

THE FLOW OF METALS, IN PARTICULAR LEAD,  
UNDER SIMPLE SHEAR.

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ABSTRACT

The creep of polycrystalline lead has been investigated under conditions of forward and reverse simple shear over a wide range of strain at different stresses and temperatures.

The forward flow was represented by a  $t^{\frac{1}{2}}$  flow at small strains succeeded by a  $t^{\frac{1}{3}}$  flow at higher strains together with a linear permanent flow until the onset of recrystallization. The transition from the  $t^{\frac{1}{2}}$  to the  $t^{\frac{1}{3}}$  law has been investigated.

The constants  $D$ ,  $B$  and  $K$ , which describe the  $t^{\frac{1}{2}}$ ,  $t^{\frac{1}{3}}$  and permanent flow respectively, were, at constant temperature, an exponential function of the stress  $\sigma$ , being represented by

$$D = H_D \exp (h_D \sigma)$$

$$B = H_B \exp (h_B \sigma)$$

and

$$K = H_K \exp (h_K \sigma)$$

where the constants  $H_D$ ,  $H_B$ ,  $h_D$  and  $h_B$  increased with rise in temperature. The stress dependence of  $K$  was obtained at only one temperature. The variation of  $B$  and  $D$  with temperature at constant stress was of the form,

$$D = P_D \exp (-Q_D/RT)$$

$$B = P_B \exp (-Q_B/RT).$$

Following recrystallization, a flow linear with time over a large range of strain was obtained. The stress and temperature dependence of the linear rate  $v$  was of the form,

$$v = v_0 \exp (h_v \sigma - Q_v/RT)$$

where the activation energy  $Q_v$  was independent of the stress. Reversal of the stress did not affect the linear rate. The relationship was valid only above a minimum stress  $\sigma_v$  which was a function of the temperature,

$$\sigma_v = W(1/T - 1/T_m)$$

where  $W$  is a constant and  $T_m$  the melting point.

The simple laws which govern the reverse flow after a pure  $t^{1/2}$  flow did not obtain when a linear component was present in the forward flow.

The stable grain diameter  $d_e$  varied with the temperature according to

$$d_e^{\frac{1}{2}} = L(T - T_0)$$

where  $L$  and  $T_0$  are material constants.

The creep below room temperature changed from the normal  $t^{\frac{1}{3}}$  flow at the higher stresses to a more complex flow at lower stresses which appeared to be a mixture of  $t^{\frac{1}{3}}$  flow and a flow of which the rate decreased with time to a limit zero.

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## I - INTRODUCTION

A material is said to creep when it undergoes progressive deformation under the action of an applied stress. In general, creep describes a time-dependent strain and may include elastic, anelastic and plastic deformations. Such deformations are particularly common in the creep of metals at high temperatures and are also observed in some nonmetals. The creep of metals, however, is essentially a crystalline phenomenon quite different from the flow of a material such as glass. The ability of metals to sustain internal stresses in crystals and crystal boundaries and the strong dependence of the creep properties on grain size, structure and previous mechanical history make the creep of metals a subject of great complexity.

Since the earliest experiments on creep attempts have been made to obtain empirical relationships between the creep strain and time. The method of measuring creep that has been generally adopted is to apply an axial load to a wire or rod and to measure the extension with time.

For experimental convenience creep data on engineering materials are often obtained under a constant tensile load. This results in a continuous increase in the true stress as the material extends, causing a marked effect on the rate of

creep. Because of the strong dependence of creep rate on stress, basic studies of creep phenomena should be performed under a constant stress.

The importance of using a constant stress was emphasized by Andrade (1910, 1914), who demonstrated that a number of pure metals showed similar creep characteristics when tested under a constant tensile stress. This led to the division of the plot of strain against time into three parts. In the first stage, which follows the immediate strain, the creep rate diminishes with time and then becomes constant during the second stage, after which it increases progressively during the last stage leading to rupture. The stages of creep are often referred to as primary (or transient) creep, secondary (or steady state) creep, and tertiary creep.

Andrade obtained a constant stress by using a weight in the shape of a hyperboloid of revolution which sank into a liquid as the wire specimen extended. A different weight was thus necessary for each load. Another constant stress device which has found extensive use is the Andrade-Chalmers (1932) beam which, for a given initial length of specimen, suffices for all creep loads.



The significance in engineering of time-dependent creep of metals was not given serious attention until after the experimental work of Chevenard (1919) and Dickenson (1922), who showed that creep fracture could occur in time at stresses much lower than the ultimate tensile stress. It was observed in the early work of Andrade on creep that metals with quite different room temperature properties exhibited similar creep behaviour at suitable stresses and temperatures. The creep was markedly affected by temperature, depending particularly on the ratio of the test temperature to the melting point of the material, known as the homologous temperature. Thus, for the same homologous temperature similar creep curves were obtained for iron and lead by choosing appropriate stresses (Andrade, 1938).

In general, the results of the early experiments on tensile creep at constant stress could be expressed by a  $t^{\frac{1}{3}}$  law which was first put forward by Andrade (1910).

$$l = l_0 (1 + Bt^{\frac{1}{3}}) e^{Kt}$$

where  $l$  is the length of the specimen at time  $t$ ,  $l_0$  is the initial length and  $B$  and  $K$  are constants. The exponential form of the  $K$  term is a trivial consequence of the increase in length, since  $\frac{1}{l} \cdot \frac{dl}{dt} = K$  if  $B = 0$ . The law represented the results closely over the range of creep up to the onset of the tertiary stage. The  $Bt^{\frac{1}{3}}$  component represents the rapidly decreasing rate of flow called transient flow and the  $K$  term is a flow linear with time called permanent flow, which describes the steady state creep. The  $K$  flow is sometimes referred to as quasi-viscous creep since  $K$  is not a linear function of the stress as would be the case for Newtonian viscosity. The creep behaviour of a particular metal is therefore determined by the relative importance of the  $B$  and  $K$  flow, which is governed by the temperature. At high temperatures the  $K$  term is predominant but at low temperatures the  $K$  term is negligible in comparison to the  $B$  flow.

Andrade (1914) suggested that the  $B$  flow was connected with intragranular glide processes and rotation of the crystal axes whereas the  $K$  flow was associated with intercrystalline movements. Evidence of the first point was obtained by Andrade and Chalmers (1932) who detected rotation of crystal planes during  $B$  flow by measuring the change in electrical resistivity. Later Gibbs and Ramlal (1934) succeeded in

measuring the rotation of the glide planes with the use of X-rays. Hanson and Wheeler (1931), working with polycrystalline aluminium, demonstrated the development of intragranular slip bands with a fast rate of strain or at low temperatures, whereas for slow extension at high temperature there was a broadening of the grain boundaries leading to the formation of intercrystalline cracks. It was emphasized by Orowan (1948) that K flow was not entirely a grain boundary phenomenon because it was observed in single crystals. Thus the B flow and the K flow appear to be interdependent phenomena. The effect of grain size on the creep of lead was studied by McKeown (1937) who found B flow to be predominant with a coarse grain size, whereas the K flow became progressively more important as the grain size was reduced. The value of K varied inversely as the grain diameter which is suggestive of a grain boundary effect.

The  $t^{\frac{1}{3}}$  law has been verified for a number of metals and alloys for, in general, strains above 2.5%, although a lower strain limit has been observed in certain cases. It was observed by Hopkin (1950) for the creep of lead over very long periods; by Feltham (1953, 1956) for iron and plain carbon steels, and for lead wires; by Feltham and

Meakin (1959) for copper; by Feltham and Copley (1960) for 90/10 brass; by Feltham and Myers (1963) for  $\beta$ -cobalt; and by Cottrell and Aytakin (1950) for single crystals of zinc. Wyatt (1953) observed a  $t^{\frac{1}{3}}$  law for the creep of pure metals (copper, cadmium and aluminium) at high temperatures and stresses, but at low temperatures and stresses the creep strain obeyed a logarithmic law. The  $t^{\frac{1}{3}}$  law is generally observed in the range of homologous temperature 0.2 to 0.7. Another form of creep called exponential creep has been obtained in the range 0.4 to 0.6 by Garofalo et al., (1963) for stainless steel and good agreement was obtained for the results of McVetty (1934) on ferritic steels. The same law was obtained by Andrade and Aboav (1964) for the creep of cadmium under simple shear stress when the stress was small. For larger stresses the cadmium obeyed the usual  $t^{\frac{1}{3}}$  law during the transient flow when the stress exceeded a certain value called the transition stress  $\sigma_c$  which decreased with increasing temperature, according to the law,  $\sigma_c = S' \left( \frac{1}{T} - \frac{1}{T_m} \right)$  where  $S'$  is a constant and  $T_m$  the melting point.

It has been observed in some cases that the transient flow could best be fitted by an exponent of the time not equal to  $\frac{1}{3}$ . Crussard (1958) analysed the flow of a

variety of substances, mostly metals, and obtained exponents somewhat larger than  $\frac{1}{3}$  which were not entirely independent of the stress. However, Andrade (1962) working with lead under simple shear, showed that an increase in the value of the exponent could be explained by the presence of a small flow linear with time in addition to the  $t^{\frac{1}{3}}$  flow.

In many tests under constant load the accelerating stage of creep is a direct result of the increase in true stress as the elongation increases. In other cases necking and various structural changes such as recrystallization (Greenwood and Worner 1939, Andrade and Jolliffe, 1952, Gilbert and Munson 1965) are responsible for the increased rate of flow. Such periods of accelerated creep are a common feature of the creep of lead which readily recrystallizes at room temperature.

Much experimental work has been done to relate structural changes during creep with the various creep stages, mainly by metallographic examination of the surface of metal specimens. In the case of creep under a tensile stress, however, the conditions of flow at the surface are not representative of the interior. The surface area of the specimen increases with strain and the rate of flow varies

across the section. The abnormal behaviour of surface grains has been demonstrated by Andrade and Kennedy (1951), by Rachinger (1952) and by Andrade and W.J.D. Jones (1962).

In previous work, (Andrade and Jolliffe, 1952, 1960, 1963) a method of investigating the flow of metals under conditions of constant simple shear has been described. The advantages of this method over the orthodox method employing rods or wires under tension are a simplification of the stress conditions, particularly with large plastic flow; the approximately constant planar surface area of the section under shear which permits useful metallographic examination of the surface during the test; the ability to reverse the shear stress at any stage in the flow process; and the large range of strain which can be attained before the specimen is ruptured.

Using both metal discs with an annulus and rectangular plates with two rectangular grooves, Andrade and Jolliffe have made a detailed study of the various stages of flow during the creep of polycrystalline lead under simple shear at 27°C. It was observed that the forward creep flow could conveniently be divided into three stages in which quite different laws applied. The first stage, FI, involved a

$t^{\frac{1}{2}}$  flow at strains above 0.01 and below approximately 0.025, which was followed by flow obeying a  $t^{\frac{1}{3}}$  law, which held until recrystallization occurred. Under favourable conditions of stress and temperature an additional permanent flow, linear with the time, persisted during stage FI. Hence,  $\gamma = C + Dt^{\frac{1}{2}} + Kt$  held for small strains and  $\gamma = A + Bt^{\frac{1}{3}} + Kt$  for larger strains, where A, B, C, D and K are constants. The second stage, FII, began when the onset of recrystallization led to an acceleration of the creep which was very marked at high stress. At a strain of 0.28 and above, depending on the stress, the flow became linear with time until the strain approached 2.0, when the specimen began to rupture. This range of the creep curve was called FIII. The constant rate of strain  $v$  varied exponentially with the stress according to

$$v = H_v \exp (h_v \sigma),$$

where  $H_v$  and  $h_v$  are constant at constant temperature.

The flow under reversed stress showed certain systematic behaviour when reversal of stress occurred following a normal  $t^{\frac{1}{3}}$  flow without the linear (K) component. The first stage on reversal, called  $R_0$ , was linear with the time

and prevailed for a constant fraction of the time of forward flow. Following this there was a normal  $t^{\frac{1}{3}}$  flow, stage RI, in which the  $t^{\frac{1}{3}}$  coefficient was proportional to and greater than that in stage FI. In stage RII there was an acceleration of creep similar to that observed in stage FII. The final stage RIII, attained only if the strain was large, consisted of a linear flow with the same creep rate as prevailed in FIII.

