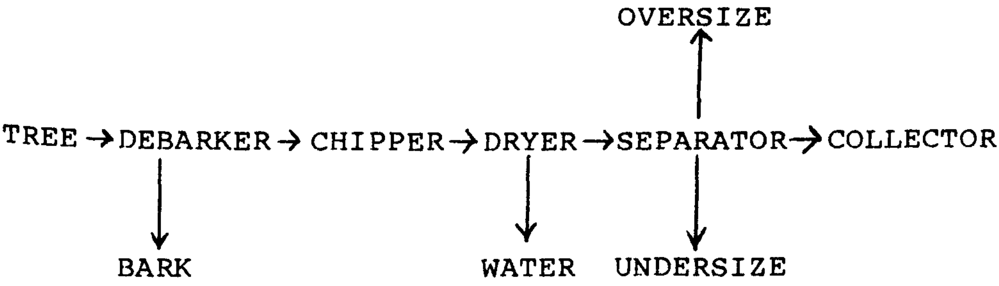
The equation for the production of wood chips from standing timber is therefore as follows:-

$$1 \text{ tonne WOOD} = \begin{array}{l} 0.50 \text{ tonne WATER} \\ + \\ 0.075 \text{ tonne BARK} \\ + \\ 0.33 \text{ tonne CHIPS} \\ + \\ 0.095 \text{ tonne WASTE WOOD} \end{array}$$

It is clear from the large amount of waste material which could be generated that there would be significant advantages if the by products could be utilised in some cost effective way. The considerations of this aspect are laid out below.

4.0.4.1.1 Utilisation of the waste

The sequence of operations in the production of a furnish is as follows:-



Since the three by products of the process are produced by separate operations it is possible to collect each as a single fraction; thus the available products are bark, water, and undersize wood particles or fines.

1) Bark possesses little strength and hence is not useful for total incorporation into products requiring mechanical strength. The principal chemical components of bark are listed in Table 4.5, (6), and over the years many workers, (7,8,9,10), have attempted to utilise or extract various of the components for commercial purposes.

The major uses in the UK (11), have been in the traditional utilisation of bark for tanning leather, and for horticultural products such as mulch and potting mixtures. Modern methods of bark stripping render the bark less suitable for leather tanning and work in this area appears to have stopped in about 1960. Horticultural applications of bark, on the other hand are still being investigated widely, and this seems to be the area most likely to provide an outlet for the waste bark from the chip preparation process. Sale of such a product would offset some of the basic raw material costs.

1) LIGNIN
   - material insoluble in concentrated mineral acids

2) CORK
   - cutose, suberin, and suberic acid (1,6 hexane dicarboxylic acid)

3) CARBOHYDRATES
   - holocellulose, total carbohydrate fraction
     a) cellulose
     b) hemicelluloses - arabans, xylans, mannans, glucosans, and uronic acid compounds

4) EXTRANEOUS MATERIALS
   - volatile acids and oils
   - non-volatile fatty oils, higher alcohols, resins and hydrocarbons
   - colouring matter
   - tannins and related water-insoluble phlobaphenes
   - polysaccharides, glucosides, pectins and sugars
   - organic nitrogen compounds
   - mineral inclusions
   - other organic components, e.g. saponins, mannitol, dulcitol

<u>TABLE 4.5</u>

PRINCIPAL CHEMICAL COMPONENTS OF BARK

2) Water, far from being a useful by product, is a major cause of expenditure in the chip preparation process. Drying of the chips is the most energy intensive and therefore costly process in the preparation chain since water makes up approximately 50% of the weight of the green timber. The latent heat of evaporation of water is 2.26 MJ kg^{-1}, and allowing for inefficiencies in the dryer, and heat used in raising the temperature of the moisture in the chips, an estimated 1600 to 2000 MJ (12), would be required for the drying of every tonne of as-manufactured chips. No profitable outlet for the water produced can be envisaged, other than for use as make-up condensate for other plant processes, and for a cost effective installation attention should be directed towards maximising the efficiency of the drying process.

3) Fines would be the third and final by product to result from the chip preparation process. In a conventional particleboard plant a proportion of these would be used in the face layers of the board to produce a fine, smooth surface. This is considered to be impractical in the case of extrusion, and a more cost effective use would be as a fuel to contribute to the energy input for the chip dryers.

An average figure for the calorific value of a typical coniferous softwood is 22 MJ kg^{-1},(using US Forest Products Laboratory Typical Values). Based on the quoted figure of 0.095 tonne of wood waste per tonne of green timber, there are thus 2100 MJ of energy available to contribute to the drying process, per tonne of raw material. Depending on the efficiency with which the fuel is burnt, therefore, the energy needs and resources of the chip preparation process are approximately equal, and utilising the waste wood in this way should be cost effective.

4.0.5 Summary and conclusions - cellulosic raw material

On the basis of the arguments and costs laid out above, it is clear that in the first instance the choice of cellulosic raw material should be specifically felled timber consisting of relatively small diameter trees. The facts suggest that most of the substance of the tree would be utilised, which not only increases the cost effectiveness of the process but is also satisfying from a conservation standpoint. It is possible that the research will indicate that the use of small twigs, branches and other forestry residue is also feasible, which is again progress in both economic and environmental senses (it is estimated (13), that tops, branches, leaves, needles, stumps and roots, and harvesting and transport losses amount to 40% of the volume of stemwood removals in Europe. In 1976 this was the equivalent of 135 million on line metres of forest residues.)

A point which has been raised in the text but which has not yet been addressed is the question of choosing a particular species of timber with which to begin the research. It was stated earlier that the majority of timber available in Great Britain is produced under the auspices of the Forestry Commission. Since 20% of the area of productive woodland administered by this is used for the production of Sitka Spruce (Picea sitchensis) a search of available literature was made (again concentrating on the particleboard theme) to assess the viability of the use of this timber. Sufficient information was gleaned during this exercise to indicate that the use of Sitka Spruce would not present any particular problems.

There was also found to be a significant amount of published research work based on this species which would be useful reference material for other parts of this thesis. It was also considered unnecessary for the research into the extrusion aspects of the work to begin the work with standing timber, and in the first instance the raw material was purchased as Sitka Spruce blocks which were reduced to woodchips as suggested earlier in this chapter, and as detailed fully in Chapter 5.

4.1 The adhesive phase

In order to assemble the prepared cellulosic particles into a strong, structural unit, an adhesive or binder system is required to bond the individual particles together. To form such an adhesive bond between wood particles there must first of all be close proximity of the surfaces involved, there must be adhesion across at least two interfaces, and in the case of thermosetting adhesives there must also be adhesive cure. The factors which influence these actions have been dealt with in some detail in Chapter 2 and there is nothing to be gained by reiterating them here. This section will therefore deal only with the choice of the particular adhesive system used for the research, and the reasons for making that choice.

4.1.1 Available adhesive systems

Again because of the close analogies between particleboard and products envisaged from this research, the particleboard industry has been used as a major source of data and information. Because the research is aimed at process characteristics and flow phenomena the possibility of using experimental adhesive systems such as were mentioned in Chapter 2 was ruled out as being an unnecessary complication at this stage. The possibility that such systems may have to be considered should the process be adopted for use in areas where more conventional alternatives are unobtainable has not been dismissed totally however.

Six possible binder systems were considered when drawing up the list of alternatives:
urea-formaldehyde resin, melamine-formaldehyde resin, phenol-formaldehyde resin, isocyanate adhesives, emulsion adhesives and thermoplastic binders.

4.1.1.1 Urea-formaldehyde resin systems

Over 80% of the world's production of dry process composition board is bonded with urea-formaldehyde adhesives, (12,14). There are three main reasons for this; they are relatively cheap, they are well-understood and therefore easy to use, and the quality of board produced is satisfactory for the majority of particleboard applications.

The manufacturing process for the production of urea formaldehyde resins is at the same time very simple and yet quite complex. The basis of all commercial processes is the heating together of quantities of urea and formaldehyde. The exact ratio of the reactants has a major influence on the properties of the resultant adhesive, and most commercial resins have been developed empirically to jealously guarded recipes. The operation can be a batchwise process, but continuous processes such as that described by Brunnmüller (15), are now the preferred manufacturing route, particularly for specialist resin manufacturers. Batchwise resin production is still commonplace in particleboard mills large enough to repay the investment in capital plant costs.

The principal disadvantage of urea-formaldehyde resins is their limited resistance to water. The cured resin is hydrolysed relatively easily in the presence of moisture, particularly at elevated temperatures, and products bonded with these resins are suitable only for indoor, low humidity applications such as furniture and flooring panels. There is also significant liberation of formaldehyde during, and even after curing, and although a great deal of work has been carried out on keeping this to a minimum (16), it still imposes limits on the application of this family of adhesives.

Urea-formaldehyde resin is generally available as a solution containing between 40% and 60% solids or as a solid powder. The UK guide price for urea-formaldehyde resins is approximately £300 per tonne.

4.1.1.2 Melamine-formaldehyde resins

The chemical similarity of urea and melamine means that most of the comments made about the urea-formaldehyde resins can also be applied to the melamine-formaldehyde resins, and only the differences between the two families need to be pointed out. The production routes are essentially similar, although until relatively recently the instability of melamine formaldehyde resin solutions meant that they were always supplied as dried powders.

Although there is virtually no increase in dry strength over urea-formaldehyde, melamine-formaldehyde resins have a significantly better resistance to thermal hydrolysis, and products manufactured using them have a limited resistance to boiling water. Although still not generally suitable for external applications, products made using these resins can therefore be used for more demanding applications than those incorporating urea-formaldehyde resins. The improved moisture resistance of the resins also confers advantages during assembly and manufacturing processes since a higher moisture content can be tolerated in the furnish. There is also less problem of toxic emissions during resin cure with melamine formaldehyde resins. The disadvantage of melamine-formaldehyde resins over the urea based equivalent is the relatively high cost of £750 per tonne, however by using blends of the two resins, acceptable compromises in terms of price and performance can be reached.

4.1.1.3 Phenol-formaldehyde resins

Phenol-formaldehyde resins are also produced by condensation reactions between the two components, and generally are divided into two distinct types; resol or one stage resins, and novalac or two stage resins.

The single stage, resol type resins are formed by reacting a mixture of 1:2, phenol: formaldehyde, in the presence of an alkaline catalyst. This forms a purely thermosetting liquid resin with a tailored shelf life of between several hours and many months.

The novalac, two stage, resins are formed by first reacting approximately equal amounts of the two components over an acid catalyst. The resulting resin can be taken up in a solvent to form a liquid resin, or dried and pulverised to form a powder.

The second stage involves the addition of extra solid formaldehyde (usually in the form of hexamethylene tetramine) which breaks down on heating and initiates resin cure.

The resins produced are almost completely waterproof being able to sustain long periods under boiling water, and can therefore be used safely for outdoor applications. Although the cost at £800 per tonne is comparable to that of the melamine-formaldehyde resins for a much improved performance, the higher press temperatures and longer press times required for the phenol based compounds has limited their use commercially. Phenol-formaldehyde resins are also very sensitive to furnish moisture content and require tight process control if the full benefit of their improved properties is to be reaped.

4.1.1.4 Isocyanate adhesives

Isocyanate adhesives are highly reactive materials whose bonding properties rely on the ability to form urethane chains. They exhibit strong chemical affinities for many functional groups, particularly those containing active hydrogen such amino, imino, carboxyl, amide, sulphonic, and most relevant, hydroxyl groups. The strong bond formed by the chemical bonding of the wood hydroxyl groups to the urethane chain makes such combinations proof against water, dilute acids and chemical liquors. The nature of the bond also means that the system contains no water, hence all of the binder applied functions as an adhesive. In addition, this property also means that the cellulosic phase need not be dried to such a low moisture content as with other adhesives. The hydrophobic nature of the urethanes also means that a degree of moisture resistance is conferred to the adherends, which in the case of particleboard makes the product suitable for external applications, [such particleboard is officially approved for building in the Federal Republic of Germany (17)].

The major drawback to the use of isocyanate adhesives is the technical difficulty in handling a material which will adhere to almost any surface. Problems have been encountered with particleboard adhering to various parts of the plant during lapses in control of the production cycle (12).

The present cost of a typical isocyanate adhesive system is approximately £1500 to £2000 per tonne.

4.1.1.5 Emulsion adhesives

These adhesives are generally based on aqueous emulsions of thermoplastic polymers, for example polyvinyl acetate, (PVA) and polyacrylates. There is a very large family of such adhesives with a range of applications each specific to an individual formulation, however all of the compounds are particularly effective in bonding to cellulosic materials.

Adhesive cure is by solvent loss or absorption into the adherend, and none of the single components systems is water resistant to any degree. Cross linking of the resins by exposure to radiation has proved beneficial in some cases, giving moderate water resistance, and recent developments in the field of two component systems (18) also show promise in this respect.

The cost of such resins depends upon the degree of sophistication in the formulation, however a UK guide price for a simple pva is approximately £600 per tonne.

4.1.1.6 Thermoplastic binders

The use of thermoplastics as "adhesives" falls into two categories; hot melt applications, and the use of materials as fillers for conventional thermoplastic materials.

Hot melt adhesives as the name implies rely on being first melted then allowed to solidify in contact with the surfaces of the adherends. The principal component of a hot melt is usually a polymer (e.g. polyethylene, vinyl copolymers, polystyrene, polycarbonate, and polyamides), compounded with plasticisers, fillers and reinforcing materials to tailor the properties of the adhesive to suit the end use. The thermoplastic nature of these materials generally confines their use to low load, non-structural applications, where ease of application and rapid attainment of working strength are important. They are relatively high cost materials with an approximate UK price of £1200 per tonne.

Conventional thermoplastics such as polyethylene and polypropylene are frequently compounded with inert fillers to improve stiffness, abrasion resistance, and particularly cost effectiveness (19). Although these fillers are generally inorganic compounds, similar experiments have also been carried out using wood flour and fine wood chips as fillers with some success (20). There are clearly fire hazards associated with the latter process and it was considered inappropriate for use in this research, particularly as the technique is still largely at the experimental stage and this was a feature considered undesirable when deciding on a binder system.

4.1.2 The adhesive system chosen

Table 4.6 summarises the main points noted in the text above. From this it can be seen that the permanence of the adhesives is roughly proportional to the cost, and that a priority must be established

TABLE 4.6 CHARACTERISTICS OF ADHESIVES CONSIDERED

SYSTEM	SETTING PROCESS	FORM	PERMANENCE	COST £/TONNE
Urea-formaldehyde	Acid catalysed. Rate temperature dependant.	Liquid or powder.	Water resistant only	300
Melamine-formaldehyde	Acid catalysed. Rate temperature dependant.	Liquid or powder	Water resistant but with limited life.	750
Phenol-formaldehyde	Can be catalysed (e.g. resorcinol) Rate very temperature dependant.	Liquid or powder	Waterproof and boil proof.	800
Isocyanate adhesives	Chemical reaction with adherend functional groups	Solution in organic carrier.	Water, boil, heat solvent, impact and fatigue resistant.	1500 2000
Emulsion adhesives	Solvent evaporation	Low solids content high viscosity	Low resistance to moisture and solvents Soften at moderate temperatures	600
Thermoplastic matrices	Solidification	Solid or granular powder	Water and solvent proof. Soften at moderate temperatures	1200

as to which attribute carries the most significance. It has already been stated that in the first instance, cost need not be of prime consideration. Nor, for the purposes of the research, is it considered vital for the product to be weather or boil proof. The processing difficulties which would accompany the use of isocyanate and thermoplastic binders legislates against their use as research materials. The use of urea-formaldehyde resin, on the other hand, has advantages in terms of simplicity, ready availablility and comprehensive documentation. These make it an ideal research material if the advantages offered by the melamine- and phenol- formaldehyde families and the emulsion adhesives are not specifically beneficial. It was considered, as was the case with the cellulosic raw material, that the use of a basic and readily available material was logical for the initial stages of the work, with the knowledge that other materials were available should the research indicate the necessity. For these reasons urea-formaldehyde resin was chosen as the adhesive system on which the initial work would be based.

On the basis of this decision, contact was made with Ciba-Geigy Ltd, who kindly agreed to supply samples of their "Aerolite" resin in sufficient quantities to satisfy the requirements of the research programme. Details of the resin used are given in Appendix I of this thesis. Although most acid salts will catalyse the curing reaction of this resin successfully, (see Appendix 1), for good reproducibility and to limit the number of experimental variables to a practical level, ammonium chloride, (British Drug Houses "Analar" grade), was used throughout the experimental work for this thesis.

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CHAPTER FIVE. MATERIALS PREPARATION AND CHARACTERISATION.

5.0

Since the starting material for the extrusion testing is made up from two individual components, the wood chips themselves and the binder system, this chapter can be divided conveniently into two sections. The first section will deal with the preparation and characterisation of the wood chips, while the second will deal in a similar manner with the adhesive binder.

5.1 The Wood Particles.

At the very earliest stage of the research it was decided to limit the investigation to one species of timber and one range of chip sizes, (see Chapters 2 and 4), in order to confine the variables to a manageable number and to those which could be reproduced consistently and readily. In order to maximise the reproducibility, all of the chip preparation and characterisation was carried out in-house using raw material of known origin and with guaranteed continuity of supply.

5.1.1 Preparation of the wood chips

Sitka Spruce (Picea Sitchensis) timber was brought in from a local timber merchant in the form of 100mm seasoned cubes. This was then reduced to large flake ($\approx$50mm x 20mm x 5mm) by the use of a chipper, designed and constructed at the Port Sunlight Laboratory of Unilever Research (URPSL). These flakes were then dried to a moisture content (see section 5.1.2.1. for details of moisture content measurement) of approximately 7.5% by weight using drying ovens, again designed and constructed at URPSL. The relatively dry flakes were then reduced to chips using a hammer mill (Miracle Mills Model 1RR) fitted with a 9mm screen. The output from the mill was divided into fine dust and useful product by the use of a high efficiency cyclone, again supplied by Miracle Mills. The wood dust was of a very small particle size and represented less than 0.1% by weight of the output from the mill. Since there was no profitable way of using the dust within the research programme it was discarded.

From the cyclone the useful fraction of the mill product was passed to a sieve (the Model SM, manufactured by Russell Constructions Limited) fitted with a number 30 (500μ) mesh screen. The material which passed through this mesh was classified as "fines" and was bagged and stored. Material retained on this mesh was passed through the sieve again, this time fitted with a coarser 10 ($\approx$2mm) mesh screen. Material passing through this screen was designated "Passed 10 Retained 30" ($P_{10}R_{30}$), and formed the wood based starting material for the research programme.

Material which was retained on the 10 mesh screen was classified as "oversize" and was subsequently recycled to the hammer mill, this time fitted with a 6mm screen. After cyclone separation the resulting mixture was sieved in the same manner as the first pass chips again to produce "fines", "$P_{10}R_{30}$", and a much lower proportion of "oversize" chips. The yields from the two operations and the total yields are listed in Table 5.1. Obviously the yield of $P_{10}R_{30}$ could be further increased by recycling the remaining 5.2% oversize chips, perhaps using a yet smaller screen, and in an industrial situation this would be likely to be the practice. For the purposes of this research, however, the continued recycling of chips to maximum yield was consuming valuable time which could be more profitably spent on other areas of the work, and the 78.3% yield of usable material was therefore considered to be acceptable.

The production of the large chips from solid timber cubes was found to be both time consuming and expensive, and when further inconvenience arose from the availability of the chipper becoming limited, an alternative source of raw material was sought.

	YIELD AS PERCENTAGE OF TOTAL BY WEIGHT		
	FINES	$P_{10}R_{30}$	OVERSIZE
First Pass On Large Chips @ 5% Moisture	10	64	26
Second Pass On Oversize From Above	25	55	20
Total From Both Operations	16.5	78.3	5.2

TABLE 5.1. YIELDS OF VARIOUS SIZE FRACTIONS OF CHIPS FROM A TYPICAL PREPARATION OPERATION.

Flakes of a suitable size and of the correct timber were found to be available in the wet (unseasoned) state from the pre-processing stocks of a Concern board manufacturing company. Drying, hammer-milling and sieving of these flakes according to the scheme used for the previous wood source gave a range of product almost identical in terms of yield and particle size to that prepared from the solid timber. This provided a much more efficient route to the starting material than previously whilst also giving more flexibility in terms of feed moisture content to the hammer-mill. This latter point is a distinct advantage when trying to produce maximum performance from the composite product, as was indicated in Chapters 2 and 4. An additional result from these trials was the indication that the nature of the product from the preparation operations in terms of particle size, shape and yield is independant of the source of the timber and is therefore probably only a function of the type of wood employed.

5.1.2 Characterisation of the wood chips.

Since many of the properties of the chips themselves and some of the characteristics of the bonding reactions are influenced by the moisture content of the chips, it was considered important to be able to measure the moisture content in an accurate and reproducible manner.

Similarly the properties of the finished product are influenced greatly by the particle size distribution within the wood chips, hence characterisation of the chips in this respect was also considered to be of prime importance.

5.1.2.1 Measurement of moisture content.

Three techniques are commonly used for the determination of the moisture content of wood or wood particles: distillation, electrical resistance, and oven drying. All of these techniques were available for use during the research reported in this thesis, and the details of the individual techniques will therefore be given.

The distillation technique is not unique to the wood particle application involved here, and the familiar Dean and Stark apparatus used is employed in so many branches of science that a detailed description is unnecessary (see BS 756 for full details). It is sufficient to say that the particular apparatus in question had a capacity of 50g of chips and that the distillation medium used was xylene. It will be appreciated that although this method is very accurate in terms of water extraction, it requires significant amounts of preparation and consumables and can take in excess of twenty-four hours to reach a steady value for moisture content it is therefore very well suited to laboratory measurement of moisture content but is less suitable in the present situation where repeated measurement of moisture content may be required at intervals as short as 30 minutes. The Dean and Stark method therefore was only used during this investigation as an absolute check on moisture content when calibrating or double checking the accuracy of one of the other two methods which were more frequently used.

The electrical resistance method of moisture content measurement relies on the fact that the electrical resistance of wood is directly proportional to its moisture content. By measuring the resistance of the piece of wood between two or more electrodes, it is possible after calibration of the instrument to use the resistance value directly as a measure of moisture content. There are several drawbacks to this method however which limit the usefulness of this technique. The electrical resistance of wood also varies in inverse proportion to its temperature and this relationship is not constant for different species. Corrections therefore have to be made if several different species are to be compared. A further source of error lies in the fact that the meter is measuring the resistance between metal probes, and this will obviously be influenced by the contact between the probes and the wood, by the depth of penetration of the probes into the wood, and by the conditions prevailing on the surface of the wood at the time of measurement. Nevertheless this method is very quick and convenient and in situations where comparative rather than absolute values are required the electrical resistance method does offer significant advantages.

The equipment used for electrical moisture content measurements during this research was the "Aqua-boy, Model HM1" moisture meter manufactured by K P Mundinger GmBH. A photograph of the equipment is shown in Figure 5.1, and as can be seen the instrument offers a choice of two probes, a penetration probe used for measurements on solid timber and on the product from the extruder, and an insertion probe used for measurements of the moisture content of the chips at various stages of the process. The manufacurers claim an accuracy of ±0.2% for the instrument which reference to Table 5.2, which gives a comparison of the values obtained by the different methods for similar samples of wood chips, will show is optimistic. Correlation between the methods and more particularly between batches using the same method, is sufficiently good to enable results obtained to be used with confidence.

The third method, oven drying, and a method using the same principle, the moisture balance, were used more frequently than any of the other methods. The moisture balance particularly was used as the mainstay technique since it is a very rapid technique but one which reference to Table 5.2 will show is

FIGURE 5.1 The "Aqua-Boy" electronic moisture meter and probes.

SAMPLE		$P_{10}R_{30}$ (1)	$P_{10}R_{30}$ (2)	$P_{10}R_{30}$ (3)
TECHNIQUE		MOISTURE CONTENT	MOISTURE CONTENT	MOISTURE CONTENT
DEAN + STARK	1st	8.50	10.90	10.00
	2nd	8.45	10.90	10.05
	3rd	8.50	10.80	10.00
OVEN DRYING	1st	8.20	10.75	9.70
	2nd	8.20	10.70	9.60
	3rd	8.20	10.75	9.65
"AQUA-BOY"	1st	7.8	11.0	10.0
	2nd	7.5	10.5	9.7
	3rd	7.9	10.5	9.9
MOISTURE BALANCE	1st	8.25	10.70	9.60
	2nd	8.20	10.70	9.50
	3rd	8.25	10.70	9.50

RESULTS FROM 5g SAMPLES THEREFORE READINGS ARE DIRECT %AGE X 2.

TABLE 5.2 COMPARISON OF RESULTS OF MOISTURE CONTENT TESTS ON THREE DIFFERENT CHIP SAMPLES USING EACH OF FOUR TECHNIQUES.

capable of yielding accurate and reproducible results.

Conventional oven drying calls first for the initial weight of a sample to be determined accurately. The sample is then dried in an oven at $103 \pm 2^{\circ}C$ until its weight is constant. A simple calculation gives the percentage moisture content derived from the weight loss. Again the method itself is common to many branches of science although the detailed technique varies from discipline to discipline. In the case of untreated, unseasoned solid timber the period of drying can be as long as 48 hours, however in the case of wood chips the much greater surface area to volume ratio and the short distance from any point within a chip to its surface mean that accurate results can generally be obtained in as little as 15 minutes.

The specific equipment used for the series of tests carried out for this work were a low temperature ($200^{\circ}C$ max) forced circulation oven manufactured by AEW Limited of Andover, and a top pan balance, the GC 62, manufactured by Oertling. The temperature of the oven was shown to be controllable to within $\pm 1^{\circ}C$ by a series of tests, and the calibrated accuracy of the balance was ± 1 digit, which in the case of the tests in question gave ± 10.0mg or 0.05% on the 20g samples used. Since the average weight loss during such tests was of the order of 1.52g (7.5 to 10% total moisture content), the accuracy of the technique was always better than 1%.

The second technique involving heat-induced-weight loss determination was carried out with the use of a commercially available moisture content balance manufactured by August Sauter KG, the MPRT 160/100. This consisted of a conventional top pan balance capable of being read to 0.0025g (or 0.025% on an alternative, direct reading percentage weight loss scale), over which a variable temperature infra-red source is fixed. Previous work, (1) had indicated that for the same sample materials (Sitka Spruce wood chips, and a variety of furnish material based on them), a value of 150V on the scale of infra red source control unit resulted in consistently accurate results without causing burning of the test sample. The duration of the exposure to the heat source is also variable, and tables 5.3 and 5.4 and Figures 5.2 and 5.3 show the results of experiments carried out to determine the optimum experimental conditions in terms of accuracy of results combined with efficient use of the time available. By striking a balance between drying to constant weight and expediency of technique, a standard operating procedure was arrived at. A 5g sample was exposed to the infra red source for a period of 20 minutes and the weight loss (or % moisture) was read immediately before the source was turned off. Examination of the data presented in

TIME ELAPSED (minutes)	SAMPLE WEIGHT (g)			
	Oversize		As Received	
	120V	150V	120V	150V
0	5.000	5.000	5.140	5.140
5	4.803	4.690	4.480	4.425
10	4.698	4.665	4.280	4.200
15	4.675	4.663	4.225	4.185
20	4.668	4.660	4.210	4.180
25	4.665	4.660	4.208	4.175
30	4.660	4.660	4.205	4.170
35	4.660	4.660	4.203	4.165
40	4.660	4.660	4.200	4.165
45			4.200	4.165
50			4.200	4.165

Table 5.3. Results from experiments to determine the effect of heater voltage on drying time using Sauter instrument

TIME ELAPSED (minutes)	SAMPLE WEIGHT (g)			
	Unsieved	Fines	$P_{10}R_{30}$	Oversize
0	5.800	5.163	5.000	5.000
5	5.475	5.000	4.695	4.845
10	5.355	4.960	4.600	4.805
15	5.325	4.945	4.580	4.800
20	5.318	4.945	4.573	4.795
25	5.315	4.945	4.570	4.795
30	5.315	4.945	4.570	4.795

Table 5.4. Results from experiments to determine optimum exposure time for moisture content determination using Sauter instrument at 150°C

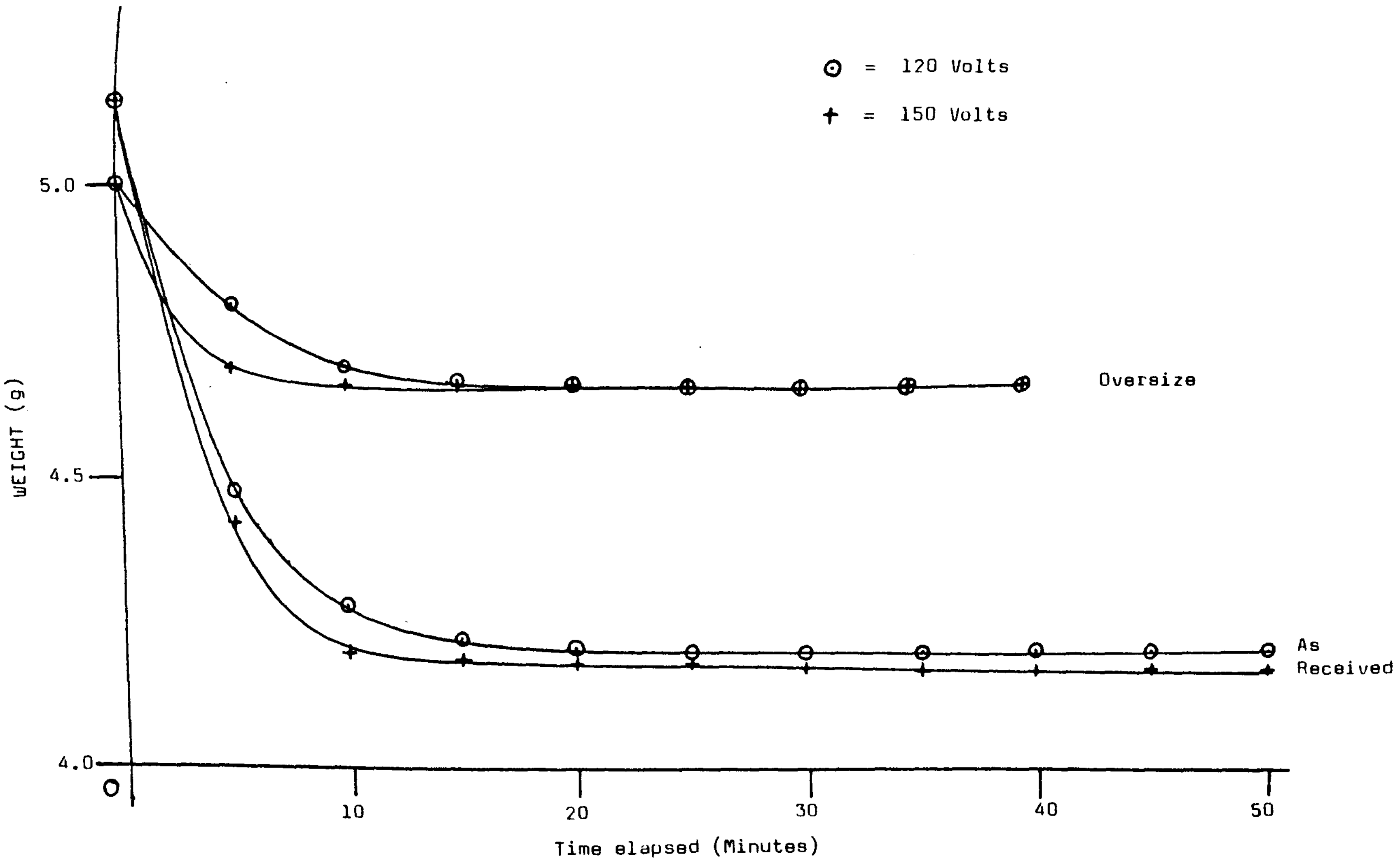
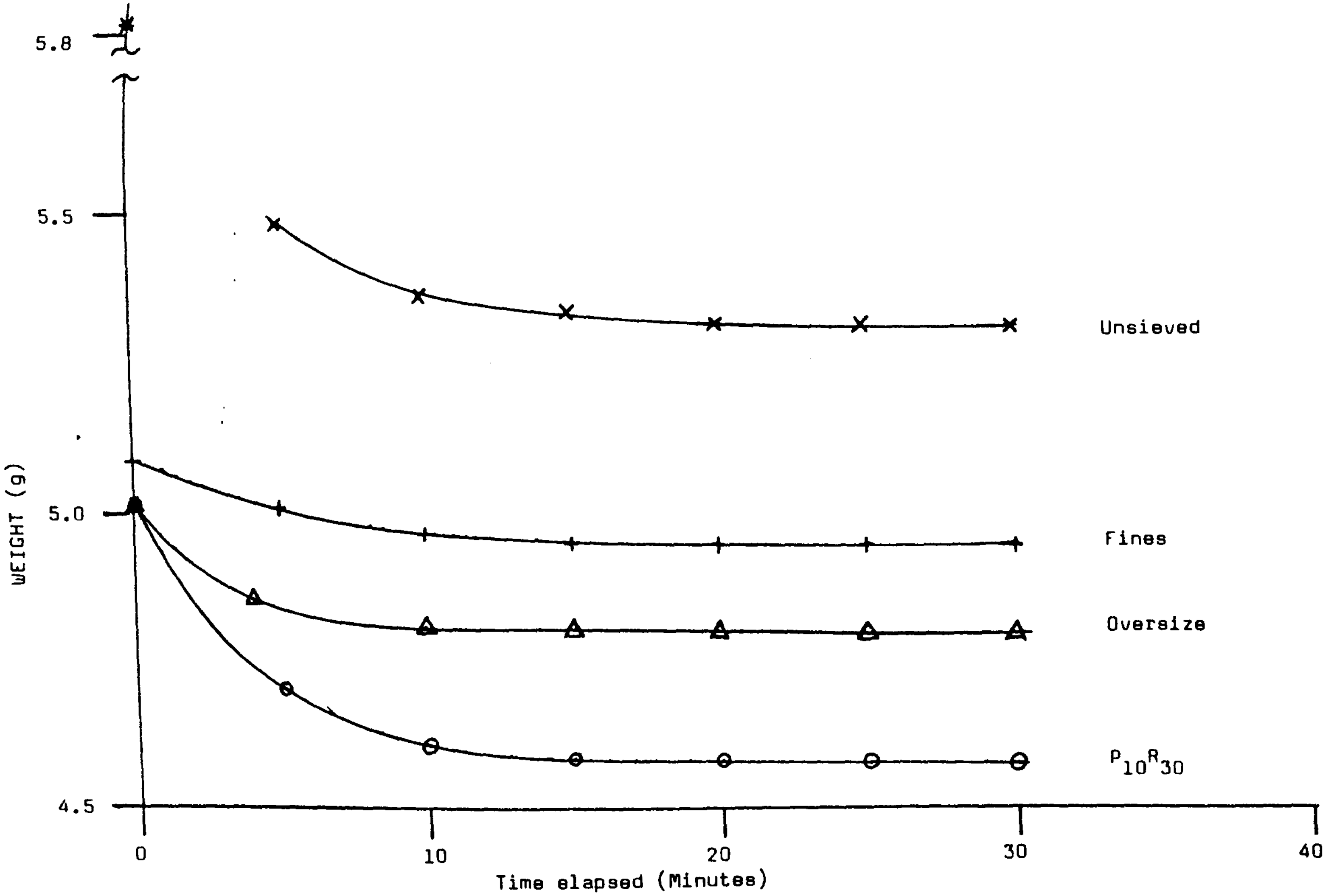


Figure 5.2. Plots of results from Table 5.3



<u>Figure 5.3</u>. Plots of results from Table 5.4

Figures 5.2 and 5.3 shows that the maximum error incurred by a fixed exposure of 20 minutes rather than drying to constant weight ($t > 50$ minutes) is less than 1.2%, which is considered acceptable.

Both of the balances mentioned were used throughout the investigation and were checked periodically for accuracy using analytical weights accurate to 0.2 mg.

5.1.2.2. Particle size analysis

The second characteristic of the $P_{10}R_{30}$ fraction which needed to be known in more detail was the particle size distribution within the broader pass 10 retain 30 limits.

A variety of techniques is available for the characterisation of the particle size distribution of such a mixture of particles, and those which were considered were the mechanical methods using graded sieves, air-classifiers, and zig-zag classifiers and the optical technique of image analysis.

The method chosen finally was an opto-electronic technique making use of the Kontron 1BAS microprocessor based image analysis system sited at URPSL, and a selection of photographic prints prepared from samples of the wood particles.

The reasons for making this choice of technique were twofold: Firstly the technique produces particle distribution data based only on the size and shape of individual particles. Reference to chapters 2 and 4 of this thesis shows that most of the published research concerned with wood composites confirms that it is particle size and shape which have the most significant effects on the properties of otherwise similar products. Secondly, the equipment was available immediately it was required and skilled personnel were on hand to assist with machine operation and interpretation of results, thus making efficient use of the limited time available.

The machine itself was calibrated using a standard 25.4mm diameter spot, and photographic prints of well mixed representative samples were positioned beneath the television camera of the instrument.

The image information from the camera is processed by the instrument software and can be presented in a variety of forms. Three of these, bar chart frequency plots, cumulative frequency plots, and class listings were chosen for this research, and two particle parameters, chip length (D_{max}) and chip aspect ratio (USER 01) are presented in each of these forms for the three fractions of chips of interest in this study. Table 5.5 gives a summary of the results obtained.

SAMPLE NAME	NUMBER OF PARTICLES	D_{MAX} MEAN + STD.DEV (mm)	USER O1 MEAN + STD.DEV (mm)
OVERSIZE	831	6.2 ± 2.2	3.9 ± 1.3
$P_{10}R_{30}$	948	3.3 ± 2.2	3.2 ± 1.2
FINES	1012	0.8 ± 0.8	1.9 ± 0.8

<u>TABLE 5.5</u>. RESULTS FROM IMAGE ANALYSIS OF TYPICAL SAMPLES OF THREE PARTICLE FRACTIONS.

5.2 The Binder System

Again at the earliest stage of the research the decision was made to confine the work on the binder to one basic system in the first instance. The number of alternative binder systems available can be judged by reference to chapters 2 and 4. The simplest system in commercial use, and therefore the one about which most information has been published, is the amine-formaldehyde system, which is typified by the simplest member of the group, urea-formaldehyde resin. Personal communication with acknowledged experts in the field (1,2), confirmed that urea-formaldehyde resin exhibits properties typical of the series and that results based on such a system would be capable of extension to other resins within the group and even binders of a different nature should this be necessary. On the basis of this information personal contact was made with CIBA-GEIGY Ltd, who generously supplied the urea formaldehyde resin used for this investigation in the spray dried form of their commercial resin "Aerolite CB". The choice of this particular resin, on which a great deal of research has been carried out and about which a great deal of technical information is available, meant that experimental work on the resin system could be kept to a minimum, a fact for which no apology is made.

The experimental work done on the binder system for this study was therefore confined to examining the effects of various additives (lubricants and water repellant agents) on the curing kinetics of the resin. The main experimental technique employed was therefore gelation time testing, carried out in the manner described in BS 2782. The equipment used was commercially available purpose designed equipment manufactured by Techne (Cambridge) Ltd., the Dri-block DB1 heater unit and the Tecam gelation timer fitted with a disc probe. Tests were carried out on samples taken from bulk batches of liquid resin to which the various additions were made to give individual sample weights of between 40 and 50g. Weighing out of the resin components was performed using an Oertling GC 62 top balance read to an accuracy of 0.01g, and mixing and blending was achieved by the use of a Hamilton Beach Model 30DM high speed mixer.

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CHAPTER SIX. DESIGN PHILOSOPHY AND CONSTRUCTION OF THE BASIC EXPERIMENTAL RIG.

Chronologically the decisions relating to raw material source, type and nature were taken prior to and independently of the discussion documented in this chapter concerning the physical characteristics of the experimental equipment. This means that when deciding on the type of equipment to use, and on the detailed mechanical specifications of that equipment, the questions of what material it would be required to handle and what characteristics this raw material would have, had already been answered. This made decisions relating to machine design easier to arrive at by eliminating some of the potential possibilities at the outset.

6.1 Introduction

Although the work described in this thesis deals only with extrusion routes to composite products, the research evolved from the much broader based objective of investigating the application of currently utilised high speed plastics processing machinery to the production of such articles. This brief historical introduciton is therefore included to outline the decisions taken during the preliminary stages which culminated in the choice of ram extrusion as the preferred process.

6.1.1 Candidate processes

The major processes currently used within the plastics processing industry are as follows:-

Extrusion
Injection moulding
Blow moulding
Vacuum forming
Calendering
Compression moulding (including transfer moulding)
Casting (including slush moulding, dip coating, powder coating and rotational moulding)

With the foreknowledge of the raw material in question, certain of these can be eliminated immediately. Blow moulding and vacuum forming as primary processes are obviously unsuitable with such a fibrous and therefore porous starting material, and although calendering is employed as a primary processing step with some particulate materials, (see Chapter 2), it was thought to be an unlikely contender for this research.

Press moulding is obviously a possible production process beginning with such material, however, as will be seen from Chapter 2 and Appendix 1, there has already been a great deal of research and commercial interest in this area, and it was thought more profitable therefore to look elsewhere for suitable processes.

Injection moulding is basically a pressurised casting process and was originally developed from the die casting techniques used for metals. The high viscosity of polymer melts precludes the use of simple melt and pour techniques and necessitates the use of high pressures to force the fluid into the cavities and ensure mould filling. Although injection moulding of reinforced thermosetting compounds is a well established technique, the size and complex geometry of the raw material particles together with the low fluid content were thought to preclude the use of injection moulding for this work, at least until further information about the "rheology" of the system had been obtained. The high cost of moulds for injection moulding, and the relative complexity of the machine also legislate against the use of this technique as a development tool for an as yet uncharacterised raw material.

Although it was stated above that casting is impractical with high viscosity fluids such as polymer melts, the process can be used successfully if the polymer is modified in such a way as to lower the viscosity to a manageable value. The casting techniques mentioned in the list above make use of this fact by employing polymer in the form of low viscosity plastisols (a dispersion of the polymer in a suitable plasticiser) or by using the polymer in the form of a free flowing fine powder which is sintered once in the mould to produce the final article. Again the nature of the raw material makes the use of such techniques for this study unacceptable, since the low proportion of fluid in the premix ($< 10\%$) is insufficient to make the mixture free flowing, and the addition of extra fluid or an alternative suspension medium would not only increase the raw materials costs to an unacceptable level, but also make the processing equipment considerably more complex and therefore expensive. The geometry and physical nature of the wood chips also mean that any product manufactured from the random assembly of chips without pressure would be highly porous and mechanically weak.

The final alternative from the original list is extrusion. Extrusion in its many forms is the most widely used plastics processing technique, and consequently one on which a great deal of academic interest has been focussed. The wide usage is due to the fact that extrusion is not only a finishing process, for example in the manufacture of pipe, tube and mouldings, but also an important mixing and pumping technique, frequently used as part of a complete manufacturing process which employs another of the techniques mentioned previously as the finishing stage.

The vast range of different designs of extruders can be divided broadly into two categories, screw extruders and ram extruders. Screw extruders as the name suggests operate on the principle of the Archimedian screw, with the product being carried along the extruder barrel by the action of the screw flights. There are further distinctions within this sub-group and multiple screw extruders, in which the screws may rotate in the same or different directions or where one screw may even be stationary, are widely used throughout the plastics processing industry. Screw geometry also varies considerably from design to design to cope with specific demands made upon extruders for different applications.

It is clear from the brief information given above that the design and specification of a screw extruder are complex subjects which rely to a large extent on practical experience. This then pre-supposes a knowledge of at least some rudimentary processing characteristics of the raw material which is to be extruded, something which is lacking for the wood chip starting material for this investigation.

At the other extreme, ram extruders tend to be relatively simple machines relying on the positive pumping action of a reciprocating piston in a cylinder to force the material through a die. This simplicity is, however, the greatest drawback to the commercial use of ram extruders since without additional equipment and/or the use of multiple units, it is impossible to obtain continuous throughput or constant pressure (1).

From the above discussion it can be seen that the only serious contenders for a research technique with possible future commercial applications are injection moulding and extrusion. Further to this, the need for comprehensive monitoring and measuring systems to fulfill the research aims on the one hand, but the speculative nature of the work and therefore an uncertain future on the other, **militate** strongly against the very capital intensive nature of injection moulding as the basis for the work, leading to the decision to pursue the extrusion route throughout the course of the subsequent research.

6.1.2 Screw vs ram extrusion for woodchip applications.

Having decided on extrusion as the basic technique for this investigation, it was then necessary to determine whether screw extrusion or ram extrusion would be the better route to follow, in order that equipment specifications might be finalised.