When the stress reversal was effected in the early part of stage FII the behaviour on reversal described above was considerably modified. The effect on the  $R_0$  stage was to diminish the duration and to increase the strain rate. The effect of prolonging the forward FII flow prior to reversal was to further decrease stages  $R_0$ , RI and RII, so that stage RIII began much earlier. Repeated reversals of the stress at intervals equal to the duration of the first forward flow produced a succession of alternate linear and  $t^{\frac{1}{3}}$  stages, with an increase of strain at each cycle.

The present work involved the shear of lead III discs at various temperatures and stresses to extend the range of observations made by Andrade and Jolliffe at 27°C. Important objectives were to find whether the  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  laws prevailed at different temperatures for a given range of strains and further to investigate the K flow and the linear time flow



in the non-Newtonian viscous state represented by stages FIII and RIII. If the laws valid at 27°C prevailed at other temperatures the variations of the constants A, B, C, D, K and  $v$  with temperature were to be found.

Extensive tests have been carried out at temperatures in the range -31° to 80°C. Measurements made at 150°C, which present certain special features, will be considered separately. Reversals of stress have been performed at different temperatures to ascertain whether a K component in the forward flow, which was absent or extremely small, in the earlier experiments at 27°C, influenced notably the reverse flow.

In order to observe the stress and temperature dependence of the flow constants it was necessary that the grain size should remain constant during the shear tests and be markedly less than the minimum thickness of the annulus. In this connection measurements were made of the stable grain diameter at different temperatures.

II - MATERIAL AND METHOD

Creep tests were performed on a lead, called lead III by Andrade and Jolliffe, which was tadanac lead with additions of 45 p.p.m. silver and 38 p.p.m. copper by weight conforming to B.S.S. 1085 (1946). The silver and copper were added to suppress grain growth and the onset of recrystallization under an applied stress. The tadanac lead contained not more than 10 p.p.m. of impurities.

The form of the specimens used for measuring the flow at temperatures above room temperature was that used by Andrade and Jolliffe. Lead discs approximately 16 cm in diameter and 1.1 cm thick were machined from blocks of the metal which had been carefully cross-rolled to avoid preferred orientation of the grains. One face of the disc was machined accurately flat and in the other face a concentric groove was cut with inner radius 4.80 cm and outer radius 6.00 cm. To keep the shearing stress constant at different values of  $\underline{r}$ , the distance from the centre of the disc, the thickness of the annulus was made to vary inversely as  $\underline{r}^2$ , a tool of special profile being used for the purpose (Andrade and Jolliffe I, 1952). The annulus thickness varied

from about 1.3 mm at the outer edge to about 2.0 mm at the inner edge.

Smaller specimens were prepared for measuring creep below room temperature to avoid the use of excessive loads. The small discs were 8.255 cm in diameter and 0.953 cm thick, and the annulus was machined with an inner radius 1.588 cm and an outer radius 2.858 cm. The thickness of the annulus was about 1.0 mm at the outer edge and about 3.2 mm at the inner edge. Four equally spaced slots, 0.953 cm x 0.318 cm, were machined in the periphery of the disc for accurate location in the apparatus.

Before testing, the specimens were normally annealed at 100°C for 16 hr, which produced an average grain size of 3 grains/mm, as measured by the linear intercept method (see Andrade and Aboav 1964, § 8). In addition samples of tadanac lead and lead III were used to determine the variation of the stable grain size with temperature.

III - EXPERIMENTAL1. Apparatusa) Method of Stressing

(i) Large Discs. The apparatus was essentially that used by Andrade and Jolliffe for the creep of lead at 27°C. (Andrade and Jolliffe I, 1952; Andrade and Jolliffe III, 1963). The outer part of the lead disc AA was firmly bolted to a steel framework CC by means of a steel ring DD as shown in figure 1. The centre portion of the disc together with a mirror attachment P was similarly fixed to a brass plate MM firmly attached to the end of a horizontal shaft ST. The shaft S was supported by two self-aligning ball-races mounted in plummer blocks bolted to a bed plate which also carried the specimen assembly. A pulley wheel 50 cm in diameter was attached to the other end of the shaft.

Loads could be applied to flat steel tapes on each side of the wheel so as to produce a known couple in either a clockwise or anti-clockwise direction. The load was applied by means of weights carried in a bucket suspended by the steel tape on each side of the pulley wheel. Each bucket stood on a screw jack which was driven by an

electric motor and the application and removal of load was achieved by lowering or raising the jack at a constant rate. A reversal of the load was effected by simultaneously raising the first load and lowering the second identical load in the opposite sense.

(ii) Small Discs. The apparatus for large discs was unsuitable for work at low temperatures because of the large loads required to obtain a convenient creep flow. The smaller disc shown in figure 2a was therefore used, as this required only about one sixth of the load on a large disc for a given stress. An apparatus was built on the same principles as the one used by Andrade and Jolliffe but the design was modified slightly. In this case the specimen AA was held in a horizontal position with its centre portion bolted to the shaped end M of a vertical shaft ST as shown in figure 2b. The periphery of the disc was prevented from rotating by locating the four metal stubs DD, bolted to the floor of the chamber CC, in the slots provided in the specimen. The chamber was welded along its rim to a rigid steel cover plate VV. The shaft S passed through a bearing KK held by the

support plate XX and was located at its lower end in a second bearing. The cover plate, support plate and lower bearing were all securely bolted to a rigid L-section steel frame which also served as a table for the strain-recording equipment.

A horizontal pulley wheel attached near the bottom of the shaft carried four steel tapes which passed over Hoffman bearings fixed to the steel frame. Two of the tapes, diametrically attached to the pulley wheel, were used to suspend the horizontal loading bar to which the desired load was added. The specimen could be stressed in the opposite sense by fixing the second set of tapes to the loading bar. The load was applied and removed at a constant rate by means of an electrically driven screw jack. In this way any possibility of the shaft bending was reduced by the application of a balanced couple.

b) Temperature Control

(i) Large Discs. To minimise heat losses an insulating plate E was inserted between C and the bed-plate, and the part of the shaft T was made of a tube of nickel steel, with the low thermal conductivity 0.030 cgs units. The whole specimen assembly was surrounded by a Sindanyo box F

packed with heat insulation material as shown in figure 1. The temperature control was such that a temperature in the region of  $27^{\circ}\text{C}$  was maintained constant to within  $0.1^{\circ}\text{C}$ . At the other extreme a temperature of  $150^{\circ}\text{C}$  could be maintained constant to within about  $\pm 1.5^{\circ}\text{C}$ .

To control the temperature a brass chamber was attached to the back of the plate which gripped the central part of the disc at MM and another chamber to the back of the plate which gripped the outer part of the disc at CC. Through these chambers a liquid was circulated by an electric pump from a large reservoir where the temperature was maintained constant by a mercury contact thermometer and relay. The liquid was heated at the required rate by means of a variable voltage applied to a 3 KW heater immersed in the reservoir. Water was employed for a temperature of  $27.5^{\circ}\text{C}$  and glycerine at higher temperatures. The temperature of the specimen was monitored by means of a chromel-alumel thermocouple clamped to the surface of the disc.

(ii) Small Discs. The specimen was heated or cooled as required by passing a liquid at the appropriate temperature through the stainless steel specimen-container F, in which

the level of liquid was constant, as shown in figure 2b. The liquid was pumped from a sealed can which was immersed in a large reservoir of the same liquid. A specimen temperature of  $27.5^{\circ}\text{C}$  was obtained by thermostatic control of water in the reservoir as described for the large discs. Temperatures down to  $-31^{\circ}\text{C}$  were obtained by replacing the water and the heater element with methylated spirit and a refrigeration unit respectively. A mercury contact thermometer was used with the appropriate range of temperature control.

The chamber surrounding the specimen container was packed with sheets of polyzote insulation E, a sheet of which was used to insulate the cover plate lid LL above the specimen. The conduction of heat along the shaft was reduced by the use of a steel tube T for the upper part of the shaft, which was insulated with bakelite RR from the support plate bearing KK. For the low temperature work the reservoir, pump and tubing were heavily lagged with cotton wool and the length of tubing reduced to a minimum. A temperature of  $-31^{\circ}\text{C}$  was maintained constant to within  $\pm 1^{\circ}\text{C}$ . The temperature of the disc was measured with a platinum/platinum -13% rhodium thermocouple clamped under the central clamping plate.



c) Measurement of Strain

The rotation of the central part of the discs was measured by recording the deflexion of a narrow beam of light from a plane mirror P in figures 1 and 2b. The axis of the mirror passed through the centre of the specimen and was coincident with the axis of rotation of the shaft. A light source I illuminated a narrow optical slit O and the narrow beam of light reflected from the fixed mirror K and the specimen mirror P, was focussed by a lens H on to a metric scale R engraved on a Perspex strip, as shown in figure 3. The scale was bent into a circular arc with the mirror P at its centre, and a strip of photographic paper was held firmly against the back of the scale. Fresh lengths of paper could be wound across the scale by means of the spools YY at each end of the scale. An image of the scale was obtained on the paper by flashing a small pea lamp J mounted in the narrow end of the camera box W. In this way errors in strain readings due to changes in size of the paper during processing were avoided. The travel of the image of the slit was recorded by intermittently illuminating the slit at suitable time intervals. For visual observation of the creep strain during the test a concave mirror was

attached to the shaft so as to produce an image of a galvanometer lamp on a scale outside the camera. The average error of reading on the photographic scale was about 0.2 mm, which corresponds to a strain of  $2 \times 10^{-4}$  in the case of the large discs and a strain of  $1 \times 10^{-4}$  for the small discs. Correction was made for slight movement of the whole apparatus by placing a mirror N alongside mirror P in figure 1 and bolting the former to the framework so that it produced a slit image on the photographic paper.

## 2. Experimental Procedure

### a) Preparation of Specimen

(i) Machining and Annealing. One face of a lead disc which had been turned from a block was machined flat, using progressively finer cuts in order to minimize the work hardening effect. The flat face was then fixed against a back plate in a jig and a circular groove of metal was removed, using a tool with a specially prepared profile. Again the depth of cut was progressively reduced down to about 0.01 mm so as to maintain the worked layer

as thin as possible. Care was taken to ensure that the tool was correctly aligned, since slight tilting of the tool would produce errors in the thickness of the metal annulus.

A pre-testing heat treatment was carried out to standardise the initial specimen state. The machined discs were annealed for 16 hours at  $100^{\circ}\text{C}$  in a muffle furnace and slowly cooled to room temperature whilst still in the furnace. This treatment resulted in a grain size of 3 grains/mm as measured by the linear intercept method. A few specimens which were to be tested at temperatures above  $80^{\circ}\text{C}$  were annealed at  $150^{\circ}\text{C}$  which gave a large grain size of 1 grain/mm.

(ii) Measurement of Specimen Annulus. The inner and outer radii of the annulus were checked by the use of vernier calipers. The thickness of the annulus was accurately measured by means of a suitably mounted dial gauge reading to 0.002 mm. Readings were taken at three different predetermined radial distances and at sixteen points on the circumference of each for the large discs and at eight points for the small discs. The annulus thickness on any one circumference was uniform to within

1%. The calculated mean stress was accurate to within 3% for the large discs and about 1.5% for the small discs.

(iii) Metallography. An area across the annulus on the flat surface of the disc was prepared for metallographic examination. A swab etch with Russell's solution was applied first to remove the thin worked layer of fine grains about  $70\mu$  in depth. The crystal structure was then revealed by flushing the surface with Worner and Worner's solution. This treatment produced a flat polished surface on which the grain and twin boundaries appeared as thin dark lines when viewed through an optical microscope. Several specimens were ruled across the etched area to form a fine grid consisting of radial lines about 0.1 mm apart and concentric circular arcs about 0.25 mm apart. For grain sizes larger than 3 grains/mm the grid lines were spaced further apart. The width of the ruled lines was approximately  $10\mu$ . Selected fields of view were photographed prior to testing so that reference could be made to the initial structure when the structure developed during creep was examined.

b) Creep Testing

The prepared lead disc was loosely bolted in position in the shear apparatus. Care was taken to align the surfaces, to which the specimen was bolted, in a single plane perpendicular to the axis of the shaft, so that the annulus was not deformed during assembly. The thermocouples and the specimen and apparatus mirrors were then attached. The large disc was enclosed in its Sindanyo box F by replacing the pieces of perspex sheet G in front of the box. The small disc was sealed off by screwing down the insulated lid LL onto the cover plate. The temperature control unit was switched on and the specimen left to reach the required temperature. When the desired temperature had been attained the disc was securely bolted in position together with its mirror assembly. In this way thermal strains during the period of heating or cooling were avoided. The temperature was recorded on a chart and periodically checked with the use of a portable potentiometer.

The mirror system was adjusted to produce images of the slit in a suitable position on the scale for the start of the creep test. The camera was loaded with a roll of film and made light tight. The film was drawn

across the back of the scale and exposed for the scale image. Zero fiducial marks were made by flashing the lamp for 10 seconds. A timing mechanism was started and the appropriate load was applied to the pulley wheel, so that the metal annulus was subjected to the required uniform shear stress. By using two Sangamo time switches, either singly or in series, strain recordings were obtained automatically at intervals which could be varied from 5 minutes to 24 hours as required. However, during the first 10 minutes after application of the load, when the creep flow was relatively fast, additional readings were registered manually at suitable intervals according to the rate of flow. When necessary the application of the stress, including reversal of stress, and the operation of the strain-recording equipment were performed automatically by pre-setting a special clock unit. In this way a fresh length of photographic paper was provided for each change in direction of the creep flow during repeated reversals of the shear stress. There was a delay period of about 30 seconds during the reversal of the load.

c) Grain Growth

Samples of lead III and tadanac lead were annealed at constant temperature and periodically examined metallographically to see if grain growth had ceased. The grain size was measured randomly across each sample using the linear intercept method. A final constant grain size was obtained at each temperature of anneal. The two leads had initial grain sizes which were stable up to  $60^{\circ}\text{C}$ . Grain growth at temperatures below  $60^{\circ}\text{C}$  was therefore studied on material with a smaller grain structure which was unstable above room temperature. Such a structure was obtained by cold rolling samples in a mixture of methylated spirit and solid carbon dioxide at  $-70^{\circ}\text{C}$ .

#### IV - RESULTS

##### A. Large Discs

##### 1. Effect of Temperature on Stable Grain Size

At a constant temperature the grain size, if less than the stable value, increases steadily to this stable value. The relationship between grain size and time was not precisely established but the existence of a stable grain size was demonstrated by maintaining a specimen at a fixed temperature for several days. After a period, which diminished with rising temperature, the grain size remained constant over an interval of days.

As shown in figure 4, in agreement with the work of Andrade and Aboav on grain growth (1964, 1966)  $d_e^{\frac{1}{2}}$  was found to be  $L(T - T_0)$ , where  $L$  is a constant,  $d_e$  being the stable grain diameter (mm) measured by the linear intercept method.  $T_0$  is a constant which is  $240^\circ\text{K}$  for tadanac lead and  $237^\circ\text{K}$  for lead III:  $L$  is 0.0068 and 0.0047 respectively, and thus is very sensitive to small impurities. The addition of 45 p.p.m. silver and 38 p.p.m. copper in lead III, which suppress recrystallization, also hinder grain growth as expressed by the stable grain diameter.

With lead III the stable grain size at  $80^\circ\text{C}$  is



given by  $n_e = 3.43$  grains/mm and thus up to this temperature the annealed discs for which  $n = 3$  grains/mm would not be expected to show grain growth. Andrade and Aboav (1966) observed that under a prolonged external stress grain growth ultimately occurred in cadmium in which, in the absence of stress, the grains were of a stable size. Such grain growth, however, was not detected in the tests on lead described here. Tests performed at temperatures above  $80^\circ\text{C}$  were not directly comparable with tests at lower temperatures because of grain growth in the former. Another important factor in this respect was the ratio of the grain size to the minimum thickness of the annulus. The occurrence of grain growth above  $80^\circ\text{C}$  produces a grain size approaching the annulus thickness and influences the flow constants in creep tests at such temperatures.

## 2. General Form of Creep Curves

The forward creep, without reversal of stress, was measured for various stresses and temperatures over a wide range of strain which occasionally reached 0.80. The three stages of forward flow, FI, FII, and FIII described by Andrade and Jolliffe were also observed in the present experiments. Even at  $150^\circ\text{C}$ , where grain growth occurred,

stages FI, which consisted of a  $t^{\frac{1}{2}}$  followed by a  $t^{\frac{1}{3}}$  flow, and FII, which was a period of acceleration and hardening, were observed. Because of the grain growth to which reference has been made the tests at  $150^{\circ}\text{C}$  were not extended and the results obtained are considered separately. A flow linear with time, K flow, was detected in stage FI at certain temperatures, but was not present at room temperature. Typical creep curves which exhibit the forward stages are shown in figure 5.

The flow constants in stages FI and FIII were obtained and their dependence on stress and temperature evaluated at temperatures up to  $80^{\circ}\text{C}$  with a constant initial grain size of 3 grains/mm.

The form of the creep curve on reversal of stress was influenced by the presence of the K flow in the forward creep and will be described later.

### 3. Forward Creep

#### (a) Stage FI

##### (i) Time laws of flow

The flow in this stage was characterized by an instantaneous strain on applying the load, followed by a  $t^{\frac{1}{2}}$  decreasing to a  $t^{\frac{1}{3}}$  flow, as found by Andrade and

Jolliffe, to which a permanent creep  $Kt$  was to be added at higher temperatures (Andrade, 1962). Measurements above  $27^{\circ}$  and up to  $80^{\circ}\text{C}$  showed accordingly that the flow could be expressed by the equations:

$$\gamma = C + Dt^{\frac{1}{2}} + Kt \quad \dots (1)$$

$$\text{and } \gamma = A + Bt^{\frac{1}{3}} + Kt \quad \dots (2)$$

At very low strains, before the  $t^{\frac{1}{2}}$  flow commences, there is an uncertain region of flow fitted by neither  $t^{\frac{1}{2}}$  nor  $t^{\frac{1}{3}}$  laws. The lower limit of strain at which the  $t^{\frac{1}{2}}$  law was valid varied somewhat with stress, temperature and grain size but was approximately 0.01. The upper limit of the  $t^{\frac{1}{2}}$  law was about 0.03 strain. For example, in Table 1, for which the stress was  $25 \text{ Kg/cm}^2$  and the temperature  $80^{\circ}\text{C}$ , the values of  $\gamma$  from 0.0107 up to 0.0343 can be represented within experimental error by the  $t^{\frac{1}{2}}$  law with  $C = -0.0004$ ,  $D = 0.00236 \text{ min}^{-\frac{1}{2}}$  and  $K = 2.5 \times 10^{-5} \text{ min}^{-1}$ . The negative value of  $C$  is a consequence of the law being invalid at very low creep strains after a small loading strain.

In the region of strain above about 0.025, where the

$t^{\frac{1}{2}}$  formula ceases to represent the creep flow, the  $t^{\frac{1}{3}}$  law is valid as shown in Table 2. This formula holds until recrystallization occurs at a strain of 0.0566. For the  $t^{\frac{1}{2}}$  law the average error is 1 part in 215 of the range and for the  $t^{\frac{1}{3}}$  law 1 part in 987 of the range. The regions of strain covered by the two formulae overlap, as in the case represented in Tables I and II, providing a smooth transition from the  $t^{\frac{1}{2}}$  to the  $t^{\frac{1}{3}}$  flow.

(ii) Determination of the transition strain  $\gamma_c$

A formula deduced by Andrade and Jolliffe (1963) enables the time and strain for the transition from  $t^{\frac{1}{2}}$  to  $t^{\frac{1}{3}}$  flow to be calculated from the flow constants. If  $\gamma_c$ ,  $t_c$  are the values of strain and time at which expressions (1) and (2) have the same co-ordinates and the same rate of creep then,

$$t_c = (2B/3D)^6 \quad \dots (3)$$

$$\text{and } \gamma_c = A + 4B^3/9D^2 + K(2B/3D)^6 \quad \dots (4)$$

$$\text{whence } C-A = 4B^3/27D^2 = 0.1481 B^3/D^2 \quad \dots (5)$$

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$$\text{thus } \gamma_c = 3C - 2A \quad \dots (6)$$

In figure 6 are plotted values of C-A against  $B^3/D^2$  for lead III at various stresses and temperatures. The experimental points lie on, or very near, the line which represents equation (5) demonstrating the general validity of the  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  formulae. Despite the fact that the values of C and A were not regular the values of C-A and  $3C-2A$  were reasonably consistent.

The value of the transition strain  $\gamma_c$  has been calculated using equations (4) and (6) at different stresses and temperatures. The values listed in Table 3 show good agreement between both methods of calculation. The average values of  $\gamma_c$  calculated from equations (4) and (6) are plotted against the shear stress  $\sigma$  for various temperatures as shown in figure 7. Some results obtained by Andrade and Jolliffe have been included which agree very well with the present work. The variation of temperature between  $27^\circ$  and  $80^\circ\text{C}$  does not have any significant effect on the variation of  $\gamma_c$  with the stress. At low stresses when the immediate strain was less than 0.02,  $\gamma_c$  appeared to be constant at approximately 0.025 strain. As the stress rises, however, the transition strain rapidly increases as shown in figure 7.

At the highest stresses the duration of flow up to the strain at which recrystallization occurs (about 0.07 for lead III at 27°C) is small. For example, for  $\sigma = 49 \text{ Kg/cm}^2$  at 27°C the duration is only 10.5 min but the  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  stages are well indicated. The immediate strain is large, being in the case just cited nearly 0.04, so that it appears that when the immediate strain exceeds the value at which transition takes place for lower stresses, the  $t^{\frac{1}{2}}$  stage is not abolished, as it would be for strains greater than 0.04 reached by creep instead of by immediate deformation, but merely postponed. This is further evidence that the mechanism of immediate strain is not that for time-dependent flow.

An experiment was performed at 60°C and  $30 \text{ Kg/cm}^2$  with metal of particularly large grain size,  $n = 1 \text{ grain/mm}$ , which showed the transition. The average value of  $\gamma_c$  was 0.0248 compared with 0.0242 for metal of  $n = 3 \text{ grains/mm}$  at the same temperature and stress. Considering the large difference in grain size and the fact that the powers of B and D involved make a precise determination of  $\gamma_c$  rather difficult,  $\gamma_c$  can be considered to be independent of the grain size.

(iii) The immediate strain  $\gamma_0$ 

The values of the constants A and C are dependent on the magnitude of the immediate strain  $\gamma_0$  and, in the case of small stresses, on the amount of creep strain obtained before the  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{5}}$  regimes are operative. Thus, at very high stresses when the  $t^{\frac{1}{2}}$  flow is very small or not detectable, the value of A approximates to the loading strain but at lower stresses, where the  $t^{\frac{1}{2}}$  flow is appreciable, A is less than the loading strain and often has a negative value. Similarly, C will represent the loading strain when the creep strain prior to the  $t^{\frac{1}{2}}$  flow is negligible, that is, when the immediate strain is about 0.01 or more. At stresses which produce smaller immediate strains, however, C is less than  $\gamma_0$  and can be negative. Andrade and Jolliffe (1962) have reported difficulty in obtaining reproducible values of A and C for a given stress and temperature and have attributed it to the fact that the immediate strain is very sensitive to the initial condition of the material.

It was not possible to record the shear strain immediately after loading but a reading was normally obtained 30 - 60 seconds after loading. Where appropriate,

values of  $\gamma_0$  were obtained from the values of A and C. When this was not possible ( $\gamma_0 < 0.01$ )  $\gamma_0$  was estimated by extrapolation of the initial creep strain to  $t = 0$ . The variation of  $\gamma_0$  with stress for three temperatures is given on a log - log plot shown in figure 8. It is clear that  $\gamma_0$  is strongly dependent on the stress, the relation being

$$X \gamma_0 = \sigma^x \quad \dots (7)$$

where X, approximately  $2.5 \times 10^8 (\text{Kg/cm}^2)^x$ , is a measure of the work hardening of the material and the index x is about 4.2. The values of the immediate strain ranged from 0.04 down to 0.001 in which range there was no marked effect of variation of temperature between  $27^\circ$  and  $80^\circ\text{C}$ . This behaviour is in direct contrast to that of the creep flow, which is markedly increased with rise of temperature.

#### (iv) Variation of Flow Constants with Stress

The variation of the constants B, D, and K with stress at constant temperature was obtained for temperatures of  $27^\circ$ ,  $60^\circ$  and  $80^\circ\text{C}$ . The experiments at  $27^\circ\text{C}$  confirmed the variation of B and D with stress obtained by Andrade and Jolliffe (1963) for Lead III. At this temperature no K flow



could be detected, even at the highest stresses employed. At the higher temperatures, however, the  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  flow were augmented by the K flow which is linear with time.