Since a key element of any research tool used for novel work is flexibility, one of the principal design criteria for the equipment for this study was that the instrument should be simply and readily adaptable in certain basic areas, specifically extrusion speed, extrudate size and shape, and internal die geometry.

The first of these is largely a question of mechanical design since it requires either a change in rotational speed for the screw machine or a change in axial speed for the ram alternative, both of which are relatively easy to accomplish in a number of ways. The second and third points concern only the die section of the machine, which could be identical in both cases, and is generally discrete from the main body and therefore readily interchangeable whichever pressure system it is used with.

The question of which system to use then becomes a question of balancing the following major factors in terms of an economic approach to satisfying the aims of the project.

1) How suited is the machine to use as a research tool?
2) How will the machine cope with the raw material provided?

3) What is the cost of the basic research scale machine?

4) How easy will the scale-up from laboratory to pilot to production machinery be?

None of these questions can really be considered in isolation since there are complex interdependencies between all of them, however a description of the simplistic answers actually used will clarify the reasons why the approach documented here was adopted.

6.1.2.1 Research applicability

It was thought at the outset that it may be possible to buy in a commercially available piece of equipment, either an instrumented extruder or a rheometer, with which to study the "rheology" of the basic raw material. The measurement and control aspects of the instrumentation would thus have been taken care of, and the machine would without doubt be capable of producing research quality data.

Unfortunately most commercial rheometers rely on measurement mechanisms totally unsuited to the raw material in question (e.g. cone and plate, co-axial cylinder, inclined plane), and on a scale totally inappropriate to the particle size and shape being considered in this work. For these reasons the details of individual such instruments will not be given here, and the reader is directed to the recent excellent reviews of the subject referred to in the bibliography (2,3). Polymer rheometry, dealing as it does with very viscous materials for the most part, approaches more closely to the type of measurements required for this work. The instruments most commonly used for polymer work are of the capillary viscometer type, in which either the flow rate at constant load, or the pressure at constant flow rate are the parameters measured. In essence, such machines could be described as instrumented ram extruders, and as such might have the potential to be adapted for the current study.

Commercial extruders, on the other hand, tend to be built to a design specification with foreknowledge of the required ranges of all controllable variables, and hence even machines designed specifically for research generally have limited operating ranges and limited monitoring of the operating parameters. They are therefore admirably suited to research on systems with well defined properties but have limited use for less well characterised systems such as the one under investigation in this research.

6.1.2.2 Machine/material compatibility

The point has been made several times that the flow of the system under investigation, particularly at high pressures, is something on which very little research has been carried out and therefore about which very little quantitative data is available. Decisions about the suitability of a processing route can therefore only be based on subjective judgements and on those characteristics of the process which do not specifically involve detailed knowledge of material flow properties.

Personal experience with screw extrusion, details of which cannot be given for reasons of commercial confidentiality, indicated that the proposed raw material might present feeding problems unless a screw with very deep flights were employed. The inference from this is that a large diameter machine would be needed to cope with the feedstock, and a research machine of such specification would be unlikely to be available "off the shelf". A further problem caused by the lack of knowledge of rheological fundamentals about the feedstock is the inability to predict, even very approximately, what the power requirements for a suitable screw extruder might be. This affects not only the overall size of the machine but also again the size of the screw, since the core diameter will determine the amount of power which can be transmitted through the screw without the danger of shearing it.

A further characteristic of screw extruders (which is a positive advantage when processing thermoplastics) is the considerable amount of heat which is generated during processing. With a cellulosic starting material such as wood chips this could cause problems of product charring, with the ever present danger of uncontrollable combustion. Although it was anticipated that some heat input would be required to accelerate the curing of the binder, it was felt that the uncertain nature and quantity of this self generated heat would be more of a risk than an advantage, and this factor alone legislated strongly against the use of a screw based machine.

Ram extrusion, on the other hand, was already being used with a similar starting material as the basis of a commercial process (see Chapter 2), which gave some indication that the process was at least viable. The lack of basic rheological information still meant that questions regarding the size and power of the machine could not be answered, but the intrinsic simplicity of ram extrusion suggested that there would be less of a problem coping with high input power requirements should this be necessary.

Again the likelihood of finding such a machine "off the shelf" was poor, although the polymer rheometers mentioned previously might be a starting point. Another possibility, and the one thought more likely, was that a ram injection moulding machine or a universal testing machine might be capable of being modified to accommodate the necessary additional equipment. The latter of these two was a particularly attractive proposition since such a testing machine was already available for use.

6.1.2.3 Cost considerations

Cost is again an area in which the lack of fundamental knowledge creates difficulties in arriving at meaningful conclusions. Without a complete specification for the requirements regarding size, power, throughput and monitoring facilities it is impossible to cost the machine accurately.

A basic single screw extruder of laboratory type dimensions and performance could be purchased for between £5,000 and £10,000 brand new. Of this basic figure, however, the screw itself can make up 20% or more of the cost depending on the degree of complexity of its geometry. It would be unwise in view of the feedstock not to have a back-up screw or perhaps a screw of alternative geometry available and this would increase capital costs considerably. Similarly it was thought unlikely that a manufacturer would be willing to loan a machine or screws in view of the high potential for causing expensive damage involved in the work.

On the other hand, a small, hand operated ram injection moulding machine was found to be available for less than £1,000, and although this would obviously have very limited power capabilities and almost no instrumentation, it was considered that it might form a feasible starting point.

The second alternative of using a suitably modified universal testing machine was even less costly in terms of capital since such a machine was available and the only additional costs would be for the design and manufacture of suitable die, and cylinder and piston assemblies. A more sophisticated route to the same point would be the purchase of the commercially available polymer rheometer attachment which although more expensive, would have the advantage of being fully instrumented from the outset.

6.1.2.4 Commercial scale-up potential

This is probably the area which carries the least influence on the overall decision, since until the results of the basic research are available, it will not be known whether an opportunity exists for the expansion of the work into a larger scale production facility.

This step need not be as fraught with problems as many such scaling up operations, however, since the size and shape of the raw material particles are established and would be no different whatever the scale of the operation. The experimental equipment will therefore need to be large enough to cope with the largest particle size anticipated. Consequently the size factors involved in scaling up will be of a low order, perhaps as low as 1.5 or 2, and this should present few problems whichever processing route is chosen for the experimental stages of this work.

6.1.3 Interim conclusions

It is clear from all of the above sections that any choice of operating mode made at this stage can only be speculative since, as has been stressed many times above, the interactions between the raw material and the machine cannot be described even loosely due to the lack of definitive data available on the subject.

On this basis alone it makes good sense to follow up the least costly (and also in this case most readily available) alternative which will yeild at least some quantitative results. Subsequent decisions on the direction of the work can then be taken on the basis of the data obtained.

For the reasons already outlined then, it was decided to carry out some initial trials using the universal testing machine fitted with suitable accessories. A description of the apparatus together with the experimental procedures and the results obtained make up the remainder of this chapter.

6.2 The design and construction of the equipment used for initial trials.

The equipment required for initial experimentation includes not only the "rheometer" itself but also the equipment used for the preparation and characterisation of the wood chip feed material, the "furnish".

Since the preparation and characterisation of both the wood chips and the adhesive binder were described in Chapter 5, the description of the furnish preparation included below deals only with the equipment used for the blending together of the two components to form the final pre-extrusion mixture.

6.2.1 The preparation of the furnish

The equipment used for the blending of the wood chips and the binder for the initial trials is shown in Figures 6.1 and 6.2. It consists of a mixing vessel (a 10 l polyethylene pail) which could be rotated at speeds between 0 and 45 rpm by means of an Azri Heidolph Model RZR 1 variable speed stirrer unit fitted with a 6 mm chuck. Up to 250 g of chips could be accommodated in the vessel at one time. The binder was sprayed onto the agitated chips using the combination of the Watson Marlow Model MHRE 88 peristaltic pump and the Spray Systems Model E2.5 external mix spray nozzle visible in Figure 6.2.

FIGURE 6.1 The mixing equipment used for initial trials.

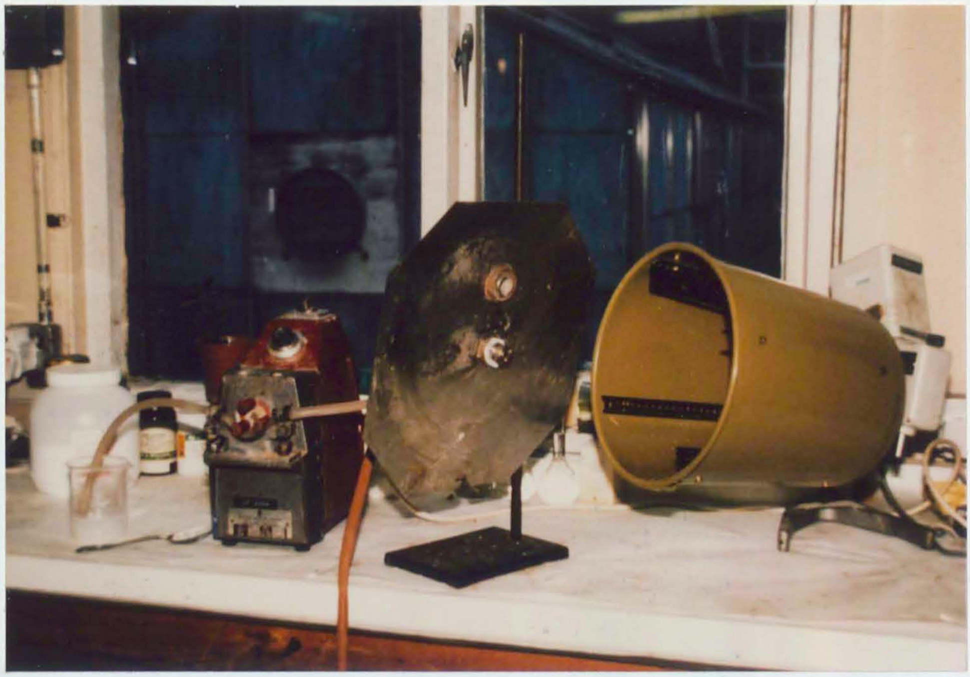


FIGURE 6.2 Detail of the equipment shown in Figure 6.1.

The air for atomising the binder in the spray nozzle was taken either from the 0.345 MPa laboratory supply or was supplied from the Aerostyle Model 238 electrically driven piston compressor seen in Figure 6.1. The maximum working pressure available with the latter system was 1.034 MPa.

When the experimental work did not require large quantities of prepared furnish, the use of a Humbrol disposable spray unit was preferred to either of the previously described spray systems both for convenience and conservation of raw material supplies.

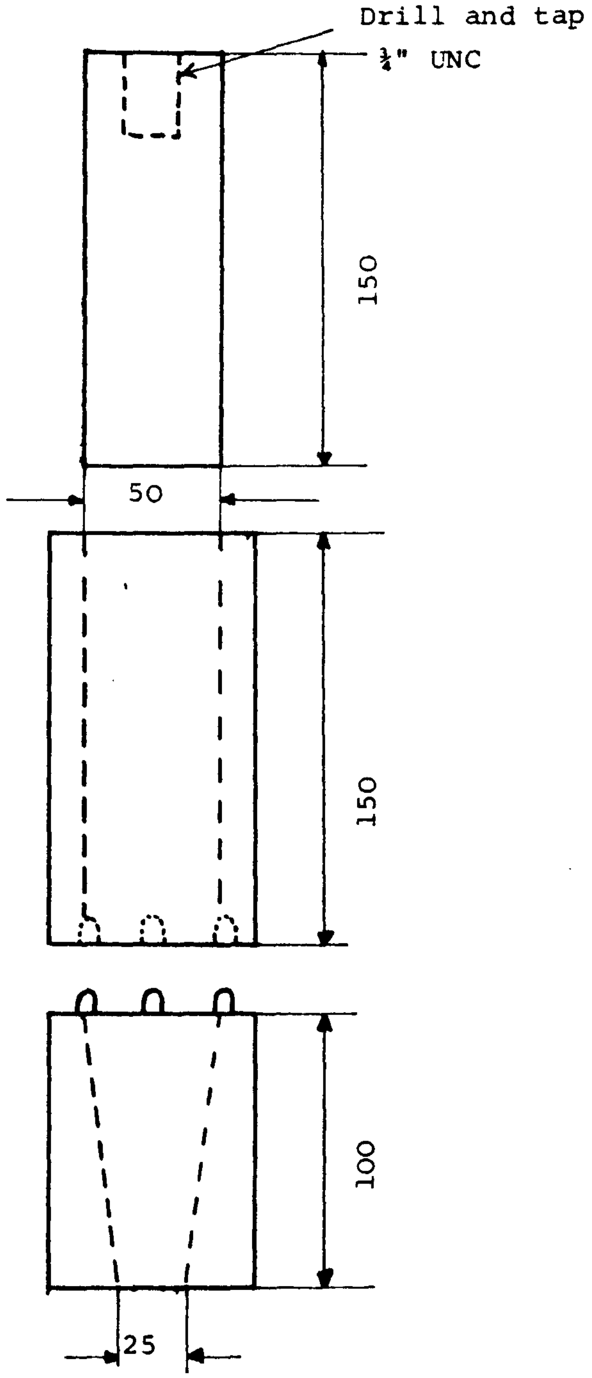
6.2.2 The rheometer

The basis of the equipment used for the initial extrusion trials was an Instron Model 1195 Universal Testing Machine fitted with an Instron Type 1193 tension/compression load cell. The machine is mechanically driven via lead screws over a pre-set cross head speed range of 0.1 to 500 mm per minute and has a maximum load capability of 100 KN. Data recording is facilitated by the built-in chart recorder, visible in Figure 6.3, which shows the complete experimental arrangement.

FIGURE 6.3 The Instron Universal Testing Machine used for initial extrusion trials.

The piston, cylinder and die assembly were manufactured from BS 970 Part 1, (1972), grade 212M36 mild steel according to the drawing shown in Figure 6.4. Standard Instron adapters were used where necessary to facilitate the fixing of the assembly to the testing machine. Furnish was fed into the cylinder by hand and loosely compacted using the fingers.

With this system it was possible to carry out limited tests over a range of piston speeds and with a variety of feedstocks. Since these initial trials were intended to establish baseline values with respect to machine/material interactions, however, no attempt was made at this stage to anticipate the range of die angles and diameters which would be necessary as the work increased in scope. The parameters measured during these initial trials were therefore ram displacement and ram force over a range of cross-head (piston) speeds. The absence of any heating facilities on this basic rig also meant that the samples were not cured during the experiments, and permanent examples of product were obtained by removing the cylinder/die assembly, complete with contents, to an oven at 140°C and allowing the binder to cure.



All dimensions in mm

External diameter
= 75 mm

FIGURE 6.4. Sketch of initial extrusion die tools, first used with the Instron Testing Machine.

6.2.3 Results of the initial experiments

The initial experiments were carried out using a die angle of 45° and an extrusion ratio of 9:1, and with this arrangement it was impossible to cause any extrusion of the product whatsoever at any crosshead speed. The 100 kN maximum load capability of the Instron was reached after 4 or 5 ram passes, and it was clear that this value was not near to any threshold value by the total absence of any extrudate appearing from the die.

6.2.4 Initial conclusions

The absence of any extrusion of product from the system demonstrated that the equipment as it stood was unsuitable for any commercial scaling up into manufacturing plant. The lack of extrusion also precluded the possibility of obtaining any experimental data with respect to the flow of the wood/resin mixture under ram extrusion conditions.

Clearly a rethink of the experimental strategy was required, and the direction this took is detailed in the following two Chapters.

REFERENCES - CHAPTER SIX

1. Westover R F; Mod Plast, March (1963).
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3. Whorlow R; "Rheological Techniques", Halstead Press, Chichester (1980).

CHAPTER SEVEN. THE ITERATIVE BUILDING PROGRAMME

From the results obtained using the equipment detailed in Chapter 6, it was clear that substantial improvements in the design of the "rheometer" would be required before more meaningful results could be obtained. It was decided quickly that modification of the Instron machine adapted for the initial work would be impractical, and that the design and construction of a purpose built piece of equipment was the logical way forward.

This Chapter details the design and construction of the first purpose built instrument, the ancillary equiment which supported it, results obtained from it, and further modifications and improvements made iteratively to the equipment as the research progressed.

7.1 Design Considerations

The most obvious conclusions to be drawn from the initial experiments were that the Instron based equipment had insufficient power for the purpose and a speed range with a low maximum limit. The new equipment should therefore have a maximum force capability well in excess of 100 kN and a ram travel speed greater than 5.0 mms^{-1}. The original hopper and die assembly would be used in the first instance, and consideration would be given to monitoring equipment when the mechanical design had been finalised.

7.1.1 The drive system

There are three possible alternatives for the ram drive mechanism:

a) screw
b) pneumatic
and
c) hydraulic

a) Screw drive. This would involve either the use of electircally driven leadscrews moving a crosshead, as in the Instron machine, or the use of a driven central pinion moving a central, threaded, ram axially. The complexity and precision of the engineering required in both of these cases legislated strongly against this mechanical approach to a new instrument design on the basis of cost. Power transmission of the level envisaged through such mechanical systems would also demand effective lubrication systems which would be difficult to incorporate within economic limits. Such a mechanically driven system would enable long ram travel distances to be designed into the instrument relatively easily, however, and would give very accurate control over axial ram displacement.

b) Pneumatic drive. Such a system would require very high pressures in order to generate the ram forces thought to be necessary, and these would cause significant problems in terms of safety and control. In addition the equipment necessary to generate such high gas pressures is very costly and somewhat difficult to procure. Such a system would have the advantage of being capable of developing very high ram speeds, and the design of the piston/cylinder/die system would be relatively simple.

c) Hydraulic drive. Again this system would involve relatively simple front end design work with the only moving part being the hydraulic ram piston itself. A framework similar to that of the Instron could be used, but without the complications of bearings and lubrication systems. The system would again be required to operate at relatively high pressure, but the intrinsic safety of using a non-compressible power transmission medium means that there would be much less of a safety hazard involved. Ram speed and stroke would be limited in this case, but the case of a sealed, gas-filled accumulator in the system would give extra speed capability. Control of the ram in such a system would be midway between that of the purely mechanical system and that of the pneumatic system. Pressure generation would also be less of a problem than in a pneumatic system, again because of the incompressible nature of the power transmission medium, and the concomitant simplicity of suitable pumps.

The advantages of the hydraulic option, in simplicity on the one hand and in safe operation on the other, were felt to outweigh the minor disadvantages of limited ram travel and perhaps limited ram speed. The decision was taken therefore to pursue this route, and design an initial instrument on the basis of hydraulic motive power.

After discussions with suppliers and manufacturers of hydraulic equipment, a complete system comprising hydraulic reservoir, pump, accumulator, control gear and bi-directional hydraulic cylinder was purchased.

7.1.2 The instrument framework

Initially it was intended to design a system in which the extrusion direction was horizontal, as this would allow for relatively unlimited extrusion lengths without resorting to mechanical bending of the product or excessively tall equipment. On balance, however, it was decided that factors such as uniform hopper filling and avoidance of particle segregation were of greater importance in terms of the research project, and that preliminary experiments should therefore be carried out with the extrusion direction vertical.

Although it would have been possible to install the hydraulic ram assembly on an existing Instron frame, it was decided that a much simpler but very sturdy purpose built frame would offer advantages in terms of adaptability and robustness. After some consideration a frame was constructed according to the rough sketch shown in Figure 7.1. The vertical sides of the frame (A1/2) were 05 by 75 mm C section steel girders 1524 mm long, the cross members (81-4) were 100 by 75 by 500 mm long C section steel girders, and the plates (C1/2) one to support the hopper and die assembly and one to support the hydraulic cylinder, were 200 x 290 by 12.5 mm thick steel plates. The assembled equipment, seen in Figure 7.2, was held together using 19.05 mm, (¾"), tensile steel construction nuts and bolts.

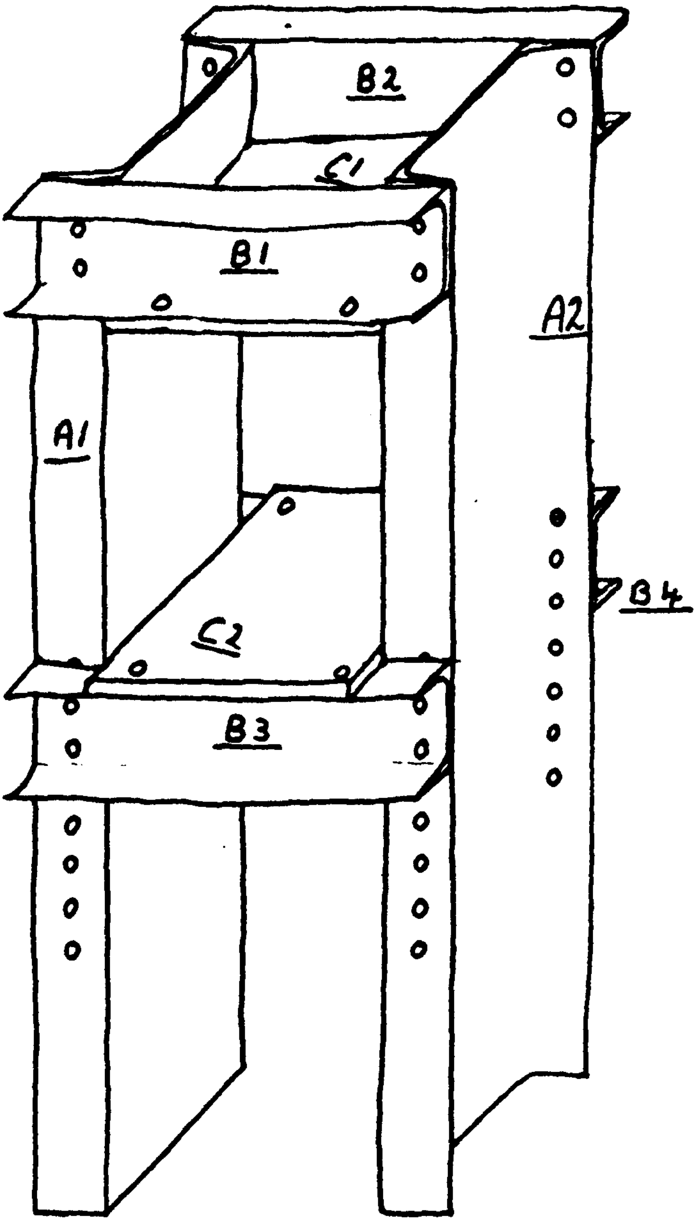

FIGURE 7.1 THE BASIC INSTRUMENT FRAMEWORK

FIGURE 7.2 The purpose built extrusion rig with associated instrumentation and control gear.

7.2 The first generation purpose built equipment

7.2.1 Equipment specifications

The hydraulic equipment purchased had been designed to function as part of a high speed filter-screen changer for injection moulding equipment, and was therefore fully specified by the manufacturer. The details are as folows:-

a) Hydraulic cylinder. Manufactured by Miller Inc the unit had a nominal 127 mm (5") diameter reversible piston with a maximum operating pressure of 24.1316 MPa, (3500 psi). The piston was rigidly coupled to a 47.625 mm diameter threaded connecting rod and had a maximum stroke of 254 mm.

b) Hydraulic power pack. Again assembled by Miller Inc this unit consisted of 30.28 litre oil reservoir in which a submerged gear pump was located. The pump was directly driven by a 1.5 kW fixed speed 3 phase electric induction motor running at 2833 rpm giving an oil delivery of 7.6 litres per minute at a maximum pressure of 16.547 MPa. The oil was delivered past an Imperially calibrated Bourdon type pressure gauge, (manufactured by Ashcroft with a maximum pressure rating of 20.684 MPa, (3000 psig) and capable of being read to an accuracy of ± 0.172 MPa (25 psi), to a 9.5 litre compressed gas bag type hydraulic accumulator. This was changed to a pressure of 13.8 MNm^{-2} with nitrogen gas. From the accumulator the oil was fed to a solenoid actuated hydraulic reversing valve manufactured by Parker Hannefin (type 11101B4NYF) and thence to the hydraulic ram via flexible hoses. The non-pressurised, return port of the valve dumped directly back into the oil tank. A "Colorflow" needle valve was incorporated in the pressure line, between the pump and the pressure gauge. This dumped directly back into the oil reservoir and therefore gave a degree of crude control over the rate of delivery and maximum oil pressure to the ram. A pressure relief valve, set at 16.547 MPa was incorporated into the oil pump.

c) Electrical control system. This was manufactured by Allen Bradley and was their standard system number 13-8886339-M. This basically consisted of relay operated contactor units which facilitated control of the on-off state of the pump motor and the direction of oil flow through the reversing valve. Both were triggered by the use of key operated switches. Also incorporated were thermal protection circuits for the pump motor, a pressure operated failsafe micro-switch and a mechanical time switch which allowed a maximum of ten minutes continuous operation of the pump motor.

The operating characteristics of the overall instrument can be calculated simply from the data given above.

At the maximum oil delivery rate of 7.6 litres per minute, the maximum achievable ram speeds are approximately 10 mm per second in the downward direction and 12 mm per second in the upward, return direction.

The maximum achievable loads in the two directions vary in the inverse ratio of the speeds, hence the maximum load on the ram during the downward stroke is 0.262 MN while that on the upward stroke is 0.220 MN.

For the initial experiments no additional monitoring equipment was added, the ram being timed over a number of strokes at a fixed valve setting to determine the ram speed, and the maximum pressure on the dial being noted from which the load could be calculated. There is a clear source of error in both values if the accumulator charging pressure of 13.8 MPa is reached, however for carrying out preliminary experiments to determine baseline conditions the arrangement was considered to be adequate.

The rate of ram travel made possible by fully charging the accumulator before releasing the pressure into the hydraulic cylinder was so high that it was difficult to measure accurately. As a first approximation the method of timing used for the slower speeds was adopted, but the number of observations was increased from 5 to 20 in an attempt to minimise any error. The figure arrived at by this method was 1.8 to 2 meters per second.

The round piston, the cylindrical hopper, and the conical die used for the experiments on the Instron machine were used for the first series of tests on the hydraulic machine. An equivalent assembly of square section was also fabricated, according to the dimensions shown in Figure 7.3, to invesigate whether extruder shape influenced the process in any way. The extruder ram in this case screwed directly onto the end of the hydraulic piston without the use of an adaptor. This equipment is shown in Figure 7.4.

7.2.2 Experimental method

For the initial extrusion experiments, the wood chips were prepared and characterised as described in Chapter 5, and the furnish was prepared using the mixing vessel and compressor driven spray system described in Chapter 6. The characteristics of the separate components and the furnish are shown in Table 7.1.

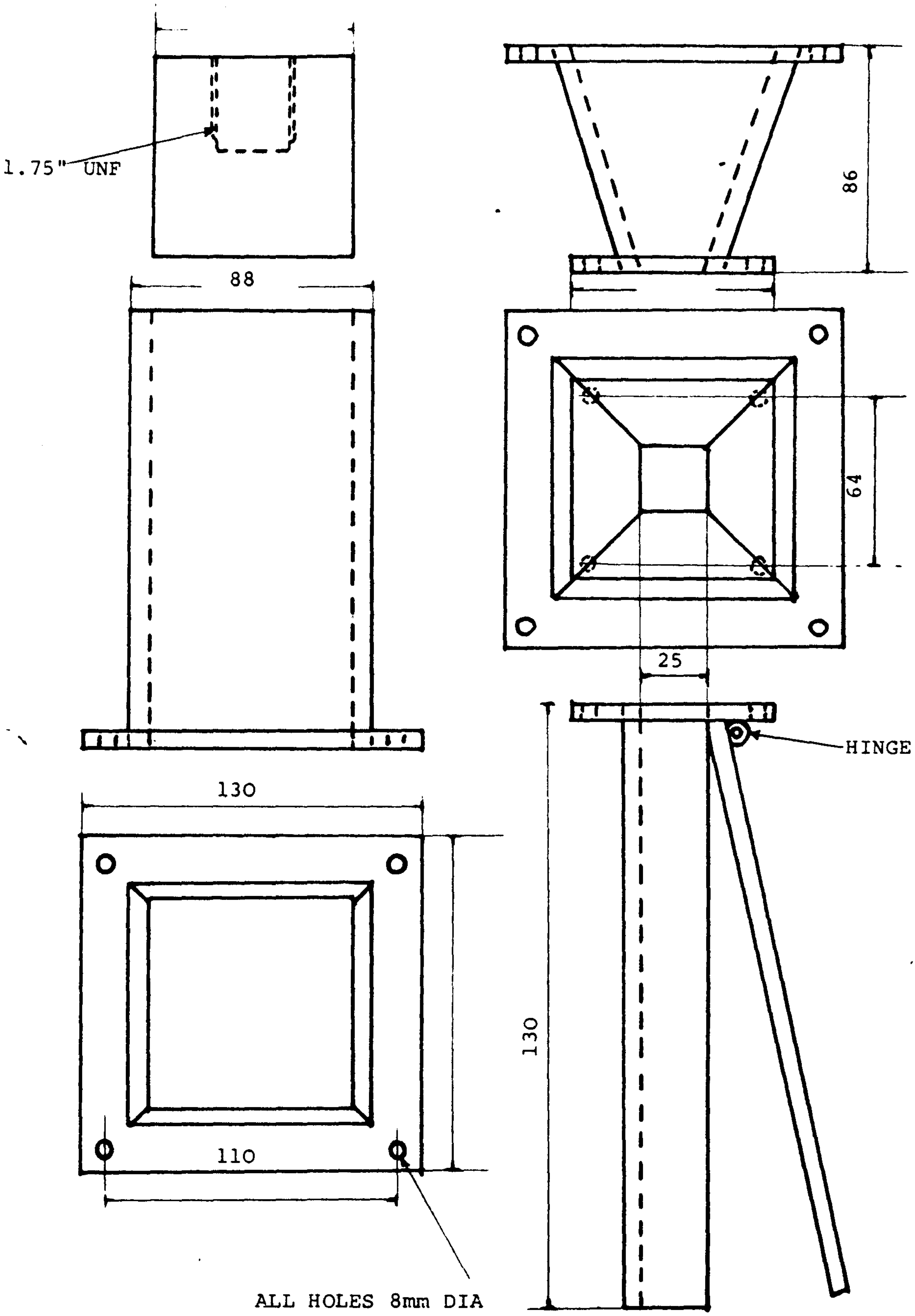

FIGURE 7.3 SQUARE EXTRUSION TOOL ASSEMBLY

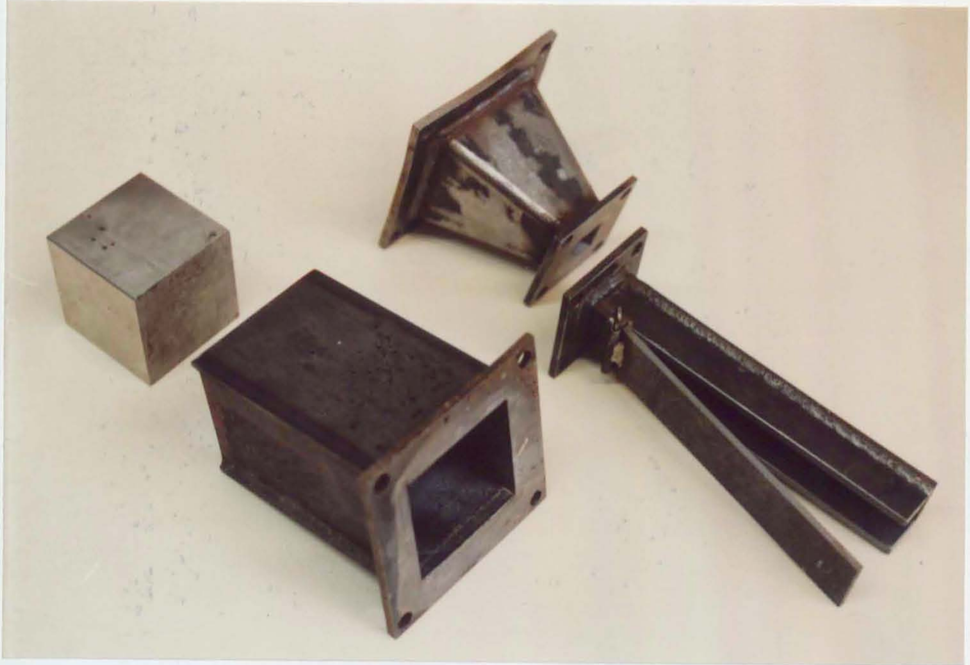

FIGURE 7.4 Exploded view of square extrusion tool assembly.

Weight of woodchip	250.0g
Fraction of woodchip	$P_{10}R_{30}$
Moisture content of woodchip	7.9% by weight
Weight of Aerolite	230g (9.989% by weight of dry wood)
Weight of ammonium chloride	0.23g
Weight of water	23.0g
Final moisture content of furnish	18.5% by weight

TABLE 7.1. CHARACTERISTICS OF MATERIAL USED FOR INITIAL EXTRUSION TRIALS.

The plate supporting the hopper assembly was adjusted such that at the full extent of its downward travel the leading face of the ram stopped 5mm short of the joint between the hopper and the tapered die section, thus preventing the ram from jamming but providing adequate compression of the furnish. The total length of ram travel within the hopper was 130 mm giving a swept volume of 7150 mm^3, and a compression ratio in the hopper of 26 to 1.

The maximum pressure during the ram travel was read directly off the pressure gauge on the power pack and thus gave the total system pressure in pounds per square inch gauge. It was expected that a small but finite hydraulic pressure would be required to overcome the frictional effects of the hydraulic ram seals and perhaps the friction between the extrusion ram and the hopper walls. It was intended that this value should be subtracted from the overall pressure reading obtained during extrusion to give a true value for extrusion pressure, however if such an effect existed, its magnitude was below the minimum pressure readable on the gauge, and hence it could not be measured. No real time measurement of ram speed could be made at this stage, however by timing the travel between the datum points at the hopper entrance and the maximum ram travel, a rough check could be made on the average ram speed for a single pass.

After the ram travel had been adjusted for the initial run, there was clearly no need for further adjustments unless the ram and hopper assemblies were physically changed for any reason.

Once the initial setting up and calibrating had been carried out, a piece of tissue was placed between the hopper and the die to prevent the furnish falling straight through, and the hopper was filled to the top with furnish by hand. Despite the relatively high compression ratio, maximum ram pressure was never developed on the first pass, and subsequent refilling of the hopper was required. Although the tissue barrier would break during the first pass, there was clearly sufficient compression to form a furnish "plug" after the first pass, and subsequent furnish additions were made by dropping fresh furnish onto this plug.

7.2.3 Experimental results

The results from the first attempted extrusion run are shown in Table 7.2, and plotted graphically in Figure 7.5.

No heat was applied to the furnish and no discernible temperature rise was detected during the experiment, hence the binder system remained uncured throughout the experiment.

PASS NO.	WEIGHT OF FURNISH (g)	RAM SPEED (mm s^{-1})	MAXIMUM PRESSURE (MPa)
1	90	5	Immeasureable
2	70	5	0.344
3	70	5	1.896
4	70	5	3.447
5	70	5	6.205
6	70	5	12.411
7	50	5*	16.547
8	40	5*	16.547
9	35	5*	16.547
10	25	5*	16.547

TABLE 7.2. RESULTS OF EXTRUSION (1). WITH 55 mm RAM AND HOPPER, 45° DIE AND 9:1 EXTRUSION RATIO.

* DENOTES ACCUMULATOR CHARGED AND PRESSURE RELIEF VALVE OPEN.

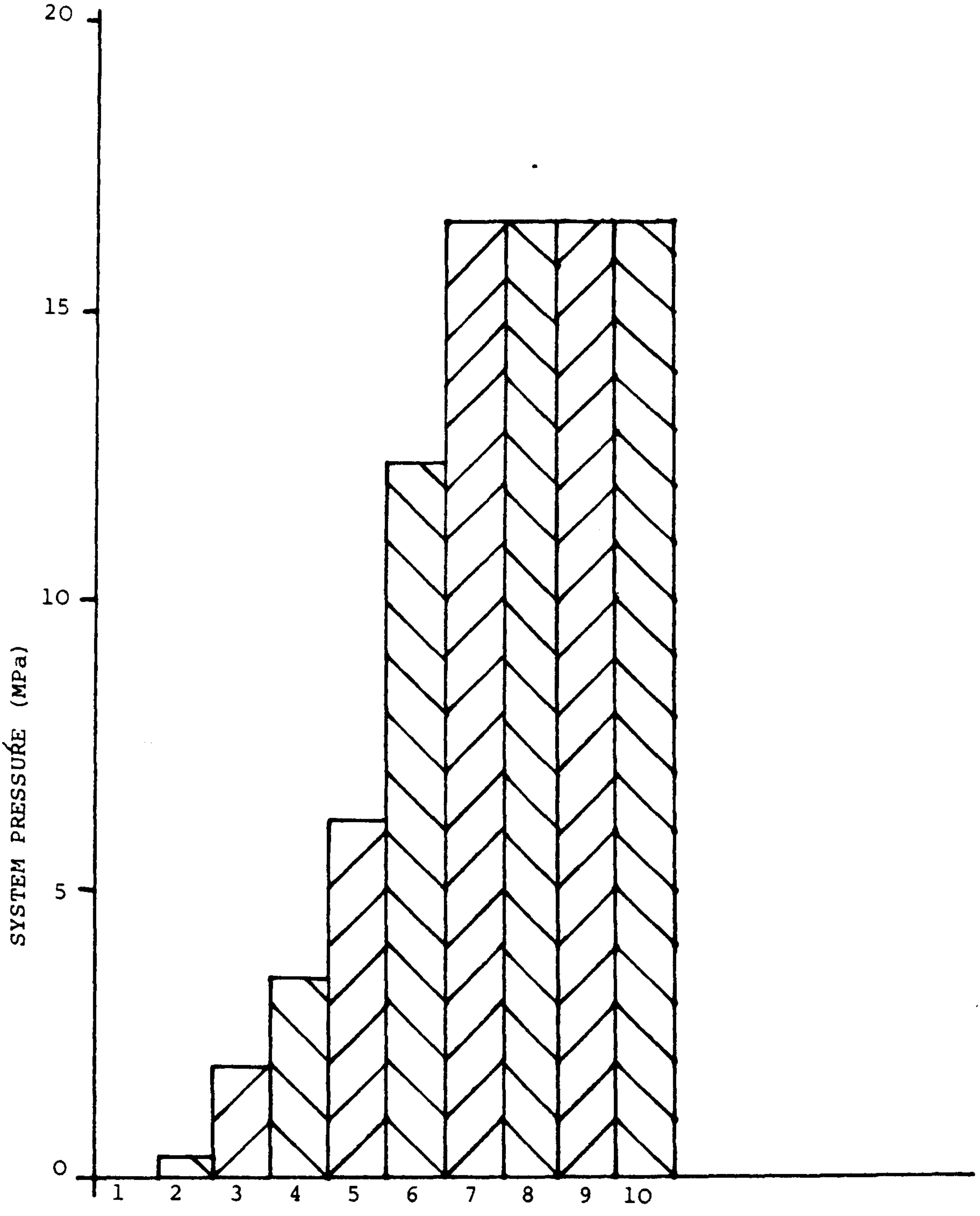

FIGURE 7.5 RAM PRESSURE vs. DISPLACEMENT. ABSCISSA FIGURE FIGURE IS NUMBER OF RUN IN SEQUENCE.

No product emerged from the die orifice and no extrusion took place, the plateau of pressure on the graph being due to the pressure relief valve in the hydraulic pump opening at the rated 16.547 MPa.

7.2.4 Conclusions

Although the experiment was inconclusive and gave no indication of any extrusion mechanism, the results are nonetheless valuable in two ways.

Firstly, the rapid attainment of maximum pressure suggests that perhaps a higher pressure system should be considered and certainly that the present system is likely to be operating near its upper limit. The accumulator is therefore likely to be the cause of more disadvantages in terms of monitoring than advantages in terms of system operation.

Secondly, the asymptotic tendency of the pressure readings suggests that with the combination of ram/hopper/die/furnish in use at present, very much higher pressures are likely to be required than can sensibly be achieved. Some attention should therefore be paid in the first instance to the possibility of altering some or all of the experimental parameters before any major system changes are carried out.

7.3 Mark II machine programme

On the basis of the experience gained in constructing and using the original equipment, and using the results of the initial experiments as a guide, modifications were made to the first instrument before any further extrusion experiments were carried out. These modifications and the results obtained using the second generation instrument are detailed in the following sections.

7.3.1 Instrument modifications

The lack of conclusive results from the first experiments indicated that improvements needed to be made in both the monitoring and the control of the instrument.

Improvements to the monitoring system were planned as follows:

1) the mechanical pressure gauge would be supplemented by an electronic pressure transducer and suitable signal conditioning unit to give a voltage output directly proportional to the system pressure,
2) a positional transducer would be incorporated on the extrusion ram, and again suitable signal conditioning equipment would be used to derive a voltage signal directly proportional to the ram travel.

Both of the above signals could then be recorded using a conventional two pen potentiometric recorder and by choosing a suitable chart speed the positional signal could be used as a measure of ram speed.

The specification of the units chosen was as follows:

Pressure transducer - Maywood electronics type p102
    strain gauge pressure transducer
    Range   0 - 2000 psig (+ 50% overrange)
    Accuracy over range + 1%
    Excitation voltage   5 v

Positional transducer - RDP electronics type D5/6000C
    linear variable displacement transformer (LVDT)
    Range ± 152.4 mm
    Accuracy ± 0.12%
    Excitation voltage 5 v @ 5 kHz

Signal conditioning units:-
    One RDP electronics type E307
    Range   1.5 mV to 20 v switchable
    Accuracy ± 0.1%
    Display ± 1999
    Excitation voltage 5 v @ 5 kHz
    Output voltage ± 2v and ± 10v
    Digital readout accuracy + 1 digit

and One RDP type E 307.2

Range	14 - 28 mV
Accuracy	0.2% full scale
Display	± 19999
Excitation voltage	0.5 - 12 v DC
Output voltage	0 - 2 v
Digital readout accuracy	± 1 digit

The pressure transducer was incorporated into the hydraulic circuit on a T piece installed immediately below the mechanical pressure gauge, as can be seen in Figure 7.6.

The LVDT was mounted using swivel bearings and was located as shown in Figure 7.7. The casing containing the coils of the instrument was attached to the vertical frame of the machine and the moving core piece was attached to the ram location adaptor.

The two transducer conditioning units were initially mounted on top of the machine framework in order for them to be visible from the instrument operating position.

Calibration of the pressure sensor was carried out independantly of the manufacturer in order to assess the accuracy of the transducer/conditioning unit combination. This was achieved away from the extruder with the use of a "Schenck" deadweight hydraulic calibration unit, and showed that between

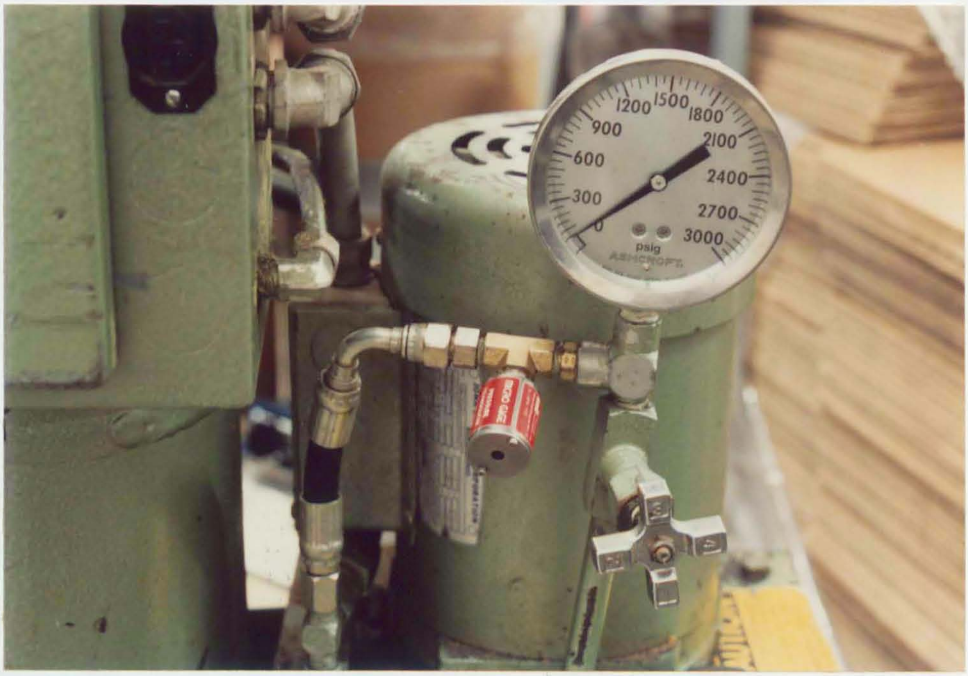

FIGURE 7.6 The strain gauge pressure transducer and its location.