The results of tests for the stress variation of the constant B, equation (2), are shown in figure 9, from which it appears that

$$B = H_B \exp (h_B \sigma) \quad \dots (8)$$

at constant temperature. The value of  $H_B$  obtained from the intercept on the semi-logarithmic plot was  $3.46 \times 10^{-4} \text{ min}^{-\frac{1}{3}}$  at  $27^\circ\text{C}$ ,  $3.75 \times 10^{-4} \text{ min}^{-\frac{1}{3}}$  at  $60^\circ\text{C}$  and  $4.70 \times 10^{-4} \text{ min}^{-\frac{1}{3}}$  at  $80^\circ\text{C}$ , and is clearly not very sensitive to an increase in the temperature. The value of  $h_B$ , given by the slope of the plot, was also observed to increase with rising temperature, being  $0.0844 \text{ cm}^2/\text{Kg}$  at  $27^\circ\text{C}$ ,  $0.104 \text{ cm}^2/\text{Kg}$  at  $60^\circ\text{C}$  and  $0.114 \text{ cm}^2/\text{Kg}$  at  $80^\circ\text{C}$ . The relationship cannot, of course, be valid at very low stresses, since it predicts a value of B at zero stress.

The constant D, equation (1), was found to vary with the stress in the same way as B, so that,

$$D = H_D \exp (h_D \sigma) \quad \dots (9)$$

at constant temperature as shown in figure 10. The value of  $H_D$  was  $9.61 \times 10^{-6} \text{ min}^{-\frac{1}{2}}$  at  $27^\circ\text{C}$ ,  $1.52 \times 10^{-5} \text{ min}^{-\frac{1}{2}}$  at  $60^\circ\text{C}$  and  $5.85 \times 10^{-5} \text{ min}^{-\frac{1}{2}}$  at  $80^\circ\text{C}$ . The slope,  $h_D$ , was  $0.157 \text{ cm}^2/\text{Kg}$ ,  $0.181 \text{ cm}^2/\text{Kg}$  and  $0.192 \text{ cm}^2/\text{Kg}$  respectively. Thus, at any particular temperature,  $D$  varies more rapidly with stress than  $B$ .

As regards the measurement of  $K$ , in suitable circumstances it was possible to obtain values, but not with great accuracy. Large stresses, which are favourable for  $K$  flow, mean a short time of flow to reach the limit at which recrystallization begins, so that the strain due to  $K$  is small compared to the  $B$  flow and hence not accurately measurable. The largest value of  $K$ ,  $5.5 \times 10^{-4} \text{ min}^{-1}$  was obtained with a stress of  $40 \text{ Kg/cm}^2$  at  $60^\circ\text{C}$ , but the time of flow was only 7 minutes and the value of  $Kt$  only 0.0038 that is about 5.4% of the forward strain, which is recorded with a possible error of  $2 \times 10^{-4}$  strain. The largest value of the  $K$  flow,  $Kt$ , was 0.0110, obtained with a stress of  $25 \text{ Kg/cm}^2$  at  $80^\circ\text{C}$ , giving  $K$  within an error of 2%. In general, with the temperature constant, the proportion of strain due to  $K$  flow in the possible range of forward strain increases as the stress decreases.

The variation of  $K$  with stress cannot, by dimensions, be the same when it accompanies  $t^{\frac{1}{2}}$  flow and when it accompanies  $t^{\frac{1}{3}}$  flow. The dimensions of  $\gamma$  (L/L) are zero, so that the dimensions of  $B$  are  $T^{-\frac{1}{3}}$  and of  $K_B$  are  $T^{-1}$ , while similarly the dimensions of  $D$  are  $T^{-\frac{1}{2}}$  and of  $K_D$  are  $T^{-1}$ .

Hence  $B^3$  is proportional to  $K_B$

and  $D^2$  is proportional to  $K_D$

When  $B$  and  $D$  are concerned, the only region in which they are simultaneously valid is the transition region, where  $B^3/D^2$  is constant. Although values of  $B$  and  $D$  can be found elsewhere, the  $B$  flow succeeds the  $D$  flow and they are not simultaneously valid, so that the simple power relation between them does not hold. The small strains involved in the  $t^{\frac{1}{2}}$  flow did not allow accurate values of  $K$  to be measured in this stage. The variation of  $K$  with stress was, therefore, obtained for the  $t^{\frac{1}{3}}$  stage at  $60^\circ\text{C}$  and is shown in figure 11. The relationship is of the form,

$$K = H_K \exp(h_K \sigma), \quad \dots (10)$$

where  $H_K$  is  $2.50 \times 10^{-9} \text{ min}^{-1}$  and  $h_K$  is  $0.307 \text{ cm}^2/\text{Kg}$ .

At 60°C it follows from the slopes of the semi-logarithmic plots that  $h_K/h_B = 3.0$ , that is, K is proportional to  $B^3$  as found by Feltham (1956) for lead wires in tensile creep. The experimental values of  $\ln B$  are shown in figure 12 against the corresponding values of  $\ln K$  which gives the following relationship,

$$B = 0.29 K^{0.33} \quad \dots (11)$$

Thus the order of increasing sensitiveness to change of stress is B, D and K.

(v) Variation of Transient Flow Constants with Temperature

Tests were carried out at a constant stress  $\sigma = 25 \text{ Kg/cm}^2$  and measurements taken over a range of temperature from 40°C to 80°C. It was found that  $\ln B$  and  $\ln D$  were linear functions of  $1/T$ , as shown in figures 13 and 14, which include values of B and D at 27°C obtained from figures 10 and 11. The plots are represented by the formulae

$$B = P_B \exp(-Q_B/RT) \quad \dots (12)$$

$$\text{and } D = P_D \exp (-Q_D/RT) \quad \dots (13)$$

$P_B$  being  $1.73 \text{ min}^{-\frac{1}{3}}$  and  $P_D$  being  $24.63 \text{ min}^{-\frac{1}{2}}$ . The values of  $Q_B$  and  $Q_D$  represent apparent activation energies for the  $t^{\frac{1}{3}}$  and  $t^{\frac{1}{2}}$  types of flow, being, at this stress, 3.83 and 6.48 K.cal/mole respectively. Thus, the apparent activation energy for the  $t^{\frac{1}{2}}$  flow is 1.69 times that for the  $t^{\frac{1}{3}}$  flow, a matter discussed later.

Since  $B$  varies exponentially as the inverse of the absolute temperature, then in the formula  $B = H_B \exp (h_B \sigma)$ , either  $H_B$  varies exponentially with  $1/T$  or  $h_B$  is a linear function of  $1/T$  or both. Values of  $h_B$  and  $h_D$  were obtained at three different temperatures only, but these were sufficient to show that both  $h_B$  and  $h_D$  could be closely represented by linear functions of  $1/T$ , as shown in figures 15 and 16. The constants  $H_B$  and  $H_D$  increased only slightly with the temperature.

Feltham (1956) observed that, for lead subjected to various constant tensile stresses,

$$h_B = m_B/RT \quad \dots (14)$$

where  $m_B$  was a constant independent of stress and

temperature. Substitution of the experimental values of  $h_B$  and  $T$  in (14) indicate that, for lead in simple shear,  $m_B$  increases with rise in temperature. It was found that  $\ln m_B$  was a linear function of  $1/T$ , as shown by the line of negative slope in figure 17: this agrees with the suggestion of Feltham and Meakin (1959) for copper. However, over this range of temperature  $27^\circ$  to  $80^\circ\text{C}$  the variation of  $m_B$  could equally well be represented as a linear function of temperature as shown in figure 18. This accords with the finding that  $h_B$  is a linear function of  $1/T$ , as described above for this range of temperature. Further values of  $h_B$  are required at other temperatures to determine which of the two functions is valid.

It seems probable that the changes in the flow constants with stress and temperature are also influenced by associated changes in the structure of the material. Thus, the experimental values of the apparent activation energies  $Q_D$  and  $Q_B$  for  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  flow are not the true activation energies for the mechanisms controlling creep. Dorn (1956) described a method of obtaining the activation energy in which the temperature was abruptly changed and the rate of flow determined immediately before and after the change in temperature, so that any structural changes

were minimised. In the present investigation the method of heating did not allow a rapid temperature change to be effected.

(b) Linear Flow in Stage III

In flow under simple shear stress, at high strain, a steady flow sets in at a rate which, at constant stress, does not vary with time. This steady flow begins at a strain in the region of 30 per cent and prevails up to strains of about double this value, the range of strain in question depending somewhat upon the temperature. In this stage III the flow under reversed stress takes place at a constant rate which is the same as that which prevails under direct stress and is characteristic of the stress and temperature, returning to the same value if the temperature, after being raised, is lowered again to its original value (Andrade and Jolliffe III). Thus the behaviour of the metal in this stage is that of a liquid of non-Newtonian viscosity. The metal is in a state very different from that which prevails at smaller strains, many of the grains being broken up and a large number of new grains formed, all grains being characterised by marked slip, the direction of which is preferentially

that of the principal stresses. Thus the results of the tests to be described have not been anticipated in the investigations carried out under tension to which reference will be made, where viscous conditions did not prevail.

The behaviour in stage III has been investigated at temperatures from 27°C, at which Andrade and Jolliffe obtained their results, to 80°C, higher temperatures not being used on account of the instability of the grains, discussed elsewhere. The results obtained at 27°C were in good agreement with those of Andrade and Jolliffe.

At a constant stress of 35 Kg/cm<sup>2</sup>, in the region of which the values of the constant velocity  $v$  are within a range which allows accurate measurement, it was found that the variation of  $v$  with  $T$  was given by

$$v = P_v e^{-Q_v/RT} \quad \dots (15)$$

giving an activation energy  $Q_v$  of 16.9 kilocal/mole, which is independent of stress.

Andrade and Jolliffe found that at 27°C

$$v = H_v \exp (h_v \sigma) \quad \dots (16)$$



where  $H_v = 4.6 \times 10^{-9} \text{ min}^{-1}$  and  $h_v = 0.30 \text{ cm}^2/\text{Kg}$  and the present finding suggests that  $H_v = v_o e^{-Q_v/RT}$ , where  $v_o$  is a constant independent of  $\sigma$  and  $T$ .

This gives

$$v e^{Q_v/RT} = v_o e^{h_v \sigma}$$

$$\text{or } \ln(v e^{Q_v/RT}) = \ln v_o + h_v \sigma \quad \dots (17)$$

When  $v$  is the secondary creep rate. The expression  $v e^{Q_v/RT}$  is known as the Zener-Holloman parameter, (Zener et al., 1944).

In figure 19  $\ln(v e^{Q_v/RT})$  is shown against  $\sigma$  for the present results and those of Andrade and Jolliffe: it is, perhaps, worth remarking that the present results are for forward creep, while those of Andrade and Jolliffe are for reverse creep. Thus, that all the points lie on one straight line not only confirms equation (17), but also the equivalence of forward and reverse creep in stage III. The value of  $v_o$  is  $1.00 \times 10^4 \text{ min}^{-1}$  and of  $h_v$  is  $0.300 \text{ cm}^2/\text{Kg}$ .

The results do not agree with those of Feltham and Meakin (1959) for the steady state creep of copper at

temperatures from 400° to 700°C. Over this range of homologous temperature, 0.50 to 0.72, which includes the range for the present work, 0.50 to 0.59, they found a marked increase with temperature of the slope of the line connecting  $\ln v$  with stress. Their experiments were carried out under tension at constant stress and the extreme strain appears to have been less than 20 per cent: further it appears that the stage of recrystallisation was not exceeded in the measurements from which the steady creep rate was deduced. It is therefore clear that they were concerned not with the metal in stage III, the viscous state, but with secondary creep, a point of great significance.

It will be seen from figure 19 that as the stress is diminished a transition stress  $\sigma_v$  is reached at which the linear law prevailing at all the temperatures concerned breaks down, being replaced by a line of greater slope. The stress  $\sigma_v$  can be determined from the point of intersection of two straight lines. As shown in figure 20, for the values so determined,

$$\sigma_v = W \left( \frac{1}{T} - \frac{1}{T_m} \right) \quad \dots (18)$$

where  $T_m$  is the melting point. That the value of  $\sigma_v$

given by the formula is 0 at  $T = T_m$  is a confirmation of the validity of the formula.

Feltham and Meakin, for their secondary creep rate with copper at high temperature, likewise obtained at a given temperature two intersecting lines to represent the variation of  $\ln$  (creep rate) with  $\sigma$ , but their transition stress shows no regularity with temperature.

The results for stage III are, then, well represented by

$$v = v_0 \exp (h_v \sigma - Q_v/RT) \quad \dots (19)$$

According to Sherby, Orr and Dorn (1954) the rate of secondary creep at constant substructure is given by  $\dot{\gamma} = S e^{-H/RT} \sinh B\sigma$  which for values of B in question is a very close approximation to equation (19). Cottrell and Aytakin (1950), however, introduce a temperature factor into the stress factor, writing  $h_v$  as  $r/RT$ , which does not agree with the present results. Everything, apparently, depends upon whether the substructure changes during the flow, which in stage III it does not, as evidenced by the behaviour on reversing the stress or cycling the temperature down and back again.

It may be noted that Andrade and Aboav (1964, figure 17) found that equation (18) governs the relation of a transition stress with temperature, but the stress in this case is one that determines, for a c.p.h. metal, the transition of the equation governing the flow from a form, called subnormal, to the  $\gamma = A + B t^{\frac{1}{3}} + K t$  form, called normal. This would suggest that the relation has a general significance.

#### 4. Effect of reversals of stress

Andrade and Jolliffe (1960, 1963) have recorded the flow under repeated reversals of stress in various circumstances and showed, among other things, that if in the forward flow the strain exceeds the limit within which the  $t^{\frac{1}{3}}$  law is valid, then the regularities which the reverse flow shows after pure  $t^{\frac{1}{3}}$  flow are modified. It seemed of interest to ascertain whether a K component in the forward flow, which was absent, or extremely small, in these earlier experiments made at room temperature, influenced notably the reverse flow.

A significant test was carried out at 60°C and  $\sigma = 33 \text{ Kg/cm}^2$ , for which the forward flow, terminating at  $t_f = 86 \text{ min.}$  and  $\gamma_f = 0.0595$ , was represented by

$$\gamma = 0.0073 + 0.0107t^{\frac{1}{3}} + 5.8 \times 10^{-5}t$$

which means that the K flow contributed the relatively large fraction 8.4 per cent to the final forward strain. The notation of Andrade and Jolliffe III, 6, is used, according to which  $p$  is the ratio of the rate of the linear reverse flow, constituting stage  $R_0$ , to the final forward rate, a ratio which was found by them to be constant for a given lead:  $q$  is the ratio  $t_r/t_f$ , when  $t_r$  is the time from the first application of stress to the end of stage  $R_0$ :  $r$  is the ratio  $B_1/B$ , where the  $t^{1/2}$  reverse stage that follows the linear reverse stage is given by

$$\gamma = A_1 - B_1 (t - t_f)^{1/2}$$

The reverse linear flow is represented by  $\gamma = a_1 - b_1 (t - t_f)$ . The value of  $t_r$  for the end of the  $R_0$  stage is determined by  $t_r - t_f = (B_1/3b_1)^{3/2}$ .

In the test now under discussion the first reverse stage was found to consist as established by Andrade and Jolliffe, of a linear stage followed by a  $t^{1/2}$  stage. The duration of the linear stage was 53 min, as shown in figure 21, the value of  $q$  being 1.62 as against 1.79 found by Andrade and Jolliffe for the same lead, lead III. The value of  $p$  was 2.15 and of  $r$  was 2.05, as against values 1.79 and 1.65 respectively found

by Andrade and Jolliffe for reversal after normal  $t_f^{\frac{1}{3}}$  forward flow. Likewise the relation  $\frac{p}{q} (q - 1)^{\frac{2}{3}} = 1$ , established by them for normal reverse flow, with two different leads, did not hold.

In the second cycle (figure 21), the forward and reverse flow resembled those found by Andrade and Jolliffe for cycles with intervals greater than  $t_f$ , that is, after a first abnormal reverse flow. Thus the K term on the forward flow entails abnormal behaviour in subsequent reversals.

In the case of a reversal at  $50^\circ\text{C}$  and  $25 \text{ Kg/cm}^2$ , where  $\gamma_f = 0.0490$ ,  $t_f = 2889 \text{ min.}$  and  $K = 1.0 \times 10^{-6} \text{ min}^{-1}$ , the K contribution to the final forward strain was 5.9 per cent. The values of p and q for the first reversal were, respectively, 1.66 and 1.60. For  $60^\circ\text{C}$  and  $20.35 \text{ Kg/cm}^2$ ,  $\gamma_f$  being 0.0314 and  $t_f$  being 2882 min, the flow obeyed the  $t^{\frac{1}{2}}$  law and  $K = 1.6 \times 10^{-6} \text{ min}^{-1}$  gave a final contribution to the forward strain of 14.7 per cent. p and q were respectively 1.48 and 1.51 for the first reversal and 1.63 and 1.50 for the second reversal. There were various minor departures from normal behaviour, such as the slight acceleration in the second forward stage shown in figure 22.

When there was no K flow at higher temperatures normal behaviour on reversal was established.

The general result is that the presence of a K component in the forward flow invalidates the simple laws which govern the reverse flow after a pure  $t^{\frac{1}{3}}$  forward flow, just as does exceeding the range of strain within which the  $t^{\frac{1}{3}}$  law holds. (Andrade and Jolliffe II, p.307). In all cases the relative duration of the linear  $R_0$  stage is markedly decreased, as it was by exceeding the  $t^{\frac{1}{3}}$  range: in the case of the high stress at 60°C, figure 21, the rate-ratio  $p$  was increased: in the case of the flow of long duration at 50°C it was decreased. The behaviour was not investigated in detail. The importance of the general result is to emphasize once more the fundamental nature of the  $t^{\frac{1}{3}}$  law: any departure from the conditions that determine its validity greatly influence the subsequent behaviour of the metal.

##### 5. Tests at 150°C

Tests were carried out at 150°C with metal that had been subjected to a preliminary anneal at this temperature, leading to a grain size of 1/mm, approaching the thickness of the annulus, which ranged from 2.1 mm to 1.3 mm, according to the distance from the axis. In figure 23 are shown results obtained at 8, 9 and 10 Kg/cm<sup>2</sup> respectively. Up to strains of about 0.045 (0.042 for 9 and 0.049 for 10 Kg/cm<sup>2</sup>), the  $t^{\frac{1}{3}}$

law, followed by the  $t^{\frac{1}{3}}$ , held, as in typical tests at 27°C, the transition strain  $\gamma_c$  being 0.0147 for 9 and 0.0142 for 10 Kg/cm<sup>2</sup>. Table 4 shows that the  $t^{\frac{1}{2}}$  law holds from the first reading,  $\gamma = 0.0046$ , to  $\gamma = 0.0195$  and the  $t^{\frac{1}{3}}$  law from  $\gamma = 0.0124$  to 0.0413, with the usual overlap (Andrade and Jolliffe III, § 3), thus showing normal behaviour in spite of the large grain size. The maximum error of  $\gamma$  in such range is 0.0002, which is the estimated error of reading. Thus, for the  $t^{\frac{1}{2}}$  law the average error over the range of strain 0.0148 is 1 part in 148 and for the  $t^{\frac{1}{3}}$  law over the range 0.0290 1 part in 312.

With  $\sigma = 10$  Kg/cm<sup>2</sup> at this temperature of 150°C, recrystallisation began at  $\gamma = 0.049$ , accompanied by a marked acceleration of the flow, which was followed by a deceleration, a transition process of which a less marked instance is given in Andrade and Jolliffe II, figure 6. This deceleration was succeeded by a short linear stage, followed in turn by a second transition process, not completed when the test ceased.

The grain size approaching the thickness of the specimen at 150°C, no great significance is attached to measurements at temperatures as high as this. The tests in question do show, however, that the normal  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  laws hold under



these extreme conditions without any  $Kt$  term, the presence of which has been established at lower temperatures. Subsequent to this normal flow, attributed to avalanches on glide bands, discontinuous grain boundary migration takes place, to which the transition accelerations and decelerations in stage FII are attributed.

#### 6. Metallographic Examination

The mode of deformation of the grains under simple shear has been studied by taking optical photomicrographs of polished and etched specimens at a number of stresses and temperatures. Marked slip bands were developed during transient creep. With increase in temperature the slip lines became less distinct and seemed to be broader, particularly if the grain size was increased to 1 grain/mm, as was the case at 150°C. At 27°C, Andrade and Jolliffe (1963) attributed the  $t^{\frac{1}{3}}$  flow to the addition of new lamellae to the established slip bands which were formed during the  $t^{\frac{1}{2}}$  flow. This mechanism is different from that of cadmium (Andrade and Aboav 1964), in which the  $t^{\frac{1}{3}}$  flow seems to be due to the creation of fresh slip bands during the flow.

A measure of the grain boundary sliding was obtained by measuring the displacement of the radial lines at grain

boundaries in the direction of the shear. It is clear that many grains would slide by amounts too small to be detected, thus furnishing a contribution which would be neglected in the measurements. Hence, the measured contribution of the grain boundary sliding would be less than that which actually occurred.

For a test at  $80^{\circ}\text{C}$  and  $25 \text{ Kg/cm}^2$  the measured contribution of boundary sliding to the shear strain was 0.0085, whereas the amount of K flow was 0.0110 strain. A test at  $60^{\circ}\text{C}$  and  $30 \text{ Kg/cm}^2$  gave 0.0042 strain due to boundary sliding and 0.0080 strain due to the K flow. Thus to prove that the shear due to the K term is to be attributed to the mechanism of grain boundary sliding demands a more accurate method of measurement, but the results offer some indication that this is the case.

It was observed that not only did the grains slide over each other in the direction of shear but they also tended to rotate to accommodate movement of neighbouring grains. Such rotation was evident by the relative displacement of adjacent portions of the circular ruled lines normal to the direction of shear.

In addition to the movement of grains relative to one another during the creep flow there was also present grain

boundary migration. In general, when the creep temperature was below the annealing temperature, the migration was slight, so that one grain grew only slightly at the expense of another or more. However, if there was a marked difference in size between a grain and its neighbours there was a tendency for a small grain to disappear, the length of the boundary diminishing until a new triple point was formed.

Photomicrographs taken after creep indicated that grain boundary migration was discontinuous with time. When a migrating boundary paused for a time it left a visible trace on the etched surface so that the progress of migration during the creep test could be seen. It was observed that when a migrating boundary became stationary, grain boundary sliding often occurred, so that a radial scribe line became segmented and displaced in the direction of shear at each boundary position. Such a case is shown in figure 24 for a test at  $150^{\circ}\text{C}$ . The approximately horizontal lines are successive positions of the boundary which is thought to have migrated during periods of accelerating creep in stage FII. There was also clear evidence of grain boundary sliding at this temperature on some photomicrographs.

When a grain became larger due to migration of the grain boundary the slip lines, already established in the

crystal, extended into the new area as it advanced into the adjacent crystal, crossing the traces of the old boundary positions. The growth of a crystal at  $60^{\circ}\text{C}$ , subjected to a stress of  $30 \text{ Kg/cm}^2$  for 320 min., is shown in figure 25. The only etching of the metal was that before stressing. The original outline of the crystal, shown by the continuous line in figure 26, was deformed by the flow under stress to the position indicated by the broken line. The growth by boundary migration must have taken place in stages, with pauses in between, as shown by the dark lines in figure 25: thus considering the top of the grain, growth started at four points, subsequently merging to two points and finally to one, as shown in figure 26. The final position of the boundary is shown by the dotted line in figure 26.

## B. Small Discs

### 1. Tests at $27^{\circ}\text{C}$

A number of experiments were carried out over the range of stress 40 to  $60 \text{ Kg/cm}^2$  in order to compare the creep behaviour with that reported for large discs at  $27^{\circ}\text{C}$ . The transient creep flow was found to be of the normal type, obeying the  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  laws. For instance, in a test at  $40 \text{ Kg/cm}^2$ , a  $t^{\frac{1}{2}}$  law was valid from  $\gamma = 0.0072$  to  $\gamma = 0.015$

from which strain a  $t^{\frac{1}{3}}$  law followed. No K flow was detected.