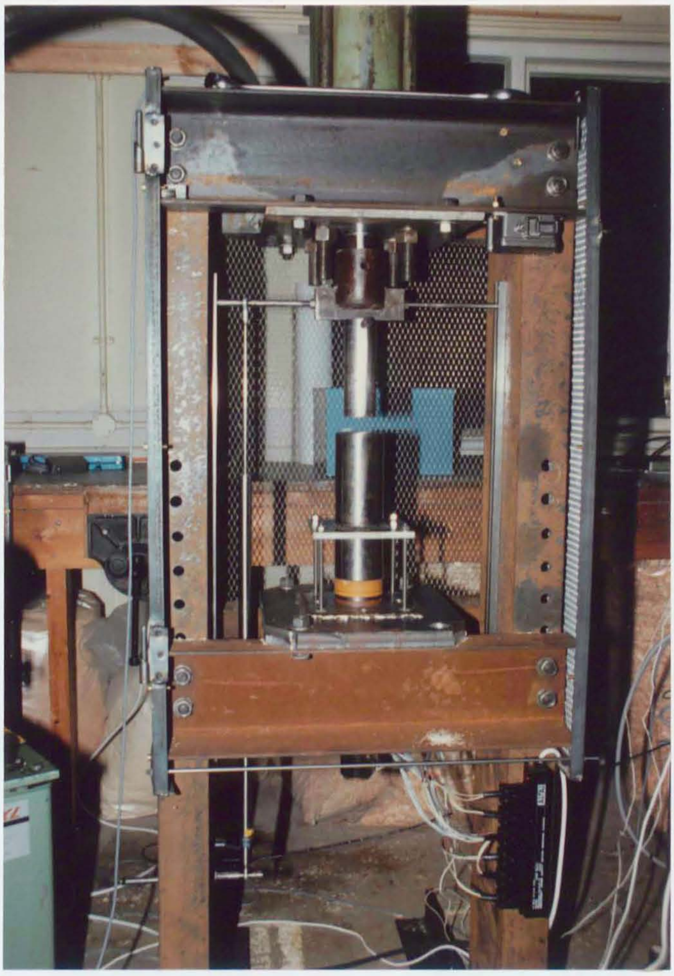

FIGURE 7.7 Location and attachments of the displacement transducer.

10 psi and 1000 psi (the upper limit of the calibration unit) the transducer/conditioning unit gave results reproducible to within 2 psi at each setpoint. The voltage output from the conditioning unit was also monitored during the calibration procedures with an AVO Model DA 116 digital multimeter (see Appendix for full specification), and was found to be consistent with the digital readout over the calibrated range. The signal conditioning unit incorporated a dummy load resistor which could be switched into circuit in place of the pressure transducer. This permitted electronic balancing and checking of the unit to be carried out, and after initial calibration of the unit this facility only was used for balancing and periodic accuracy checks.

Calibration of the LVDT was carried out on the extrusion rig itself with the use of feeler gauges. The ram was first moved down to a known position relative to the top of the hopper, achieved by sandwiching a piece of flat bright mild steel bar stock between the ram and the hopper top at a pressure of 0.103 MPa as read on the digital pressure indicator. Feeler gauges of a range of thicknesses were then incorporated between the ram and the steel bar at the same pressure, and the conditioning unit of the LVDT adjusted to give a linear scale in millimetres over the range tested. Once this operation had been carried out the LVDT/conditioning unit combination gave a repeatable accuracy of ±0.1mm at any point in its range, the zero point of which could be located at any point in the 300mm travel of the transducer. Again the output voltage of the conditioning unit was monitored using the AVO DA 116 during this calibration, and was shown to be totally consistent with the digital reading obtained from the conditioning unit.

The chart recorder used to take a permanent record during the experiments was a Ventura Servoscribe Model RE 520.20 two pen recorder. The stated accuracy of the unit on the fixed ranges used was 0.1% fsd, however the chart could only be read to an accuracy of 1 division, (by far the most inaccurate part of the total monitoring system), thus giving a maximum accuracy for pressure recording of ± 0.010MPa and for position of ± 2mm.

In addition to the refinements carried out on the instrument monitoring system, a further improvement to experimental accuracy and reproducibility was gained by taking the accumulator out of the hydraulic system. Since the instrument control panel was physically mounted on the accumulator it was clearly not possible to remove it altogether, and the break in the circuit was achieved by manufacturing and then hard brazing a suitable steel plug into the barrel union connecting the accumulator to the reversing valve. This then gave direct positive control over the ram movement up to the maximum system pressure of

16.547 MPa, although at the same time it removed the possibility of the use of ram speeds in excess of 10 mm per second.

7.3.2 Experimental method

Since none of the extrusion tools had been altered from those used in the first experiments, the same experimental technique was used for this set of experiments, including the composition and blending of the furnish. The details are included in Section 7.2.2. of this chapter.

7.3.3 Experimental results

Again no extrusion of product was observed during this set of experiments. This was a predictable result based on the experience of the previous set of experiments, since no changes had been made to either the maximum system pressure or the hopper/die assembly. The time taken for the pressure to reach its maximum value was predictably shorter, however, with the accumulator removed from the hydraulic circuit.

The performance of the electronic data recording equipment was verified during these experiments and the system produced results which were both higher in quality and more comprehensive than had previously been obtained.

7.3.4 Conclusions

Although the main objective of extruding a product has yet to be achieved, the equipment has shown itself to be capable of producing good quality data which should be adequate when optimum conditions for extrusion are found. Clearly more modifications are necessary to the basic design of the equipment before this state can be achieved, however.

7.4 Mark III machine programme

Since the modifications to the hydraulic system had failed to produce an instrument which could extrude the material in use, further changes to the programme were clearly needed if the research was to progress.

There were three options open in this respect:

1) to change the hydraulic system for a more powerful unit
2) to change the geometry and/or the dimensions of the extrusion tools
3) to change the formulation of the feedstock in use during the trials

Of the three options, number 1) would clearly be the most expensive and would take the longest time to accomplish. Number 2) could be carried out quickly and at minimal cost since the parts involved were relatively small. Number 3) had the severe disadvantage that the formulation in use had already been assessed as being likely to give optimum properties in the final product, and any change could potentially reduce the quality of this product. On the basis of the arguments above it was decided to opt for alternative number 2) in the first instance.

7.4.1 Instrument modifications

The details of the die set in use during the previous experiments were given in section 6.2.2. The calculated extrusion ratio of the arrangement was 9:1 (i.e. the ratio of the cross-sectional area of the ram to the cross-sectional area of the die exit was 9:1) and the die half angle was 45°. The first modification decided upon was to lower the die angle to 30°, which, according to the theories outlined in chapter 3, should lower the force required for extrusion. This was achieved by turning the 45° die down on a centre lathe to give the lower angle, without altering the extrusion ratio of the equipment in any way.

7.4.2 Experimental method

The techniques of furnish preparation and operational procedure outlined in section 7.2.2 were also used for the experiments carried out with the modified apparatus.

7.4.3 Experimental results

Apart from the predictable effects of a slight increase in the unswept volume of the die section (shot weight decreased more slowly and maximum pressure for the initial and second ram pass was slightly lower due to the decrease in volumetric compression of the furnish), there was no obvious difference between this and any of the previous sets of experimenst. Maximum system pressure was reached without any extrusion taking place, and there was no indication of any increasing likelihood of such an event occurring.

7.4.4 Conclusions

Clearly die angle changes of the order of the one carried out do not affect the ability of the furnish to extrude through the system. As there was no indication of any effect from reducing the die angle, by 15° (33%), the choice of direction for the next equipment alteration must lie between lowering the die angle even further, or changing some other fundamental operating parameter such as system pressure or extrusion ratio.

7.5 Mark IV machine programme

Of the three alternative directions suggested in the previous section for the next experimental modification, the first, to lower the die angle still further, requires only a minimum of machining to accomplish and is therefore the most economical. It can be shown using simple goemetry, however, that a proportional decrease in extrusion ratio will accompany any reduction of more than 15°. This would give rise to experimental inconsistency and make direct comparison between runs to investigate the effect of variation in die angle invalid. Cost therefore ceases to be of primary importance. A redesign of the whole extrusion "front-end", that is piston, cylinder, and die, could include modifications to allow both a wider range of die angles and a change of extrusion ratio and would still be less expensive than a change of hydraulic unit. This course was therefore chosen for the next set of experimental equipment modifications.

7.5.1 Instrument modifications

Since the whole of the front end of the system was being modified, the opportunity was taken to investigate a number of alternative designs in terms of mechanical assembly, availability of materials, and ease of interchangeability of parts. The design chosen finally is shown in Figure 7.8. The ram was

ALL DIMENSIONS IN MILLIMETRES

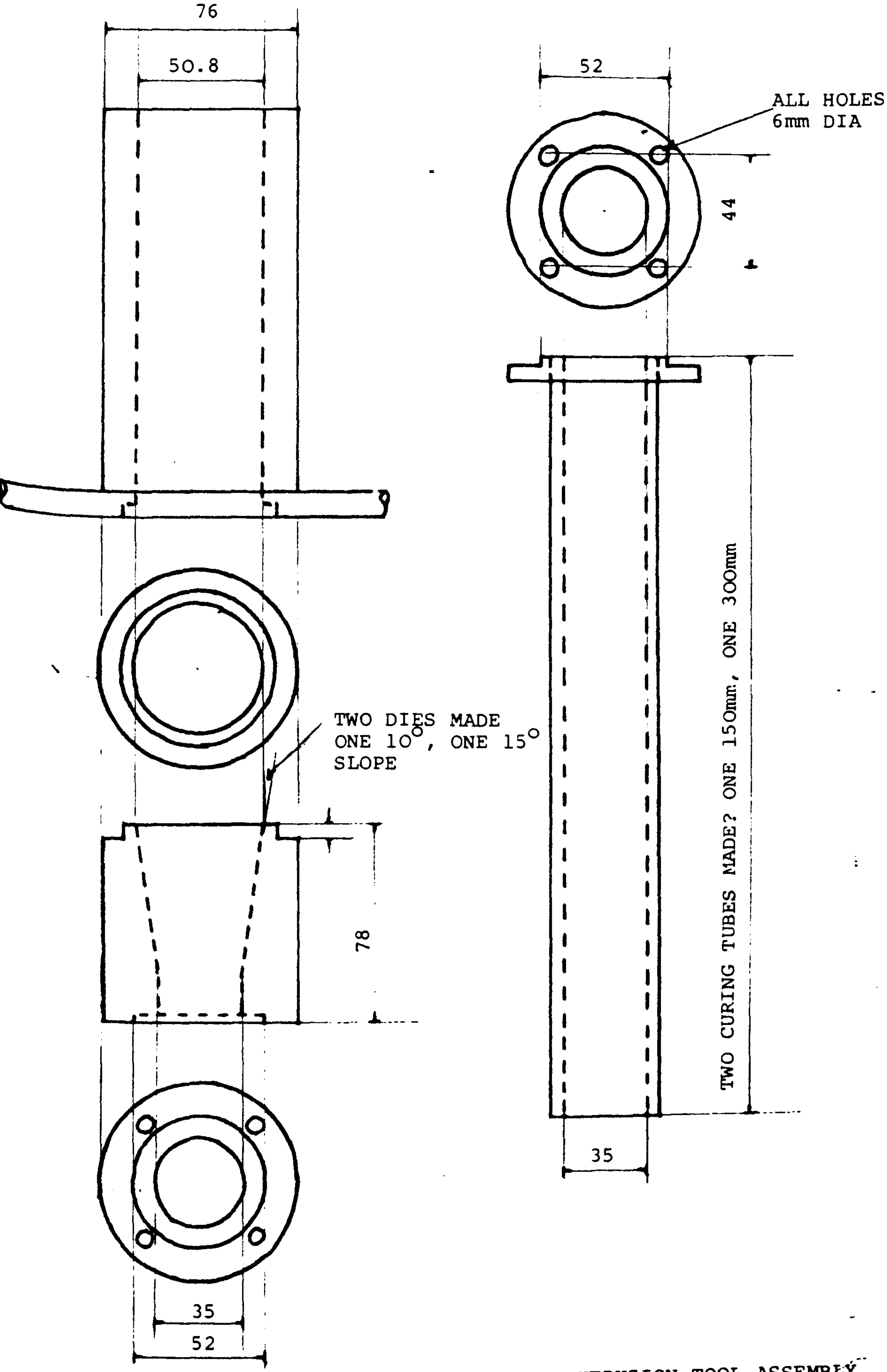

FIGURE 7.8 MODIFIED EXTRUSION TOOL ASSEMBLY

manufactured from bright drawn mild steel bar 50.8mm in diameter, and the hopper and die sections were turned from hot forged hollow bar to match the piston diameter exactly. The die exit diameter was increased to 35mm resulting in a decrease in extrusion ratio from the original 9:1 to a much lower 2.05:1.

The section labelled "curing tube" was added to allow investigation of in-line curing of the resin in order to manufacture a continuous product, if extrusion can be made to occur. This section was fabricated from a turned mild steel flange to which a section of thick walled mild steel tube was welded. The whole assembly was then bored out to the required 35mm diameter in a centre lathe to eliminate any welding distortion and/or eccentricity induced during the fabrication process. 240v band heaters, made to specification by Elmatic of Cardiff, were used to heat the tube. These were all connected to a common distribution board to which the power was supplied by an Ether "Mini" thermostatic temperature controller. The thermocouple probe from the controller was located in a groove filed into the outer surface of the curing tube and was held in place by a split full length sheath made from 1mm thick copper sheet, over which the heater bands were fitted. The sheath also helped to minimise the effect of the dead spaces between the heater bands. Because all of the heater bands were controlled from a single Eurotherm unit, heater bands of various powers were specified to enable crude temperature profiling along the curing tube to be achieved. A Comark Model 160C battery operated thermocouple meter fitted with a type I (iron-constantan), thermocouple was used to check the accuracy of the temperature control exercised by the Ether unit. The actual temperature and the set temperature were found to agree to within 2.5°C, which was the accuracy to which the scale on the Ether unit could be read.

7.5.2 Experimental method

Again the techniques of furnish preparation and equipment operation outlined in Section 7.2.2 were used when conducting experiments with this modified equipment.

7.5.3 Experimental results

The increase in the unswept volume of the die again affected both the rate of decrease of shot weight and the number of ram passes required before maximum pressure was reached.

Although the point in the material furthest away from the ram face was actually forced further down the die/tube assembly than on any previous occasion, maximum system pressure was still attained without any result approaching well-defined "extrusion" of the product. Removal of the curing tube and operation of the system with only the die in place did not make any noticeable difference other than that material which did protrude through the bottom of the die formed into a mushroom shape whose diameter was greater than that of the die exit. Still no extrusion occurred.

Heating the curing section proved the efficiency and accuracy of the heating system, and enabled in-situ curing of the material in the die to be carried out due to conduction of the heat back through the system. It had no noticeable effect on extrusion, however.

7.5.4 Conclusions

The equipment as it stands is clearly still unacceptable since no extrusion of a viable nature can be performed. The modifications performed in an effort to obtain extrusion have clearly failed, although the equipment as it now stands lends itself more easily to further modification than the previous design. The additional feature of the curing tube has been shown to function correctly, although its full potential has yet to be realised.

7.6 Mark V machine programme

The three options for machine modification which were outlined in section 7.4 are still valid for the next stage of the work. These are:

1) increase the power of the hydraulic power pack
2) make further changes to the geometry of the system
3) change the formulation of the feedstock material

Option 1) is still the most expensive route by a considerable margin. The changes in geometry made thus far have still not allowed extrusion to be carried out, but the new design of the hopper/die sections mean that further changes can be made in the same direction, i.e. the die angle can be lowered still further and if necessary the extrusion ratio can be changed again. Modification of the formula of the feedstock still remains an option although the previous objections to this course of action still exist.

Again option 1) was considered to be a "last resort" and therefore discarded at this stage. It was decided, however, that to increase the changes of successful extrusion after the modifications had been carried out, both of the remaining options would be used.

7.6.1 Instrument modifications

Because the combination of a reduction in both die angle and extrusion ratio was seen to have some positive effect on the amount of material actually passing through the system, and because the effects, if any, of a change in feedstock are unknown, it was decided to maintain the present die at an angle of 15°. An additional die of identical external design and with the same extrusion ratio of 2.05:1 but with a die angle of 10° was therefore manufactured. The curing tube assembly was maintained in its existing form.

Although the chart recorder used for data collection had been the obvious first choice when considering a monitoring system, it had become clear by this stage of the work that it was not the ideal system to use. Though simple, it was not ideal for data collection since accurate raw numerical data for subsequent manipulation was not available using this method. Increasing unreliability in the operation of the recorder also added weight to the argument for a new, and better system to be considered.

Factual information from the literature, noted in Chapter 3, regarding the influence of radial pressure, via wall friction, on the extrusion pressure required for any given system, indicated that the addition of strain gauges at some future

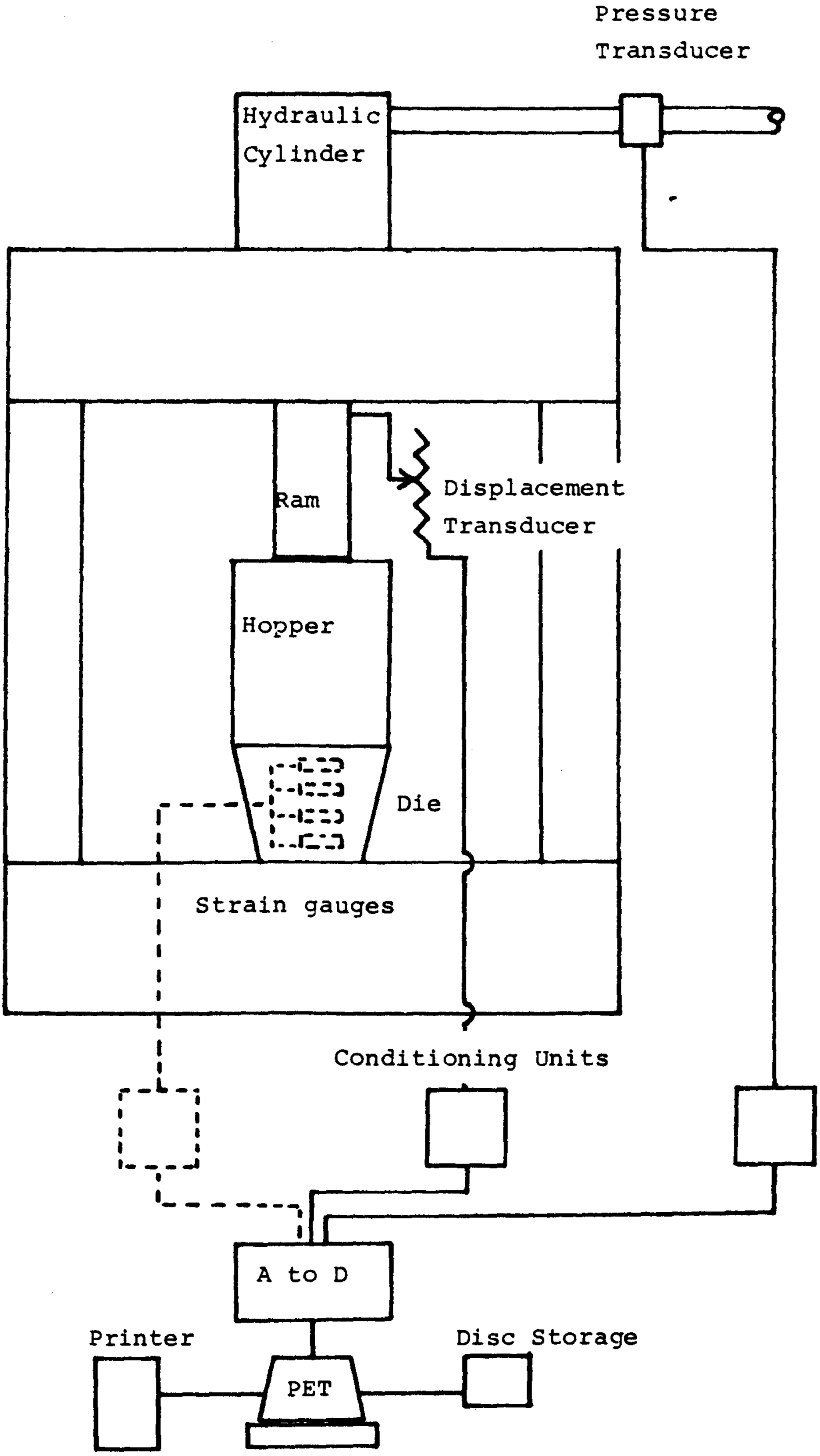

FIGURE 7.9. ARRANGEMENT OF COMPUTERISED MONITORING SYSTEM.

PAGES
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(239 + 240)

TALC

GRAPHITE

POLYTETRAFLUORETHYLENE

MOLYBDENUM DISULPHIDE

COMMINUTED CELLOPHANE

POLYETHYLENE GLYCOL

POLYOLEFIN WAXES

TABLE 7.3 Possible lubricants for inclusion in the feedstock mixture.

assume, therefore, that the value of yield strength calculated for the chip mass is very optimistic, since no permanent deformation of the steel components was detected. The second explanation offered above is therefore the more likely of the two, with the frictional components dissipating the pressure over the area of the wood/steel interface.

Modifications to the feedstock which in some way reduce the level of friction are thus more likely to have beneficial effects than any which merely reduce the yield strength of the material. Reduction of strength is also something which should be avoided if the final product is intended to have structural applications. A list of possible lubricant systems was therefore assembled as shown in Table 7.3 below.

Any lubricant added to the system must satisfy certain basic criteria in order to be acceptable;

1) it should confer a degree of surface slippiness to the mixture
2) it must be compatible with all other components in the system and cause the minimum possible disturbance to the material properties
3) it should be inexpensive and readily available

Of the possible choices listed in Table 7.3, polyethylene glycol and polyolefin waxes are well established as additives for the furnish in the particleboard industry and thus fulfill criterion 2). By their nature they are also likely to fulfill criterion 1), although their cost is comparable to that of the adhesive in the system and the on-cost involved in their use will therefore be very dependant on the addition level.

Because talc is relatively inert it is widely used as a filer in both thermo-setting and thermo-plastic systems and is likely to meet criterion 2). Its use as a lubricant, however, relies on the ease with which individual particles move across each other, i.e. a low internal friction, and this might be compromised by the presence of the adhesive. If this proves to present no problem, however, the cost of bulk quantities is relatively low, although this may be offset by the need for increased adhesive addition to compensate for the inevitable absorption losses.

Polytetrafluoroethylene (PTFE), is well known as a lubricant, particularly in the area of "non-stick" surface treatments. Because of its extreme slippiness and the inert nature of its surface, this material is likely to interfere with the bonding together of the system components, and its potential lies more in possible uses for surface treating the internal steel surfaces as a separate process than in additive applications. The cost of PTFE is also likely to be disadvantageous in this instance.

Graphite and molybdenum disulphide rely on their particle geometry for lubrication properties, and are generally used in a carrier medium for this reason. Their use in this application would therefore be dependant on the identification of a suitable carrier, and this would add to the already heavy experimental schedule. Both of these systems also have the possible disadvantage of being strongly coloured which might cause unacceptable effects in the final product.

Comminuted Cellophane has a basic chemical structure not dissimilar to wood and is therefore likely to be totally compatible with the other components in the system. Its non-porous nature, however, means that a potential two-fold increase in the quantity of binder may be required, since its thickness is very much smaller than that of the woodchips and a significant increase in surface area will result from the addition. Again any lubrication effect is likely to be purely mechanical and the system will need to be tried before an assessment of its efficacy can be made.

On the basis of the above arguments then, it was decided to test the lubrication effects of three additives, 1) comminuted Cellophane, 2) polyolefin wax and 3) polyethylene glycol.

7.6.3 Experimental methods and results

Of the three alternatives chosen, the comminuted Cellophane was the system about which least was known, and work was therefore begun on this system first.

7.6.3.1 Comminuted Cellophane

The raw material for this trial was obtained locally as a roll-end from a film packaging operation. As no references could be found to any similar work, it was decided to use the hammer mill described in Chapter 5 fitted with the smallest screen available. (3mm diameter circular holes), to comminute the Cellophane. If these particles appeared to be of a potentially useful size, they would be incorporated into the furnish, if not then an alternative preparation procedure would be sought. The full particle size analysis as carried out on the wood component was considered unnecessary for the cellophane for this exploratory work.

The particle as produced by this method were polygonal in shape and predominantly 3 to 4mm across the face. The consistency of the product vindicated the lack of particle size analysis, for at least the early stages of the work.

For the first experiment, the Cellophane particles produced were added to the mixture as described in Table 7.1 in an amount equal to half the quantity of Aerolite by weight. The wood chips and the cellophane were premixed dry before the adhesive solution was sprayed on, in order to ensure an even distribution of the Cellophane throughout the mixture. It was felt that adding the very light Cellophane after the adhesive had been applied to the chips would have caused an uneven distribution of the plastic over the woodchips exposed at the surface of the mix at the time of addition.

The results of the extrusion trial, labelled CELLO.01, were recorded as a value of ram displacement and a corresponding system pressure in a single sequential file on the Commodore floppy disc unit. These results were then transferred to a DEC PDP 11-44 mainframe computer in order to plot the graph shown in Figure 7.10. The test was carried out without the curing tube in place and therefore wholly at room temperature, and the 15° die used in previous experiments was fitted to the hopper assembly.

A second trial using identical feedstock was subsequently carried out in a similar manner but with the new, 10° die fitted to the hopper. The results from this trial were also recorded and displayed in the same manner as those for the first trial.

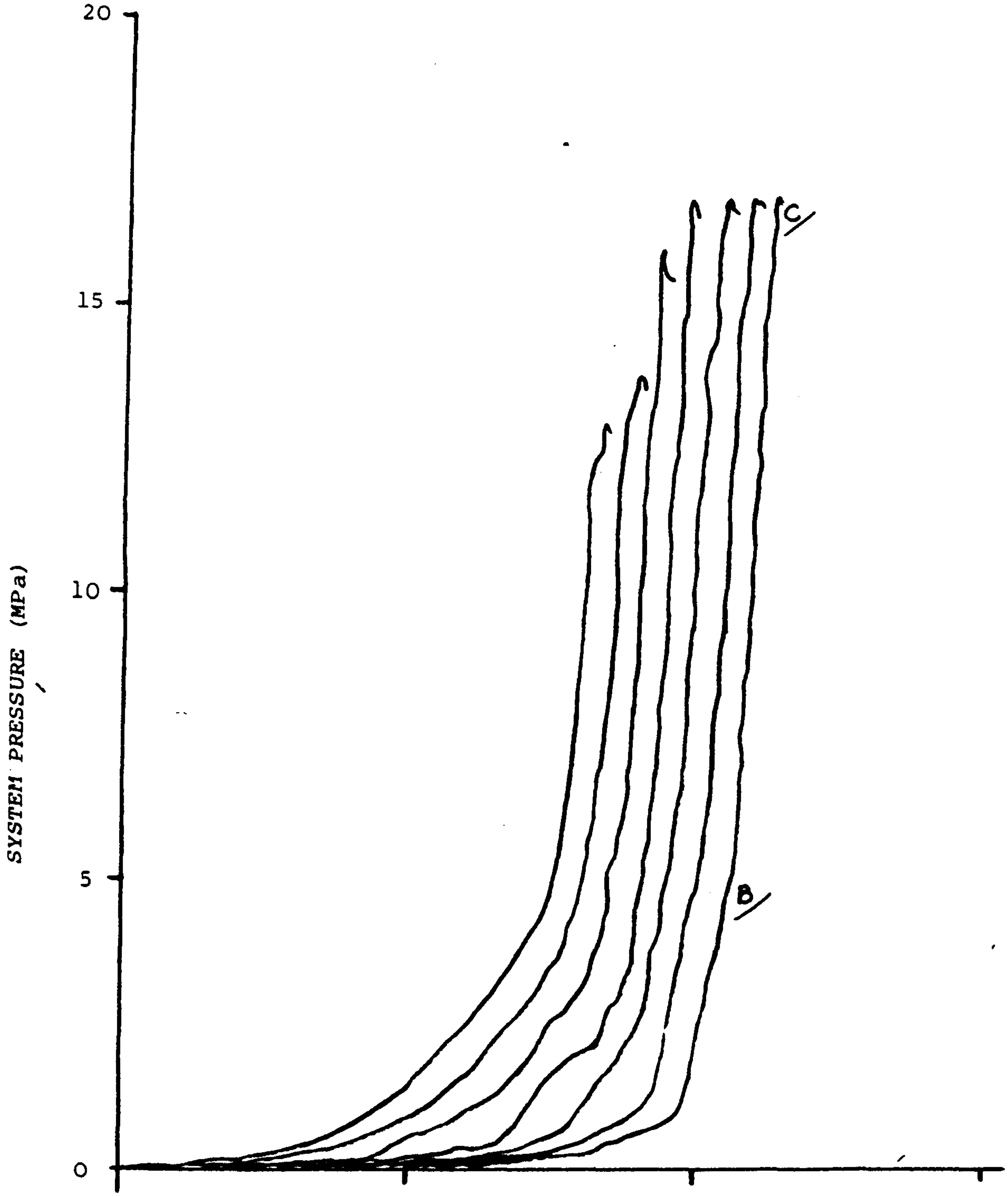

FIGURE 7.10. RESULTS FROM EXTRUSION TRIAL USING 15° DIE AND CELLOPHANE LUBRICANT.

7.6.3.1.1 Experimental results

The results from the two trials with the Cellophane addition are shown in Figures 7.10 and 7.11 for the 15° die and 10° die respectively.

At first sight these appear very similar to the results obtained in all previous experiments, no extrusion occurred and the maximum pressure recorded corresponds to the lifting pressure of the hydraulic safety valve. The number of ram passes required to reach this pressure in both cases is significantly lower than in any previous experiment, however, and the difference is greater than can be attributed to experimental error.

7.6.3.1.2 Discussion

The rapidity with which maximum pressure was reached could be explained by one of two mechanisms, or perhaps a combination of both.

1) The comminuted Cellophane has increased the yield strength of the compressed mass significantly.

2) The comminuted Cellophane has altered the surface characteristics of the furnish in such a way that the friction between the chip mass and the steel surface has risen dramatically.

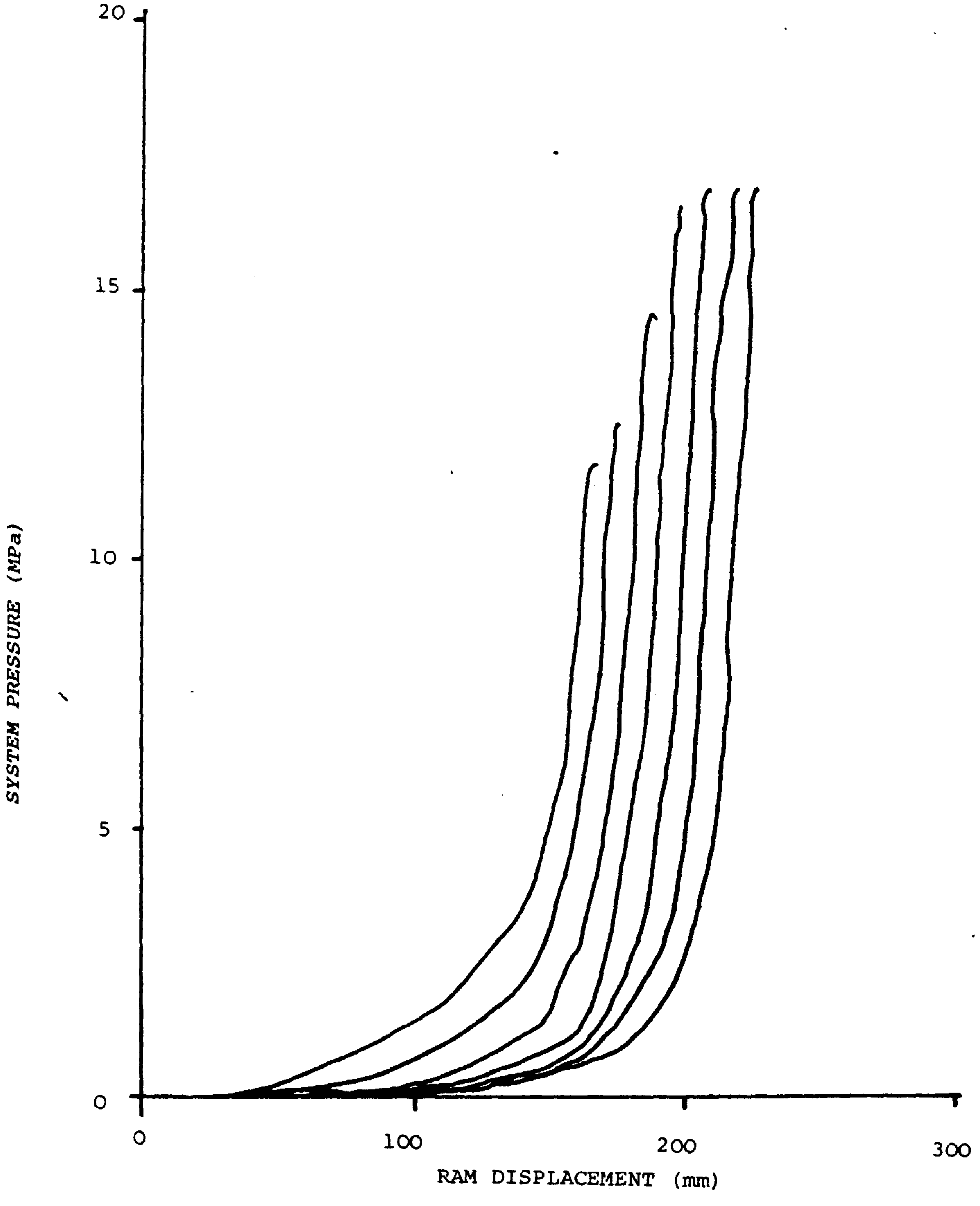

FIGURE 7.11. RESULTS FROM EXTRUSION TRIAL USING 10° DIE AND CELLOPHANE LUBRICANT.

Measurement of the density of the product gave value of 0.9 g cm^{-3} which is lower than has been achieved previously, (values between 1.15 and 1.2 g cm^{-3} had been measured for previous products). This indicates that the chip mass is not being compressed to the same degree as on previous occasions and suggests that the internal friction between particles is higher with the addition of the Cellophane.

This would be expected to cause a decrease in the radial forces transmitted to the steel/composite interface.

This in turn should result in a decrease in the axial friction contribution to the extrusion pressure, (τ_n in equation 3.4-8), and a concomitant decrease in the pressure required to cause extrusion. This combination of contradictions would suggest that the addition of cellophane to the chip mass actually increases all frictional properties of the feedstock, contrary to the stated aim of providing lubrication.

It is feasible that a significant decrease in the Cellophane particle size might produce a decrease in friction as the particles behave like ball or roller bearings between the wood particles. Such an addition would almost certainly require a significant increase in the adhesive content to compensate for the increase in surface area, and since similar results are conceivable with the use of wood dust or wood flour which is available as a by product from the particle production, it was decided to proceed no further with Cellophane as an addition.

A possible explanation as to the reason Cellophane caused these effects might be a catalytic action on the curing of the resin system under conditions of high pressure. No effect was observed at atmospheric pressure, as the excess feedstock remained tacky for many hours after the extrusion tests had been completed. No further attempts to qualify this theory were made, although if such an effect does exist it could have commercial applications within the particleboard industry.

7.6.3.2 Polyethylene glycol (PEG)

Polyethylene glycol is available in a number of chain lengths and hence molecular weights from commercial chemical suppliers (e.g. Hopkin and Williams, British Drug Houses), and many experiments on the use of the chemical as a wood preservative have been documented (see for example references 9, 10, and 12, Chapter 2). Stamm, in reference 10, Chapter 2, cites a molecular weight of 1000 to be the optimum in terms of preservation properties, and quotes levels of up to 30% by weight on dry wood being used for the face veneers of plywood. The incorporation of the PEG is carried out before the veneers are assembled, which suggests that even such a high level as 30% does not interfere significantly with the adhesive bonding processes. On the basis of this information it was decided that for the initial lubrication tests, the use of two systems, one having a 20% addition by weight on dry wood of PEG 4000 and a second having 10% of PEG 6000 would be standardised upon. Conveniently, PEG 4000 is a liquid at or slightly above room temperature, and could therefore readily be incorporated in the adhesive solution and subsequently sprayed onto the chips. PEG 6000 was obtained in crystalline form and was melted in a warm water bath before incorporation into the solution.

Again the furnish was as described in Table 7.1 but with the addition of the appropriate quantity of PEG. Both the 10° die and the 15° die were used, and the curing tube was not fitted to the hopper/die assembly. Results were recorded using the system described in section 7.6.3.1.

7.6.3.2.1 Experimental results

It can be seen from the results plotted in Figures 7.12 to 7.15 that in all of these experiments, successive hopper loads of furnish material could be caused to extrude through the system. The system pressure required to cause the extrusion varied with the experimental conditions, but in all cases was significantly lower than the relief valve pressure which has been the limiting pressure in all previous

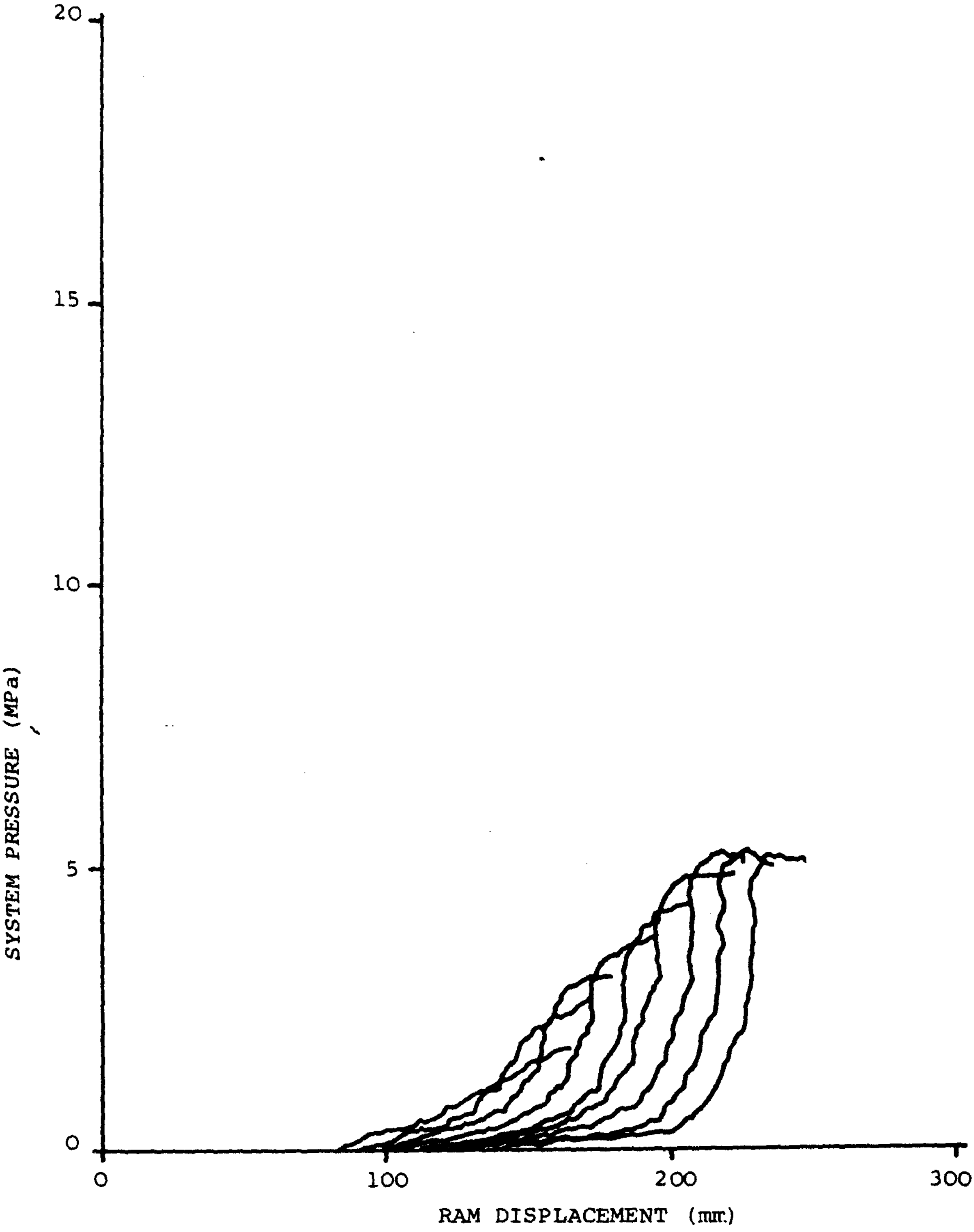

FIGURE 7.12. RESULTS FROM EXTRUSION TRIALS USING 15° DIE AND 20% PEG 4000 LUBRICANT

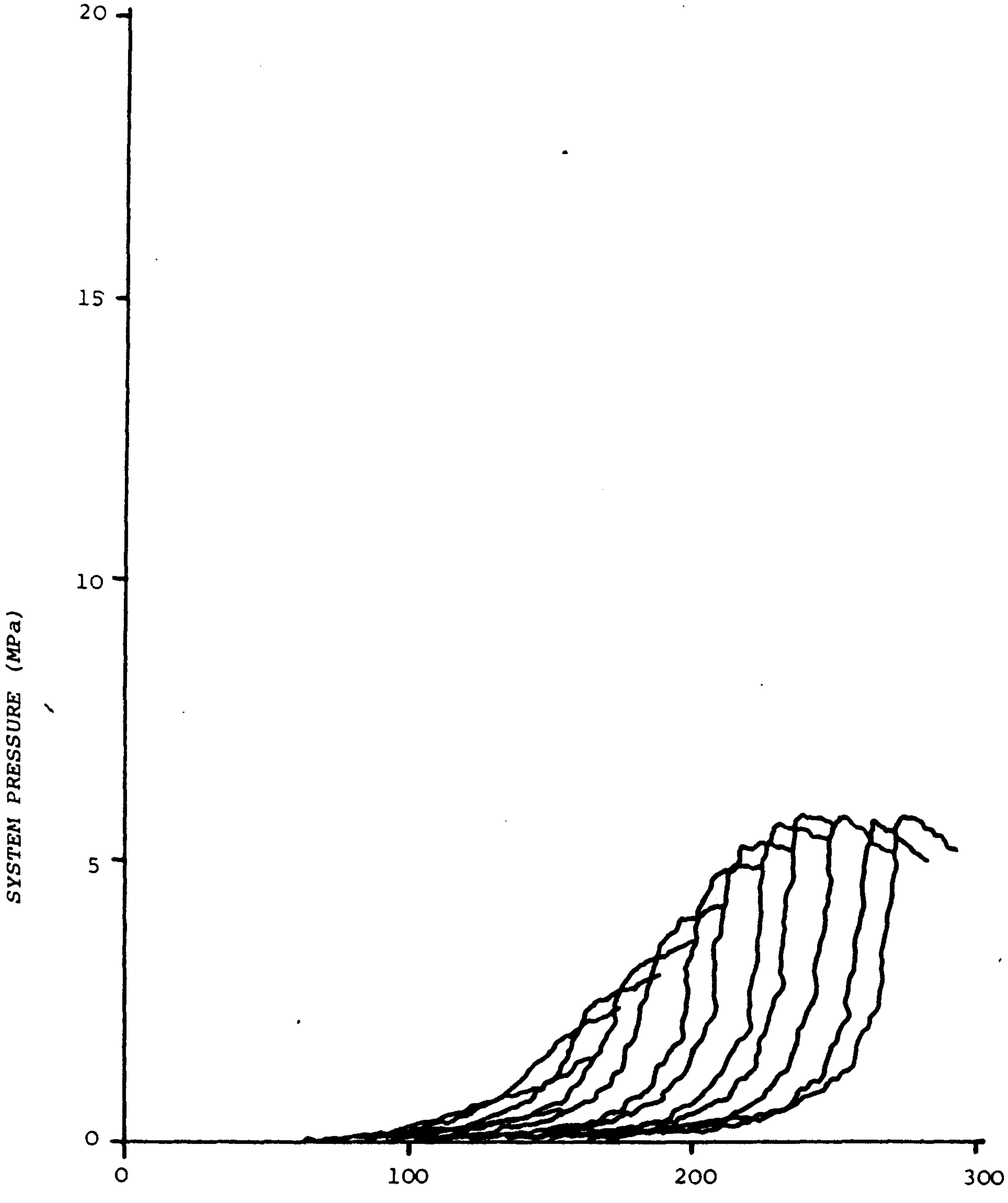

FIGURE 7.13. RESULTS FROM EXTRUSION TRIALS USING 10° DIE AND 20% PEG 4000 LUBRICANT.

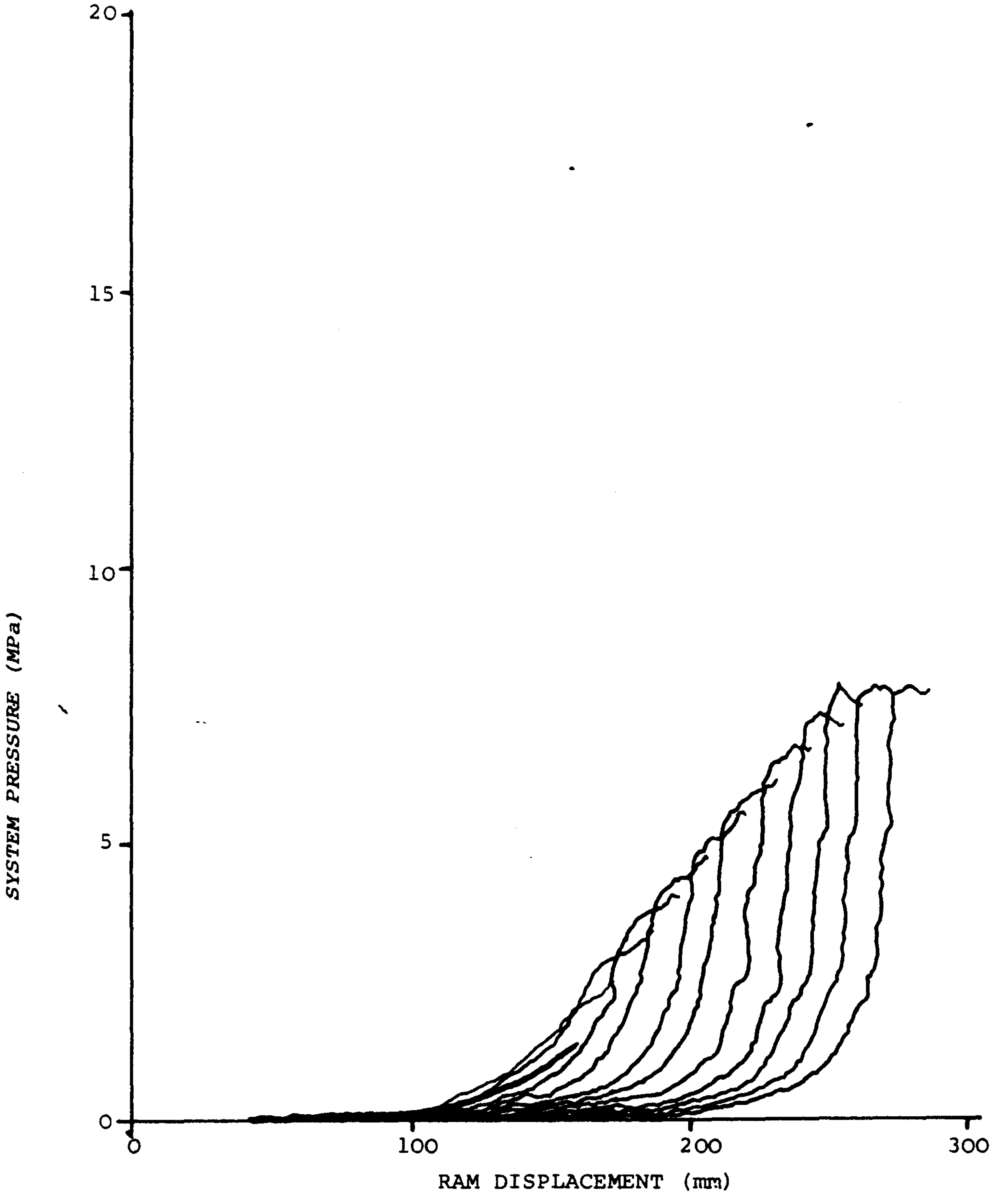

FIGURE 7.14. RESULTS FROM EXTRUSION TRIAL USING 15° DIE AND PEG 6000 LUBRICANT.

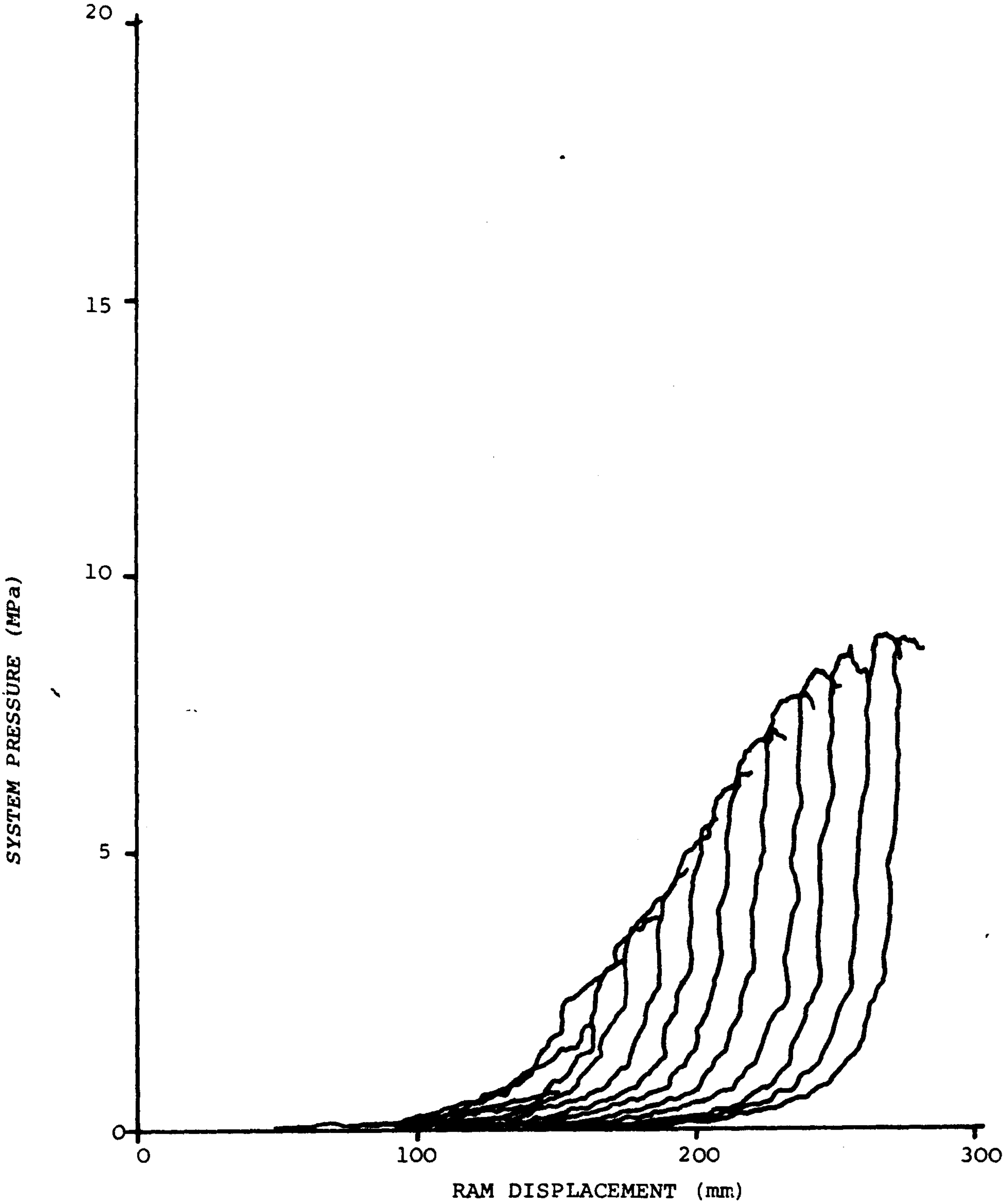

FIGURE 7.15. RESULTS FROM EXTRUSION TRIALS USING 10° DIE AND PEG 6000 LUBRICANT.

experiments. The furnish composition, experimental conditions, and maximum recorded pressures are shown in Table 7.4.

7.6.3.2.2 Discussion

Examination of the tabulated figures in Table 7.4 reveals certain specific trends:-

1) Lubrication clearly aids the process of extrusion. This suggests that in the specific case under study, friction is the predominant source of power dissipation and not plastic deformation of the chip mass as was first thought. Although by changing the molecular weight of the PEG additive as well as its concentration the issue is subject to some uncertainty, it appears that the extrusion pressure is very sensitive to the amount of lubricant present.

2) Over the limited range of die angles tested, there is consistent evidence that the die angle does not influence the extrusion pressure significantly.

If the comminuted cellophane could legitimately be called a lubricant, then the nature of the lubricant clearly has a significant effect on both the 'extrudability' of the furnish, and on the axial

FIGURE	INITIAL MOISTURE CONTENT (%)	FINAL MOISTURE CONTENT (%)	DIE ANGLE	CURING TUBE LENGTH	ADDITIVES	MAXIMUM SYSTEM PRESSURE (MPa)
7.10	8.0	17.6	15°	-	11.5% Cellophane	16.547
7.11	8.0	17.6	10°	-	11.5% Cellophane	16.547
7.12	7.2	17.0	15°	-	20% PEG 4000	7.005
7.13	7.2	17.0	10°	-	20% PEG 4000	7.819
7.14	7.8	15.6	15°	-	10% PEG 6000	11.376
7.15	7.8	15.6	10°	-	10% PEG 6000	12.169
7.16	6.2	10.0	10°	300	20% Mobilcer 739	12.962
7.17	6.2	10.0	10°	150	20% Mobilcer 739	6.840
7.18	6.2	10.2	15°	150	20% Mobilcer 739	6.350

TABLE 7.4 EXPERIMENTAL CONDITIONS AND MAXIMUM RECORDED PRESSURES.

pressure required to cause extrusion. This suggests that there is a need for an independant method of assessing the efficiency of a possible lubricant system. This would allow a pre-extrusion ranking of alternatives to be carried out, and would also enable the data from this set of tests to be interpreted in a more meaningful manner.

The data from the tests involving a change of die angle can be assessed with the use of equation (3.4-7). in that equation, the quantity f is defined as the "friction factor", and can be calculated from the formula:

$$f\alpha = \frac{\tau_n}{k} \qquad 7.6\text{-}1$$

As described in Chapter 3, τ_n is the friction stress along the wall, k is the yield stress of the extrudate in pure shear, and α is the ratio between the real and apparent areas of contact. For a soft material in contact with a hard tool at high normal pressures, α can be assumed to be approximately equal to unity. This then gives a worst possible value for f of 1, which represents the case when the softer material is adhered to the wall of the tool, and the friction stress is therefore the stress required to cause shearing of this material adjacent to the wall.

Avitzur's basic equation for work done on internal deformation, from which be developed general equation (3.4-7), is as follows:

$$W_I = 2\pi\sigma_o V_f R_f^2 F(\beta) \ln \frac{R_o}{R_f} \qquad 7.6\text{-}1$$

where W_I is the internal work of deformation, Vf is the velocity of the emerging extrudate, Rf is the radius, and R_o is the radius of the unworked billet.

$F(\beta)$ and σ_o have the same significance as in equation (3.4-7). It can be seen from this that if all other extrusion conditions are kept constant, then the work of deformation, and hence the extrusion pressure required to cause it, is only dependant on the die half angle β. For the experiments reported above, if the change in extrusion pressure were due to the effect of die angle on work of deformation alone, then the factor of change would be expected to be:

$$\frac{P_{10}}{P_{15}} = \frac{F\ (10°)}{F\ (15°)} \qquad 7.6\text{-}2$$

$$= \frac{1.00064}{1.00146} = 0.99918$$

This implies a decrease of only 0.08% for a reduction in die angle of 5°. The measured change was actually an increase of 11.6%, (7% for PEG 600), and therefore cannot be explained on the basis of work of deformation alone, since this is well outside the error limits of the observations.

Using equation (3.4-7) to calculate the change in extrusion pressure, assuming that f = 1 as stated above, gives the following results:

$$\frac{P_{10}}{\sigma_o} = 3.1844$$

and

$$\frac{P_{15}}{\sigma_o} = 2.4548$$

where P_{10} and P_{15} are the extrusion pressures for a 10° and 15° die respectively. The prediction is therefore that moving from a 15° die to a 10° die will result in a 29.7% increase in extrusion pressure. Although this predicts the direction of the change in pressure accurately, the figure is almost a factor of 3 too great.

It is the second term in equation (3.4-7) which is generating the difference, and this term was included by Avitzur to take account of both the redundant work done on the material, and the increase in the length of the material/die interface as the die angle decreases. Apart from the die angle the only other variable in this term is the friction factor f, which was assumed in the above example to have the worst case value of 1. If, as was intended, the PEG in the furnish has acted as a lubricant, then the die wall/material friction will have decreased and the approximation $f \approx 1$ will be pessimistic.

By substitution it can be calculated that a value of $f = 0.28$ gives a predicted extrusion pressure increase of 11.46% which is close to the experimenal value obtained.

Since the value of f is obtained from equation (7.6-1);

$$f\alpha = \frac{\tau_n}{k}$$

and none of the three remaining variables from that equation are known accurately, it is still impossible to show any conclusions from the results.

It is likely that the differences in extrusion pressure measured will result in slightly different degrees of compaction of the furnish, thus space need not have the same value in each case. Similarly, there will be differences in the normal pressures within the die which will affect both the real area of contact and the friction stress.

To base any theories on the present results would further be invalid because:

1) Avitzur's equation is based on a simple landless die, consequently the term dealing with the increase in contact length for diminishing die angle does not fit the experimental equipment in use. Simple geometry indicates that for the design of landed die shown in Figure 7.9, the change in contact length is negligible for the range of die angles chosen.

2) No attempt has been made to allow for axial friction in the hopper section by the use of equation (3.4-8). For a compressible starting material such as the one under investigation, all three variables in the integral will be changing continuously which makes evaluation very inaccurate if not impossible.

It is therefore necessary to conduct further experiments in an effort to quantify the unknown, or at least to build a sound base on which to make any further assumptions.

7.6.3.3 Polyolefin wax (Mobilcer 739)

Polyolefin waxes are widely used as additions throughout the particleboard industry where the use of up to 1% on the weight of dry timber confers a degree of moisture resistance on the product. There is general agreement, (see references 10, 39 and 64 of Chapter 3), that addition levels in excess of 1% impair the adhesive bond and thus detract from the mechanical properties of the finished product. In spite of this knowledge it was felt that, in the light of the results from the PEG experiments, it would be fruitless to attempt extrusion with such low levels of lubricant. Rather than set a value for wax alone, it was decided to use the same addition rate as for the successful PEG experiments. This was added in the form of a 50% by weight emulsion in water of Mobilcer 739, a commercially available particleboard additive manufactured by the Mobil Oil Company, which was mixed with the adhesive solution and sprayed onto the chips using the equipment described in Chapter 6.

The 15° and 10° dies were used for the experiments, and in order not to duplicate directly the experiments with PEG, curing tubes of 150mm and 300mm length were added to test the effect on extrusion pressure. Results were recorded in the same manner as for the PEG experiments. It was noticed before the extrusion trials that the furnish prepared for the experiment had an overall waxy feel, presumably due to the high addition rate, and was subjectively more compactible when squeezed in the hand.

Since no cure of the binder was expected at this level of wax addition, no heat was applied to the curing tubes for these experiments.

7.6.3.3.1 Experimental results

The results of the three runs with this material are shown graphically in Figures 7.16 to 7.18 and the details are given in Table 7.4.

As was the case with the PEG additive, the material was extruded successfully in all cases without maximum system pressure being reached, and again trends in the effect of the equipment variations on system pressure were identifiable.

7.6.3.3.2 Discussion

Although there were insufficient runs for the data to be regarded as conclusive, there were several points which need further investigation:

1) There is only a small difference between the 10° and 15° die under the same conditions.

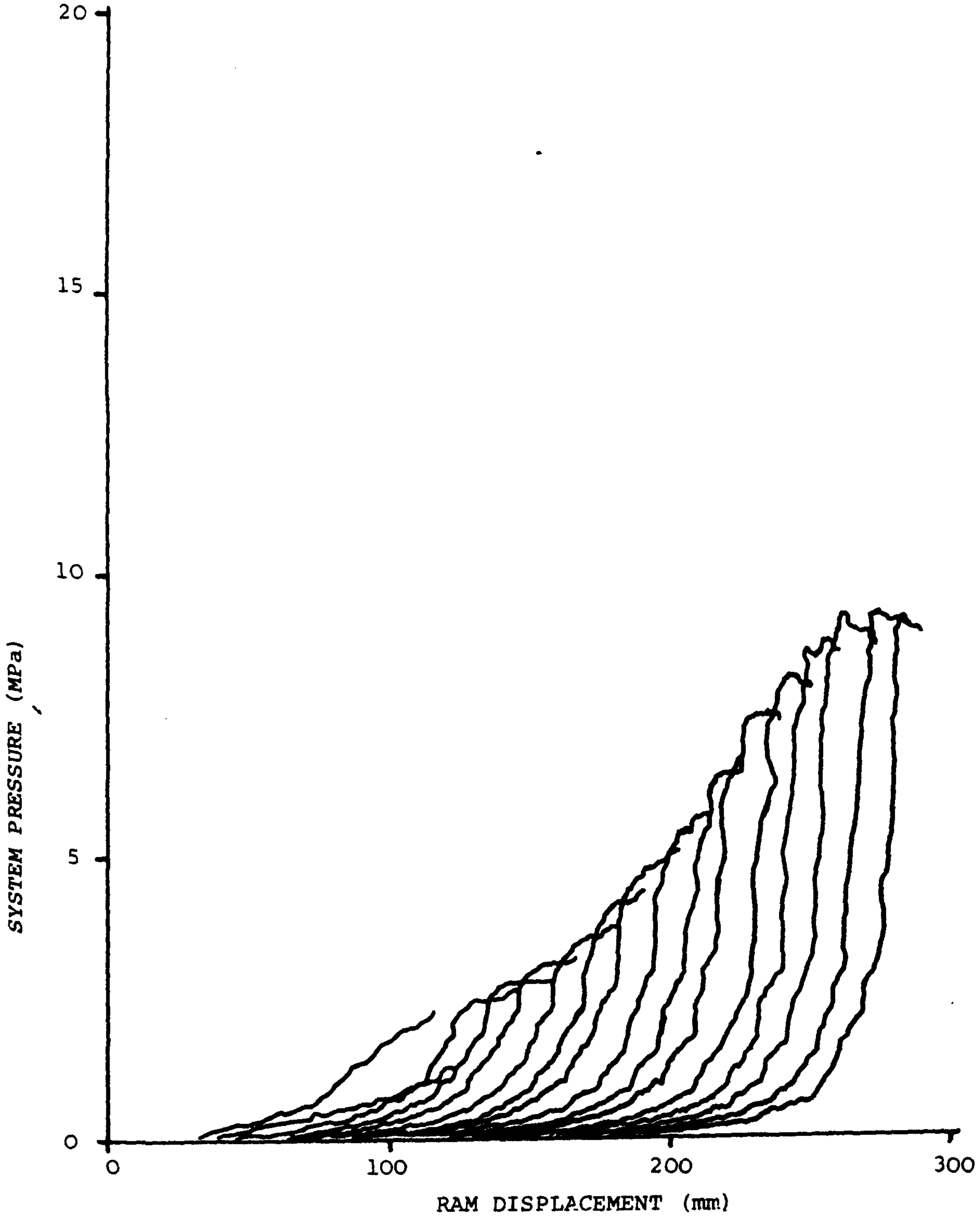

FIGURE 7.16. RESULTS FROM EXTRUSION TRIALS USING 10° DIE, 300mm CURING TUBE AND MOBILCER 739 LUBRICANT.

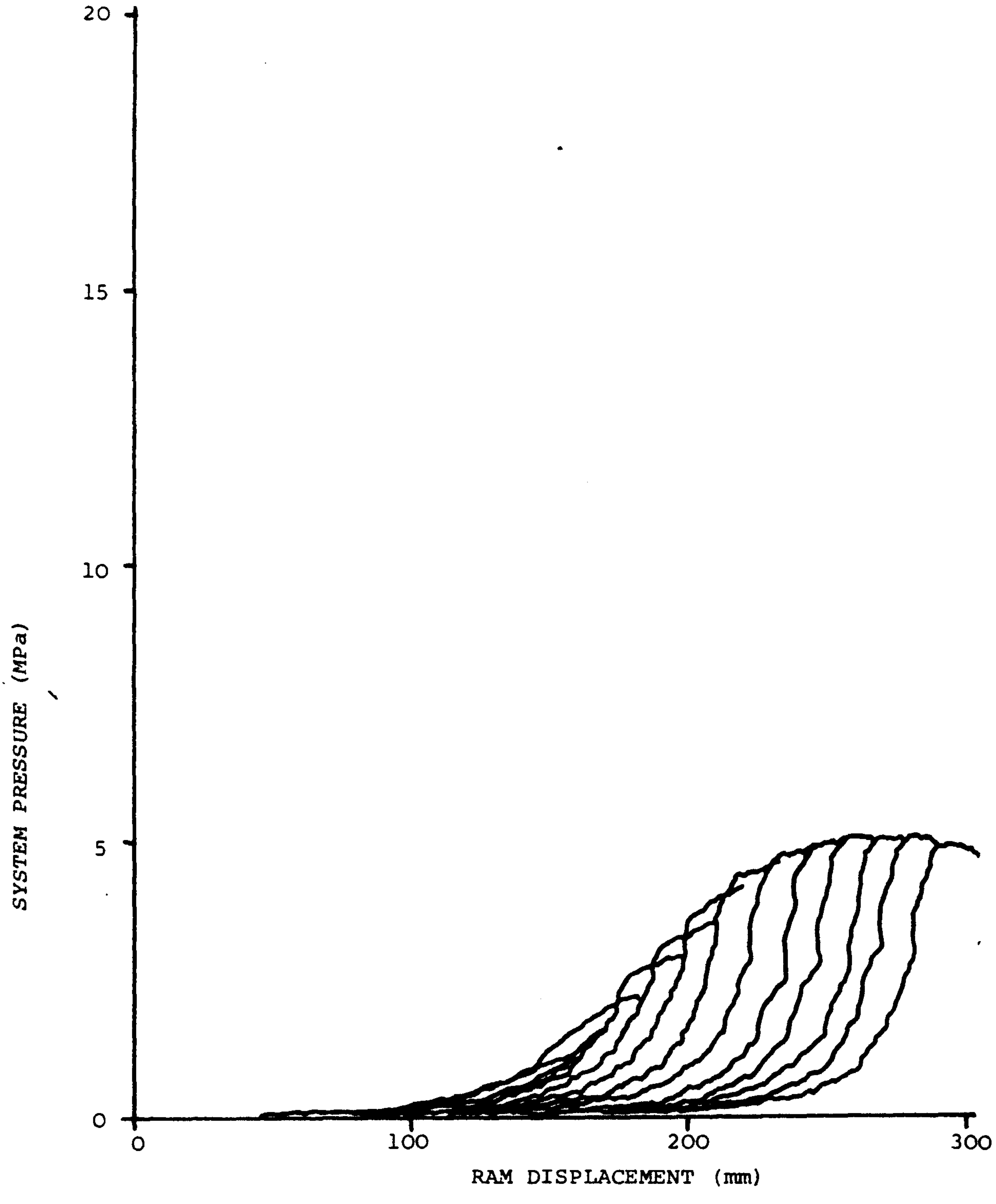

FIGURE 7.17. RESULTS FROM EXTRUSION TRIALS USING 10° DIE, 150 mm CURING TUBE AND MOBILCER 739 LUBRICANT.

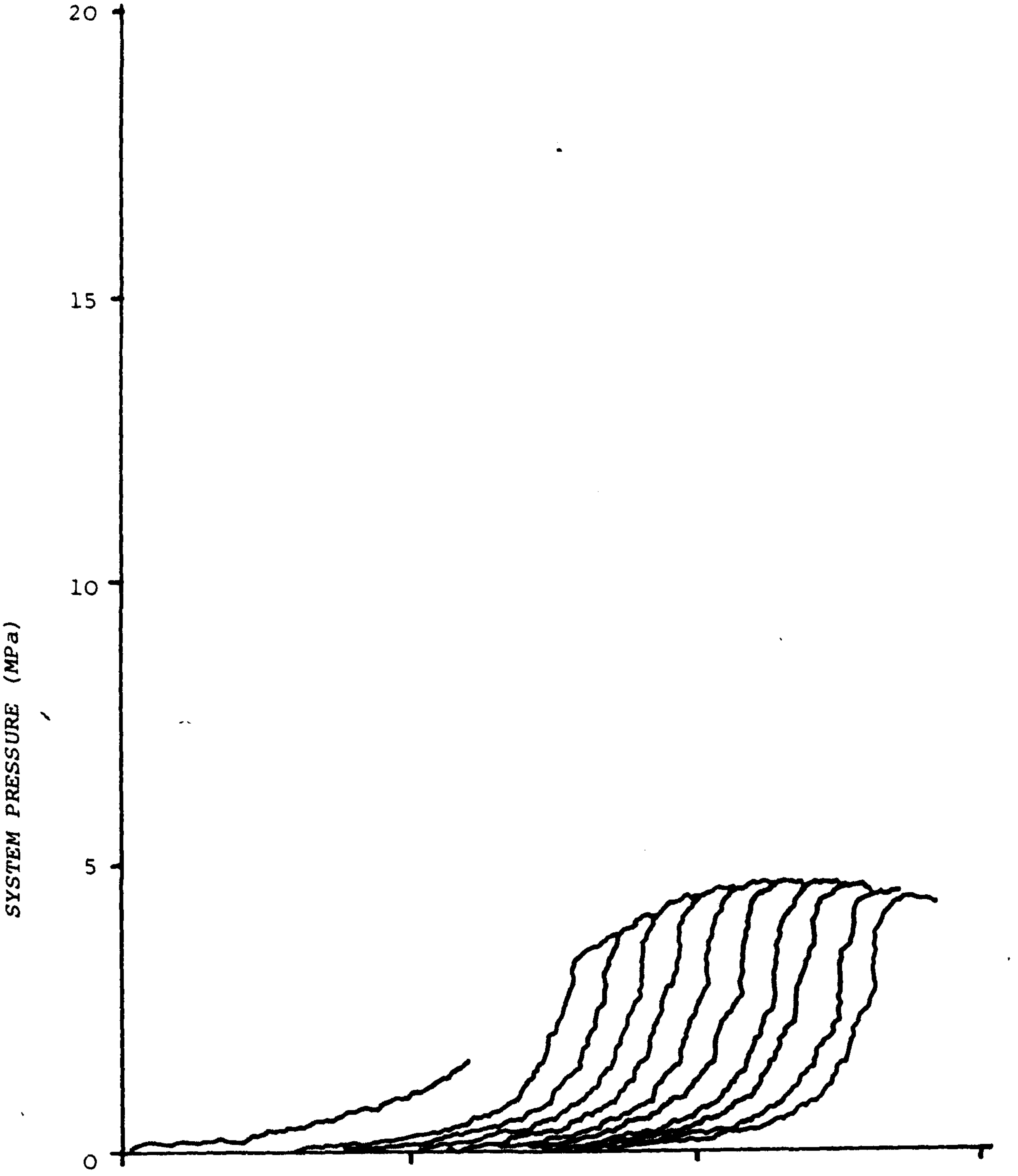

FIGURE 7.18. RESULTS FROM EXTRUSION TRIALS USING 15° DIE, 150mm CURING TUBE, AND 20% MOBILCER 739 LUBRICANT.

2) The results from the runs with the alternative curing tubes indicate that the length of the tube has a significant effect on the extrusion pressure.

The difference between the two runs using the 150mm curing tube and 10° and 15° dies is 7.7%. This is an increase in pressure moving from the 15° die to the 10° die as was observed with the PEG lubricant, however the magnitude of the difference is smaller than in the previous experiments. Since equations (3.4-7) and (3.4-8) do not include terms to cover the effect of post die friction it is impossible to compare the results with theoretically predicted values. Intuitively, if wall friction is the predominant energy sink, it is likely that the contribution of the curing tube to the extrusion pressure would be greater than that from the die, and thus the effect of the die angle would be expected to be less of the total in this case.

The increase in extrusion pressure moving from the 150mm curing tube to the 300mm curing tube suggests that the assumptions regarding the significance of the friction contribution are correct. Doubling the length of the tube from 150mm to 300mm on the 10° die increased the extrusion preessure by 89%. If the landed section of the die is included in the calculation, then the increase in post die contact length is 82.2%, and although the correlation is far from exact, the inference is that there may be a linear relationship between post die contact length and extrusion pressure. This further substantiates the assumptions regarding the influence of friction on extrusion pressure. It is also clear from these results that the wax lubricant is significantly more effective than PEG 4000 when present in the same concentration.

7.6.4 Conclusions

The role of friction between the material and the equipment walls has been shown to be significant in determining the extrusion pressure. From the results of these early successful extrusion attempts it is not possible to quantify system variables such as internal friction, yield strength, or degree of surface contact since they all change during the course of an experiment due to the nature of the feedstock. Because post-die friction appears to play such a major role in determining the extrusion pressure, and because no theoretical treatment of its effect can be found, there is an obvious need for further experiments to incorporate some means of quantifying this parameter.

Comminuted Cellophane has been shown to be ineffective as a lubricant and will be discarded for future experiments. Although there are indications that the wax additive is a more efficient lubricant, weight for weight, than the PEG, the addition level used is far in excess of that which could be tolerated without impairing bond strength. Since the product has a requirement to be commercially viable and adequate bond strength must be maintained, and it was felt that this could be achieved more effectively by accepting the less efficient lubrication properties of the PEG in the knowledge that the addition is unlikely to influence bond strength to any significant degree. Mobilcer was not used, therefore, for any further extrusion experiments.

In the graphs the ordinate is pressure in MPa and the abscissa displacement in millimetres. The results of each ram pass are displaced 10 units along the abscissa from those of the preceding pass. There appear to be three identifiable stages in each of the curves, and a tentative hypothesis for the observed shape, as labelled in Figure 7.15, is as follows:-

A-B this part of the curve represents the low pressure stage of the process and is thought to be that period of ram travel during which the furnish is compressed from its free bulk density of 140 Kgm^{-3} to a substantially solid plug of material at close to the theoretical density of about 800 Kgm^{-3}. At this stage the individual chips probably have the same orientation as is found in conventional platen pressed particleboard. This stage of the process involves a reduction in the volume of the material to about 15% of the original charge volume.

It can be seen that once the total volume of charge has reached a value which enables pressure transmission through the medium, the point B occurs at approximately the same pressure in each pass.

B-C over this part of the curve the system pressure increases considerably for only a small amount of ram travel. The head of the charge does not move along the barrel during this stage and all the piston movement must therefore be taken up with further furnish compaction. This stage is thought to involve plastic deformation of individual chips with a consequent reduction of the pore space within the mass and a concomitant increase in density. Although this portion of the graph is essentially linear and might therefore suggest some sort of elastic behaviour, tests on the compressed material have shown that deformed chips do not recover their original shape, even after prolonged periods. The linearity might therefore be a characteristic of the hydraulic power system.

C-D this portion of the curve represents the stage when the entire compressed charge moves through the apparatus. A detailed explanation for the observed behaviour has not yet been arrived at, but the fact that the pressure reaches a peak then begins to decline might be connected with the transition between the static friction coefficient and the sliding valve. Very local temperature increases at the wood/steel interface could also contribute to the reduction in friction. Quantification of these aspects are of course required since it is also during this stage that the reduction in diameter of the chip mass takes place.

The stages of the curve can therefore be summarised as: A-B compaction, B-C compression, C-D extrusion, although the actual processes occurring during each stage are complex and require further work for clarification.

Samples of the extrudate from each experiment were taken once dynamic equilibrium appeared to have been reached, and were subsequently assessed. Although there was no resin cure in any of the experiments, the extrudate developed sufficient physical strength, simply from the compression, to maintain the as-extruded structure. No valid strength measurements could be made at this stage.

In every case, regardless of feedstock formulation or equipment configuration, the chips in the extrudate had developed the same orientation. This took the form of a cone and the included angle in every case was approximately 90° as measured with an engineering protractor. It was noticeable that the extrudate had regular weaker spots, identified as corresponding to the inter-shot interfaces. The fracture surface at these locations, in addition to the characteristic 90° cone angle, had a radially corrugated appearance for which no hypothesis has yet been formulated.

Clearly since none of the experimental parameters so far varied have had any measureable effect on the chip orientation, it is likely that this is linked to the extrusion ratio of the system. This is therefore another variable which requires further investigation. The fact that the orientation is developed at all is encouraging, since one of the major criticisms of extruded particleboard is the poor mechanical strength associated with the normally observed two-dimensional chip orientation. This aspect of the current process might therefore be of some importance to the commercial application of the project work.

In the light of the results from the experimental programme documented in this section, considerable redesign of the extrusion instrument and the peripheral and support equipment was carried out. The resulting equipment formed the basis of the rest of the experimental work involved in the project, and the description of the apparatus and the results obtained are therefore drawn together and presented as a coherent entity in the following chapter.

CHAPTER EIGHT. THE FINAL INSTRUMENT AND RESULTS OBTAINED.

A summary of the conclusions reached with the previous experimental equipment, and areas which as a consequence have been identified as requiring further work is as follows:

1) The effect of extrusion die angle on extrusion pressure is minimal compared with other influences. Should die angle prove to be a variable which it might be advantageous to change for any reason, then this should be achievable without major changes in any of the other variables being required.

2) Friction appears to play a very major part in determining the extrusion pressure. The relative contributions of internal friction and product/machine friction have not yet been identified, nor have these quantities themselves been evaluated. This is an area therefore which should receive considerable attention in subsequent work.

3) Chip orientation appears to be unrelated to any of the parameters investigated thus far. There are only two other experimental variables which would be likely to have any influence, extrusion ratio and extrusion rate, and these should also be considered for investigation.

To extend the scope of the investigation to cover these new areas of interest requires a considerable increase in both the complexity of the central extrusion instrument and in the background and peripheral work necessary to support it. The work described in this Chapter therefore falls under four main headings, 1) Equipment modifications, 2) Support activities, 3) Experimental, 4) Results and discussion.

8.1 Equipment Modifications

Before any more work could be carried out on the extrusion aspects of the project, it was necessary to improve the raw material preparation equipment to cope with the increased quantities of feedstock required now that the extrusion process could be made semi-continuous.

A one metre diameter stainless steel bowl coater was obtained to contain and agitate the wood chips during coating. This was modified by the manufacture and addition of a replacement mounting boss assembly and when tests showed that friction between the smooth bowl interior and the wood chips was inadequate to give thorough agitation, four baffles were also added to the internal surface of the bowl. Tests were carried out by placing a batch of natural chips and a batch of chips stained with potassium permanganate in discrete piles in the bowl then observing the time taken to achieve thorough visual mixing. At a rotational speed of 22 rpm the contents of the bowl were thoroughly mixed after a maximum of one minute.

The adhesive application system was also judged to be inadequate for the work and a Volumair Type T2C portable spraying outfit was obtained. The range of nozzles available with this system was adequate to cover the range of adhesive viscosities it was envisaged would be utilised. A crude but very effective viscometer supplied with the unit, together with tables published by the manufacturer, made it possible to select the correct nozzle and needle to ensure that the droplet size was consistent, regardless of the mixture being sprayed. In order to verify this claim, a number of tests were carried out using solutions of various viscosities coloured with Alizarin Red dye. Chips were sprayed with the resin, using the nozzle combination obtained from the data, and were allowed to dry thoroughly. The size and distribution of the adhesive droplets on the woodchips were then assessed visually under a Leitz Metallux microscope using incident illumination. Although no quantitative data is available from these tests, a qualitative assessment of all samples indicated that only a small variation in droplet size and distribution existed. This was deemed to be acceptable for the work being undertaken.

The equipment is shown in Figure 8.1

FIGURE 8.1 The second generation coating and mixing equipment.

8.1.1 Modification of the extrusion instrument

As the equipment was constructed at this time, there were three major deficiencies in its operational characteristics:

1) the ram speed could only be varied very crudely and the maximum extrusion speed and pressure were severely limited

2) there was no instrumentation on the machine to allow the direct measurement of strains within the apparatus

3) only one extrusion ratio had been used with the entire range of die angles

An additional problem had also been identified, since the rig constituted a power press in its current form yet it had no form of safety guarding or interlocking fitted. As such it was contravening University safety rules and could no longer be operated.

Redesign and modification of the entire system was therefore of the highest priority and the details of the work are laid out below.

8.1.1.1 The basic mechanical system

The changes under this heading fall into three categories:-

a) A new hydraulic power pack consisting of a gear pump driven directly by a Brook Motors 7.5 kW three phase electric motor was obtained. This was coupled to the existing hydraulic cylinder via a Husco Model 3311 manually operated two way shuttle valve with a pressure limiter. A Sensotec Model A10/743 pressure transducer coupled to the existing RDP E307 signal conditioning and display unit was installed at the inlet to the control valve to monitor the system pressure. Location of the transducer any nearer to the hydraulic cylinder to eliminate piping pressure drops was physically impossible. Calibration of the pressure transducer/indicator combination was carried out in the manner described in Section 7.3.1. This new hydraulic equipment had a maximum working pressure of 34.47 MPa and an oil delivery rate of 40 l min^{-1}, giving a theoretical maximum working pressure at the ram tip of 571.03 MPa. Since the hydraulic cylinder was only certified to 24.1316 MPa, however, the pressure relief valve in the Husco control unit was set to limit the pressure to 22 MPa. The maximum piston velocity of the new system was calculated to be 52.63 mm min^{-1} at the rated speed of the electric motor (1440 rpm).

This new assembly was considered to be adequate, at least as the next progression toward a working system.

b) The need to be able to control the velocity of the extrusion ram was addressed next. There are two alternatives which could be used in a system with a fixed displacement pump such as this one.

i) An electronically controlled servo hydraulic flow control valve can be incorporated in the pressure line of the system. Such a unit was available from Moog Controls Ltd, but the cost was high and there would be a need for very stringent control over the quality of the hydraulic oil if this system were to be used.

ii) The speed of rotation of the motor and therefore the pump can be controlled, thereby controlling the volumetric rate of oil delivery and hence the piston velocity. Three phase AC 7.5 kW motor speed controllers are also expensive, and this system would have the drawback of decreasing pump efficiency, and therefore falling maximum pressure, with decreasing rotational speed.

Since there was a large margin of over design in the maximum potential system pressure, and because of the much greater tolerance of the oil system to contamination, the second of the options above was chosen.

A Danfoss Model VLT 10 frequency converter unit and a Model VT 20 control unit were therefore obtained. Motor protection in the form of a Telemecanique Model 75 over-current thermal protection relay was also incorporated in the control circuit of the motor. Because of the uncertainty regarding the effectiveness of this protection under reduced voltage/variable frequency conditions, the existing control cabinet for the original hydraulic power pack was incorporated into the input circuit of the Danfoss unit and the limit controls within the unit were reset to appropriately larger values.

With the system assembled as above, motor speed control between 20% and 115% of the rated speed at 50 Hz was obtainable. Maximum ram speed attainable was therefore further increased to 60.52 mms^{-1}. Although closed loop feedback control of the system was available and would make ram speed controllable directly from the computer, it was felt that this was an unnecessary complication at this stage. It would also have posed certain safety problems since the 6V DC control voltage was superimposed on a 440v 50Hz AC waveform. Manual control was therefore retained.

c) It was felt that with the substantial increase in power and ram speed which had been obtained, the existing framework might be too flexible to produce usefuly accurate results. Improvements were therefore made as follows:-

Support beams B3 and B4, (Figure 7.1), were replaced with more substantial 100 x 150 x 500mm beams to support the die and hopper unit.

Plate C2 was replaced by a similar sized plate of twice the thickness (25.4 cf 12.7 mm). The upper plate C1 and the two beams B1 and B2 were not replaced as it was felt that the substantial end plate of the hydraulic cylinder (50mm thick x 200mm square), which was bolted solidly to the upper plate, provided sufficient mechanical reinforcement to make change unnecessary. During the course of the replacement of plate C2, it was realised that because of the point of attachment of the fixed end of the LVDT, any elastic deformation of plate C2 would register in the same manner as piston travel on the monitoring system. This might explain the highly reproducible linear portion of the load/displacement curves, labelled B-C in figure 7.10, which could simply be an indication of the bending characteristics of 12.7mm thick mild steel plate. To eliminate this effect from future experiments, the point of attachment of the LVDT was moved to a bracket attached to the side of plate C2. Any bending of the plate would now cause a corresponding downward movement of the LVDT body which would not register as ram travel on the monitoring instruments.

d) In order to render the instrument safe to operate, a cage was manufactured from galvanised expanded metal mesh and 20mm angle iron, and fitted so as to enclose the working area of the machine, (bounded by cross-members B1-4 and uprights A1+2 in figure 7.1). Access to the hopper for loading and adjustment purposes was by means of a hinged door which made up one face of the cage. The door was electrically interlocked to the hold-on circuit of the local motor contactor using a Mobrey magnetic switch, thus it was impossible to move the ram with the door open, and the ram would stop moving almost instantaneously if the door were opened during a run. Rising butt hinges were also used, so that the door would tend to close under its own weight. In addition to this, two of the relay channels on the C.I.L. interface were incorporated as safety features. One was wired in series with the main contactor in the original control cabinet, and the other was wired in series with the low tension on/off control of the phase inverter, without either of which the motor would not run. Movement of the ram was therefore only possible whilst the computer programme was running for either logging or set-up purposes.

8.1.1.2 The monitoring system

Since friction appears to dominate any other factors which contribute to the extrusion pressure, there was a clear requirement to be able to quantify this parameter. The obvious way to achieve this is to install strain gauges at strategic points on the system and monitor them during extrusion runs. Strain gauges were therefore mounted in pairs to measure both axial and hoop stresses at the points marked in Figure 8.2. In addition, a pair of strain gauges were also mounted on the shaft of the hydraulic cylinder in order to measure the actual force on the extrusion ram itself, (monitoring of the hydraulic system pressure gives false maxima at the ends of the stroke when the piston butts against the

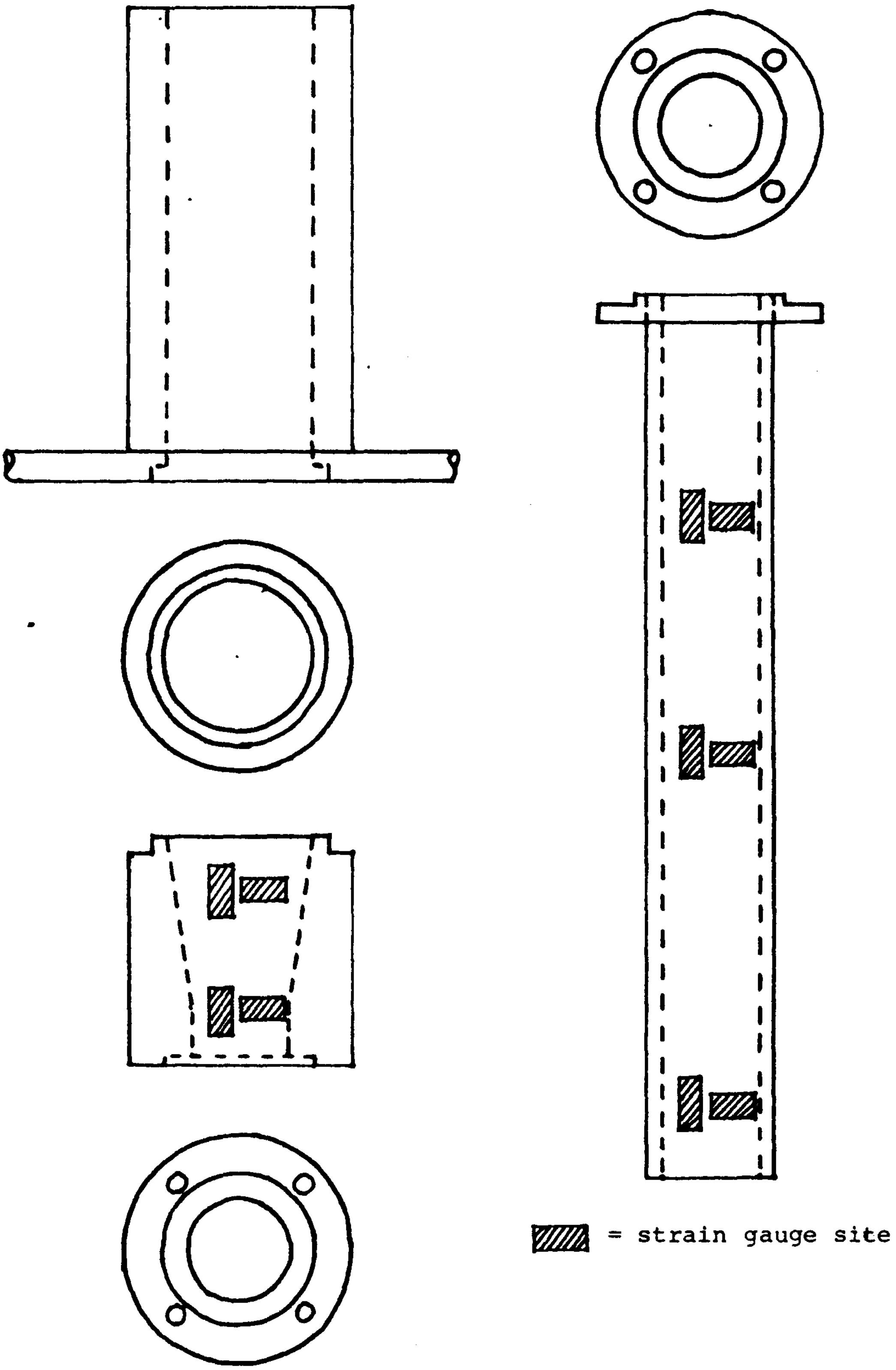

FIGURE 8.2. Positions of strain gauges on rig components shown schematically.

mechanical stops within the cylinder).