Values of the  $t^{\frac{1}{3}}$  constant B were derived from the creep curves for several stresses and compared with the corresponding values of B for the large discs. In all cases the large discs gave a value of B approximately 2.4 times greater than that for the small discs. Thus the rate of change of  $\ln B$  with  $\sigma$  was the same for both types of disc although the actual values of B differed for the same stress.

## 2. Tests Below Room Temperature

Several tests over a range of stresses were performed at  $0^\circ$  and  $-31^\circ\text{C}$ . A notable feature of the flow at these temperatures was the existence of a critical stress  $\sigma_c$  below which the normal  $t^{\frac{1}{3}}$  behaviour was not obtained. The values of  $\sigma_c$  were not precisely determined but were estimated to be about  $65 \text{ Kg/cm}^2$  and  $95 \text{ Kg/cm}^2$  at  $0^\circ$  and  $-31^\circ\text{C}$  respectively. Andrade and Aboav (1964) observed similar behaviour with cadmium which exhibited subnormal flow below a certain stress  $\sigma_c$ , provided that the test temperature was below a critical value  $T_c$ . The subnormal flow for cadmium was accurately represented by an equation of the form,

$$\gamma = M (1 - e^{-mt}) + K_M t. \quad \dots (20)$$

In the case of lead III, however, the observed abnormal flow was not easily fitted and appeared to be a mixture of normal  $t^{\frac{1}{3}}$  flow and subnormal flow. For example, a comparison of the observed results for a stress of  $55 \text{ Kg/cm}^2$  at  $0^\circ\text{C}$  with those calculated from the formula,

$$\gamma = A + Bt^{\frac{1}{3}} + M(1 - e^{-mt}) \quad \dots (21)$$

is given in table 5. The error obtained by this fit approximates to the experimental error for the strain readings.

It was shown by Andrade and Aboav (1964) that  $\sigma_c$  for cadmium was a function of the temperature such that,

$$\sigma_c = S' \left( \frac{1}{T} - \frac{1}{T_m} \right) \quad \dots (22)$$

where  $S'$  is a constant and  $T_m$  is the melting point of the metal. In the case of lead III the estimated values of  $\sigma_c$  also decreased as the temperature was raised but more values are required to establish the relationship with temperature.

## V - DISCUSSION

### 1. Stable Grain Size

It has been shown that, in the absence of an external stress, a constant stable grain size can be obtained at a particular temperature of anneal. The time required to achieve a constant grain size diminished with increasing temperature but a precise law relating grain size and time was not established. Such a law has, however, been demonstrated by Andrade and Aboav (1966) in a detailed investigation of grain growth in hexagonal close-packed metals.

It is important, when studying creep behaviour, that the test temperature should not exceed the temperature corresponding to stability of the grain size of the specimen. At higher temperatures grain growth occurs and modifies the creep behaviour. For instance, in tests at 150°C, with a grain size of 3/mm, the final grain size was about 1 grain/mm. The normal  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  creep laws were obeyed but the creep constants did not vary in a regular manner with the stress. Andrade and Aboav (1964) found, for cadmium, that at low stresses, for which what they called the laws of subnormal flow prevailed, an abnormal increase in the rate of flow took place at a temperature which, if exceeded, led to

rapid grain growth. Below this temperature, with grains of stable size in the absence of stress grain growth occurred in cadmium after a long period of creep but did not affect the laws governing the initial stages of creep. This phenomenon was not observed in lead.

A comparison of the grain growth in lead III with that in tadanac lead showed that small additions of copper and silver in lead resulted in a finer final grain size at various temperatures of anneal.

## 2. Flow in Stage FI

At strains greater than about 0.01 the flow has been fitted with  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  laws and in some cases with an additional flow linear with time, K flow, over a range of temperatures and stresses. The  $t^{\frac{1}{3}}$  law was valid from a strain of about 0.03 until recrystallization occurred at a strain in the neighbourhood of 0.07 at 27°C, which caused an acceleration of the creep flow. The smooth transition of the  $t^{\frac{1}{2}}$  flow to the  $t^{\frac{1}{3}}$  flow has been verified experimentally at temperatures from 27° to 150°C. These results confirm the observations of Andrade and Jolliffe (1963) for similar tests at 27°C.

The mechanism of the  $t^{\frac{1}{2}}$  flow is that the number of



visible slip lines increases regularly with strain, while for the  $t^{\frac{1}{3}}$  flow the intensity and width of the individual bands increases, as established by Andrade and Jolliffe (1963). At transition, therefore, fresh bands formed during  $t^{\frac{1}{2}}$  flow cease to appear but all bands increase with intensity. From Andrade and Jolliffe, figure 11, it would appear that with an immediate strain of about 0.01 no visible slip lines appear, whereas for strains increasing from this value through the machinery of the slow  $t^{\frac{1}{2}}$  flow, lines appear in number proportional to the strain. This indicates that the immediate strain does not cause the appearance of fresh slip bands in the way that time-dependent flow does. The persistence of the  $t^{\frac{1}{2}}$  flow at high stresses when the immediate strain exceeds the value at which transition takes place for lower stresses is further evidence that the immediate strain is due to a process quite different from that responsible for creep flow. This is, perhaps, confirmed by the irregularity of the immediate strain produced by a given stress as compared to the regularity of the  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  flow constants (Andrade and Jolliffe III, § 5). Possibly the immediate strain is associated with the imperfectly understood microslip.

Feltham (1956) found that the immediate strain in lead wires in tensile creep was related to the applied

stress by the law of parabolic work hardening for metals of cubic structure. For the present lead III under shear it was found that the instant strain was proportional to a power of the stress, but the power was about 4.2 instead of 2 which characterises the parabolic law. The disparity is much too large to be accounted for by the experimental error in measuring the initial strain. However, the shear stress was applied rather more slowly than in the case of the tensile stress in Feltham's experiments, which attained its full value in about 2 seconds. Thus, it is possible that creep may be occurring in the shear tests in question during the application of the load, which was controlled by a constant speed screw jack, the load taking somewhere about 5 sec. to reach its constant value.

Developments of the theory of rate processes adopted by Kauzmann (1941) for the interpretation of the variation of the secondary creep rate with stress,  $\sigma$  and temperature,  $T$  yield the relationship,

$$d\gamma/dt = K_0 \exp(-E_K/RT) \exp(m_K \sigma/RT) \quad \dots (23)$$

for high stresses and temperatures. According to Dorn (1956) the parameter  $E_K$  is equivalent to the activation energy for

volume self-diffusion in pure metals. The  $t^{\frac{1}{3}}$  coefficient B would be expected to obey a similar law, since  $B^3$  is proportional to K, the flow linear with time, thus,

$$B = B_0 \exp(-E_B/RT) \exp(m_B \sigma/RT) \quad \dots (24)$$

$$\text{where } E_B/E_K = m_B/m_K = \frac{1}{3} \quad \dots (25)$$

Hence, the value of the activation energy for the mechanism of flow in the  $t^{\frac{1}{3}}$  stage should be  $\frac{1}{3}$  of the activation energy pertaining to steady state creep. In the present work, however, the variation of B with temperature was influenced by structural changes in the metal so that an apparent activation energy  $Q_B$  was obtained instead of  $(E_B - m_B \sigma)$ . Thus, according to equation (24)  $\ln B$  should be a linear function of  $1/T$  at constant stress  $\sigma$ , and a linear function of  $\sigma$  at constant temperature T. The experimental results have been shown to satisfy both conditions for the B flow but did not allow the temperature dependence of K flow to be established, because K was too small to be precisely determined except at 60°C. The  $t^{\frac{1}{2}}$  coefficient D varied with stress and temperature in a similar manner to B, but was more sensitive to changes of the stress and the temperature.

The apparent activation energy  $Q_D$  (13) for  $t^{\frac{1}{2}}$  flow is greater than that for  $t^{\frac{1}{3}}$  flow  $Q_B$  (12), yet the  $t^{\frac{1}{2}}$  flow always occurs first. During the  $t^{\frac{1}{2}}$  flow the number of slip bands increases and during the  $t^{\frac{1}{3}}$  flow the number remains constant. The  $t^{\frac{1}{3}}$  mechanism is the formation of fresh lamellae on existing slip bands. For this the energy of activation is smaller than for the formation of separate bands. The formation of slip bands, however, is a necessary preliminary to the formation of lamellae.

The rates of change of  $\ln B$  and  $\ln D$  with stress,  $h_B$  and  $h_D$ , have been shown to vary with the temperature so that  $h = m/RT = f(T)$ . It has been observed, however, that for constant structures and for a limited range of temperature  $h = m/RT$  is independent of temperature (Sherby et al., 1954) for secondary creep flow. In annealed metals and alloys  $h$  generally increases with temperature (Feltham 1956, 1957; Garofalo, 1963). Results for various metals (Feltham, 1957) are found to agree well with the relation,

$$m_K = m_0 \exp(-Q_m/RT) \quad \dots (26)$$

where  $m_0$  and  $Q_m$  are constants. For the  $t^{\frac{1}{3}}$  flow in lead III the variation of  $m_B$  with temperature also satisfied this

relationship. At present little is known with regard to the significance of the activation energy  $Q_m$ .

It has been shown in this investigation that the constant  $H_B$  in the relationship (8) between  $\ln B$  and  $\sigma$  at constant temperature increases only slightly with increasing temperature. A comparison with the theoretical relationship (24) shows that  $H_B$  can be equated with the term  $B_0 \exp(-E_B/RT)$ . It follows, therefore, that  $B_0$  must decrease with the temperature, since the exponential term increases with increasing temperature. Similarly, for secondary creep flow in  $\alpha$ -brass, the term  $K_0 \exp(-E_K/RT)$  in equation (23), expressing the steady flow, was found to be virtually independent of the temperature (Feltham and Copley, 1960).

An interesting feature of the forward creep was the permanent K flow, which at  $60^\circ$  and  $80^\circ\text{C}$  accompanied the normal  $t^{1/2}$  and  $t^{1/3}$  creep flow. No K flow had been detected in lead III at  $27^\circ\text{C}$  although a comparatively small linear flow was observed at high stresses in lead I (Andrade, 1962), which was thought to be due to the relative movement of adjacent crystals. The tests on lead III at  $60^\circ$  and  $80^\circ\text{C}$  showed that the proportion of the K flow in the forward creep increased with rise in temperature and decrease in stress. This suggests that the K flow may be related to grain boundary sliding

which behaves similarly to K flow with stress and temperature. The largest permanent strain obtained, 0.011, was for a test at 80°C and 25 Kg/cm<sup>2</sup> when the K flow constituted almost 20% of the flow in FI. Measurements of grain boundary sliding at offsets on marker lines could only account for about half the observed K flow, probably because of the difficulty in detecting small displacements. Conditions which favour B flow would be expected to be unfavourable to grain boundary sliding, since deformation of the grains would affect the topography of the sliding grain interfaces. A smoothing of grain interfaces can occur by grain boundary migration, a phenomenon which has been observed at the temperatures at which K flow prevails. Thus, the migration of a grain boundary can facilitate further grain boundary sliding by relieving the internal boundary stresses which oppose further sliding. Evidence of such a mechanism of shear has been observed in photomicrographs (figure 27) in which alternate periods of grain boundary sliding and migration are observed.

### 3. Reversals of Stress

Andrade and Jolliffe (1963), working with lead III at 27°C, showed that the creep behaviour after a stress reversal was precisely determined by the constants in the forward

flow and the time  $t_f$ , whatever the stress, provided that the forward creep was of the  $t^{\frac{1}{3}}$  type. When a permanent flow is present in the forward creep, as in the tests at higher temperatures, the regular behaviour of the reverse flow following a  $t^{\frac{1}{3}}$  flow is modified.

The  $R_0$  stage after reversal is a linear flow which restores the material to a condition approaching that prior to loading, so that the subsequent flow in RI obeys a  $t^{\frac{1}{3}}$  law. The presence of the K term in stage FI causes a reduction of the time of flow in stage  $R_0$  and changes the value of p. It is suggested that the internal stresses obtained during forward flow are greater when the grain boundary sliding is present in addition to the intragranular slip. Thus, on reversal of the applied stress, the local stresses are considerably larger, giving a faster rate of flow and a shorter time for the material to achieve a condition favourable for  $t^{\frac{1}{3}}$  flow.