Two types of strain gauge were used, Micro Measurements miniature gauges, and R S Components strain gauges, stock number 308-102. All gauges mounted on the extrusion tools were 120 ohm nominal resistance with a gauge factor of 2.1, while the pair mounted on the shaft were of the 350 ohm type.

These latter gauges were both mounted parallel to the axis of the shaft but on diametrically opposite sides. By wiring these into opposite quarters of a half bridge circuit, an increased output per unit strain is obtained while any strains due to bending of the shaft are cancelled out. With the exception of the shaft gauges, the elements were connected in quarter bridge configuration to a purpose designed and built 16 channel strain gauge conditioning unit. Each channel was based on the standard R S Components strain gauge amplifier, part number 308-815, and the circuit as published in the relevant data sheet was used as the basis for the modified design. The standard circuit is shown in Figure 8.3. For this application any one of four resistors was capable of being switched into position R1 to give fixed gains of 10x, 100x, 1000x and 10000x the input signal. Output was calibrated to give 0-10v for each range and engineering values were converted to actual values in the computer software.

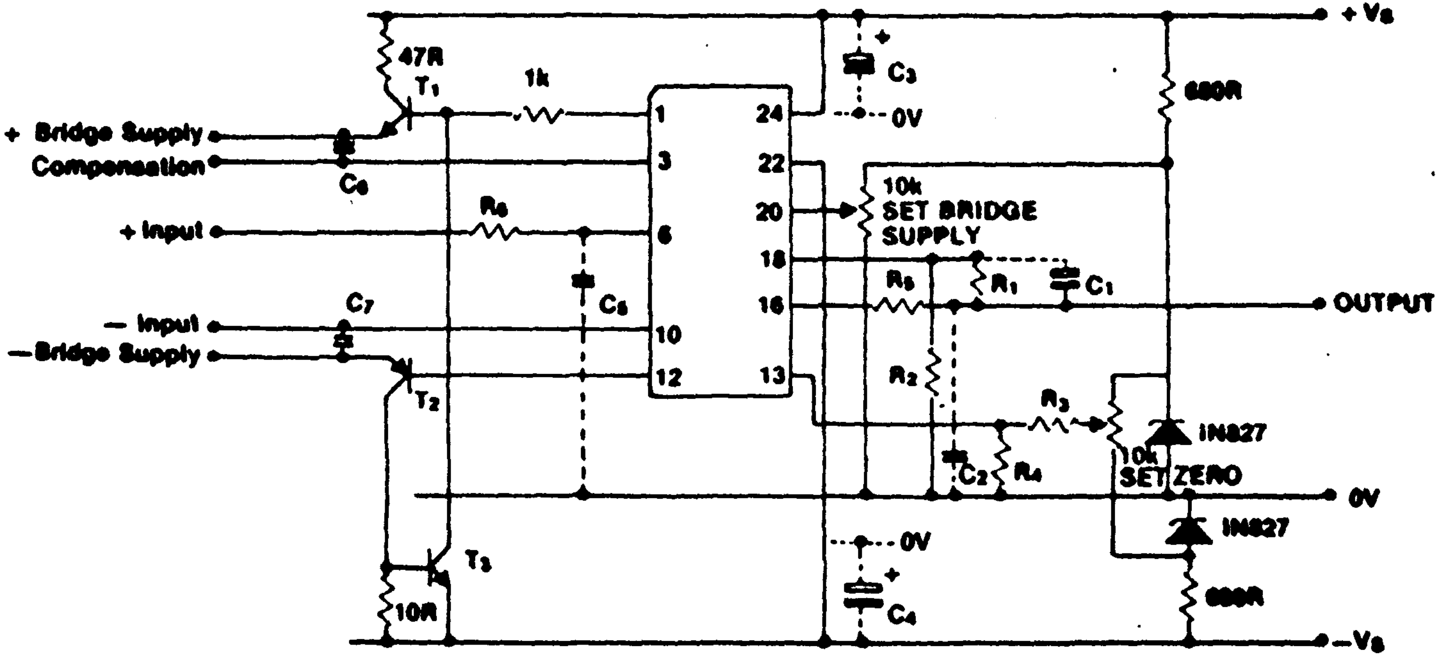

FIGURE 8.3. The basic circuit of the strain gauge conditioning amplifiers.

The output from each amplifier was fed into a discrete channel on a CIL 1281, 12 bit, 16 channel intelligent A to D convertor and the digitally coded values were passed over an IEEE 488 bus to the Commodore Pet computer. As with the monitored values from the oil pressure sensor and the ram position LVDT, the data from the strain gauges was then stored in sequential files on 5¼" floppy discs for subsequent interrogation and manipulation.

In order to accomplish the additional data acquisition the Basic program was extensively revised and rewritten, incorporating both the safety measures mentioned above, and facilities for the setting up and calibration of the sensors, conditioning units and control hardware of the modified equipment. A hard copy of the program is included in Appendix 1. No specific information regarding strain gauge mounting, or the theory behind their operation is included here since this information is widely available, and the reader is guided to references (1) and (2) for further details.

A Digitron digital thermocouple meter was fitted to the system in addition to the Ether unit already in use to control the band heaters. This allowed more accurate monitoring of the system temperature at one location, (to ± 0.5% and ± 1 digit) and by means of a 1 mV per degree Celsius output interfaced with the original CIL unit, (Model PCI 6300), enabled a log of the temperature to be recorded during extrusion trials.

8.1.1.3 The extrusion tooling

In order to provide information on the effects of changing extrusion ratio, a further set of dies was manufactured similar to the existing dies but with the overall length increased to 95 mm, and the exit diameter increased to 47.5 mm. This resulted in an extrusion ratio of 1.1:1 compared with the existing 2.05:1. Because the results of previous experiments suggested that there is little effect of die angle on extrusion pressure, the range of angles tested was limited to two, 1° and 5°, although a section of equivalent length but having parallel sides, and therefore zero die angle was also manufactured for completeness.

Curing tubes of 300 mm and 150 mm length were also manufactured to attach to the dies in the same way as those for the smaller diameter dies. The dies and tubes were eventually strain gauged in the same manner as their smaller diameter equivalents, (figure 8.2).

The hole in the centre of plate C2, (figure 7.1), was enlarged to accommodate the increased diameter of the larger curing tubes.

8.2 Support activities

The areas which require further background work are as follows:

1) The properties of the chip mass, particularly internal friction and chip/wall interface friction, need to be quantified.

2) The effects of the lubricant additives on the adhesive system need to be investigated to ensure that the product can remain commercially viable.

3) The influence of process variables on chip orientation and hence product strength must be quantified. A better knowledge of the way chips move during the extrusion process would also be valuable in understanding the mechanics of the process.

The following sections cover the work carried out in each of these areas.

8.2.1 Friction studies

As a starting point, the technique of measuring internal friction using mound formation was assessed. Very simply, chips were released in a controlled stream from the mouth of the small mixing vessel and allowed to form a mound. The angle of the apex of the mound was then measured using an engineering protractor. As Jenike found (Chapter 3, reference 28), the angle of the mound was totally independant of the formulation of the furnish, and was governed by the height from which it was dropped and by local environmental conditions. Despite numerous experiments with a wide range of materials, no correlation could be found which would suggest a link between mound angle and internal friction.

On the basis of this failure it was decided to assess the possibility of using a shear cell, after either the form used by Jenike, or that quoted by Nielsen(3) as being suitable for granular materials. Because of the large size of the wood chips, any shear cell used would be required to have generous dimensions in order to avoid complications and inaccuracies due to bridging effects.

Although a motorised rotary unit similar to that suggested by Nielsen was available, initial tests showed it to be unsuitable both for the reason above, and because the range of normal loads available was limited.

The cells used by Jenike were very simple and were based on standard soil-mechanics test procedures. Although these cells would be much too small for the wood-chip material, it was decided that the principles were likely to be applicable, and a large scale version of the Jenike equipment was designed and built as shown in Figure 8.4. The 75mm deep rings were cut from 300mm diameter, 25mm wall thickness, drawn steel pipe. Mating faces were machined to flatness on a surface grinder and then polished with 1000 grit emery cloth to remove any directionality. They were subsequently degreased and coated with PTFE lubricant in order to reduce sliding friction to a minimum. The motive power was provided by a Citenco 240 1/8 h.p. electric motor and by interposing a 0 to 50 kg Salter spring balance in the cord between the motor and the ring, it was possible to measure the force required to cause the contents of the ring to shear. Normal loading was varied by the use of a large series of weights of different values, up to a total of 75.65 kg.

8.2.1.1 Experimental procedures

Before the start of a series of tests, the mating surfaces were recoated with PTFE and buffed with a soft cloth to minimise friction. The cell was assembled empty and sheared at this stage, and the value obtained was noted and subsequently subtracted from recorded values for material tests.

The cell was then filled to the brim with the test material and a piece of 6mm plywood was placed on top to ensure even loading. The motor was started and

FIGURE 8.4 Purpose built shear box apparatus, (after Jenike, reference 28, Chapter 3).

the load registered on the balance observed closely. The load would rise to a peak then drop slightly to a steady value as the material sheared. This steady value was recorded and used as the value for the shear force, as recommended by Jenike.

The cell was then emptied and refilled, and the normal load incremented by the addition of a known weight on top of the plywood. The shearing operation was then carried out as before and the shear force and normal load noted. This procedure was repeated with small increments in normal load up to the maximum value obtainable.

The results from 5 such series of tests using various test materials are documented in Table 8.1 and the results are plotted graphically in Figure 8.5.

A second set of experiments was carried out to assess the level of friction between the wood chips and the instrument walls. Although under ideal conditions, an apparatus with two balanced opposing rams, such as that described by Benbow (4), would offer the best chance of comprehensive, accurate results, it was decided that a much simpler assembly would be more expedient.

NORMAL LOAD (kg)	SHEAR FORCE (kg) 1	2	3	4	5
1.96	49.03	44.13	39.23	39.23	44.13
21.57	85.81	76.00		73.55	
41.19	138.84	100.52	122.58	122.58	
60.80	164.26	134.84	142.20	152.00	
80.41	193.68	149.55	166.71	196.13	
100.02	198.58	174.07	191.23	201.04	
119.64	220.65		215.75	220.65	
139.25	240.26	220.65	225.55	250.07	
158.87	274.59	242.71	245.17	304.01	
178.48	289.30	269.68	279.49	294.20	
224.57	343.23	316.26	333.43	343.23	
244.19	399.62	333.43	367.75		
263.80	411.88	355.49	362.85		426.59
283.41	456.01	387.36			
303.03	470.72	404.52	426.59		
322.64	500.14	402.07			
342.25	509.95	429.04	436.40	446.20	509.95
361.87	588.40	456.01			
381.48	558.98	453.56	465.82	470.72	
401.09	578.60	480.53			
430.51	617.82	544.27	549.17		
467.29	686.47	549.17	568.79		642.34
486.90	715.89	568.79			
506.51		549.17			
526.13			647.24	598.21	627.63
565.35		578.59	666.85	615.37	696.27
594.77		627.63			701.18
604.58			676.66	671.76	
614.39		755.11			
643.81			701.18	725.69	735.50
653.61		794.34			
673.23		813.95			
692.84		862.99			
722.26		848.28			
741.87		931.63			

TABLE 8.1. Shear force vs normal load for the following systems:-

1) $P_{10}R_{30}$ chips untreated
2) $P_{10}R_{30}$ + 20% Mobilar 739
3) $P_{10}R_{30}$ + 5% PEG 6000
4) $P_{10}R_{30}$ + 10% PEG 6000
5) $P_{10}R_{30}$ + 20% PEG 6000

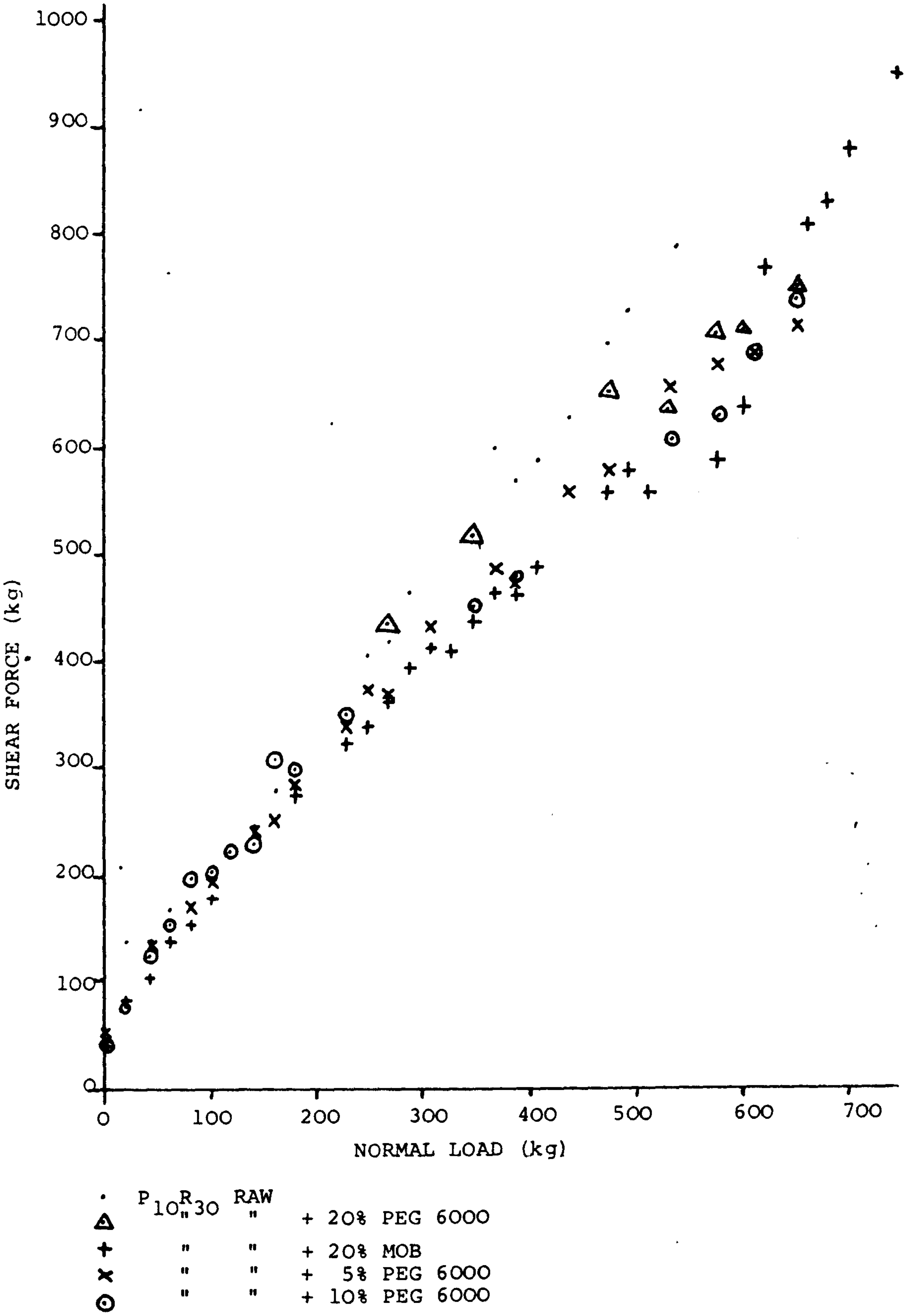

FIGURE 8.5. Plot of shear force vs. normal load obtained using the Jenike type equipment.

Two alternatives were considered:

1) an apparatus in which a sample could be compressed under a known pressure between pistons in a tube to a given thickness, and then the force required to move this slug along the tube using a driven ram could be measured

2) a device consisting of half of the shear cell used above which could be filled with a sample and loaded normally with a given weight. The force required to move this assembly across a variety of surfaces could then be measured.

In the event, since both tests were relatively straightforward it was decided to carry out both and assess whether the results were in any way similar.

The results of the tubular compression tests are listed in Table 8.2 and plotted graphically in Figure 8.6. The tests were carried out using a 40mm diameter piston in a close fitting tube. In every case the sample was compressed to a thickness of 12mm, and by varying the quantity of material used, different compression pressures were obtained. As might be predicted, the pressure to overcome static friction and cause the slug to move was greater than the force required to maintain motion against the lower value of dynamic friction. The value used to plot the graph in Figure 8.6 was this latter force,

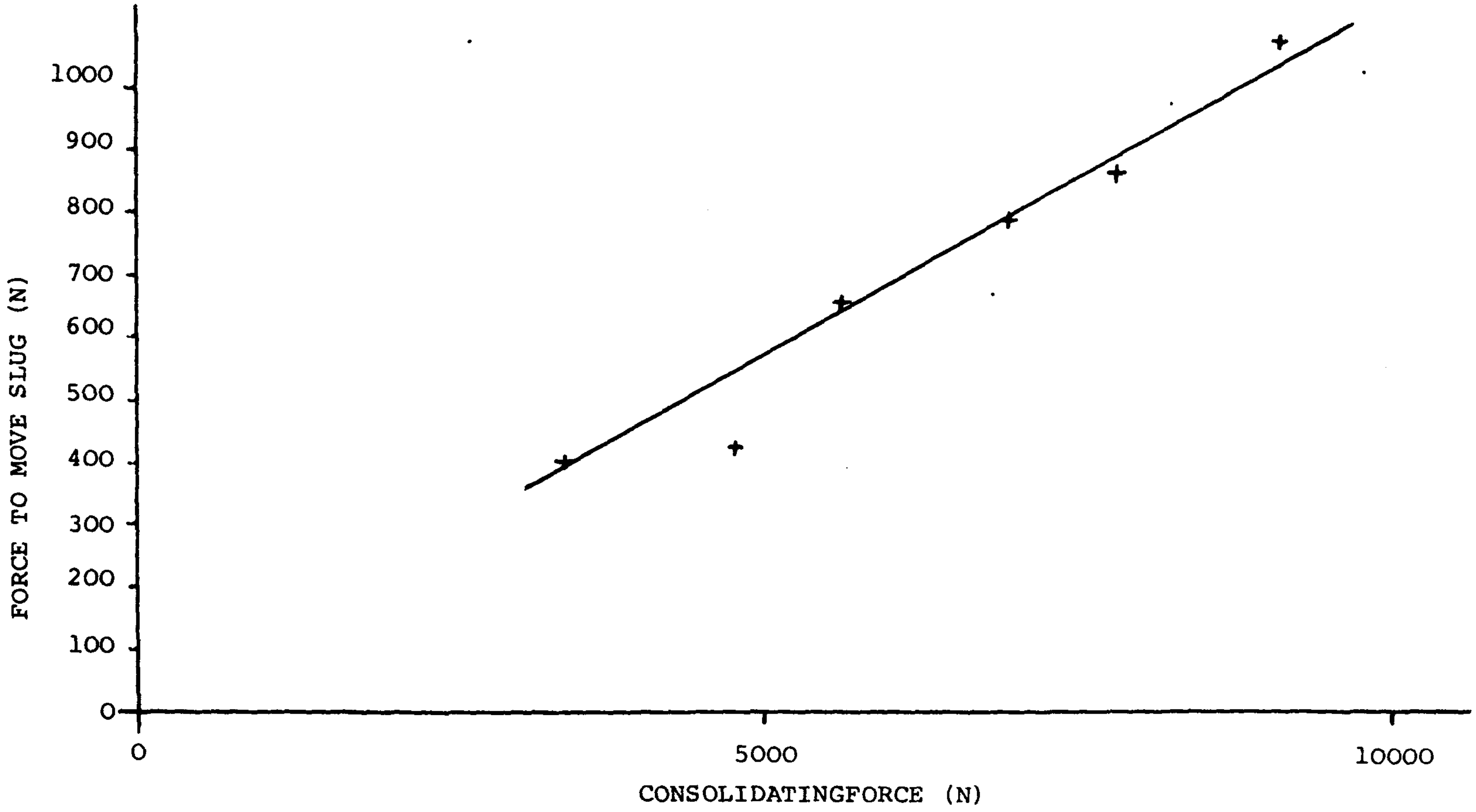

FIGURE 8.6. Plot of consolidation force vs force to move slug along barrel.

COMPRESSION PRESSURE (MPa) (40 mm dia RAM)	PRESSURE TO SUSTAIN MOVEMENT OF 12 mm PLUG (MPa)
17.25	2.00
23.69	2.13
28.00	3.25
34.50	3.94
38.75	4.31
45.25	5.38

Average = 0.111 = 0.010

TABLE 8.2. Results from piston type friction test using various weights of raw chips at 8.3% moisture, compressed to a plug 40 mm diameter, 12 mm thick.

measured once equilibrium appeared to have been established.

The alternative series of tests, carried out using the upper half of the shear box linked to the straining apparatus described previously, had the advantage that the effect of changes in the metallic component could be investigated without time consuming machining operations. Its major disadvantage was the limited maximum normal load achievable. Three sets of experiments were carried out using different sample materials and one different metallic substrate. The procedure was the same in all cases.

First of all the ring was coated with PTFE, polished and drawn across the surface empty to obtain a baseline value. The ring was then filled to the brim with wood chips and a known normal load applied by the use of deadweights.

On starting the motor, the force read from the balance increased to a maximum immediately before sliding began, then fell away to a lower, but steady value. This value was recorded for the sliding load. Once the steady value had been recorded, the ring was stopped, emptied, and the process repeated again with a different normal load.

The results of the tests carried out are listed in Table 8.3, and plotted graphically in Figure 8.7.

8.2.1.2 Discussion of results

On the basis of the exploratory experiments with mound forming mentioned above, there is clearly nothing to be gained by pursuing this line of testing. It was therefore discarded.

All of the alternative tests, however, yielded interesting results.

The results of the shear tests appear to confirm that the application of lubricant to the chip surface does have the effect of lowering internal friction. It is unclear which of the additions works most efficiently at low normal pressures, however at higher pressures all three samples treated with PEG 6000 begin to deviate from a straight line and the slopes decrease. The order of the effect does not follow the trend suggested by the extrusion experiments detailed in the previous chapter, however, since the lowest level of addition appears to have maximum effect on the shear force. This is interpreted as indicating that although internal lubrication must have some effects on the movement of the chips during compression, this is probably confined to the lower pressure, "consolidation" phase of the extrusion process, and the chip mass/wall friction becomes

	SLIDING FORCE (N)		
NORMAL FORCE (N)	RAW P_{10} R_{30} ON 254 SMO*	RAW P_{10} R_{30} + 10% AEROLITE ON 254 SMO	RAW P_{10} R_{30} ON ALUMINIUM
49.03	14.71	14.71	17.16
88.26	24.52	24.52	26.97
127.49	32.36	34.32	36.77
186.33	47.07	53.94	53.94
222.61	56.39	56.39	58.83
271.64	67.67	68.65	
320.68	79.43	80.90	80.90
369.71	93.16	95.61	93.16
408.94	100.52	100.52	102.97
448.16	110.32	110.32	110.32
573.20	138.27	139.74	139.74

Average = 0.254 = 0.019, = 0.262 = .019, = 0.274 = 0.034

<u>TABLE 8.3</u>. Results of sliding tests using shear cell apparatus.

* 254 SMO is an austenitic/ferritic stainless steel made by Avesta.

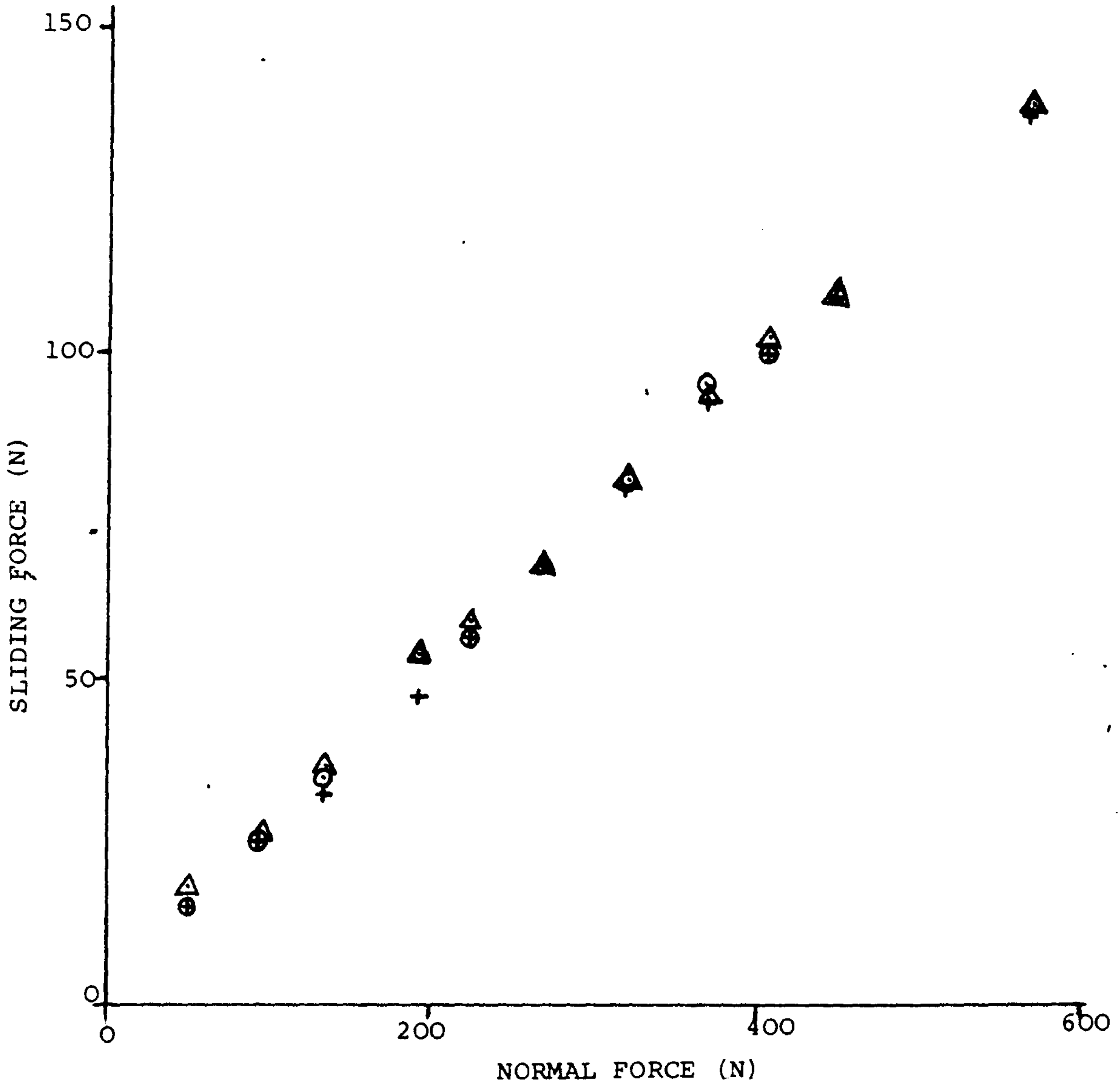

FIGURE 8.7. Results from friction tests using shear cell.

+ = RAW $P_{10}R_{30}$ chips
⊙ = $P_{10}R_{30}$ chips + 10wt % AEROLITE ON 254 SMO
△ = $P_{10}R_{30}$ chips + 10wt % AEROLITE ON ALUMINIUM

dominant before and during the actual extrusion of the chip mass. This inference cannot be taken as conclusive, however, since not only are there relatively large unavoidable uncertainties in the measurements of shear force, but the maximum normal load used, 741.87N, only gives a pressure over the surface of the material of 0.011 MPa. Compared with a typical maximum normal pressure during a successful extrusion run of perhaps 44 MPa, it is clear that the shear test results only represent the very low end of the curve obtained during an extrusion run. This is consistent, however, with the observation made earlier that in the initial stages of each ram pass, all of the pressure versus displacement curves are virtually identical. This in turn suggests that the monitoring system produces valid results, even at the low pressure end of its range.

The normal pressures used for the experiments with a consolidated plug in the steel barrel, on the other hand, varied between 17.25 MPa and 45.25 MPa thus covering a range representative of the observed experimental conditions.

The major experimental inaccuracy with this technique is the potential for axial recovery, and hence stress relief, in the sample, which the more complex arrangement used by Benbow would have avoided. By carrying out the friction pressure measurement as soon after initial compression as was possible, however, any influence of this effect was kept to a minimum.

In all sets of experiments, the value of the coefficient of friction (μ), which has been calculated is consistent within a set. Interestingly the values obtained for two different feedstocks on the same metallic surface did not differ significantly, while changing the metallic surface with the same feedstock not only resulted in a higher value for μ but also an increase in the standard deviation of the results. Such a small change may not be significant, however, since all the values obtained during ½ shear box test were within the broad range given by Bowden and Tabor (reference 35, chapter 3), of 0.2 to 0.6 for wood on dry metals.

The values of μ obtained in the piston test were significantly lower than the shear box values, however, and although Bowden and Tabor do refer to a decrease in the coefficient of friction at high normal loadings, it is felt that the decrease in this instance is not due to this effect alone. The effect of elastic recovery of the plug is likely to be a decrease in radial pressure when the compression stage is complete, and this will manifest itself as an apparent decrease in coefficient of friction. Similarly, although the 12mm thickness chosen was considered to be sufficiently thin in terms of the cylinder diameter to ensure that the transmission of compressive forces throughout the chip mass would be complete, it is possible that the effect noted by Rankine (reference 27, chapter 3) of decreasing radial pressure with increasing distance from the material surface, is also affecting the measured values.

The use of strain gauges during extrusion trials should enable the magnitude and distribution of radial forces, and therefore friction effects, to be carried out, with consequent clarification of the issues raised during the course of the experiments described above.

8.2.2 Effects of additives

Although qualitative data on the effects of additives on bond formation is available, no quantitative results could be found. Since most references also quoted addition levels considerably lower than those found to be effective for lubrication, it was considered necessary to carry out a series of simple experiments to provide background information on the potential viability of the products.

These tests were divided into two areas:

1) Gelation time tests. Using the standard laboratory gelation timer described in Chapter 5, the effect of various changes to the adhesive formulation on gelation time were assessed.

2) Bond strength tests. A very limited number of simple transverse tests were carried out on samples of compressed and cured furnish, with and without lubricant, in order to assess the extent of any deterioration in bond strength.

8.2.2.1 Gelation time tests

Three sets of tests were carried out in this part of the work to verify

a) the effect of temperature on the standard resin formulation already in use (see Table 7.1)
b) the effect of increasing the quantity of hardener, ammonium chloride, contained in the adhesive
c) the effect of varying quantities of lubricant (PEG 6000) addition to the basic formulation in a) above.

In all cases the quantity of material used was an aliquot sufficient to fill the beaker of the test apparatus, approximately 20-25 g of liquid. This was taken from a larger quantity of adhesive solution, made up on the basis of a fixed 25 g of resin with other additions in the required proportions, in order to minimise any errors on material losses during the weighing and mixing stages.

The details from the individual series are given in Table 8.4.

a) Effect of temperature

In order to minimise errors introduced by the need to heat the solution from room temperature to the test temperature, the whole batch of resin, without hardener, was heated to the required temperature as a first stage. The sample aliquot was then weighed out and transferred to the apparatus, where it was again allowed to equilibrate to the test temperature. The required quantity of hardener was mixed in immediately before timing was begun.

The results from the experiments are given in Table 8.4, and plotted graphically in Figure 8.8. These confirm statements found in the literature regarding the effect of temperature on curing time of urea-formaldehyde resins. The graph shows clearly that the curing time is still decreasing above 60°C. In practise, however, the limiting factor in curing such an adhesive once incorporated into a furnish is the rate of heat transfer through the furnish and not

TEMPERATURE (C)	RESIN (g)	HARDENER (g)	PEG 6000 (g)	GELATION TIME (minutes)
20	25.00	0.20	0	190.0
20	25.00	0.63	0	111.3
20	25.00	1.25	0	109.9
20	25.00	2.50	0	108.0
20	25.00	5.00	0	108.8
20	25.00	6.25	0	103.0
20	25.00	8.33	0	97.6
20	20.00	0.20	0	132.0
40	20.00	0.20	0	44.0
50	20.00	0.20	0	14.6
55	20.00	0.20	0	5.5
60	20.00	0.20	0	3.0
65	20.00	0.20	0	1.9
70	20.00	0.20	0	1.4
75	20.00	0.20	0	0.8
80	20.00	0.20	0	0.6
85	20.00	0.20	0	0.4
90	20.00	0.20	0	0.3
95	20.00	0.20	0	0.2
100	20.00	0.20	0	0.1
50	25.00	0.25	0.00	10.2
50	25.00	0.25	0.03	10.9
50	25.00	0.25	0.05	8.9
50	25.00	0.25	0.10	8.9
50	25.00	0.25	0.15	12.5
50	25.00	0.25	0.20	9.3
50	25.00	0.25	0.25	10.1
50	25.00	0.25	0.38	12.0
50	25.00	0.25	0.50	10.3

TABLE 8.4. Effects of formulation variations on curing time of resin solutions.

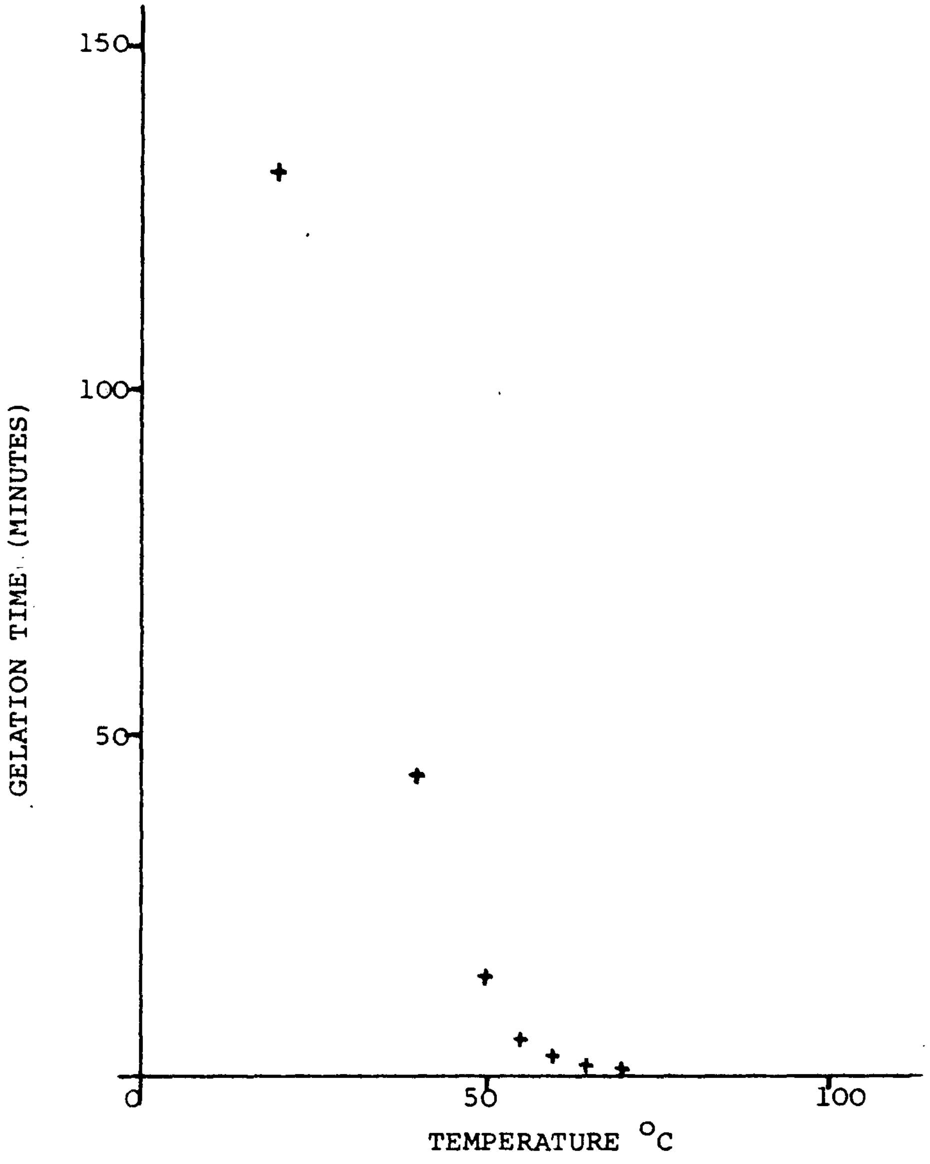

FIGURE 8.8. Gelation time vs temperature 50% resin solution + 1% NH_4Cl.

the curing rate of the adhesive. Any attempt at curing the extrusion product from the main stream experiments of this investigation will also be governed by the same constraints, and the resin system as formulated should therefore be adequate.

b) Effect of hardener quantity

Since the previous experiments showed that at elevated temperatures, the resin formulation in use would be adequate, these tests were carried out at room temperature. Clearly a lowering of the curing temperature to around ambient would have advantages in terms of machine simplicity, but the possibility of resin pre-cure would be a distinct disadvantage.

The results of the experiments are again given in Table 8.4, and are plotted graphically in Figure 8.9. Although the results do suggest a trend for decreasing curing time with increasing hardener addition, the effect is slight, and the predicted quantity to produce times equivalent to those achieved by raising the temperature exceeds the weight of resin by several orders of magnitude. It is therefore unrealistic to change the formulation on the basis of these results.

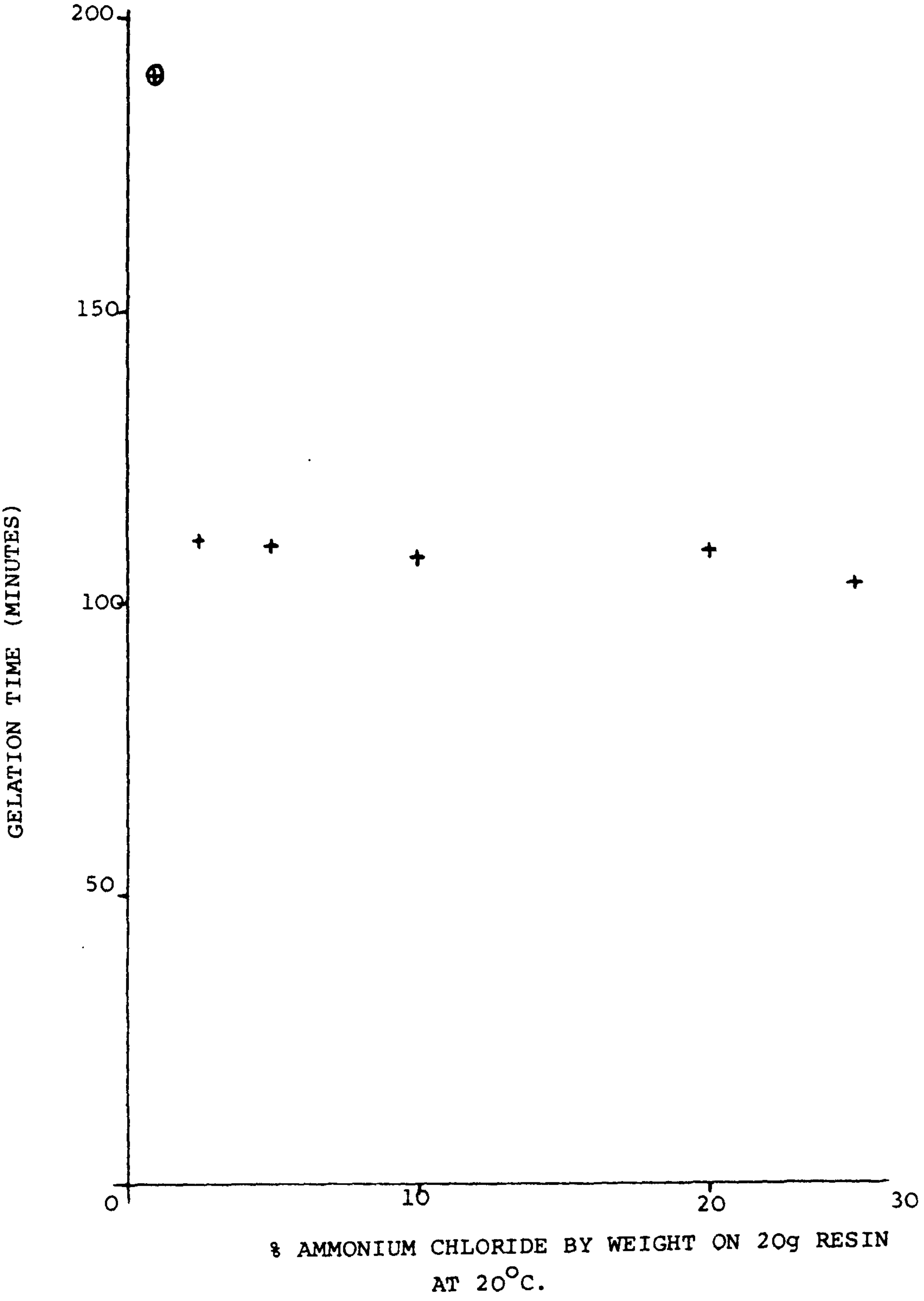

FIGURE 8.9. Effect of ammonium chloride concentration on gelation time of standard resin mixture.

There are two positive aspects to the results obtained, however. Firstly it would seem that precure of the resin is unlikely to occur within the relatively short time scale of the low temperature parts of the extrusion experiments. Secondly, it was noticed that ageing of the unhardened resin appeared to decrease curing time more effectively than increasing the hardener content, but not to an extent that would suggest that the pre-preparation of quantities of resin sufficient for several runs would cause operational difficulties.

c) Effect of PEG additions

The assessment of this effect is crucial to the progress of the extrusion experiments, and in order to obtain relevant data, a test temperature of 50°C was shown. The results from these experiments could then be judged against the simple resin formulation used in a).

Again to minimise experimental error, the hardener was added immediately before timing commenced.

The results from the experiments are again shown in Table 8.4, and are plotted graphically in Figure 8.10. It can be seen that there is considerable scatter in the results, but in no case was the gelation time as great as for the resin in test a). Even the control sample containing no PEG displayed a shorter gelation time than the resin under the same

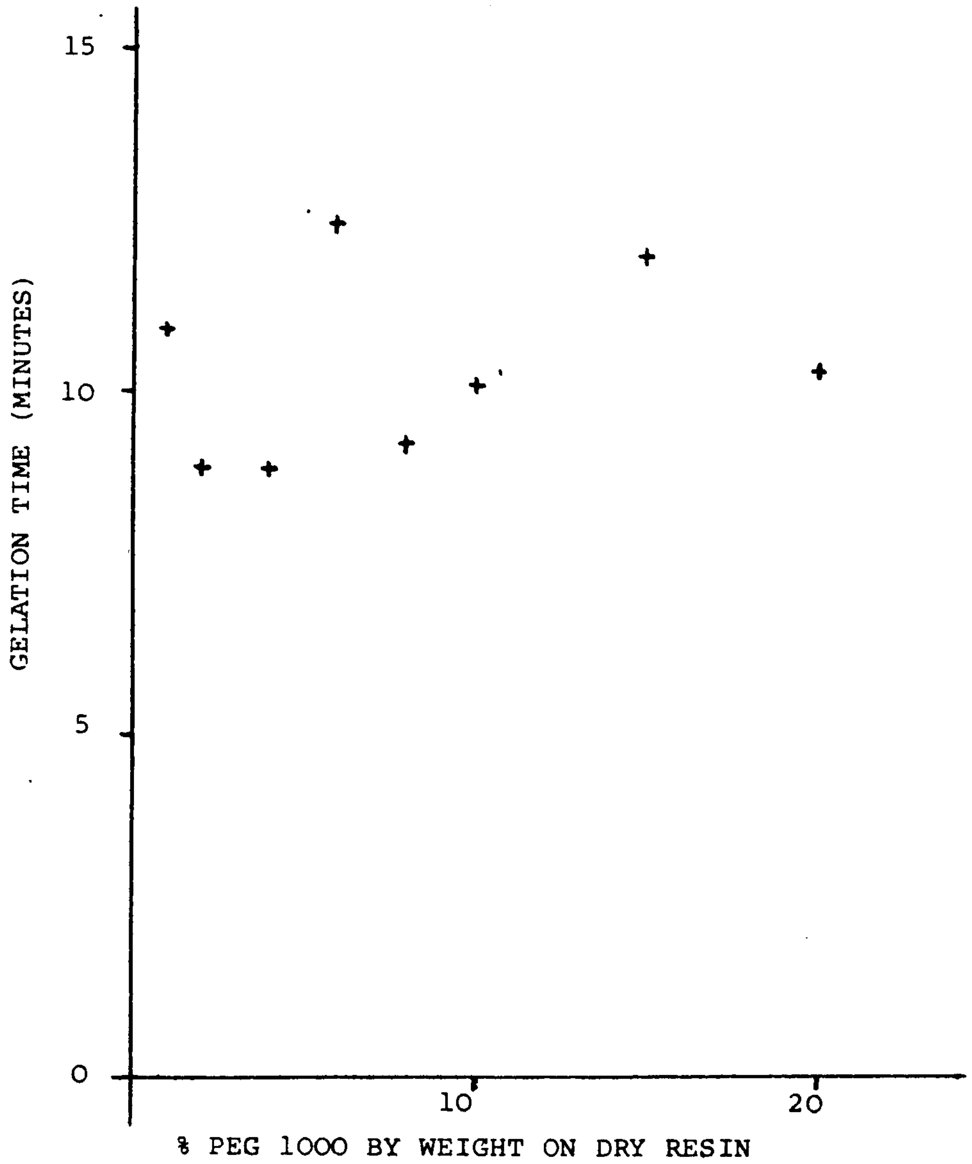

FIGURE 8.10. Effect of PEG 6000 concentration on gelation time of resin mix containing 1% ammonium chloride at 50°C.

conditions as in test a). Since the resin itself was identical, it is likely that the discrepancy is due to temperature effects, as the experiments in a) show a marked temperature dependance, 30% decrease in curing time for a 5° rise in temperature. The overall conclusion which can be drawn from the results is that PEG additions of up to 20% by weight do not affect the resin cure adversely, thus if the mechanical properties are also unaffected, then the use of PEG 6000 as an extrusion lubricant is perfectly feasible.

8.2.2.2 Bond strength tests

Since it was shown in Chapter 7, Table 7.4, that decreasing the PEG content from 20% to 10% caused a significant ($\approx$ 70%), increase in extrusion pressure, and since 20% PEG has been shown to have no detrimental effects on resin cure, it was decided to limit this series of tests to 6 samples. Three of these would be made with the simple resin/woodchip formulation given in Table 7.1, and three would contain 20% by weight on dry wood of PEG 6000.

Samples were made by compressing the furnish, prepared using the equipment described in section 8.1, in the tube and piston assembly used for the friction tests. This was then raised to approximately 60°C with the use of "Isopad" heating tape connected to a "Variac" variable transformer, and the whole assembly held under pressure at this temperature for a period of 30 minutes. The same weight of sample and the same compression pressure were used for all experiments with both furnish types.

Samples were then allowed to equilibrate for 7 days before being glued to the adaptors, seen in Figure 8.11, with Araldite adhesive (also made and supplied by CIBA-GEIGY Ltd). Following a further period of 7 days to ensure total cure of the Araldite, the samples were subjected to tensile loading by gripping in the chucks of the Instron Universal Testing Machine used for the initial extrusion trials, and stressed to failure. The maximum load recorded was taken as a measure of the strength of the bond within the composite.

The results of the experiments are given in Table 8.5. Although the average tensile stress for the samples containing lubricant is slightly lower than that for the original resin formulation, the scatter within the two groups would suggest that there is no significant difference in strength between the two.

8.2.2.3 Conclusions

From the results of all the experiments carried out, it would appear that the use of PEG 6000 at an addition level of 20% will have no deleterious

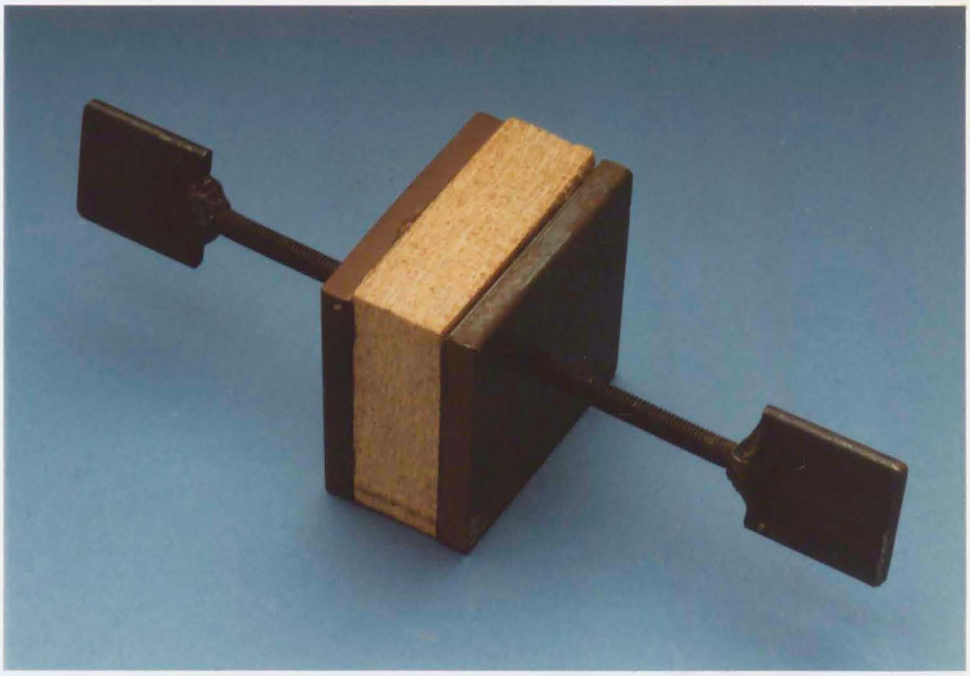

FIGURE 8.11 Typical adaptors used for tensile testing of product.

SAMPLE NO.	% PEG 6000	TENSILE STRENGTH (MPa)
1	-	0.42
2	-	0.38
3	-	0.41
4	20	0.40
5	20	0.40
6	20	0.34

Table 8.5 The result of Transverse tensile tests carried out to assess the effect of PEG 6000 on bond strength.

effects on either the curing of the resin system or the mechanical properties of the final product. Since it has also been shown that this type and level of addition contribute in a very positive way to the ease with which extrusion of the material described in Table 7.1 can be accomplished, the remainder of the experimental extrusion programme will be carried out using this basic formulation.

8.2.3 Chip orientation

That orientation effects in the form of 90° included angle fracture faces had been observed, was noted at the end of chapter 7. Since favourable chip orientation is likely to confer improved mechanical properties on the extruded product, this is clearly an area requiring further investigation. Although no continuous extrudate has yet been subjected to resin cure, the cured samples from the very early unsuccessful extrusion attempts were still available. To test the possibility of visual assessment of chip orientation, several of the samples were sectioned along the central axis, and polished on successively finer grades of emery paper. The result of one such attempt is shown in Figure 8.12. The technique can be seen to be effective, although the angle of the face to the incident light is critical for optimum contrast. There would also be considerable difficulties in bringing any sample much larger than the one shown to

FIGURE 8.12 Polished section through die area showing particle orientation.

the required state of polish, since the process is very laborious.

The most obvious alternative to the method above would be to use penetrating radiation of a kind which could differentiate between individual particles, either in real time or with the use of photosensitive film. It was thought unlikely that differences in orientation alone would provide sufficient contrast between neighbouring particles to reveal any detail, and that a tracer particle of some kind would therefore have to be used. From experience it was known that even light metals might provide sufficient contrast, but it was thought more likely that a heavier particle such as iron or tungsten would be necessary. The effects of incorporating such particles into the furnish would be unpredictable, however, and it was felt that more acceptable alternatives would be to use a much denser species of timber, or to dope a proportion of the Sitka spruce particles with a heavy metal salt, at least as a preliminary step.

To assess the suitability of the technique, a composite wedge was constructed, as shown in Figure 8.13. This consisted of three individual wedges cemented together using Aerolite adhesive. The wedges were as follows:

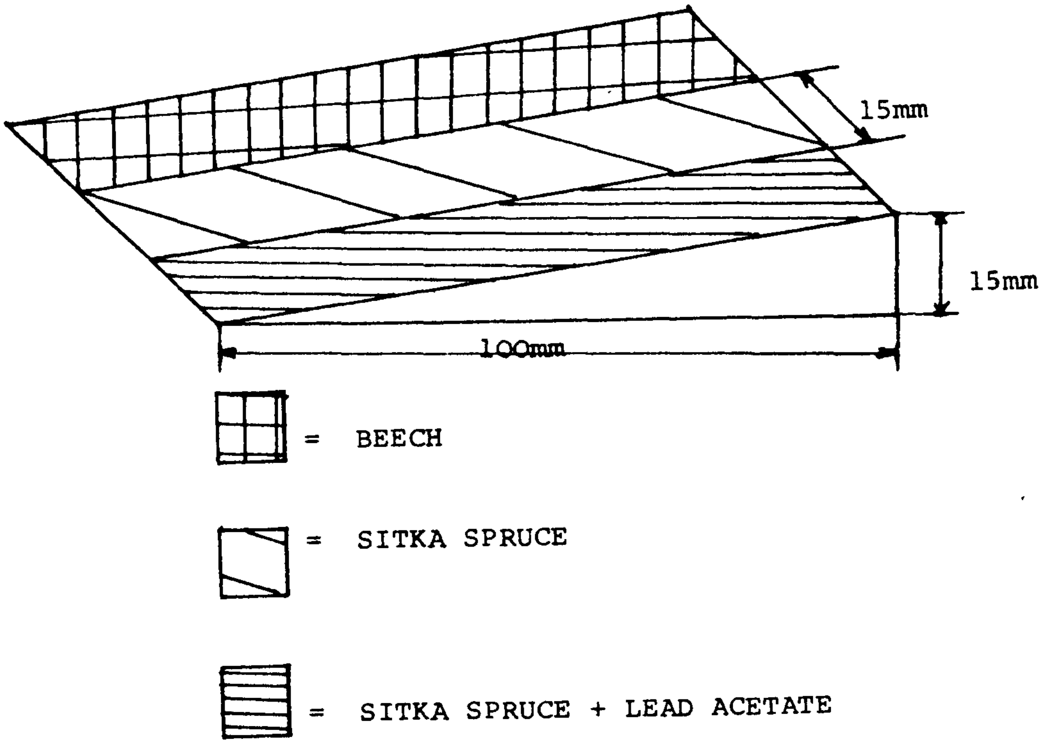

FIGURE 8.13. SCHEMATIC DIAGRAM OF TEST WEDGE.

1) solid beech timber wedge - untreated

2) solid Sitka Spruce wedge - untreated

3) Sitka Spruce wedge, soaked under vacuum in a saturated solution of lead aceatate for 525 hrs

It was felt that low power focussed X rays would be the most appropriate penetrating radiation to use, and a suitable installation was located in the University.

Exposure tests were made using Kodak Industrex CX X-ray film in 18 x 24 cm ready packs. Four exposures were given, one in each quarter of the film and the anode voltage and tube current were noted. The skiagraph in Figure 8.14 shows the exposures for 2, 4, 8 and 12 seconds at 50 kV and 13mA. As a result of these tests an exposure at 40 kV for 2 seconds was standardised upon. Figure 8.14, shows a skiagraph of the composite wedge sawn into lateral sections, and illustrates how the lead acetate solution has penetrated the structure of the Sitka Spruce. It is quite clear that penetration along the grain is more advanced than that across the grain, as mentioned in Chapter 2, and it is also clear that the sapwood layers are more easily penetrated than the heartwood.

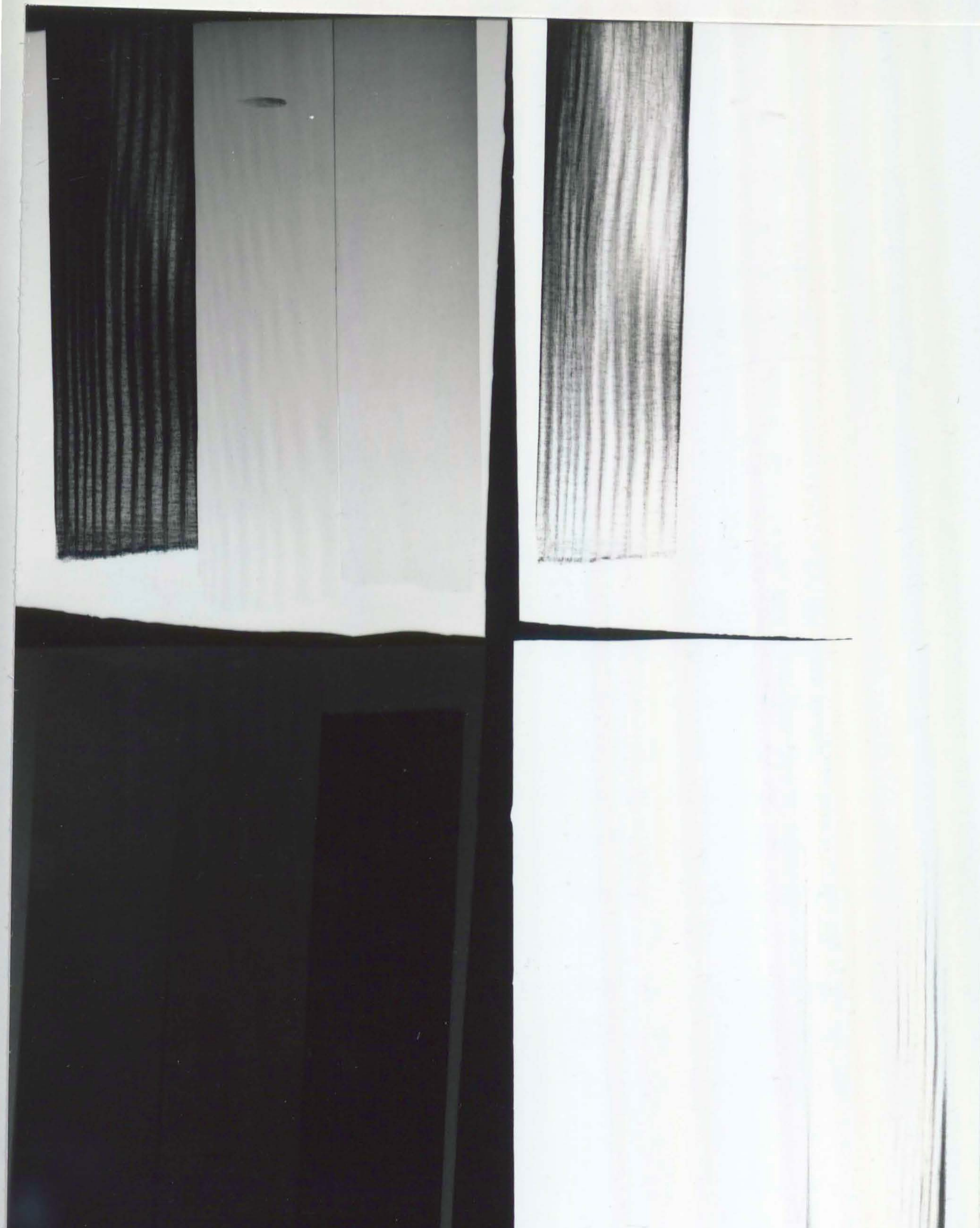

FIGURE 8.14 Skiagraph of composite test wedge for X-ray trials.

It is interesting to note that the grain structure of the untreated spruce wedge is perfectly visible, as is the much closer grained and dense structure of the beech. Although this suggests that the X-ray technique may work without resorting to doped chips, the extra contrast and definition afforded by the doping was felt to be worthwhile.

In addition to the external X-ray technique, it was thought that a similar technique using autoradiography with chips doped with a weak β-emitter might also be useful. A sample 100g of spruce chips were soaked in a saturated solution of Thorium Nitrate under vacuum for 48 hrs, and after drying were spread thinly directly onto a strip of the Industrex film used for the X-ray experiments. After 48 hrs the film was developed to reveal an unprintably pale image which was also far too diffuse to yield any detail of the individual chips. In view of the success of the X-ray technique, no further use was made of autoradiography, although the thoriated chips were used successfully as tracer materials for the subsequent X-ray work.

8.3 Experimental Techniques

Since the equipment for both the preparation of the furnish and the extrusion experiments had been extensively reworked and modified, new procedures for both aspects of the work were laid down. The details are given below.

8.3.1 Furnish preparation

The wood chips used for the experiments were prepared from the same material and in exactly the same way as outlined in Chapter 5.

The adhesive was mixed to the original formula given in Chapter 7, (Table 7.1), with the addition of the required quantity of PEG 6000 as lubricant. The solution, less the hardener, was warmed to 50°C in a water bath in order to assist the dissolution of the PEG 6000, and was allowed to return to room temperature, taking about 15 minutes, before the hardener was added. The mechanical mixing was carried out using the aluminium reservoir of the spray-gun unit as the mixing vessel, and the Hamilton Beach mixer described in Chapter 5 was used as before.

Before the wood chips were charged into the bowl mixer, their moisture content was measured using the Moisture Balance described in Chapter 5, and the value noted for subsequent calculations of furnish total moisture content.

Tests showed dramatically that the jet from the spraying equipment caused forcible ejection of wood chips from the bowl during coating, resulting in loss of both material and accuracy. A low density polyethylene cover with a hole at its centre through which the spray nozzle could be inserted was used to prevent this, and although a certain amount of adhesive was lost as overspray onto this sheet, the quantity was so small compared with ejection losses that its loss was ignored.

In order to ensure optimum droplet cover on the wood chips, the adhesive was sprayed into the bowl using a large number of short bursts, synchronised with the rotational position of the bowl and consequently with the chips showering from the baffles within it. Tests using the alizarin red dye, mentioned earlier, showed that the results produced in this way were consistent between runs.

Although the tests documented in Section 8.2.2.1 indicated that deterioration of the furnish due to resin precure was very slow at room temperature, to avoid any inadvertant or unforeseen problems which might be caused by allowing the prepared furnish to stand, the chip preparation was carried out after the setting up and calibration of the extrusion apparatus, as detailed below, and used as soon as possible after it was ready.

8.3.2 Equipment preparation

With the significant increases in ram speed and power obtained by the incorporation of the new hydraulic system detailed in Section 8.1.1, alignment of the extrusion ram, hopper, and die was now more critical than in previous experiments. A section of the computer program was written to allow independant operation of the ram for setting-up purposes, and the siting of the hopper and die was adjusted manually to a position giving minimum ram pressure at maximum ram speed. Once this position had been determined the assembly was clamped in place using four 8mm studs and lock-nuts.

Since computer control of ram speed could not be used on safety grounds, the speed was set by using the VT 20 controller to control the motor speed, and monitoring the time taken for the ram to travel between two set-points using the LVDT and the PET computer clock. Again a sub-routine of the main computer programme was written specially to allow this operation to be carried out. Because different ram speeds would result in different time periods for ram travel during a run, a further, associated sub-routine to allow selection of an appropriate logging frequency was also incorporated in the main programme.

The pressure and position transducer/conditioning amplifier combinations were energised at the beginning of any test period and allowed to equilibrate for at least 30 minutes. After this time the zero points of both instruments were checked and adjusted if necessary, and the range calibrations checked by means of the internal shunt resistors of the amplifiers.

Since temperature was the least critical of the parameters monitored, after initial checking and calibration of the appropriate instruments following installation, their accuracy and reliability were assumed to be constant.

The strain gauge conditioning amplifiers were calibrated before assembly, but because of their inherent high sensitivity and the consequent potential for error, each channel was recalibrated individually after the entire monitoring system had been assembled. The bridge voltage was monitored using the AVO DA 116 digital test meter described in Section 7.3.1, and adjusted to a value of 9.000v for each channel. Dummy resistors of 0.05% tolerance were used in parallel with unstrained strain gauges at room temperature to simulate the range of strains listed in Table 8.6, and again a sub-routine written into the main computer programme was used to monitor the value after conditioning, converting and being passed through the A to D to the PET. This operation

RESISTANCE	STRAIN SIMULATION STRAIN	GAUGE FACTOR
300 + 0.05%	199.92	2.00
	195.04	2.05
	190.40	2.10
90 + 0.05%	665.78	2.00
	649.54	2.05
	634.08	2.10
10 + 0.05%	5928.85	2.00
	5784.24	2.05
	5646.52	2.10

TABLE 8.6. Dummy resistances used in calibration of the strain gauge amplifiers.

indicated that an accuracy of better than ±2% was obtainable on the two lower ranges, with ±5% on the x 10000 range and ±10% on the x 100000 range. For the experiments, the two higher ranges were never required, and the lower ranges were calibrated at the analogue stage prior to each run using the Avo meter at the input to the A to D.

The strain gauges on the equipment were connected to the conditioning amplifiers using 3 core unshielded cable and were temperature compensated. Although the signal cables between the amplifiers and the A to D were carrying much higher signal voltages and would therefore be less prone to interference, because of their close proximity to the assembly of mains powered equipment, these connections were made with shielded 50 ohm co-axial cable.

8.3.3 Experimental method

The extrusion experiments themselves were carried out either immediately after the furnish had been prepared, or after a specific time interval when the effects of delay were being assessed. As outlined above, the equipment was calibrated and all parameters including ram speed, equipment temperature, logging rate and number of log cycles, set using the appropriate sub-routines from the main programme before the furnish was prepared. It was therefore necessary only to charge the hopper and set the ram in motion in order to begin each experiment. The passage of the LVDT reading beyond a value corresponding to the ram face entering the hopper triggered the logging programme to begin automatically. By means of the control relays built into the safety circuit, the hydraulic pump was switched off automatically at the end of each ram pass, and required a deliberate keypress to re-energise it in order to return the ram to its starting position. At this stage the door could be opened in order to refill the hopper for the next pass.

Because the alignment of the hopper and die assembly was critical and the equipment was therefore fixed rigidly once alignment had been achieved, a technique was sought which would expel the product from the equipment completely at the end of an experiment thus leaving the equipment immediately ready for the start of a subsequent experiment. The method chosen finally was to use a piston of a diameter slightly smaller, 30mm, than that of the die exit, and push product clear of the equipment using the hydraulic ram. The programme subroutine for setting ram speed was used to permit control of the ram without the logging programme running.

Because this technique did not require the hopper and die to be disassembled, it was no longer possible to use a membrane of tissue paper to prevent the first charge falling through the equipment, and a ball of crumpled computer printout, lodged in the die exit, was used instead.

Since the strain gauges used were unsuitable for high temperature operation and therefore could not be used when the curing tube was being heated, experiments using this apparatus were divided into two types each with a specific aim.

Without the strain gauges the equipment was used to investigate the routes to curing the binder system, and therefore manufacture a product.

With strain gauges the work was intended to shed more light on the influence of radial pressures and friction first tackled during the work described in Chapter 7. During this series of tests, the presence of binder was unnecessary in terms of the analysis of the underlying processes, and therefore with one exception, which will be mentioned later, all of these tests were carried out using furnish made up from wood chips, water, and 20% PEG 6000 only.

In order to facilitate X-ray analysis of samples from both series for particle orientation, in some cases 10% of the total timber content was made up of chips which had been modified by vacuum impregnation of thorium nitrate or lead acetate. These chips were dried to constant weight after impregnation and then allowed to equilibrate under the same storage conditions as the untreated chips in order to provide a relatively uniform starting moisture content.

No quantifiable difference in density between the treated and untreated chips could be measured, hence it was assumed that during the mixing operation no segregation occurred and that the resulting furnish was therefore homogeneous.

8.3.3.1 General extrusion experiments

One of the principal reasons for modifying the original extrusion equipment was to facilitate tests using increased ram speed. The effect of the increase on extrusion pressure was unknown, and this was therefore the first variable it was attempted to quantify with the modified apparatus. A series of tests was carried out using the 7.5°, 10° and 15° 2.05:1 extrusion ratio dies over a range of ram speeds from 10 mm s^{-2} to 50 mm s^{-1}. A mixture of natural and stained chips was used in the otherwise standard lubricated furnish, since it was thought worthwhile to assess whether any evidence could be found of a speed related orientation effect. The test conditions and results are laid out in Table 8.7.

8.3.3.2 Production experiments

In all, 55 individual sets of experiments were carried out with the purpose of manufacturing an extruded product. With few exceptions, which will be mentioned later, the experiments were carried out using the 35 mm diameter dies and the PEG lubricated doped feedstock described in Section 8.2.2.3. The numerical results from the experiments confirmed the findings of the work described in Chapter 7, but because of the elevated temperatures used, also provided some additional information.

a) Effects of temperature

In order to attempt to cure the resin binder, curing tube temperatures between 50°C and 150°C were used. A reproducible trend emerged which suggested that increasing tube temperatures resulted in decreasing extrusion pressures. There are three possible explanations for this observation:

DIE ANGLE (°)	RAM SPEED ($mm\ s^{-1}$)	MAXIMUM SYSTEM PRESSURE (MPa)	ORIENTATION (HALF ANGLE °)
7.5	10	9.54	45
	20	8.96	45
	30	8.60	45
	40	8.80	45
	50	9.01	45
10	10	8.22	45
	20	7.91	45
	30	7.80	45
	40	8.61	45
	50	8.40	45
15	10	8.13	45
	20	7.98	45
	30	7.60	45
	40	7.44	45
	50	8.10	45

TABLE 8.7. Test conditions and results for a range of die angles and ram speeds using the standard furnish formulation.

i) The coefficient of friction between wood and steel decreases with increasing temperature. Although this is the case with certain polymers on steel, this is as a result of thermal softening and a reduction in yield strength, and would not be expected to apply to the rigid cellulose structure of wood. Were the phenomenon only to occur above 100°C, then softening of the wood structure by steam could be responsible, (see Section 2.2.3.4), however the effect can also be observed at temperatures as low as 50°C. A second counter argument to the plasticisation theory is that as plasticisation increases, so the furnish would be expected to behave increasingly more in the manner of a fluid. Radial pressure, and consequently longitudinal friction, would therefore be expected to increase and not decrease if plasticisation were dominant.

ii) The increase in temperature causes the liberation of a fluid from the furnish which behaves as a boundary lubricant, (see Section 3.3.3). It is possible that the combination of elevated temperature and high pressure could cause substances from within the wood structure to exude onto the surface of the wood and thence into the interface between the wood and the steel.

Two routes were taken to investigate this possibility. Firstly, immediately following a series of ram passes with lubricated chips, during which steady extrusion was obtained, a hopper full of otherwise similar but unlubricated chips was compressed then followed with further amounts of lubricated chips. If boundary lubrication existed, then a certain amount of the lubricant would be expected to be deposited on the equipment walls. This should then be sufficient to lubricate the passage of one hopper load of unlubricated chips, and the extrusion pressure required should remain relatively constant. The result of the experiment was that the extrusion pressure rose to a level at which the hydraulic relief valve opened without the material extruding. This indicated not only that boundary film lubrication is unlikely to exist, but also that the lubrication which does exist is embodied in the chips, and that the area over which the retarding friction acts is relatively small.

Secondly, a series of 0.5 mm diameter holes were drilled at intervals down the hopper and the extension tube. If any fluid film was developed during extrusion, then the ram should develop sufficient hydrostatic pressure to cause some of the fluid to flow into or through one or more of the holes. Even following repeated successful extrusion runs, no evidence of any deposit or fluid of any kind could be detected in any of the holes. Again this would seem to indicate that no boundary film of a lubricating fluid is formed.

iii) The increase in temperature causes an increase in the diameter of the tube, thus diminishing radial normal wall pressure and consequently wall friction. This theory does fit the observed results, in that the decrease in extrusion pressure that is evident at all temperatures above the minimum tested would be predicted if it were due to this effect. Without constructing a series of curing tubes, accurately sized to produce a constant diameter at a variety of temperatures, no test of the theory was obvious. Theoretically, since the thermal expansion of steel is linear with temperature change, the effect on extrusion pressure would also be expected to be linear, which practise has shown it is not. It is possible that there are competing processes involved and that these change at different rates, however the evaluation of such possibilities is a task beyond the scope of this thesis and no further work was carried out on this aspect.

At temperatures near the upper experimental limit of 150°C, there was clear evidence of steam formation, and also of the formation of formaldehyde gas as a product of the resin curing reaction. Depending on the rate of extrusion, and consequently on the extent of resin cure within the product, this internal pressure could be sufficiently high to overcome both the bond between successive hopper loads of furnish and the wall friction and cause explosive ejection of the product from the end of the curing tube. The product from one such run is shown in Figure 8.15. By experimentation with the extrusion conditions it was possible to prevent this from occurring, principally by lowering the curing tube temperature. This resulted in longer curing times being needed which naturally slowed the process down considerably. For commercial applications this may be unacceptable, and it was decided to examine alternatives to direct conduction from the hot tube at this stage of the investigation.

b) Alternative heat sources

Radio-frequency (RF), curing is in wide use in the particleboard industry, and since the formulation of the test furnish is close to that of a conventional commercial furnish it was thought that RF might offer a suitable alternative. Tests on board, which was platen pressed from furnish prepared for extrusion

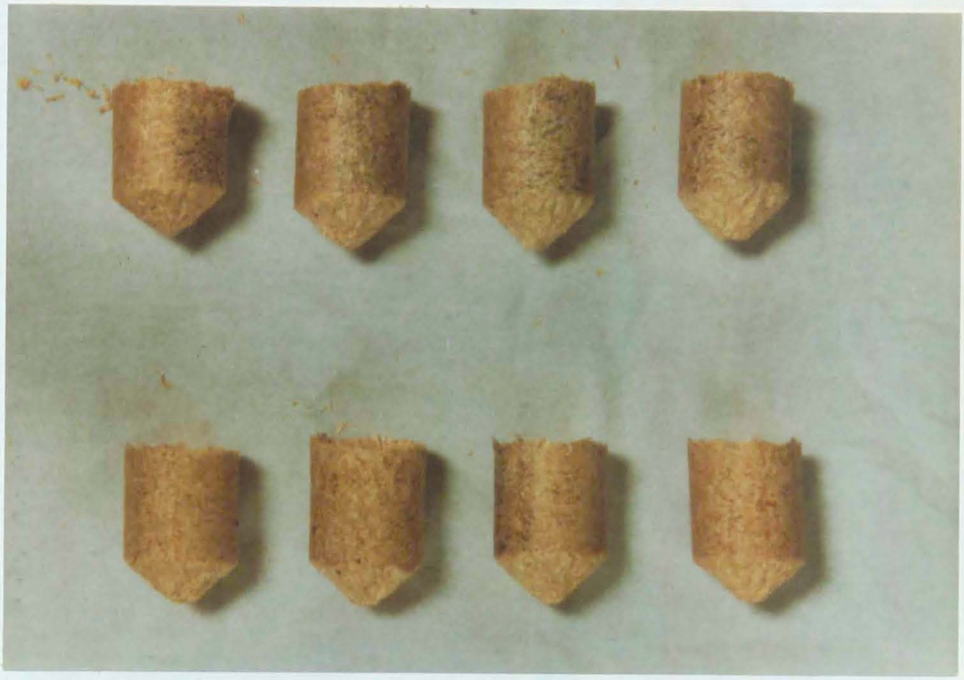

FIGURE 8.15 "Exploded" product caused by evolution of steam and formaldehyde during resin curing stages.

trials and then subjected to RF from a Radyne 3.5 kW RF generator confirmed that the technique was viable. Incorporation of suitable electrodes into the extrusion equipment was far from simple, however, and following discussions with Radyne personnel, this line of work was discontinued on the grounds of both time and safety.

Resistive heating was also considered as a possibility. By passing a current through a body, heat can be generated according to the formula:

$$W = I^2R \qquad (8.3\text{-}1)$$

where W = heat energy in watts, I is the current in amperes and R the path resistance in ohms. Since most of the conduction through the compressed furnish would be likely to be through the binder matrix because of its low electrical resistance relative to that of wood, the application of heat in this manner would be very efficient, with only those areas requiring the heat being affected. Tests were carried out to determine the resistance of the furnish through the various stages of compaction. Using a PTFE lining in the 40 mm tube used for the friction tests and insulating the bottom plug from the steel framework with PTFE sheets, the resistance of a quantity of furnish under varying amounts of compression was measured using a Twenty Million Megohm Meter made by EIL of Cambridge. The results of such a test are given in Table 8.8. Although, as can be seen from these results, the resistance of the furnish remains relatively high even under maximum system pressure, it was decided that experiments using the extrusion system could be justified. To ensure that the electrical path passed through the furnish, the curing tube was electrically isolated from the extrusion die and the apparatus framework by means of "Tufnol" washers, specially manufactured for the purpose from 12.5 mm thick sheet. Tests on the assembled equipment using the EIL instrument mentioned above indicated that the resistance through the washers was of the order of 1000 times greater than the minimum resistance of the furnish, which would ensure that some current would pass through the product. Electrical energy was provided by the use of a British Oxygen Company "Transarc 100" portable a.c. welding transformer.

Extrusion trials using the standard lubricated furnish, the 2.05:1 extrusion tools, and the modified apparatus as described above were curtailed prematurely before the application of current when the Tufnol washer between the die and the curing tube burst open under the radial pressure of the product, as seen in Figure 8.16. There were two conclusions which could be drawn immediately from this:-

SYSTEM PRESSURE (MPa)	LENGTH OF CHIP COLUMN (mm)	RESISTANCE (megohms)
0.00	142.0	0.41×10^4
0.05	105.0	0.95×10^3
0.10	95.0	0.47×10^2
0.50	38.0	0.63×10^2
0.75	27.0	20.00
1.05	23.0	18.00
1.95	19.0	12.00
3.10	18.0	8.50
3.90	16.5	6.50
5.18	16.0	4.80
6.73	15.5	3.20
10.35	14.0	1.50
20.06	12.0	1.05
22.00	9.5	0.75

TABLE 8.8. Height and electrical resistance of a column of furnish prepared according to the proportions given in Section 8.2.2.3, and subjected to a range of compression pressures.

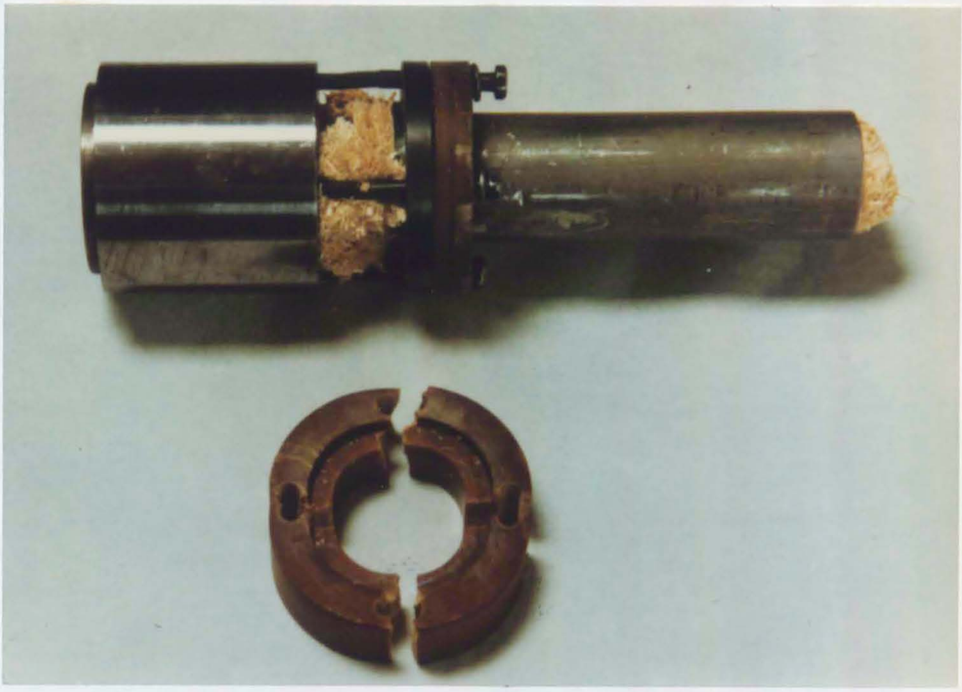

FIGURE 8.16 "Tufnol" washer which failed due to excess internal pressure during extrusion trial.

1) The radial pressure exerted by the furnish persists even after the reduction in area has been accomplished. This must be due to energy of elastic deformation remaining within the product. Strain gauges on the wall of the curing tubes should enable these stresses to be quantified.

2) If experiments using this form of heating are to be continued, then more substantial insulators will be required.

The second point was addressed immediately, and new Tufnol washers were made with 3 mm thick steel reinforcing rings fitted around their circumference.

Because of the incompatibility between the strain gauges and the elevated temperatures, no stress levels were measured at this stage of the work. Knowing that the tensile strength of Tufnol is 145 MPa in the plane of the sheet, however, and by using the formula for hoop stress in thick walled brittle cylinders, (Lamé's equation).

$$t = \frac{D}{2} \quad \frac{S + p}{S - p} \quad - 1 \qquad\qquad 8.3\text{-}2$$

where t = wall thickness (mm), D = inside diameter of cylinder (mm), S = tensile stress in wall (Pa), and p = internal pressure (Pa), then an approximate figure for the minimum normal stress can be calculated.

Taking t = 9.5 mm (the ring broke across the locating holes where clearly the cross section is smallest), D = 35 mm and S = 145 MPa, then by substitution in equation 8.3-2, p = 84.1 Pa.
This is clearly an overestimation of the pressure involved since no allowance has been made for the stress raising effect of notches, (bolt holes), and end faces, nor any bending or assymetric distortion stresses introduced by the clamping process.
Nevertheless, even if a factor of 5 or 10 is involved, the level of stress which this would indicate is transferred from the vertical to the horizontal direction is significant and should be easy to detect using strain gauges.

The equipment was reassembled using the reinforced Tufnol washer, and on this occasion extrusion was achieved without mishap. Despite using the maximum current available from the welding transformer, (150 Amperes), however, no temperature rise sufficient to initiate rapid binder cure could be achieved within the furnish. Making the following assumptions:-

Wt. of resin in current path = 1.5g
Specific heat of resin = 1
Temperature rise required = 60C

then by using Ohms law it can be calculated that the potential difference required to initiate resin cure under ideal conditions is of the order of 8kV. Since the use of this would clearly cause safety problems, this line of investigation was curtailed at this point, and attention was focussed on improving the operation using direct heating techniques for the remainder of the experiments.

8.3.3.3 Strain gauge experiments

A series of 15 experiments was carried out using the fully strain gauged equipment described above to extrude samples of the lubricant containing furnish described in Section 8.3.3. The results of the experiments are given in the next section. One of the experiments was also used to investigate the effects of process parameters on chip orientation by using a furnish containing doped chips and Aerolite binder. In this case, curing of the binder was achieved by wrapping the entire extrusion section with the Isotape mentioned earlier and holding the equipment at 50°C during the course of the experiment. Such a low curing temperature did not affect the strain gauges or their adhesive in any permanent way, but did enable the product to be sectioned for later X-ray analysis. The assembly was split open whilst still warm, but was allowed to cool before the product was expelled using the hydraulic ram as described above.

In some of the experiments, a mixture of equal parts of natural chips and chips stained with potassium permanganate was used to enable visual assessment of chip movement to be carried out in addition to the X-ray technique.

The product was sectioned using a vertical bandsaw, and where necessary, faces were again polished using emery paper.

8.4 Results and discussion

The results of the first stage experiments to assess the effect of ram speed on extrusion pressure appear at first sight to be rather inconclusive. The range of values obtained for each die angle are relatively consistent, varying only by a maximum of about 11%, however there is no clear pattern relating ram speed to system pressure within any group.

The trend relating system pressure to die angle, first observed during the experiments described in Chapter 7, can be seen in these results. The change in moving from a 15° die to a 10° during these experiments was significantly lower at 4.3% than the 11.6% change observed for the 20% PEG 4000 addition in the previous experiments. The change between the two dies for the lower percentage addition of PEG 6000 in the previous experiments is closer to the most recent value at 7%. The magnitude of the pressures involved was almost 40% higher than in the later experiments, however, and the most recent figures are much closer to those obtained using PEG 4000 at the 20% addition rate. The standard deviations from the mean values in the latest set of experiments are, 3.9%, 4.2% and 3.9% for the 7.5°, 10°, and 15° dies respectively. These values are sufficiently small to suggest that the results may be taken as significant, even though the sample population is small, and it is likely that the discrepancies are due to inaccuracies in the logged results from the earliest experiments. The modifications detailed in the early part of this chapter should have reduced this source of error significantly.

At this stage it was also thought possible that the surface finish on the die walls might be having an effect on the extrusion pressure, as outlined in Chapter 3, and in order to check this possibility, the internal surface finish of each of the dies was measured using a Rank-Taylor-Hobson Talysurf instrument. The centre line average roughness of each of the dies was measured at four points around the circumference and there was little difference between the values for all of the dies. It is thought that with the very "viscous" nature of the material being extruded, there will be no measurable effect on extrusion pressure as a result of these slight changes in surface finish.

It can be seen from Table 8.7 that no discernable effect of ram speed on particle orientation had emerged. Once again this could be due to the relatively crude, and hence inaccurate, measuring system used. If the effect is so small as to be undetectable in this way, however, then there is little to be gained, from a practical point of view, from a more accurate study of the subject.

Since both sets of results point to the fact that ram speed has no discernable effect on any of the parameters being monitored in this study, all subsequent experiments were carried out at constant speed. The value chosen, 40 mm s^{-1}, represents the speed at which it is still possible to effect resin cure and produce a continuous product, yet which allows relatively rapid operation of the experiments thus avoiding problems related to pre-cure or ageing of the resin.

8.4.1 Production experiments

The results of the variety of tests carried out with the aim of manufacturing an extruded product efficiently were:-

1) Extrusion pressure decreases as temperature increases. This is probably due to expansion of the curing tube, perhaps in combination with

some undefined temperature dependant changes in the lubrication regime.

2) Although both radio-frequency and resistive heating have been shown to be possible in theory, incorporation of such systems is difficult on the pilot scale equipment in use.

3) It is possible to manufacture finished, extruded product from the system using conducted heat as the curing initiator. The balance between extrusion rate and temperature is critical in ensuring that the product has attained a high degree of strength before emerging from the machine. Steam blowing, common in non-optimised particleboard production, has also proved to be a problem in this extrusion process.

It is clear from these results and from those of the individual experiments that the aim of achieving continuous extrusion of a wood composite article is possible. The use of efficient heating regimes such as radio-frequency and direct resistive heating has been shown to be feasible in theory, and although impossible to refine to a working level in the time scale of this investigation, should be possible to apply in a full-scale production plant. The use of direct conductive heating has been maintained for the purposes of this investigation, however, and by achieving a careful balance between extrusion rate and heat input, a continuous product which is consistent, repeatable, and of adequate quality can be manufactured. Although the incorporation of the PEG 6000 lubricant has increased the cost of the finished product to a considerable degree, it is clear that manufacture of the desired product without it would be difficult, if not impossible, and the cost margins quoted elsewhere in this thesis are sufficiently large for this extra cost not to be a serious problem.