In the absence of a permanent component in FI, repeated reversals of the applied stress at intervals equal to the time of forward flow produced a  $t^{\frac{1}{3}}$  flow in the forward direction and a linear flow in the reverse direction. This process was shown by Andrade and Jolliffe (1960) to be a forward and backward movement involving the same lamellae.

The presence of the K term in FI, however, resulted in a linear stage being followed by  $t^{\frac{1}{3}}$  stage in the reverse direction and also in the forward direction. Again the duration of linear flow is curtailed, so that a  $t^{\frac{1}{3}}$  flow commences before the next reversal. This process is repeated after each reversal of the stress. Such behaviour is very similar to that in figure 10, Andrade and Jolliffe III, for repeated reversals with the duration of each reversal equal, but greater than  $t_f$ . It is assumed that the presence of grain boundary sliding increases the internal stresses because of the geometric restraint arising from the different shapes of randomly oriented crystals. Such internal stresses may be relieved under certain conditions by the mechanism of grain boundary migration.

#### 4. Linear Flow in Stage III

This linear flow prevails after the grain structure has been thoroughly broken up and extensive recrystallization has occurred. A unique feature of this linear creep is that the flow is of the same nature and at the same strain rate,  $v$ , when the stress is reversed. The material in this condition behaves like a non-Newtonian liquid.

The variation of the linear flow,  $v$ , with stress and



temperature is analogous to that of the secondary creep rate  $dy/dt$  at high stresses,

$$v \exp(Q_v/RT) = v_0 \exp(h_v \sigma) \quad \dots (27)$$

$$d \gamma/dt \exp(E_K/RT) = K_0 \exp(m_K \sigma/RT) \quad \dots (28)$$

where the Zener-Hollomon parameters are functions of structure and stress. The value of  $h_v$  was independent of stress and temperature and the structure parameter  $v_0$  was also constant indicating that the relationship is applicable to a constant structure. For secondary creep the stress coefficient  $m_K$  varies with temperature and the stress function is independent of temperature if  $m_K/RT$  is constant. Thus the two equations are directly comparable if  $m_v = h_v RT$ . The form of the equation describing linear flow appears to be dependent on whether the substructure changes during creep, which in stage III it does not. At constant structures, therefore, the activation energy for  $v$  flow is independent of the stress.

The stress dependence of  $v$  at various temperatures has been shown to apply to stresses above a minimum value,  $\sigma_v$ , which is a regular function of the temperature. The law

could not hold at lower values of stress since it predicts a finite value of  $v$  at zero stress. The functional form of the stress dependence below  $\sigma_v$  was not determined but in this region  $v$  was more sensitive to change of stress at constant temperature. The  $v$  flow appears to be a balance between work hardening under the action of the applied stress and recovery due to recrystallization.

#### 5. Tests below room temperature

A region of subnormal flow has been established below a stress,  $\sigma_c$ , which was a function of temperature, increasing as the temperature was decreased. Above this stress the normal  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  laws of flow prevailed. Feltham and Meakin (1959) observed a similar transition stress for the variation of the equilibrium creep rate of copper with stress at constant temperature. The region of flow at the low stresses was quite distinct from that at the higher stresses. Similarly, Andrade and Aboav (1964) showed that the behaviour of cadmium under simple shear changed from the normal  $t^{\frac{1}{3}}$  flow to a subnormal exponential flow as the stress was decreased. At stresses close to the critical stress both normal and subnormal types of flow occurred

together, making accurate analysis rather difficult. In the case of the tests on lead III, however, it was not possible to isolate a pure exponential flow even at stresses much below the transition stress. For instance, in a test at 0°C and 55 Kg/cm<sup>2</sup>, about 12 Kg/cm<sup>2</sup> below  $\sigma_c$ , the ratio of the strain due to  $t^{\frac{1}{3}}$  flow to that due to exponential flow was about 4 : 1.

The exponential flow in cadmium was attributed to a process of grain boundary sliding in the absence of intragranular glide which obtains above  $\sigma_c$  on the basal slip planes. Lead, being a metal of F.C.C. structure, possesses several slip systems which allows intragranular slip to occur rather easily compared with cadmium. Thus, only at very low stresses would the resolved shear stress along all the various slip directions be unfavourable for slip, which is the necessary condition for creep occurring by the relative movement of adjacent grains.

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ACKNOWLEDGEMENTS

The advice and encouragement given by Professor E.N. da C. Andrade, F.R.S., who suggested the investigation described in this thesis, is gratefully acknowledged.

I wish to thank Professor J.G. Ball for his interest and the laboratory facilities he kindly provided.

I am indebted to Mr. L. Walden for his generous help and technical advice, and to the Central Electricity Generating Board for financial support during this investigation.

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- Figure 23. Creep flow at 150°C.

- Figure 24. Grain boundary migration and sliding at 150°C.
- Figure 25. Grain growth at 60°C.
- Figure 26. Movement of grain boundary during creep at 60°C.
- Figure 27. Grain boundary migration and sliding at 80°C.

Table 1

Validity of  $t^{\frac{1}{2}}$  flow above  $\gamma = 0.01$  for test at  $80^{\circ}\text{C}$   
and  $25 \text{ Kg/cm}^2$

$$\gamma_{\text{calc}} = -0.0004 + 0.00236t^{\frac{1}{2}} + 2.5 \times 10^{-5}t$$

t(min)	$10^4 \gamma_{\text{obs}}$	$10^4 Kt$	$10^4 (\gamma_{\text{obs}} - Kt)$	$10^4 (\gamma_{\text{calc}} - Kt)$	$10^4 (\gamma_{\text{obs}} - \gamma_{\text{calc}})$
4	57	1	56	47	9
7.5	68	2	66	61	5
10	77	3	74	70	4
20	107	5	102	101	1
30	132	8	124	125	-1
40	156	10	146	145	1
50	174	13	161	162	-1
60	193	15	178	178	0
80	227	20	207	206	1
100	258	25	233	231	2
140	308	35	273	274	-1
160	331	40	291	293	-2
170	343	42	301	302	-1
180	353	45	308	311	-3
240	411	55	356	360	-4

N.B. Transition strain  $\gamma_c = 0.0248$

Table 2

Validity of  $t^{\frac{1}{3}}$  law for test at 80°C and 25 Kg/cm<sup>2</sup>

$$\gamma_{\text{calc}} = -0.0118 + 0.00754t^{\frac{1}{3}} + 2.5 \times 10^{-5}t$$

t(min)	$10^4 \gamma_{\text{obs}}$	$10^4 Kt$	$10^4 (\gamma_{\text{obs}} - Kt)$	$10^4 (\gamma_{\text{calc}} - Kt)$	$10^4 (\gamma_{\text{obs}} - \gamma_{\text{calc}})$
40	156	10	146	140	6
60	193	15	178	178	0
80	227	20	207	207	0
100	258	25	233	232	1
140	308	35	273	273	0
180	353	45	308	308	0
220	391	55	336	337	-1
260	429	65	364	363	1
300	460	75	385	386	-1
340	493	85	408	408	0
380	523	95	428	428	0
420	552	105	447	447	0
460	583	115	468	464	4
500	622	125	497	481	16

Table 3

Comparison of values of  $\gamma_c$  derived from equations (4) and (6)

$$\gamma_c = A + 4B^3/9D^2 + K(2B/3D)^6 \quad \dots (4)$$

$$\gamma_c = 3C - 2A \quad \dots (6)$$

Temperature (°C)	Stress (Kg/cm <sup>2</sup> )	10 <sup>4</sup> $\gamma_c$ from (4)	10 <sup>4</sup> $\gamma_c$ from (6)	10 <sup>4</sup> Average $\gamma_c$
27	29	277	275	276
	39	382	392	387
	49	574	574	574
40	25	267	268	267
50	25	275	271	273
60	20.35	244	220	232
	25	212	196	204
	30	246	238	242
	33	242	247	244
80	21	238	230	234
	25	248	222	235
	31	284	283	283
60 *	30	258	239	248

\* Specimen of large grain size  $n = 1/\text{mm}$ .

Table 4

Validity of  $t^{\frac{1}{2}}$  and  $t^{\frac{1}{3}}$  laws for lead at  $150^{\circ}\text{C}$

and stress  $9 \text{ Kg/cm}^2$

$$\gamma_{\text{calc}} = 0.0024 + 0.00221t^{\frac{1}{2}}$$

$$\gamma_{\text{calc}} = -0.0036 + 0.00586t^{\frac{1}{3}}$$

time (min)	$10^4 \gamma_{\text{obs}}$	$t^{\frac{1}{2}}$			$t^{\frac{1}{3}}$	
		$10^4 \gamma_{\text{calc}}$	$10^4 (\gamma_{\text{obs}} - \gamma_{\text{calc}})$		$10^4 \gamma_{\text{calc}}$	$10^4 (\gamma_{\text{obs}} - \gamma_{\text{calc}})$
1	46	46	0	23	23	
2	57	55	2	38	19	
5	72	73	-1	64	8	
10	93	94	-1	90	3	
20	124	123	1	123	1	
30	145	145	0	146	-1	
40	162	164	-2	164	-2	
60	194	195	-1	193	1	
80	218	222	-4	217	1	
100	237	245	-8	236	1	
130	260			261	-1	
160	281			282	-1	
200	306			306	0	
250	333			333	0	
300	356			356	0	
350	378			377	1	
400	397			396	1	
450	415			413	2	
500	434			429	5	
550	452			444	8	
600	470			458	12	

Table 5

Combined normal and sub-normal flow at 0°C and 55 Kg/cm<sup>2</sup>

$$10^4 \gamma = 217 + 42t^{\frac{1}{3}} + 51(1 - e^{-0.142t})$$

time (min)	$10^4 \gamma_{\text{calc}}$	$10^4 \gamma_{\text{obs}}$	$10^4 (\gamma_{\text{calc}} - \gamma_{\text{obs}})$
$\frac{1}{2}$	253	252	1
$1\frac{1}{2}$	275	275	0
3	296	296	0
5	315	316	-1
$7\frac{1}{2}$	332	332	0
10	346	346	0
14	362	360	2
20	379	377	2
$25\frac{1}{2}$	391	390	1
30	397	398	-1
40	412	413	-1
50	423	423	0
60	432	433	-1
80	449	449	0
110	469	472	-3