In all cases where the emerging product had fractured or when a complete sample showed weaknesses or broke, the line of the fracture surface always formed approximately the same included angle of 90°. In addition the fractures always occurred at the regular intervals which could be seen to be the inter-shot interfaces. These are clearly points of weakness which are not compensated for by the semi-axial orientation of the wood particles. The most likely cause of the weakness is the flat smooth surface which results on the top of each hopper load as the ram compresses and then forces the material through the equipment. Although the orientation in the final product indicates that this surface undergoes considerable further deformation as it passes through the equipment, the lack of any surface "key" with which the succeeding batch of furnish can interlock means that at this point in the product the strength will be entirely dependant upon the strength of the adhesive present at the interface.

There are two obvious ways to overcome this problem:

1) a much stronger adhesive can be used between successive charges of furnish

2) the surface of the material at the end of a ram stroke can be disrupted mechanically before the succeeding charge is fed into the hopper.

Neither of these two options is attractive since the former would increase costs further, and both would disrupt the flow of the process.

Although no attempt was made to investigate option 1), a modified ram was manufactured to investigate the plausibility of option 2). By cutting a slot 2 mm wide across a diameter of the base of the ram a section of hacksaw blade could be mounted normal to the face with the teeth protruding. At the end of the ram travel during the compression stroke, the ram could be rotated with a lever whilst still under pressure, thereby disrupting the surface.

Tests using this technique did not produce any measurable difference in the propensity for breaking at the join, nor in the load required to cause fracture between these samples and samples produced using the standard ram. Although the level of engineering required to incorporate such a ram rotation system on a production scale unit would be relatively trivial, and rams with tips which cause significantly more surface disruption might be more effective, no further investigations along these lines were carried out.

At the end of the series of experiments, the alternative die and curing tube with an extrusion ratio of 1.1:1 were fitted to the equipment in order to assess the effect of extrusion ratio on extrusion pressure and particle orientation.

The results of one test using a 1° die and a 150 mm curing tube are shown in the skiagraph of Figure 8.17. The maximum extrusion pressure recorded during the run was 6.0 MPa. It is clear from Figure 8.17 and by inference from the low extrtusion pressure that the product extruded very easily and consequently is barely compressed at all. The density could not be measured accurately due to the high porosity but appeared to be approximately 0.6 - 0.7g cm^{-3}. The product was weak and friable, being barely able to support its own weight, and therefore totally unsuitable for service in almost any end use.

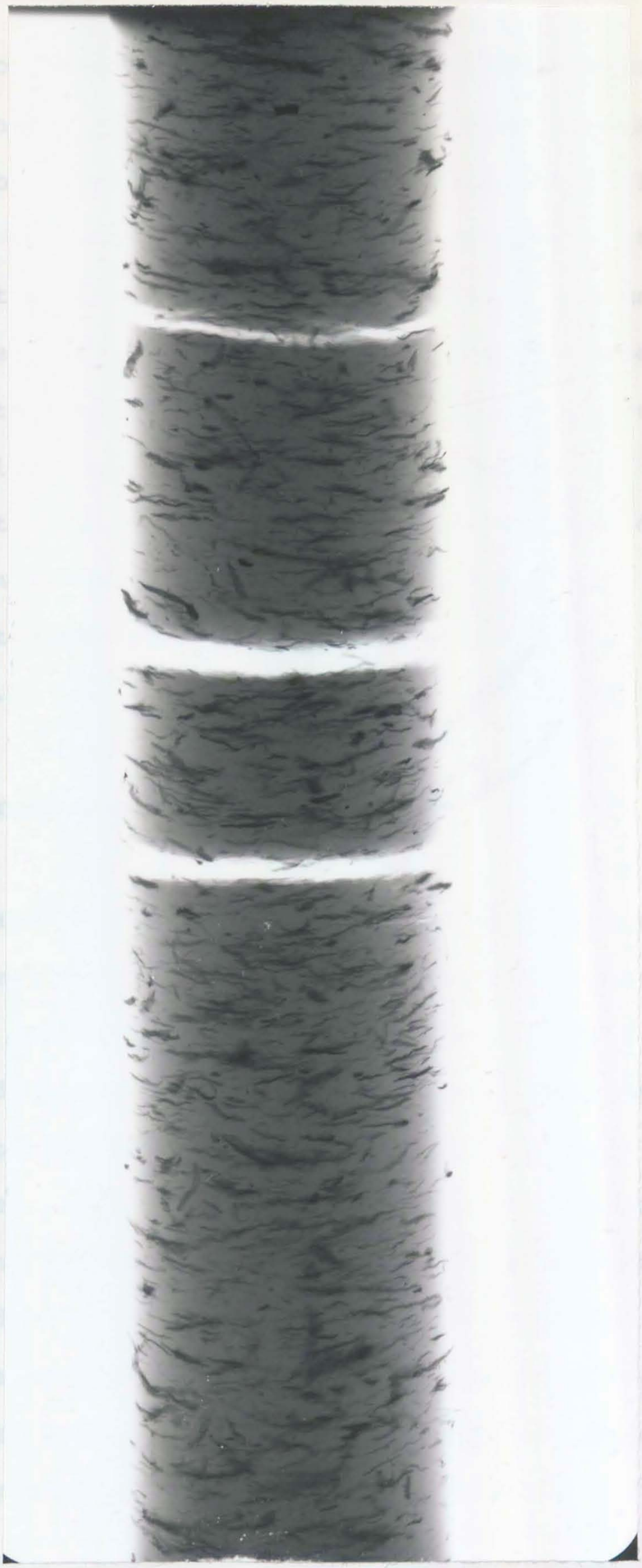

FIGURE 8.17 Skiagraph of extrudate produced using a 1° die and an extrusion rate of 1.1:1.

It was not possible to section the product because of the weakness, and this explains the poor contrast and resolution of Figure 8.17. Sufficient detail is visible, however, to indicate very clearly that there is almost no orientation present even in the material below the die. This supports the view expressed earlier that orientation is a function of extrusion ratio, although whether this is due to the constriction of the die, or to the frictional drag forces on the outer layers of the product will not be clear until the strain gauge test results are examined.

8.4.2 Strain gauge experiments

Because all of the information from each of the process monitors was logged and recorded by computer, each run generated a very large amount of data. All of this data has been examined and analysed, and general trends have been identified throughout the programme. In order not to increase the size of this thesis any further, only one full set of results will be included to illustrate the format used, and reference will be made to specific points and values from other relevant runs wherever they are necessary to verify arguments used and statements made.

Table 8.9 illustrates the format in which the results were recorded, and Tables 8.10, and 8.11 summarise the conditions used and the results obtained for each of the first twelve runs.

8.4.2.1 Comments on results

On first examination of the full results, large discrepancies were found between the values of ram strain measured, and theoretical values calculated from the hydraulic pressure readings. Since the electronic hardware was checked and calibrated at the beginning of each set of tests, the software used to record the data was examined. This was found to contain an error of a factor of 3 in the value of strain, and the results shown in Table 8.10 contain the corrected values.

Although logically the maximum system pressure and the axial strain on the ram as measured with the strain gauges should vary in the same ratio between runs, inspection of the results indicates that this is not the case. As has been stated earlier, both measurement systems were checked and calibrated regularly and neither showed significant drifts in accuracy between runs, or even from day to day. Since the accuracy of the analogue stages of both systems was of the order of ± 2%, the accuracy of the correlation of the two results should be to within ± 4%. There is one obvious exception to this case,

```
NEWCHIP 5

09 11 84

P10R3006.4%MC+150WAT+100PEG6000+100AERO+3.75NH4CL-90DIE-150EXT

RAM SPEED = 40 MM/S

LOGGING RATE 4 PER SECOND

S 1=RAM AXIAL
S 2=DIE 20MM HOOP
S 3=DIE 20MM AXIAL
S 4=DIE 55MM HOOP
S 5=DIE 55MM AXIAL
S 6=TUBE 40MM HOOP
S 7=TUBE 40MM AXIAL
S 8=TUBE 140MM HOOP
S 9=TUBE 140MM AXIAL
NO  DISP PRES  TEMP   S1     S2     S3     S4     S5     S6     S7
********************************************************************
1   64.3  11.1  19.2 1991   1      0      -2     8      -2     3
                     6      1      9      10     8      4

2   74.4  9.6   19.5 1994   1      0      -2     9      -2     2
                     6      1      9      10     8      5

3   84    15.6  19.2 1992   1      0      -2     8      -1     2
                     6      1      10     10     8      4

4   94.1  22.8  20.1 1988   1      0      -2     7      0      2
                     5      1      9      10     8      5

5   104.2 36.4  19.2 1983   0      0      -2     4      5      6
                     5      1      9      10     8      4

6   114.5 38    19.8 1982   0      0      -2     3      7      7
                     5      1      9      10     8      4

7   123.8 42.5  20.5 1988   -1     0      -2     3      7      8
                     5      1      9      10     8      5

8   134.3 47.8  19.8 1985   -1     0      -2     3      7      8
                     5      1      9      10     8      5

9   144.6 42.8  19.5 1984   -2     0      -2     3      6      7
                     5      1      9      9      8      4

10  154.3 42.9  20.8 1983   -2     0      -1     2      7      7
                     5      1      9      10     8      4

11  163.9 56.5  19.2 1983   -4     0      -2     1      8      5
                     5      1      9      9      8      4

12  173.9 69.3  19.5 1982   -4     0      -2     1      8      3
                     5      1      9      10     8      5

13  183.4 67    19.2 1979   -6     0      -3     0      7      2
                     5      1      9      10     8      5
```

TABLE 8.9 Example of the format of results obtained showing layout of information

RUN TITLE	*INITIAL* MOISTURE CONTENT (%)	*INCLUDED* DIE ANGLE (DEGREES)	*CURING* TUBE LENGTH (mm)	*MAXIMUM* SYSTEM PRESSURE (MPa)	*MAXIMUM RAM* STRAIN & POSITION (microstrain/ mm)	*MAXIMUM TUBE* HOOP STRAIN & POSITION 1 (microstrain/ mm)	*MAXIMUM TUBE* HOOP STRAIN & POSITION 2 (microstrain/ mm)
NEWCHIP 1	8.3	7.5	300	16.74/186	396/184	1091/189	554/189
NEWCHIP 2	8.3	7.5	150	14.39/186	332/186	649/195	644/189
NEWCHIP 3	8.7	7.5	150	12.60/188	289/187	579/188	625/199
NEWCHIP 4	8.7	7.5	150	14.75/186	319/188	703/196	722/197
NEWCHIP 5	6.4	90.0	150	9.94/188	240/188	544/189	412/199
NEWCHIP 6	8.4	90.0	150	17.84/174*	267/188	287/*	71/*
NEWCHIP 7	8.2	90.0	-	5.45/190	122/180	142/200[3]	112/200[4]
NEWCHIP 8	8.3	10.0	-	8.53/189	189/189	203/189[3]	-
NEWCHIP 9	8.3	7.5	-	7.54/189	171/189	180/189[3]	-
NEWCHIP 10	8.3	90.0	-	9.12/187	139/199	92/199[3]	44/187[4]
NEWCHIP 11	8.3	7.5	-	7.73/188	162/188	108/199[3]	-
NEWCHIP 12	8.3	15.0	-	9.04/183	205/183	369/183[3]	-

TABLE 8.10. Conditions and results from extrusion tests with strain gauge monitoring.

1 Circumferential strain gauge at top of curing tube

2 " " " at exit from curing tube

3&4 " " " on outside of die

* Included adhesive in formulation for assessment of orientation, results not comparable.

MAXIMUM STRESS VALUE AT EACH STRAIN GAUGE SITE (MPa) (t = tension, c = compression)

RUN TITLE	APPLIED EXTRUSION PRESSURE	DIE NORMAL STRESS 20 mm	DIE AXIAL STRESS 20 mm	DIE NORMAL STRESS 55 mm	DIE AXIAL STRESS 55 mm	TUBE NORMAL STRESS 40 mm	TUBE AXIAL STRESS 40 mm	TUBE NORMAL STRESS 140 mm	TUBE AXIAL STRESS 140 mm
NEWCHIP 1[3]	104.63	-	-	225.7[a]	-	114.6[b]	-	58.4[c]	-
NEWCHIP 2	89.94	-	-	-	-	132.2 t	-	134.5 t	-
NEWCHIP 3	78.75	-	-	-	-	119.8 t	18.0 t 9.0 c	129.7 t	11.5 t
NEWCHIP 4	92.19	-	-	-	-	94.3 t	23.5 t 1.0 c	101.4 t	5.5 t
NEWCHIP 5	62.13	99.9	34.1 c	36.1 t	52.3 c	112.5 t	31.4 c	85.2 t	6.2 t
NEWCHIP 6	111.50	100.7	45.1 c	37.9 t	110.5 c	59.4 t	27.7 c	14.9 t	3.1 c
NEWCHIP 7	34.06	29.4	14.7 c	22.9 t	38.7 c	-	-	-	-
NEWCHIP 8[1]	53.31	42.0	58.5 c	-	-	-	-	-	-
NEWCHIP 9[2]	47.13	37.2	50.9 c	-	-	-	-	-	-
NEWCHIP 10	57.00	19.0	12.4 c	9.1	34.5 c	-	-	-	-
NEWCHIP 11[2]	48.31	21.1 t	19.2	-	-	-	-	-	-
NEWCHIP 12[2]	56.50	76.3	92.3 c	-	-	-	-	-	-

TABLE 8.11. Stresses measured during extrusion tests by the use of strain gauges.
Positions of strain gauges vary from table as follows:

1 Readings were at 30 mm from throat of die, not 20 mm as labelled
2 " " " 40 mm " " " " " 20 mm " "
3 Readings taken on curing tube only at a) 40 mm b) 140 mm and c) 240 mm

and that is Newchip 6 in which cure of the binder was induced in order to prepare specimens for orientation investigations. This has clearly influenced the results.

Some alternative explanation must exist for the discrepancies between the other results, however, and after considering all possibilities it is felt that the logging frequency is likely to be the major cause. Most runs were monitored at a logging frequency of 4 Hertz, which at a ram speed of 40 mm per second allows 10 mm of ram travel between readings. Examination of the results from Newchip 12, which were logged at 8 Hz, shows that pressure changes of almost 70% can be seen over a ram travel of 5.6 mm. Furthermore, over that part of the ram travel where maximum pressure is generally observed, 175 - 190 mm, the pressure can rise by that 70% and then fall again to its previous value in the space of 0.375 seconds. Over that same period the ram strain gauge readings also vary by some 45%, thus it is easy to see how the discrepancies in the results can occur. Although the logging process was made automatic to minimise errors in the timing of the logging process, it is clear that a small variation in the time at which each log is made can cause some considerable error in the parameter values obtained. Similarly the differences in the ram position at maximum pressure/strain gauge reading are of the order of the distance travelled between log cycles and therefore could also be attributable to the same cause. Clearly in any future work of this nature, much faster logging times, and the consequent changes in computer hardware and software will need to be considered.

8.4.2.2 Observations from the results

Notwithstanding the considerable uncertainty regarding the accuracy of the results, each experiment has contributed some unique feature to the overall picture of the process detail. There are also some observations common to most runs which can form the basis of a discussion of the mechanisms operating within the system.

1) The influence of curing tube length, and thus wall contact area, observed during earlier experiments was confirmed during this later work.

   Taking Newchips 1, 2, 3, 4, 9 and 11 using the 7.5° die, it is clear that increasing contact length results in increasing extrusion pressure. The internal consistency between the individual subgroups is sufficiently good to place some reliance on the results, although the single value for the 300 mm tube cannot be regarded with any certainty.

If it is assumed that the major force to overcome before extrusion can begin is that of static friction, then the maximum recorded pressure will occur at the point of incipient movement when the coefficient of static friction gives way to that of dynamic friction. By the use of simple geometry, and by knowing the dimensions of the equipment, the total contact length can be calculated. Table 8.12 below shows the contact lengths and the respective extrusion pressures recorded.

Contact Length (mm)	Extrusion Pressure (MPa)
86	47.72 ± 0.59
236	86.96 ± 5.87
386	104.63

TABLE 8.12. Contact length and corresponding extrusion pressure using the 7.5° die and standard lubricated furnish.

Although the results are ranked in the expected order, there is no obvious linear relationship between them as might have been predicted. This could be due to the differing maximum pressures causing different degrees of compaction within the furnish. This would then result in variations in yield strength of the solid plug and therefore add a second variable term to the pressure equation. Although this hypothesis would best be tested by determination of the density of the samples at a fixed point, this was impossible since the samples concerned contained no binder and were therefore not dimensionally stable once removed from the instrument. The hypothesis is supported, albeit somewhat less convincingly, by the results obtained from the circumferential strain gauges sited near the top of the curing tubes. These suggest that although the extrusion pressure only increases by an average of 20.3% with the increase in curing tube length from 150 mm to 300 mm, the radial stresses in the tube wall at corresponding distances from the die/tube interface increases by an average of 75.2%. The inference drawn from this is that at the higher applied normal pressure, the furnish transmits the force more efficiently and thus behaves more in the manner of a fluid. This in turn suggests that the voids and interstices which differentiate a granular mass from an homogeneous mass are becoming smaller or are disappearing and the density of the material is therefore increasing. It might be argued that an increase in frictional force should accompany any increase in radial force, thus making extrusion more difficult and not easier as is observed. The counter-argument to this is that with increasing compaction and therefore decreasing void space, the lubricant phase is likely to be forced to the surface of the mass, where it can act most efficiently in aiding extrusion. This is borne out by Figure 8.18, in which a substance can clearly be seen to have been squeezed from the interior of the wood particle removed from a sample of part of Newchip 1.

The feature of the axial strain against ram displacement results of Newchips 3 & 4 indicating that surface stresses fluctuate between tension and compression during a single ram pass is also present in the results of Newchip 5. In this latter case the change from tension to compression and back again is only relative and no value for the compressive stress is given therefore. Nevertheless this does mean that the effect occurs consistently

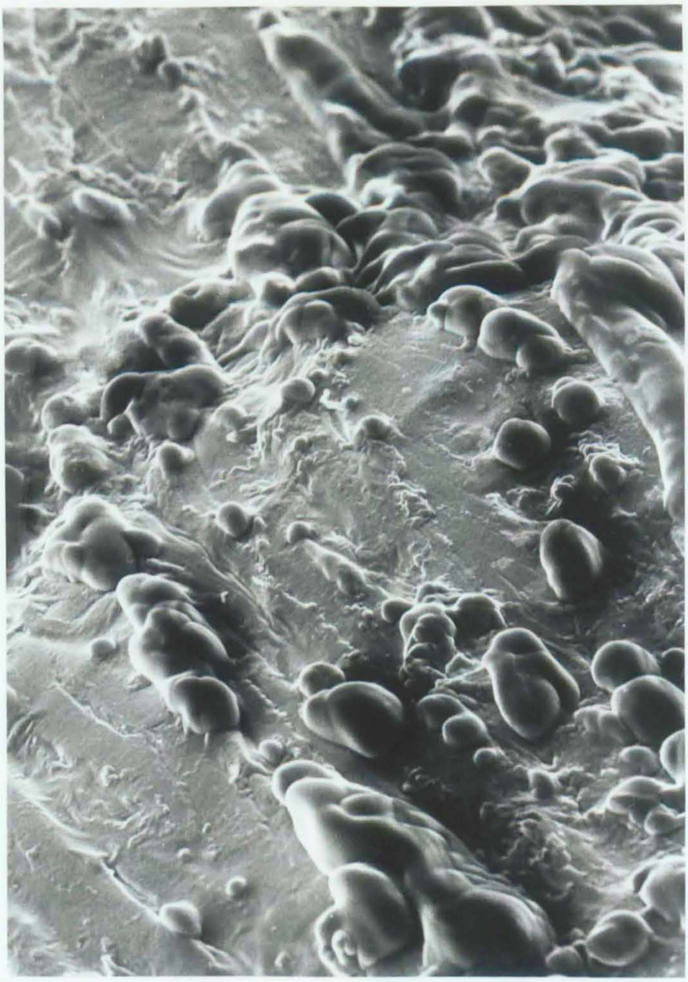

FIGURE 8.18 Electron micrograph of chip surface after extrusion showing substance exuding from cell structure.

in three comparable runs. Calculations based on ram position at the time of the change indicates that the compressive stress, which always precedes the build-up to maximum tensile stress, occurs immediately before the interfacial region between two hopper loads of material passes the measurement position. Since the magnitude of the effect cannot be explained by Poisson's ratio alone, this is interpreted as an indication that a bulge or bulges travel down the curing tube corresponding to interface region(s). The compressive strain would then occur as the concave outer surface immediately before the bulge passed the measurement position. The predictable peak tensile stress then follows as the convex outer surface passes the measurement position, but this is then immediately followed by a second, higher compressive stress. This clearly suggests that the bulge is almost hump shaped, and therefore that the interface region alone is responsible for the bulging, and not the entire slug of freshly extruded material. The residual stress in the wall after the interface has passed remains compressive however, but is then at a level which, allowing for the data logging inaccuracies, can be explained by the effects of Poisson's ratio. By the time the hopper has been refilled and the logging of the subsequent ram pass has been initiated (a period of between 20 and 30 seconds), this residual stress has once more reverted to a tensile stress. The explanation offered for this is that subsequent to the pressure of the ram being removed, the chip mass undergoes some form of elastic recovery. Since the residual stress does not merely subside to zero, but actually acquires a tensile value, the inference is that the recovery involves axial straining of the chip mass which not only relieves the radial pressure but also exerts an axial tensile stress on the tube walls, presumably via friction. Although the magnitude of the changes varies from run to run, it is felt that this should be associated much more with the logging inaccuracies than with the changes in die angle. This recovery theory also offers the most plausible explanation for the results of Newchip 6 not following the general pattern. For this run hardener was included in the adhesive formulation, and heat was applied carefully to the non strain gauged sections of the die by means of a hot air gun. Curing of the resin would therefore be initiated within the die, but from the information on resin cure contained in Table 8.4, and from a knowledge that the external die temperatures never exceeded 60°C, the final cure would almost certainly take place towards the end of the die or at the beginning of the curing tube. On this basis the explanations of the observed results are as follows:-

Although the exact temperature of the die in the area of the strain gauges was unknown, all gauges used were guaranteed to have a temperature/strain coefficient of less than 0.2 strain per degree centrigrade, thus over the range employed, 17°C to 60°C the maximum expected error would be of the order of 9 μstrain or 12.5% in the worst possible case. This was considered small enough to be neglected in view of the other inaccuracies involved in the monitoring system. This is especially true since all gauges would be at approximately the same temperature and should therefore be subject to the same temperature offset.

The values for radial stress in the pre-contraction section of the die are comparable in runs Newchip 5 and 6, which were carried out under similar conditions except for the presence of hardener and the application of heat in Newchip 6. This would suggest that the redistribution of the applied axial force is dissimilar in the two materials, since the axial pressure is 83% higher in Newchip 6 than in Newchip 5. A similar effect can be seen at the second radial measuring point, which corresponds to a level midway between the change in section and the exit of the die. Quantitative analysis of these results using Mohr's circle (see Section 3.2.2), or any of the other formulae noted in that section is not possible at a simple level because the material in these experiments is still undergoing a reduction in volume, a feature which none of the simple techniques can cope with. If the two systems are compared qualitatively on a parallel basis however, then from Rankine's formula (equ 3.2-4) it can be inferred that the angle of internal friction of the curing system is significantly greater than that of the non-curing system. This could be explained by incipient curing of some of the glue "spot welds" between the chips which would give the material a much higher level of internal bond strength, i.e. the material would act much more in the manner of a cohesive solid as described in the same section. Again this is not a property which can be quantified easily, since none of the traditional test methods can cater for a dynamic system such as this one.

There is some evidence from the measurement of the axial stresses in the upper part of the die that there is a difference in the frictional characteristics of the chip/metal interface. The results show a 32.25% increase in the axial stresses in this area for the curing over the non-curing system. Since the radial stresses are almost identical this cannot be attributed to a change in the normal force (see equation 3.3-1) and must therefore be a result of a change in the coefficient of friction. This is disappointing from a processing viewpoint since the inference is either that the efficiency of the lubricant is decreased at elevated temperatures, or, more likely that its action is interfered with by the curing of the resin. Nevertheless, the fact that an extruded product was obtained during this set of experiments indicates that the lubrication benefits are not completely lost.

The increasing internal friction factor, and the concomitant increase in apparent yield strength could also form part of the explanation of the results obtained from the axial strain gauge on the lower portion of the die. The stress measured here is over twice as great for the curing system as for the non-curing system. Although this will be due in part to the increased coefficient of friction mentioned above, it could also be explained by the transmission of axial forces through the particles to the ledge formed by the change in diameter, which would then exert a direct compressive force on the metal in the area below the change of cross-section. This aspect will be dealt with more fully in the next part of this chapter.

If, as was assumed earlier, the curing of the material is almost fully complete by the time the material enters the curing tube, then the conversion of axial to radial forces will be much reduced, and any tendency of the material to recover by either axial or radial straining will be inhibited by the binder system. These hypotheses fit the observed facts that the radial stresses in the curing tubes are much lower for the cured material than for the uncured, and therefore still elastic material. The axial stresses are also reduced for the cured material, and the value for the axial strain at the point most distant from the die is compressive and of the order of that which might be explained by the effect of Poisson's ratio alone.

The fact that residual stresses are present in the cured product is amply demonstrated in Figure 8.19, which shows a length of the product from Newchip 6 that has been slit slightly off the longitudinal axis. The shape of the resulting piece clearly indicates that axial residual stresses are present in the product. It is difficult to conceive how tensile stresses could exist in the surface layers except as a result of residual compressive stresses in the core, and such a stress system can easily be reconciled with the recovery activities described above.

2) The effect of die angle on extrusion pressure is no more consistent in these experiments than it was in the previous sequence, reported in Chapter 7.

Although the results using the 10° die in both sets of experiments give extrusion pressures within 10% of each other, the significance of this must be questionable since the lubrication additives are different in each case. In addition, decreasing the die angle from 15° to 10° in the second set of experiments has the opposite effect on extrusion pressure to that observed in the first experiments. The magnitude of the change was also lower than the 11% observed originally. Decreasing the angle

FIGURE 8.19 Demonstration of the residual stresses present in an extruded sample.

even further to 7.5° resulted in a further decrease in extrusion pressure of 11.7% compared with that measured when using the 10° die. The direction of both changes is predicted by Avitzur's basic equation (7.6-2), in which redundant work and the effects of friction are ignored. The magnitude of the total change predicted in this way is a decrease of only 0.12% however, compared with the observed value of 14.49%. Incorporation of the other terms into the equation would only serve to widen the gap further, as the discussions in Section 7.6.3.2.2 indicate that these terms cause a reversal in the direction of the predicted change from that observed here. On the other hand it is totally unrealistic to believe that conditions of zero friction and zero redundant work exist, and the cause(s) of the observed phenomena must therefore lie elsewhere.

Equation (7.6-2) is reiterated below:

$$WI = 2\pi \, \sigma_o \, V_f \, R_f^2 \, F(\beta) \ln \frac{R_o}{R_f} \qquad (7.6\text{-}2)$$

WI = internal work of deformation,

σ_o = equivalent yield stress of material to be extruded, V_f = velocity of emerging extrudate, R_f = radius of extrudate, and R_o = radius of original billet. F (β) has the same significance as in equation (3.4-7).

Inspection of this equation shows that within the terms of reference for the experiments detailed here, only $_o$ and V_f can be described as variables at any fixed die angle. It is also likely that these two variables would be inter-related, since the yield strength will determine the degree of compaction of the furnish immediately before it enters the die, and hence will determine the velocity of the final extrudate. The yield strength of the material surface, which need not be related directly to that of the bulk material, will also influence extrusion pressure through the level of wall friction generated, providing that the conditions of soft material/hard wall, as described earlier, prevail. Unfortunately the values obtained for ram position at maximum pressure are subject to the same inaccuracies as the pressure readings, due to the long logging interval. Inspection of Table 8.10 does show that the position does not appear to vary in any predictable way with die angle, however, and also that it is unaffected by the level of pressure observed.

Taking the three significant ram positions as d_1, the top of the hopper, d_2, the position at which maximum pressure occurs, and d_3 as the end of the ram stroke, then the compression experienced by the furnish is given by:

$$\text{compression} = \frac{d_3 - d_1}{d_3 - d_2}$$

d_1 and d_3 are constant for all runs at 65 mm and 202 mm respectively, thus the range of apparent compression ratios varies from 11.4:1 to 7.2:1. Although it would be possible to draw a considerable number of conclusions from the results and the apparent trends embodied in them, it is felt that in view of the large errors of timing in positional and hydraulic measurements it would be unwise to do so. It is thought far more likely that the observed differences in compression ratio are due to the monitoring system, and that there is unlikely to be any measurable difference between runs. This is borne out to some extent by the results of density measurements made on product from experiments carried out before the extended monitoring system was fitted. These showed no significant differences in the densities of product manufactured using different die angles.

One interesting set of results which was obtained during this series of tests, however, was that using the 90° die. The results of Newchips 5 and 6 were discussed in the previous sub-section, however it will be seen from Table 8.10 that Newchip 7 and Newchip 10 were also carried out using the same die and a similar furnish composition as in Newchip 5, but without any curing tube in place.

As would be expected in the light of previous discussions, the absence of the curing tube resulted in a drop in extrusion pressure. The magnitude of the change and the associated strain gauge readings are not as would have been predicted, however, and therefore do require some additional explanation.

Again the inaccuracies introduced by the logging speed make drawing absolute conclusions difficult and the results and conclusions must be viewed with this in mind. It does appear, however, that the results of the experiments can be grouped into two pairs, with Newchips 5 and 7 as one group and newchips 6 and 10 as the other.

The runs in the first of these groupings, 5 and 7, were carried out using freshly prepared furnish containing no adhesive material and were therefore typical of the runs carried out with other die angles. Althugh it was expected that the abrupt change of section would result in extrusion pressures greater than those observed with lower die angles, it can be seen from Table 8.11 that in fact the opposite is true.

From the elementary extrusion theory outlined in Chapter 3, it is clear that with such a sharp die angle, considerable redundant work would be anticipated, or a "dead zone" of non-extruding material would form in the right angle thus effectively lowering the die angle from the nominal 90°. Which of these two would occur would depend largely on the wall friction, according to Kalpakjian (5), with high friction favouring the formation of the dead zone and low friction a high proportion of redundant work. Pearson (6), carried out considerable work on this subject in 1952/3 and produced broad ground rules describing the action of various materials and with varying levels of friction. The basic results of this work are summarised in Figure 8.20. In order to compare these results with the behaviour of the materials under test in this investigation it was necessary to examine the flow of material within the die region of the equipment. The X- ray technique described in Section 8.2.3 was used to examine the product from Newchip 6, and the material in the die region was deliberately allowed to harden in-situ at the end of the run so that the flow pattern in this area could be assessed.

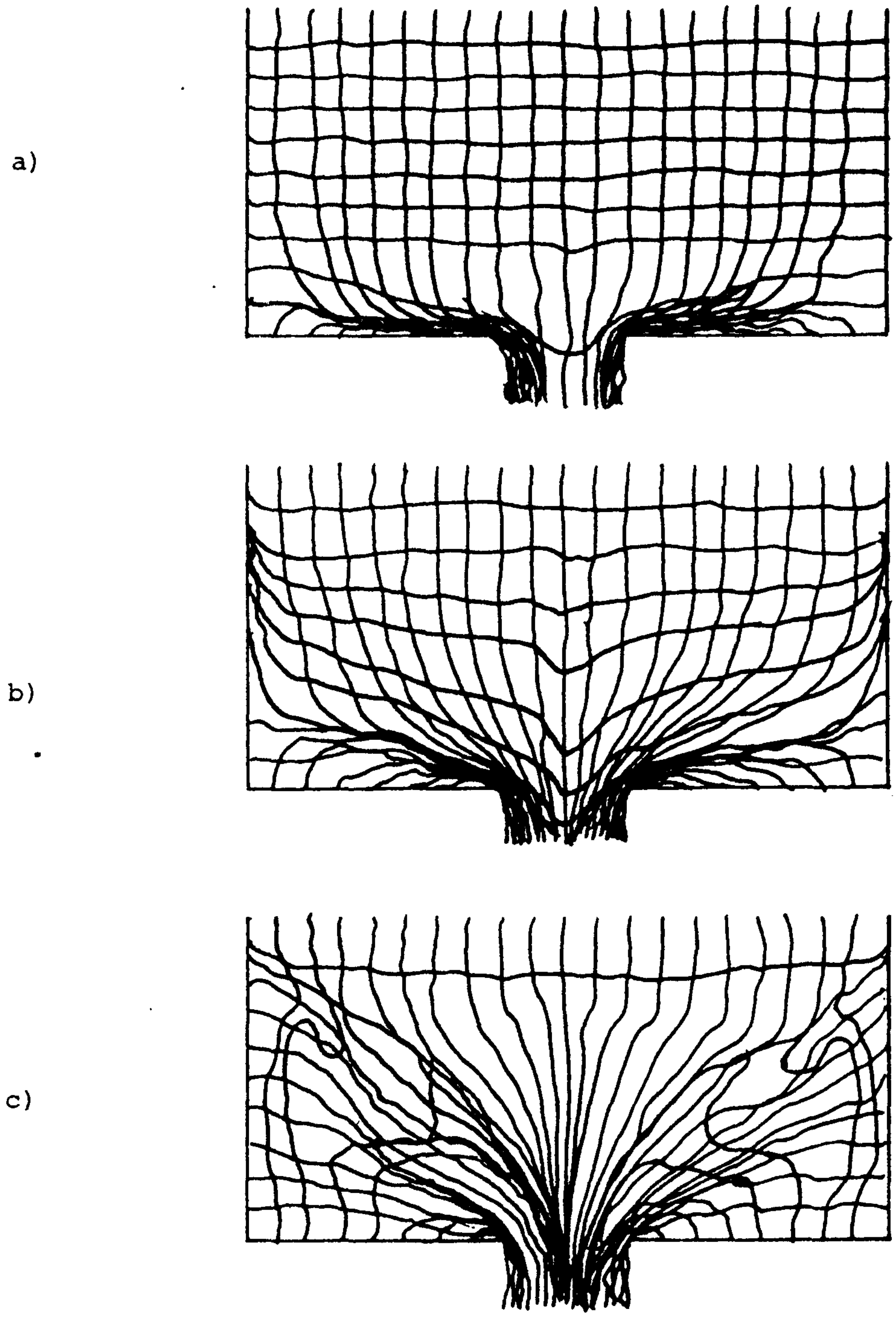

FIGURE 8.20. Grid deformation patterns for a variety of friction conditions. a) homogeneous material - low wall friction; b) homogeneous material - high wall friction; c) non-homogeneous material - high wall friction (Pearson (5))

Figure 8.21 shows the material from the die section as removed and before sectioning. There appears to be no dead zone and the angle at the change of section can be seen to be sharp. During closer examination, however, the portion of material shown in Figure 8.22 became detached from the main body, leaving the material shown in Figure 8.21 in the state illustrated in Figure 8.23. It is perfectly clear from this photograph that a dead zone has formed at the change of die cross-section, and that the shape of the true "die" surface at this point is almost an arc of a circle. What is not clear from the photographs is that the radius of the curve is not constant around the circumference of the material, and this effect is shown more clearly on the skiagraph of a section through the material shown in Figure 8.24. The reasons for this effect are unclear since the die, hopper, and ram are all almost perfectly symmetrical, and the surface finish of the contact areas is the same at all points. That the asymmetry persists through the die and into the product can be seen from Figure 8.25 which shows the surfaces at the split line between the die and the curing tube, and also from the skiagraph of the product which appears later in this Chapter.

FIGURE 8.21 Die section of extrudate produced using 90° die and 2.05:1 extrusion ratio.

FIGURE 8.23 Extrudate from Figure 8.21 with section from Figure 8.22 removed.

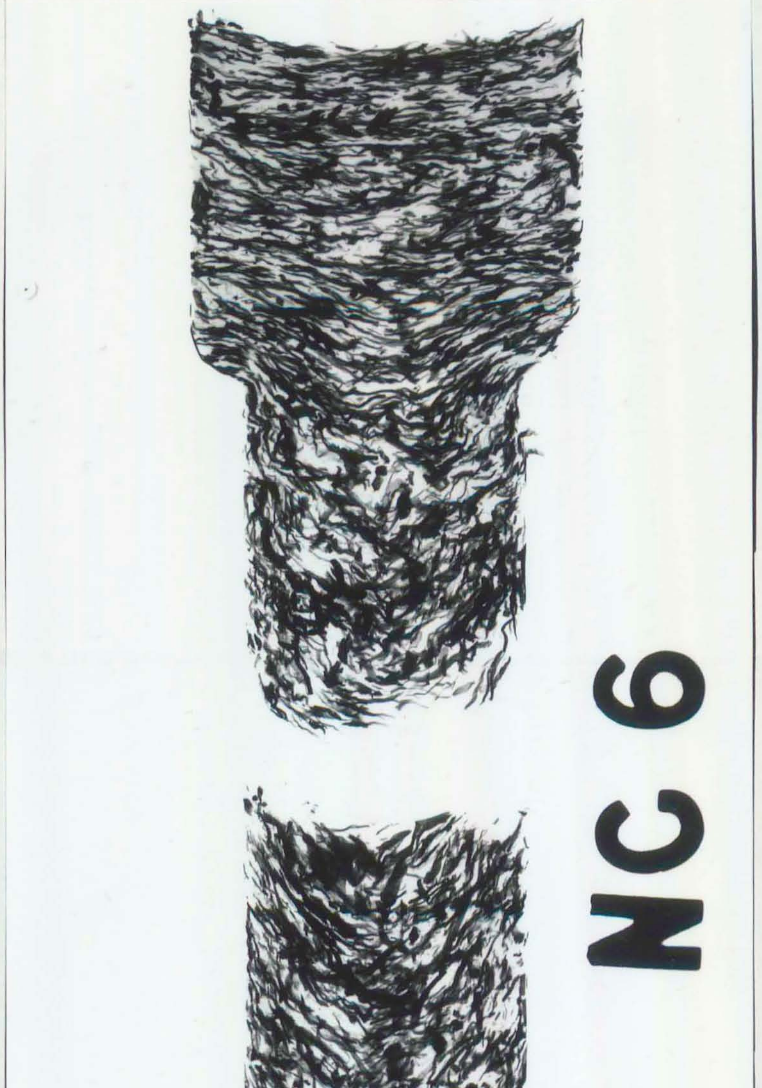

FIGURE 8.24 Skiagraph of sample shown in Figure 8.23

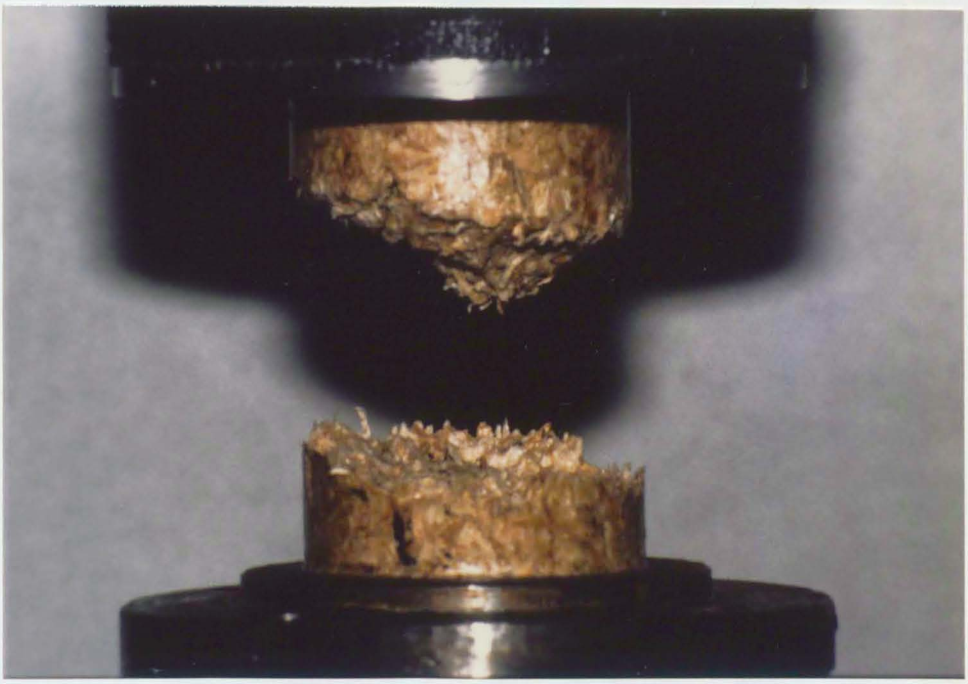

FIGURE 8.25 Section at split line of extrudate shown in Figure 8.21, illustrating formation of offset conical interface.

The visual results mentioned above all compare favourably with the results of Pearson, (Figure 8.20.b), which suggests that the material at this point in the equipment is behaving as a homogenous plastic material under conditions of high wall friction.

If this is the case, and since the density measurements mentioned earlier indicate that the material is probably in the same state no matter what experimental die angles are used, then this offers an explanation for the lower extrusion pressures observed with the 90° die. If the friction between material and die wall is high, as suggested, then the formation of a zone in which the friction is lower, (if the internal friction were not lower than the wall friction then the formation of such a zone would be energetically unfavourable), will clearly lower the resistance to flow through the die, and thus the overall extrusion pressure. With more accurate and more complete data, it ought to be feasible at this point to test the fit of the observed results with theoretical predictions. Since the data is to be considered at best only very approximate however, it is felt that such an exercise at this stage would be of no advantage to the investigation.

For an explanation of the abnormal behaviour of Newchip 6, the description of that run offered earlier to explain the anomalous strain gauge results can be called upon again. The curing of the binder within the furnish is likely to alter both the yield strength of the chip mass and the frictional characteristics of the material/die interface. These changes in turn would be reflected in the value of extrusion pressure developed, although the position of the die strain gauges may not be such as to enable any redistribution of forces to be detected directly. Newchip 10 might be expected to exhibit similar characteristics to a lesser extent, since the furnish used for this run was deliberately allowed to age for 40 minutes before being used for the extrusion tests. Although the furnish contained no adhesive and there would therefore be no effects of binder cure, the water/lubricant solution would have considerable opportunity to be absorbed into the wood structure. This would be expected to affect both the yield strength of the mass of material, (since this is related to internal friction), and the external frictional characteristics of the chip mass, and thus have a similar, but lesser, effect on extrusion pressure as does binder cure.

3) Effects of the various parameter changes on chip orientation were assessed using the doped-chip radiographic approach described earlier in Section 8.2.3. Owing to the difficulties presented in carrying out strain gauge measurements at elevated temperatures, only Newchip 6 was prepared in this way. Three subsequent runs, titled Woodchip 50, 51 and 52 were carried out using the same furnish as for Newchip 6, but using the 15° and 10° dies and curing tubes of 150 and 300 mm length with the strain gauges removed. No data was collected from these runs but care was taken during each run to ensure that sufficient product of an adequate quality and consistency was produced to enable X-ray examination to be carried out. The results of these three runs are shown in Figures 8.26 to 8.28.

Although the use of an engineer's protractor for the measurement of orientation angle was only accurate to ±1°, there do appear to be measurable differences between the chip orientations in the products from runs using different die angles. Woodchip 50 shows the results using the 15° die and 150 mm curing tube. Although further errors are introduced by attempting to choose representative areas of

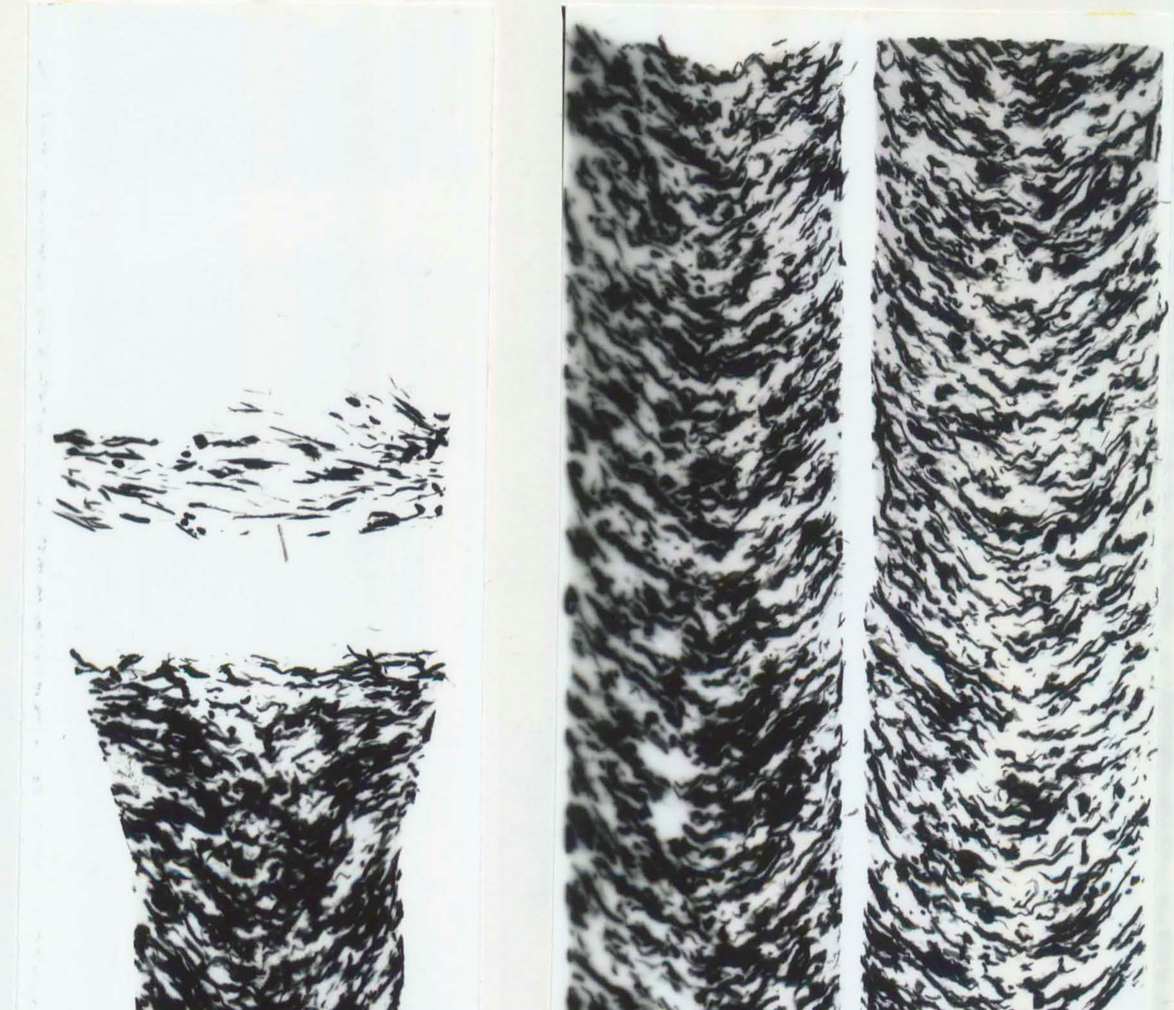

FIGURE 8.26 Skiagraph of extrudate from Woodchip 50.

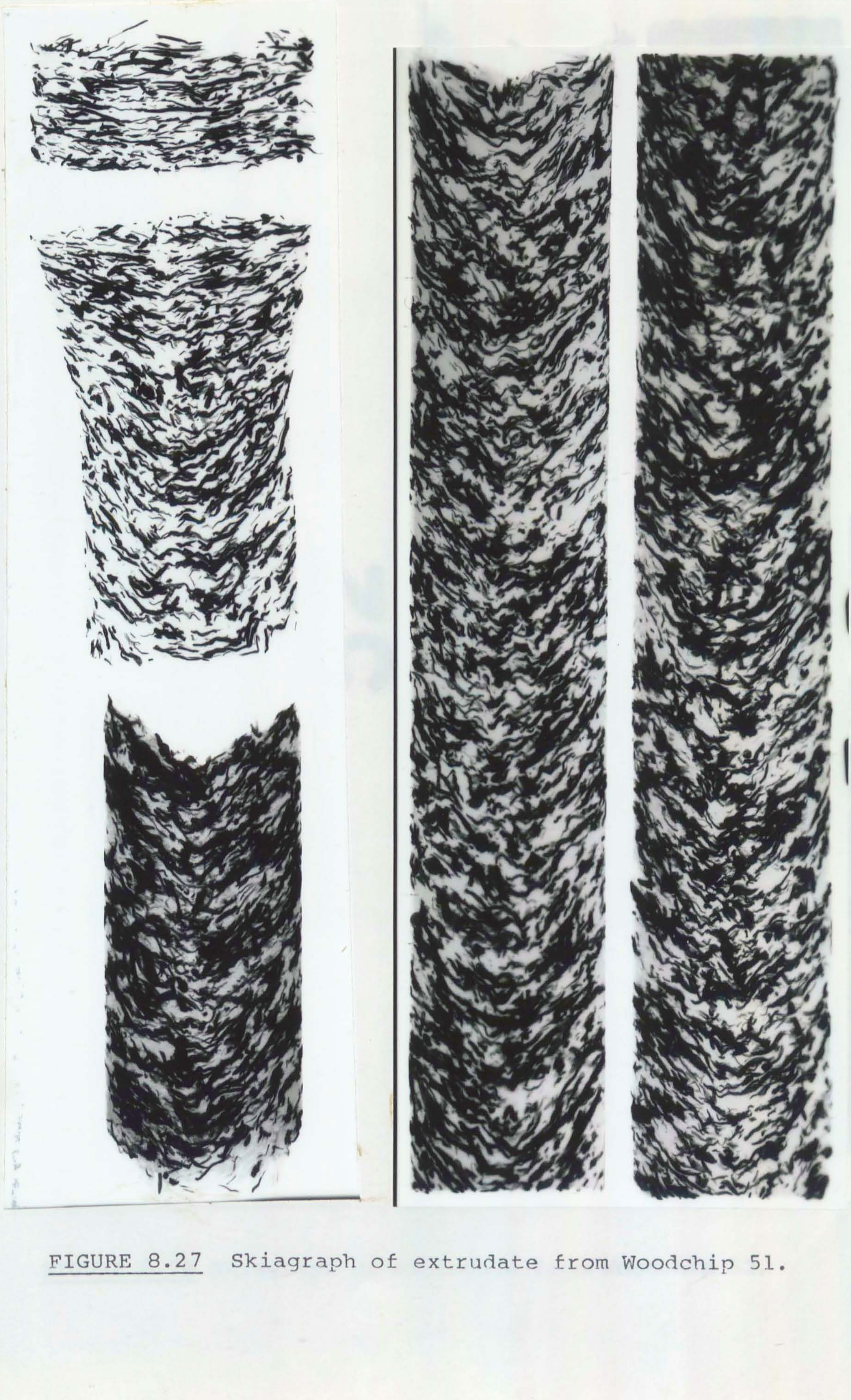

FIGURE 8.27 Skiagraph of extrudate from Woodchip 51.

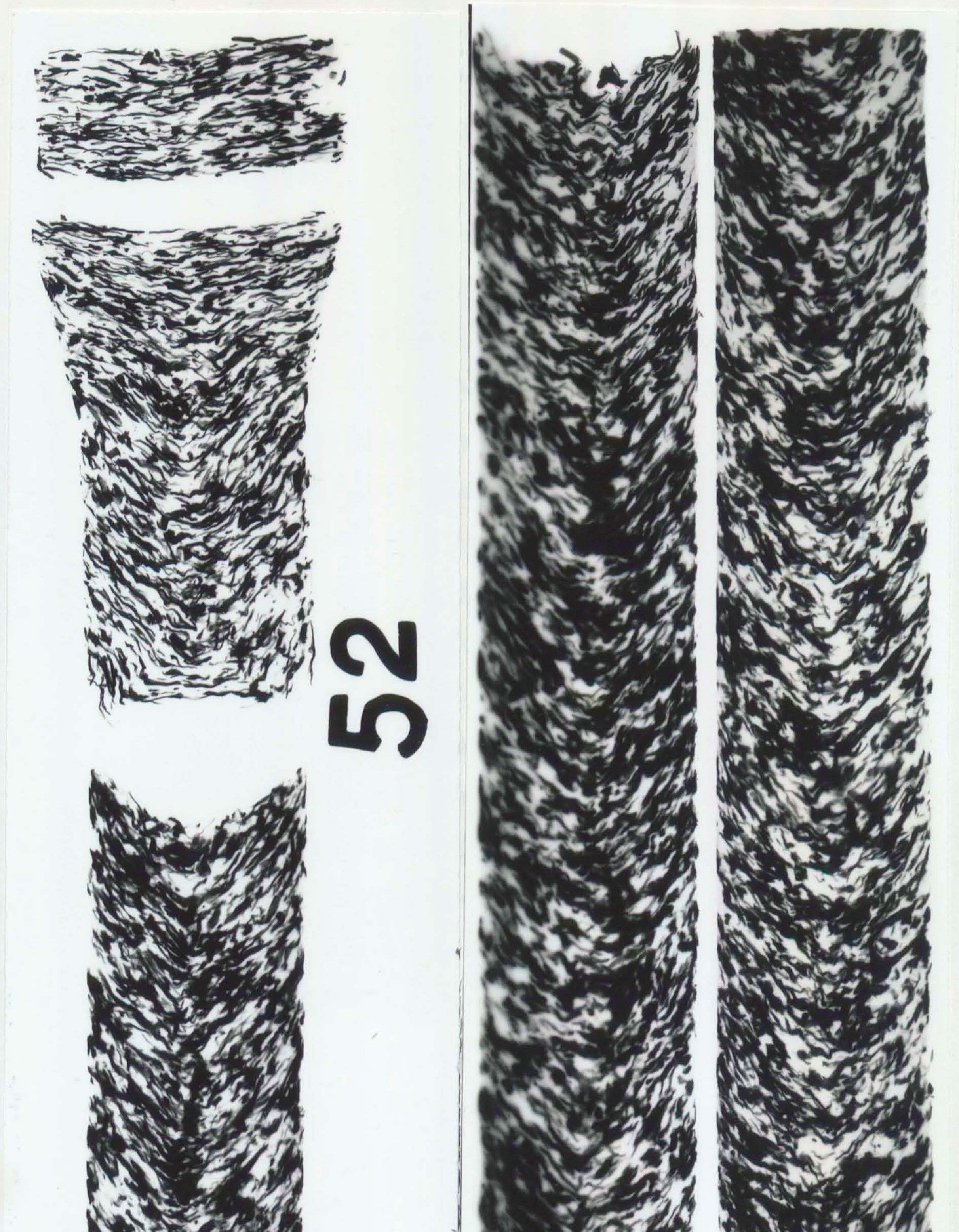

FIGURE 8.28 Skiagraph of extrudate from Woodchip 52.

the print on which to take measurements, it would appear that the average angle of orientation is of the order of 95°. In comparison, the products from Woodchip 51 and 52, both manufactured using the 10° die, appear to contain a chip orientation angle of about 90°, again subject to the actual measurement location. These results are contrary to results presented previously in which no relationship between chip orientation and die angle had been observed. Although the quality of the X-rays is not perfect, it is felt that the measurement technique is no less accurate than the technique used previously, and that the results must therefore be regarded as valid. Furthermore because the tests were carried out specifically to investigate orientation effects and therefore care was taken to ensure that the product was manufactured in a wholly reproducible manner, there is reason to suppose that these latter results are more representative than those obtained previously. Obviously further investigation of this aspect of the work in isolation is required before any formal mathematical description of the mechanisms involved can be produced.

In spite of this the results of these experiments illustrate very clearly how the orientation within the product develops as the material progresses through the die.

Difficulties in extracting the hopper portion from the equipment after the Woodchip 50 run resulted in the poor correlation of that part of the result with those from the second and third tests. These difficulties arose because the ram pressure was inadvertantly released before the resin cure was complete, and lifting of the ram caused delamination of the plug structure. Nevertheless three discrete stages of compaction can be observed on the corresponding skiagraphs. At the end of each ram stroke there is a plug of material between the end of the ram and the mouth of the die. This has been fully compacted and the chip orientation is uniformly parallel to the ram face. This orientation is representative of that observed in commercially produced extruded particleboard, and is responsible for the low longitudinal bending strength shown by such products.

As this pre-compacted material is forced into and through the die by subsequent charges of furnish, it can be seen that re-orientation of the chips begins almost immediately the chip mass enters the tapered region. It appears that from a relatively uniform density profile (as judged from the distribution of X-ray opaque chips in the skiagraphs), the central core of the material becomes somewhat denser as the orientation takes place while the surface annulus appears less dense. The most plausible explanation for this observation is that as the orientation begins, chips in contact with the walls are retarded by friction, while those which are only in contact with other chips are freer to move under the radial compressive stress exerted by the converging die walls. This mechanism corresponds well with the observations regarding residual stresses and relaxation mentioned earlier.

This inward radial motion of the chips towards the axis of the die could also explain the phenomenon, visible in all three products, of a region at the centre of the extrudate in which chips appear to be distorted longitudinally and even to travel backwards against the extrusion direction. An explanation in which chips converging radially meet at the axis and then buckle under the continued radial stress would fit the observed patterns.

Stage three of the orientation process occurs over a length of several tens of millimetres from the exit of the die through the start of the curing tube. In this region the buckled central core appears to undergo further re-orientation, perhaps as a result of recovery processes, or alternatively as a result of further retardation of the surface layers by friction effects at the wall. The buckling becomes very much less obvious as the material passes on through the curing tubes. Evidence of both processes was supplied by the results of the strain gauge experiments documented earlier, and it is therefore likely that the observed patterns represent a combination of both effects in some as yet undetermined proportions. As might be expected in a curing system, no further re-orientation effects are observed as the material passes through the equipment.

There are several other observations which can be made regarding the results of these experiments:-

a) Although the final product exhibited the inter-shot weaknesses described previously, there is no evidence from the skiagraphs of any regularly occurring structural differences which might correlate with such a feature.

This suggests that although the phenomenon is thought to result from the lack of mechanical interlocking at these points, it might also be due to differences in the distribution or curing of the binder at the interface caused by the action of the ram surface.

b) It is clear from the skiagraphs that although the products from runs 50 and 51 appear to have a similar density, that from run 52 has a noticeably higher density. This confirms observations made earlier that die angle appears to have no influence on product density. Since the length of the curing tube affects extrusion pressure, and therefore compaction, it would be expected that product density would be affected in a similar way. This observation was confirmed by measuring the apparent density of the product from the three runs, the results of which are shown in Table 8.13.

RUN	RELATIVE DENSITY
WOODCHIP 50	0.8
WOODCHIP 51	0.8
WOODCHIP 52	0.9 - 1.0

TABLE 8.13

Apparent relative densities of extrudate from three runs using a curable binder system. Results determined by weighing samples of material whose volume had been calculated from measurement of dimensions.

c) There are portions of samples from each run which appear out of focus in the skiagraphs. Since the X-ray cabinet used was of totally fixed dimensions and layout, this artefact must be a property of the material and not of the photographic process. There is no apparent pattern to be found in the results, hence the cause must also be a random feature of the experiments. For the extrusion process the only totally random event is the taking of the sample quantity from the mixing vessel and the subsequent filling of the hopper. The furnish preparation stages were rigorously controlled as detailed in Chapter 7, and it is felt unlikely that there would be any variation in the quality of the furnish produced at any one time. The only other explanation for the artefacts is that some part of the product sectioning and sample preparation process is responsible. Again the process was carefully controlled at every stage with the exception of the initial use of the bandsaw to section the extrudate and cut the samples to length. It is therefore possible that the randomly generated heat produced during this process caused some diffusion of the lead salts through the material across adhesive interfaces or possibly even a chemical reaction(s) which resulted in migration of lead from doped chips into adjacent areas of the product.

Both explanations are feasible since a certain amount of water would be available as the carrier medium even after cure was complete. However since the mechanism, and the result, are unimportant in terms of the studies documented in this thesis, and any elaboration of the hypotheses would require considerably more experimental work to be carried out, further progress in terms of an explanation was thought unnecessary and the subject was not pursued.

Although there are clear indications of positive links between processing conditions and process/ product performance, the analytical data collected which it was hoped would be utilised to formulate rigorous mathematical descriptions of the mechanisms involved was disappointly inadequate. The process of extrusion has been shown to be effective in terms of the manufacturing aims of the project, however, and it is felt that relatively minor improvements to the monitoring system would be required in order to fulfill the second aim.

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CHAPTER NINE - CONCLUSIONS AND RECOMMENDATIONS FOR FURTHER WORK

The work documented in this thesis had two complementary aims, viz:-

1) to investigate the potential for, and the viability of, the use of a novel processing approach to the production of useful composite end products from "waste" cellulosic starting material,

2) to attempt to gain an understanding of the physical processes occurring during such an operation.

Although the work in raw materials is common to both of these aspects, it will be dealt with under the production section, since this provided the set of constraints with which the materials were defined.

9.1. Conclusions - production aspects

Based on information from the literature and from discussions with colleagues, a set of guidelines was drawn up which defined the nature of the raw material being sought:-

1) it must be readily available since $6.5m^3hr^{-1}$ of wet material was considered to be the minimum for

a realistic commercial venture.

2) it must be cheap, since the product will probably be competing with sawn, dried, machined timber.

3) for structural uses the product would require adequate tensile and longitudinal bending strengths. Taking particleboard as the closest analogy, this implies that the particles used should have a relatively high aspect ratio, and some inherent strength of their own.

4) to keep pre-processing to a minimum the raw material should be as clean and pure as possible.

In Europe, North and South America, Australia and parts of Africa, the most likely sources of material fulfilling these criteria are roundwood, (unprocessed timber), and wood waste from woodworking operations. These and other alternatives were considered in depth in Chapter 4, and a decision was made to pursue the standing timber option on the basis of both cost and opterational efficiency.

The second component required in order to assemble the wood particles into a structural material was a suitable binder or adhesive. Again a set of constraints was imposed on this material:-

1) the material should preferably be relatively low cost.

2) it should be efficient in its operation so that addition levels can be kept as low as possible.

3) it should form bonds which meet with the durability requirements of the end-use envisaged for the composite material.

Again the options considered are detailed in Chapter 4, and on the basis of the information contained there, urea-formaldehyde resin in powder form was chosen as being the most appropriate for this study.

Having established outline raw material specifications, the processing route was then considered in detail. Based on experience with particleboard manufacture and considerable information from published literature and patents, the particle preparation process detailed in Chapter 5 was arrived at. The particle size chosen was typical of that used in medium density particleboard, and was shown by experimentation to be capable of reproducible manufacture from the raw materials available.

The various processing routes from such a mixture of raw materials to a finished product were then considered in detail, as documented in Chapter 6. Since compression moulding was already a well-established technique, and all other original alternatives were impractical for a variety of reasons, extrusion was considered to be the only process suitable for further work. Both screw extrusion and ram extrusion were considered and the decision to follow the ram extrusion route was based on the arguments laid out in Chapter 6.

Learning from the very many failures which occurred during the course of experimentation the raw material formula given in Table 7.1 was shown to display adequate characteristics. Despite the use of larger and more powerful extruder systems it was found that with this standard formulation, extrusion was consistently unattainable. Details are included in Chapter 7 of the attempts to rectify this situation with the use of lubricant additives. The lubricant finally chosen, Poly Ethylene Glycol of molecular weight 6000, was shown to have no detrimental effects on binder properties, but did enable extrusion to take place at manageable pressure levels.

Again, following practices common in the particleboard industry, a number of techniques were asssessed for raising the temperature of the product within the machine, and thus achieving resin cure. Although all three methods investigated, direct conductive heating, direct resistive heating, and radio frequency heating, proved capable of effecting resin cure with the formulation in use, technical difficulties with the application of the second and third techniques meant that only direct conductive heating was used to manufacture the product. Both of the other techniques would be suitable for a large scale production installation, but the costs of overcoming the problems on the pilot scale equipment were prohibitive. The direct heating equipment described in Chapter 7 was used therefore to produce samples of cured product which were subsequently subjected to necessarily very limited product evaluation testing.

Predictably, a relationship was found to exist between product density, hardness, and strength, and product density could be controlled in a coarse manner by varying the length of the curing tube and therefore the extrusion pressure in the system.

Although compression test results and values for nail holding and axial tensile strength were encouraging, a problem of product breakage at inter-shot boundaries was identified. During the course of the study this problem was alleviated somewhat by mechanical alterations to the machine, but the weakness was never eliminated completely, and would be a significant drawback to the use of the product in structural applications.

The orientation of particles within the product, which is presumed to give the product its high bulk strength and the absence of which has been a drawback in previous attempts at extrusion, (references to which can be found in the literature), was found to be largely independant of all of the process parameters with the exception of extrusion ratio. There are clearly limits at both extremes of extusion ratio which can be used, nevertheless further work in this direction might be beneficial in reducing the effects of inherent inter-shot bond weaknesses.

Up to this point detailed financial analysis of the process has not been entered into, however, the materials and the process are now sufficiently well defined to allow a simple costing exercise to be carried out.

Using the basic process described in Chapters 4 and 5 as models, the cost of raw materials and of a chip preparation plant can be evaluated.

Table 9.1 gives a breakdown of the major capital plant required, its cost, source, capacity, and power requirements for the production of 5880 tonnes of P_{10} R_{30} chips per year. Added to this is an estimated cost of £25000 for the actual extrusion equipment itself, resulting in a total capital investment of £354000.

Table 9.2 gives a breakdown of the running costs of the equipment per tonne of prepared furnish. It can be seen that the estimated cost of the finished product is £114.42 per tonne. If as was suggested in Chapter 4, the undersize fines from the chip preparation are used as fuel for the driers, then the electricity costs will be reduced and the cost of the finished product will be proportionately lower. In any event, it is clear from Table 9.2 that since labour, electricity, and raw materials costs make up over 70% of the cost of the finished product, plant location with its significant influence on these variables will be vital in determining the cost effectiveness of the operation.

Since bark makes up almost 17% of the total solid products from the raw material preparation, a brief survey of potentially profitable uses for this otherwise waste material was carried out. Although the traditional uses of bark for tanning leather are no longer viable, the use of enriched bark as a nutrient-containing soil conditioner had received considerable research attention. By treating the dried bark with the same urea-formaldehyde resin used in the extrusion process in addition to conventional nutrient additives, a slow release general fertilizer/soil conditioner is obtained. The costs

ITEM	SOURCE	CAPACITY	POWER	COST(£)
1) Hog-mill	Klochner	7.5m^3/hr	75kW	16K
2) Hammer mill + cyclone + ducting	Miracle Mills	1 tonne/hr	30kW	8K
3) " with 6mm screen	"	0.5 tonne/hr	30kW	8K
4) "	"	"	30kW	8K
5) 3mm screen "	"	"	30kW	8K
6) Resin/bark blender	Draisewerk	"	15kW	5K
7) Screen sifter	Locker Ind. Ltd	1 tonne/hr	1kW	6K
8) "	"	0.5 tonne/hr	1kW	3K
9) Chip drier	APV Mitchell	2 tonne/hr	5kW	80K
10) Bark drier	"	0.5 tonne/hr	3kW	50K
11) De-barker	Local	10m^3/hr	20kW	15K
12) Bagging Machine	"	300kg/hr	1kW	15K
13) Resin plant	"	40kg/hr	1kW	10K
14) Separators	"	-	-	6.2K=12K
15) Installation (inc. electrical & steelwork)				35K
16) Waste heat boiler + heat exchangers				50K
		TOTALS	242kW	£329000

TABLE 9.1 - Breakdown of production plant for chip preparation.

CAPITAL COST OF PLANT = £354000

OUTPUT OF PLANT = 5880 tonnes/year

RAW MATERIALS		COST (per tonne)£
Wood		11.00
Resin (for product and bark)		40.00
DIRECT EXPENSES		
Labour (10 men - 100K per annum)		17.00
Electricity (345 kWhr @ 5p per unit)		17.25
Depreciation (7% on capital)		4.21
Insurance (1% on capital)		0.60
Maintenance (5% on capital)		3.01
Direct factory cost	=	93.07
Factory indirects (10% of directs)		9.31
Total	=	102.38
COST OF CAPITAL		
Add to depreciation to make 20% return after tax over 10 years (from tables)		12.04
Total product cost	=	114.42

SENSITIVITY

a) Raw material cost + 50%,

product cost = 114.42 + 13.46

b) Plant cost + 50%, prod cost = 114,42 + 7.44

TABLE 9.2 - Costs involved in production of composite material.

of the extra resin and of the plant to dry, treat and package the bark were included in the figures given in Tables 9.1 and 9.2, thus any revenue obtainable from the sale of the product would reduce the cost of the finished extruded product still further. It is estimated that the selling price could be as high as £150 per tonne, and at a production rate of 400kg/hr, this represents a saving of approximately £60 on the cost of the extruded product, bringing the total cost down to roughly £54 per tonne.

Since the problem of inter-shot weakness had not been overcome, potential areas of use for the product were limited to those in which structural strength was of secondary importance. Initial product ideas included window and door frames where the uniform cross section lends itself to production by an extrusion route. The cost of these items produced conventionally from natural timber is of the order of £1000 - 1200 per tonne, which leaves a very signfificant profit margin based on the costings given above. The production of simple picture frame material which could either be left with the natural extruded finish, or could be stained or painted was also financially attractive since the conventionally produced article sells for around £2000 per tonne. This latter idea was considered in some detail by potential commercially interested parties, but could be taken no further for organisational reasons.

The ability to tailor the physical and chemical properties of the product by means of chemical modifiers incorporated at the blending stage also prompted commercial interest. The potential for rot proof decorative moulding aroused interest both for kitchen and bathroom use and for marine outfitting applications. Again organisational difficulties prevented the projects being taken any further than the conceptual stage.

At least as important as any financial implications of such a process are the potential benefits to conservation regimes. Since the margins on the product are so large, the usual financial constraints which prevent the use of very small diameter roundwood, due to the high proportion of bark involved, do not apply. In areas where timber resources are scarce, or the trees are only slow growing, the technology developed could be used to meet the demand for non-structural timber constructions with a significant increase in the efficiency of the use of the felled lumber. The horticultural/agricultural bark by product would also have potentially valuable applications in such areas. The possibility of tree cloning to produce forests from which the now acceptable small branches could be harvested without felling the trees themselves could also be very advantageous where trees are required for purposes other than straightforward timber production. It is also clear from the background literature quoted in Chapter 4 that useful products could be manufactured from other starting materials such as bagasse or flax shives where their abundance makes these more attractive raw materials.

9.2 Conclusions - Fundamental Physical Process.

The major conclusions reached at each stage of the research are documented and discussed in the appropriate chapters of this thesis, but an overall conclusion that can be drawn is that the shortcomings of the monitoring system mean that it is impossible to make meaningful interpretations based on the results obtained. The techniques employed have been shown to be relevant and useful in themselves, however, and only relatively minor alterations to the hardware and software would be required to obtain data of a much higher quality. Unfortunately, higher quality would inevitably mean greater quantity and the peripheral data manipulation and presentation systems would also require uprating as a consequence.

Notwithstanding the somewhat disappointing overall quality of these initial results, there are some significant trends which can be identified and which give insights into the behavour of the wood chip system under extrusion conditions.

Friction in all its forms plays a major role in determining the outcome of the extrusion process. Although there are numerous references in the literature to the effects of friction in both metal and plastic extrusion and in granular flow, no reference could be found to its effects in any system similar to that under investigation in this work. The major complication of this system is the change in the nature of the feedstock as it progresses through the process. Although by substituting values for friction into Avitzur's equation (ref. 56, Ch.3, and equation number 7.6.2) theoretical values close to those obtained experimentally can be calculated, the lack of knowledge regarding other terms in the equation renders this result of little direct use. Again it is felt that the dynamic nature of the material properties poses the greatest obstacle to successful evaluation of these parameters. Experiments to determine both the internal friction of the chip mass and the effective wall friction between the uncompacted material and the steel of the instrument gave results agreeing acceptably with previously published values. The applied pressure used during the experiments was too low to give results which could be substituted realistically into the extrusion equations, however.

Although it is very clear from the experimental results that the presence of a lubricant has a very marked effect on the extrusion pressure for a given system, and therefore that lubrication is clearly taking place, no evidence of a product/wall boundary layer could be found.

The X-ray examination of extruded product demonstrated dramatically that even at maximum density there was relative motion of chips within the compacted mass, which suggests that the lubricant effect was also significant at this level. It is felt that since the chips themselves deform early in the compaction process, this relative motion between chips might be equatable to a pseudo "yield strength" property and if quantified might form the basis of a more useful "constitutive equation" than exists at present.

Despite the fact that a moderate fit has been obtained between one set of results and Avitzur's equation, the other major variable contained in that equation, die angle, has been shown to have no predicable effect on extrusion pressure. All classical and theoretical work on this subject has been based upon materials of constant, known properties, and although mention is made of non-uniform plastic deformation in several instances, (references 52, 56 and 57 of Chapter 3), no attempt at describing the changing properties observed in this work could be found. In one of the references dealing with extrusion of a system very similar to this one, Mataki and Nakamura(1), found that the variation in vertical pressure across the die plane followed that predicted by the basic extrusion equations for rigid plastics(2,3), i.e. peak pressure at the centre and falling away towards the walls. They also claimed evidence of the effect of die angle on extrusion pressure as predicted by these theories. The die used in their experiment was of rectangular section and only produced plane strain in the system, nevertheless although the properties measured were different in each case, the vertical pressure profile across the die face could explain the observed orientation effects. The complicated interchip movements observed during this work by the use of the X-ray technique are not so simply explained, however, and clearly require more work before a satisfactory explanation can be given. The differences between the results of Mataki and those of this thesis with regard to the effect of die angle are major, and although the results of this work suffer from the logging rate induced inaccuracies, it is felt that this is insufficient to explain the observations and that the difference in shape of the die, or subtle differences in experimental parameters are more likely to be the cause.

Changes in die angle also appear to affect chip orientation to a slight degree, but the effect is very marginal and the magnitude of the effect so small that there are significant measurement errors involved. The direction of the change is as would be predicted from fundamental studies, i.e. an increase in die angle causes a decrease in the included angle of chip orientation, but moving from a die angle of 90° to one of 10° appeared to make less than 10° difference in chip orientation.

Extrusion ratio, on the other hand, appears to have a marked effect on chip orientation. From the 90° to 100° included angle generated with a ratio of 2.05:1, the included angle for an extrusion ratio of 1.1:1 drops to almost 0°. Extrusion pressure also falls dramatically moving from the higher ratio to the lower, and because the contact length of the system does not change significantly from one to the other, the extrusion ratio must have a direct bearing on the build up and transmission of radial forces during the experiments.

Although the few conclusions offered above are disappointingly general, and the goal of the theoretical section of this thesis - the production of usable constitutive equations describing the wood chip system - has not been achieved, the results are sufficiently informative to suggest avenues of future work which could be more profitable. These are detailed in the following section.

9.3 Suggestions for further work

On the theoretical side, the lack of published work on any closely related topics has indicated that a considerable gap exists between the use of mechanical techniques for the manipulation of the raw material and the knowledge of the underlying physical principles. Of the literature reviewed, that from the fields of civil engineering and soil mechanics comes closest to describing a similar system. In all cases, however, the particles in question have been rigid and incompressible and this has been the major stumbling block to using established theory as the basis for this work. The technique of finite element analysis has been used with some success in the investigation of elastic/plastic deformation of mechanical structures, and since this application is not far removed from the wood chip work, this would seem to offer the most potential as the logical way forward with this study.

Clearly some additional background work to determine material parameters more accurately would be required as a precursor to any finite element studies, and improvement of the data logging/manipulation system would be a necessary first step if the existing equipment design is to be utilised.

At the boundary between the fundamental physical processes and the realistic manufacture of a useful product there are several other observations which pose questions for future work.

The observation that the peak of the cone of fracture of the product is not on the machine axis requires an explanation, since nothing in the process, equipment, or raw material would be an obvious cause of the phenomenon. There is nothing in any of the results of Mataki et al, (1,4) to suggest that the effect might be pressure related, although non-uniformity of particle size distribution or moisture content might conceivably cause the effect.

Similarly no references have been found to the fluted or rippled nature of the fracture surface just visible in Figure 8.15. It is possible that this is the most energetically favourable means of incorporating the plastic deformation into the composite structure, but none of the results of this thesis offer evidence of the truth of such an explanation. This aspect could have significant commercial implications, since if better understood, the fluting/rippling effect might make some positive contribution to the problem of inter shot bonding.

The topic of inter shot bonding is in itself worthy of further investigation since this is an obvious weakness in the case for the exploitation of the process on a commercial scale.

The other area of work which could prove to have practical significance is the nature of the binder, specifically routes to faster, more efficient curing or perhaps the wider issue of whether the binder used is the optimum solution it was thought to be. Clearly the technologies of both conductive and radio frequency heating could be exploited more fully in the search for a more efficient process, particularly if scale up were possible. The ability to use a more natural adhesive product, perhaps a suitable timber resin, would doubtless be attractive for applications in those areas of the world where it could be supplied locally.

Thermoplastic binders such as polyvinyl acetate and copolymers based upon it might also offer an alternative to the current system. This would require an inversion of the current curing system, i.e. the wood chip/resin mix would have to be preheated prior to extrusion and cooled following passage through the die. Preliminary experiments of this type were carried out successfully, although the details of this work are not included in this thesis. The omission is deliberate since no records of the experiments were retained for security reasons.

As stated above, the work of Mataki et al does mention effects on pressure transmission of particle size and geometry, and although the chips used in this study were carefully chosen from experience with platen pressed particleboard production, it is feasible that research on the effects of particle geometry and size on extrudability would be beneficial to both the commercial and the theoretical aspects of the work.

Finally, and on a very much more speculative note, in the light of reported successes in the field of tree and plant cloning to produce rapid growing plants with specifically tailored properties, it is possible that the techniques could be applied to produce timber perfectly suited to use as extrusion raw material and which grows best in the conditions prevailing in the area chosen for the site. This would not only give commercial benefit, but could also be environmentally beneficial to the area in which such raw material was grown.

APPENDIX I

```
 100 GOTO190
 110 PRINT"❏":REM,DATALOG 1281CH BY J C WAHLERS 18 05 1984.
 120 REM,THIS PROGRAM IS WRITTEN TO RUN WITH THE 16 CHANNEL A TO D ONLY!!
 130 FORLL=0TO5:PRINT"❏":OPEN10,10:PRINT#10,"R00,R10,R20,R30":CLOSE10
 140 PRINT"THIS PROGRAMME IS DESIGNED TO LOG TO A  SEPARATE DATA DISC IN D1
 150 PRINT"S":PRINT"QQQQQQQQQQQQ":PRINT"PLEASE MAKE SURE SECOND DISC IS PRESENT"
 160 PRINT"OR YOU WILL LOSE ALL YOUR DATA!"
 170 FORFF=0TO500:NEXTFF:NEXTLL:PRINT"QQQ"
 180 PRINT"S":PRINT"QQQQQQQQQQQQQQQQ":PRINT"RPRESS ANY KEY TO CONTINUE":GETAA$:
FAA$=""GOTO180
 190 PRINT"❏QQTO SET THE RAM SPEED YOU MUST SELECT"
 200 PRINT"RMAINTENANCE█ BEFORE YOU SELECT RRUN█!
 210 PRINT"SQQQQQQQQQQ":PRINT"DO YOU WANT A RR█UN OR RM█AINTENANCE":GETQ$
 220 IFQ$=""GOTO210
 230 IFQ$="R"GOTO260
 240 IFQ$="M"GOTO270
 250 GOTO210
 260 RU$="RAM SPEED NOT CHECKED":GOTO630
 270 OPEN10,10:PRINT#10,"R01,R11":PRINT"❏RALL ELECTRONIC SAFETY INTERLOCKS ARE█
 280 PRINT"RNOW OVERIDDEN---BE CAREFUL!█"
 285 OPEN9,9:PRINT#9,"A,F1,I0"
 290 PRINT"SQQQQQQQQQ":PRINT"DO YOU WANT TO CHECK THE RAM SPEED Y/N":GETQ$
 300 IFQ$=""GOTO290
 310 IFQ$="N"GOTO340
 320 IFQ$="Y"GOTO360
 330 GOTO290
 340 RU$="RAM SPEED NOT CHECKED":GOTO520
 360 PRINT"❏QQQQQQQQQQQQQQQQOKAY--TAKE THE RAM TO THE TOP OF ITS"
 370 PRINT"TRAVEL , PRESS RR█ THEN REVERSE THE RAM
 380 GETQ$:IFQ$<>"R"GOTO380
 390 INPUT#9,X:X1=-X
 400 IFX1<1638THENGOTO390
 410 LT=TI
 420 INPUT#9,Y:Y1=-Y
 430 IFY1<4912GOTO420
 440 FI=TI:DU=(FI-LT)/60:RA=(INT(1000/DU))/10:RA$=STR$(RA)
 450 RX$="RAM SPEED ="+RA$:RU$=RX$+" MM/S"
 460 PRINT"Q                              ":PRINT"❏"RU$
 470 PRINT"QQ":PRINT"AGAIN RY█ OR RN█?"
 480 GETQ$:IFQ$=""GOTO480
 490 IFQ$="Y"ORQ$="N"GOTO510
 500 GOTO480
 510 IFQ$="Y"GOTO360
 520 PRINT"❏"
 530 PRINT"SQQQQQQQQDO YOU WANT TO SET THE LOWER RAM TRAVEL LIMIT ?":GETQ$
 540 IFQ$=""GOTO530
 550 IFQ$="N"GOTO600
 560 IFQ$="Y"GOTO580
 570 GOTO530
 580 PRINT"SQQQQQQQQOKAY - TAKE THE RAM TO THE REQUIRED POSITION THEN PRESS RS█
 590 GETQ$:IFQ$=""GOTO580
 592 IFQ$="S"GOTO594
 593 GOTO580
 594 INPUT#9,A:LS=-A
 600 CLOSE9:CLOSE10
 610 PRINT"❏QQQQQQQQQQWHEN YOU WANT TO START THE RUN PRESS RC█"
 620 GETQ$:IFQ$<>"C"GOTO620
 630 PRINT"❏":INPUT"WHAT IS THE NAME OF THIS RUN";N$
 640 PRINT"S":PRINT"QQQQ":PRINTTAB(6)"RDD MM YY█"
 650 PRINT"S":PRINT"QQQQ":INPUT"DATE";DD$
 660 PRINT"S":PRINT"QQQQQQQQ":INPUT"COMMENTS";CC$
 670 PRINT"SQQQQQQQQQQQQQQ"
 680 INPUT"APPROXIMATE LOGGING RATE REQ'D(4,6,8,10,12,14,16,18 /SEC ONLY)";DE
 690 IFDE=4THENQD=196:GOTO790
 700 IFDE=6THENQD=113:GOTO790
 710 IFDE=8THENQD=72:GOTO790
```

```
730 IFDE=12THENQD=31:GOTO790
740 IFDE=14THENQD=19:GOTO790
750 IFDE=16THENQD=10:GOTO790
760 IFDE=18THENQD=3:GOTO790
770 PRINT"▣▓▓▓▓▓▓▓▓STOP MUCKING ABOUT !! 4,6,8,10,12,14,16,18 AND NOTHING ELSE"
780 GOTO670
790 QD$=",QD"+STR$(QD)
800 PRINT"▩▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓"
810 INPUT"NO. OF LOG CYCLES REQ'D (I SUGGEST  RAM SPEED*LOGGING RATE)";NC
820 QS$=",QS"+STR$(NC):NC$=STR$(NC)
830 FL$=",D1,W"
840 SCRATCHD1,""+N$
860 SCRATCHD1,"DD"+N$
870 DOPEN#7,""+N$,D1,W:DOPEN#8,"DD"+N$,D1,W
880 PRINT#8,DD$CHR$(13)CC$CHR$(13)RU$CHR$(13)NC$CHR$(13)
890 DCLOSE#7
900 II=NC:TL=1
910 APPEND#7,""+N$,D1
930 PRINT"▣":PRINTTAB(10)"▓▓▓▓▓▓▓▓▓▓▓":PRINT"▨PRESS ANY KEY TO START LOGGING■"
940 OPEN10,10:PRINT#10,"R01,R10,R20,R30":CLOSE10
950 GETZ$:IFZ$=""GOTO950
960 AL=TI
970 PRINT"▣":PRINTTAB10"▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓":PRINT"▨LOGGING NOW■"
980 PRINT"▩":PRINTTAB10"▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓":PRINT"PRESS ▨S■ TO STOP!"
990 PT=TI
1000 PRINT"▩▓▨ DISPL     PRESS       TEMP      LOG NO■"
1010 TK=TI:AT=TK
1020 OPEN9,9
1030 CM$="AF1PA5000,I0I1I2"+QS$+QD$+"QE"
1040 PRINT#9,CM$:BE=TI
1050 PRINT#9,"A":IFST<>0GOTO1050
1060 EN=TI:LT=NC/((EN-BE)/60)
1070 BEEP1000,150
1080 PRINT#9,"PA5000,QI":CLOSE9
1090 FORTT=1TONC
1100 OPEN9,9:INPUT#9,A,B,C:CLOSE9
1110 Z$=CHR$(13):GOTO1140
1120 IF-A<6000GOTO1140
1130 OPEN10,10:PRINT#10,"R00":CLOSE10:GOTO1200
1140 PRINT#7,TT,Z$,A,Z$,B,Z$,C,Z$
1160 PRINT"▩":PRINT"▓▓▓▓":PRINT"                                                  "
1170 PRINTA,B,C,TT
1180 NEXTTT:PRINT"LOGGING RATE = "LT
1185 K$=CHR$(42):PRINT#7,K$,Z$,K$,Z$,K$,Z$,K$,Z$
1190 AT=AT+DL
1200 AM=TI:RT=(T-50)*60/(AM-LT):II=II+NC
1210 PRINT"▩▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓"
1220 PRINT"DO YOU WANT TO ADD TO THE FILE-▨Y■ OR ▨N■":GETQ$:IFQ$=""GOTO1210
1230 IFQ$="N"ORQ$="Y"GOTO1250
1240 GOTO1220
1250 IFQ$="Y"THENTL=TL+1:GOTO930
1260 PRINT"▣":TL$=STR$(TL):PRINT#8,TL$:DCLOSE#7:DCLOSE#8:DCLOSE#77
1270 PRINT"▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓▓":PRINT"DO YOU WANT A HARD COPY-▨Y■ OR ▨N■"
1280 GETQ$:IFQ$=""GOTO1280
1290 IFQ$="Y"ORQ$="N"GOTO1310
1300 GOTO1280
1310 IFQ$="Y"GOTO1410
1320 PRINT"▣":PRINT"▓▓▓▓▓▓▓▓▓▓▓":PRINT"DO YOU WANT ANOTHER RUN-▨Y■OR▨N■"
1330 GETQ$:IFQ$=""GOTO1330
1340 IFQ$="Y"ORQ$="N"GOTO1360
1350 GOTO1330
1360 IFQ$="Y"THENGOTO110
1370 GOTO1380
1380 XX=(S-S1)/60:II$=STR$(II):TL$=STR$(TL):APPEND#8,"DD"+N$,D1
1385 PRINT#8,II$
1390 DCLOSE#7:DCLOSE#8
1400 PRINT"▣":PRINT"SOD YOU THEN,I'M OFF.BYE BYE.":END
1410 PRINT"▣":PRINT"▨PRESS ANY KEY TO START PRINTING■"
1420 LR=60*I/(AM-PT)
```

```
1440 PRINT"■":PRINT"■■■■■■■■■":PRINT"■PRINTING NOW■"
1450 OPEN1,4:PRINT#1,"■"
1460 PRINT#1,CHR$(1)N$:PRINT#1,"■──────────────────────────────────────────────
1470 PRINT#1,DD$:PRINT#1:PRINT#1,CC$:PRINT#1:PRINT#1,RU$:PRINT#1
1480 PRINT#1,"LOGGING RATE";DE;"PER SECOND":PRINT#1:PRINT#1:PRINT#1:CLOSE1
1490 OPEN1,4
1495 PRINT#1," NO  DISP   PRES  TEMP
1500 PRINT#1,"***************************************************************":CLOSE
1510 OPEN2,4,2:OPEN1,4,1
1515 PRINT#2,"999  999.9  9999  999.9
1520 DOPEN#7,""+N$,D1
1525 FORJL=1TOTL
1530 FORJJ=1TONC
1540 PRINT"■":PRINT"■■■■■■■■■■■■■■"
1550 PRINT"I HAVE RETRIEVED "JJ*4" PIECES OF DATA"
1560 INPUT#7,AX,A,B,C:GOTO1580
1565 INPUT#7,AF$,BF$,CF$,DF$:GOTO1595
1580 GOSUB 1630
1590 PRINT#1,AX,T,P,TP:IFAX=NCGOTO1565
1592 NEXTJJ
1595 PRINT#1,AF$;BF$;CF$;DF$
1597 NEXTJL
1600 PRINT"END AFTER"II"ROWS OF DATA":DCLOSE#7:DCLOSE#77:CLOSE1:CLOSE2
1610 OPEN1,4:PRINT#1,"I HAVE PRINTED "II"ROWS OF DATA":PRINT#1,"■":CLOSE1
1620 GOTO1320
1630 T=-(INT((A/32.76411)*10))/10
1640 P=(INT((B/7.2860)*10))/10
1650 TP=(INT((C/3.36)*10))/10
1660 RETURN
```