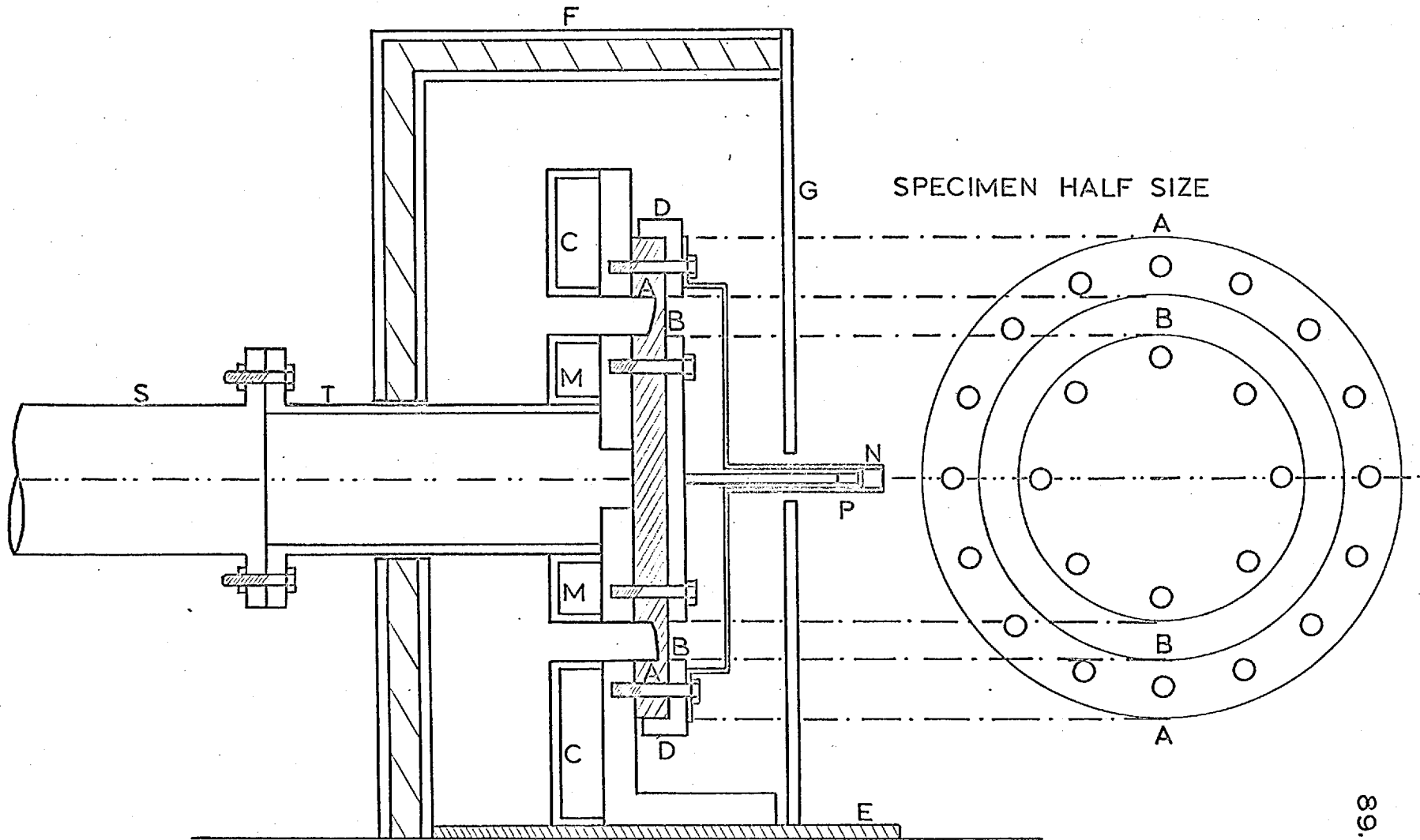
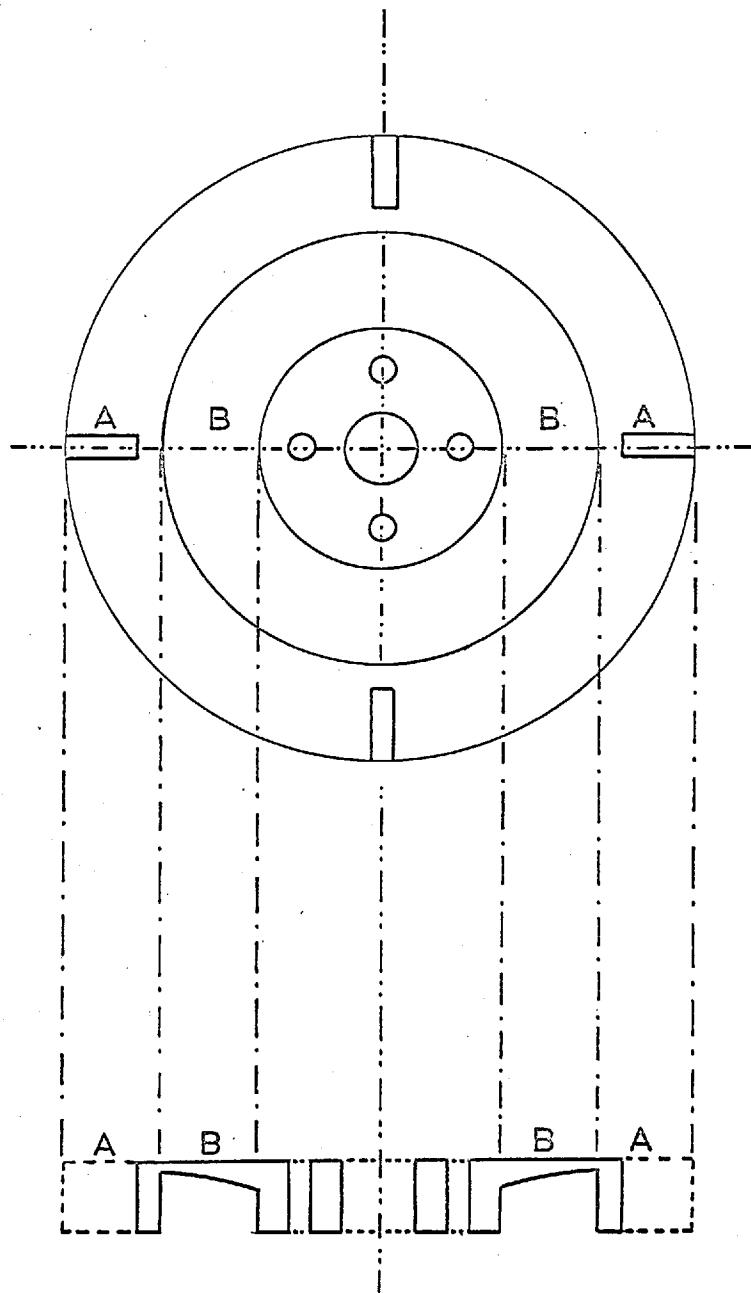


Figure 1



FULL SIZE

Figure 2a

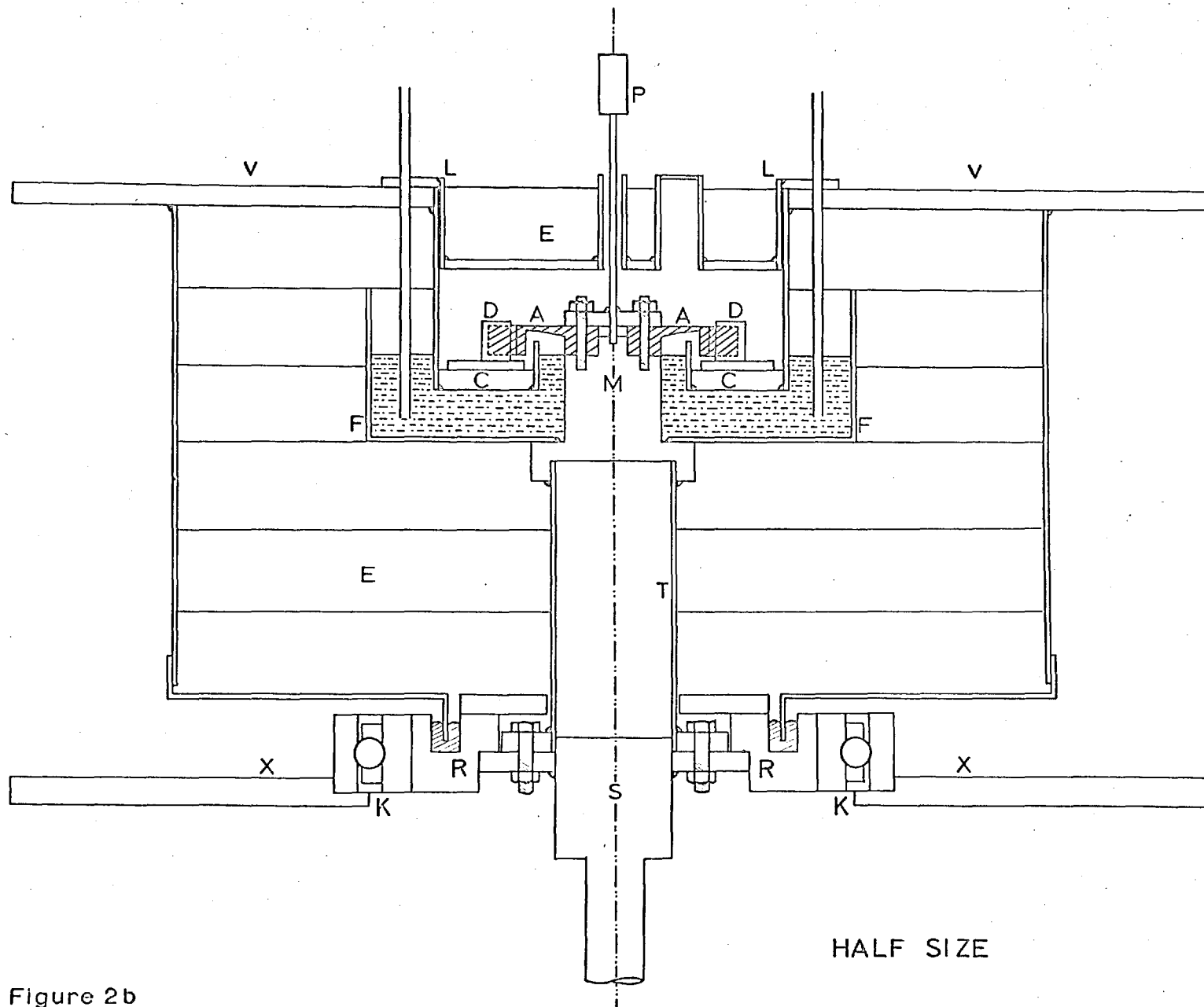


Figure 2b

HALF SIZE

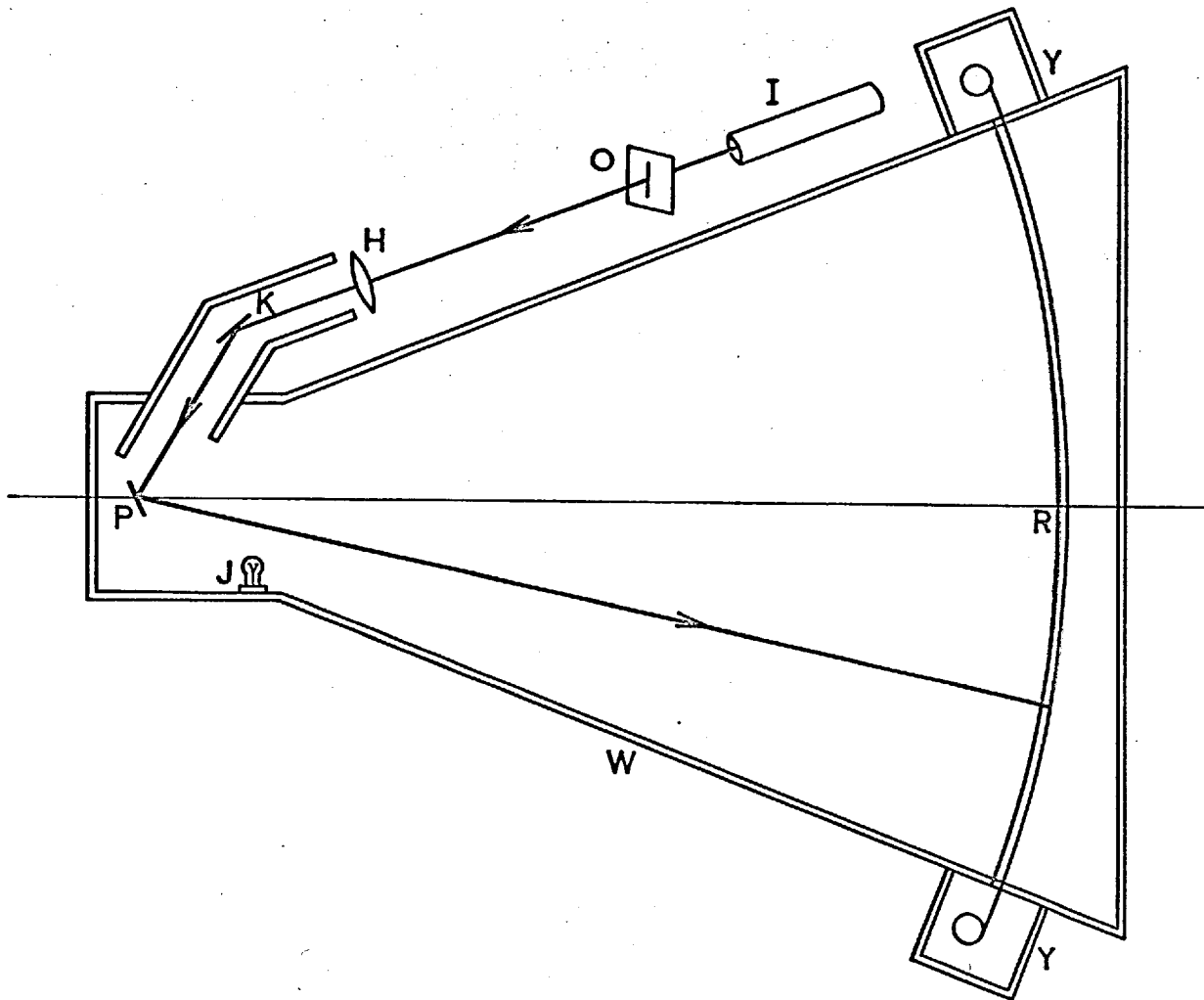


Figure 3

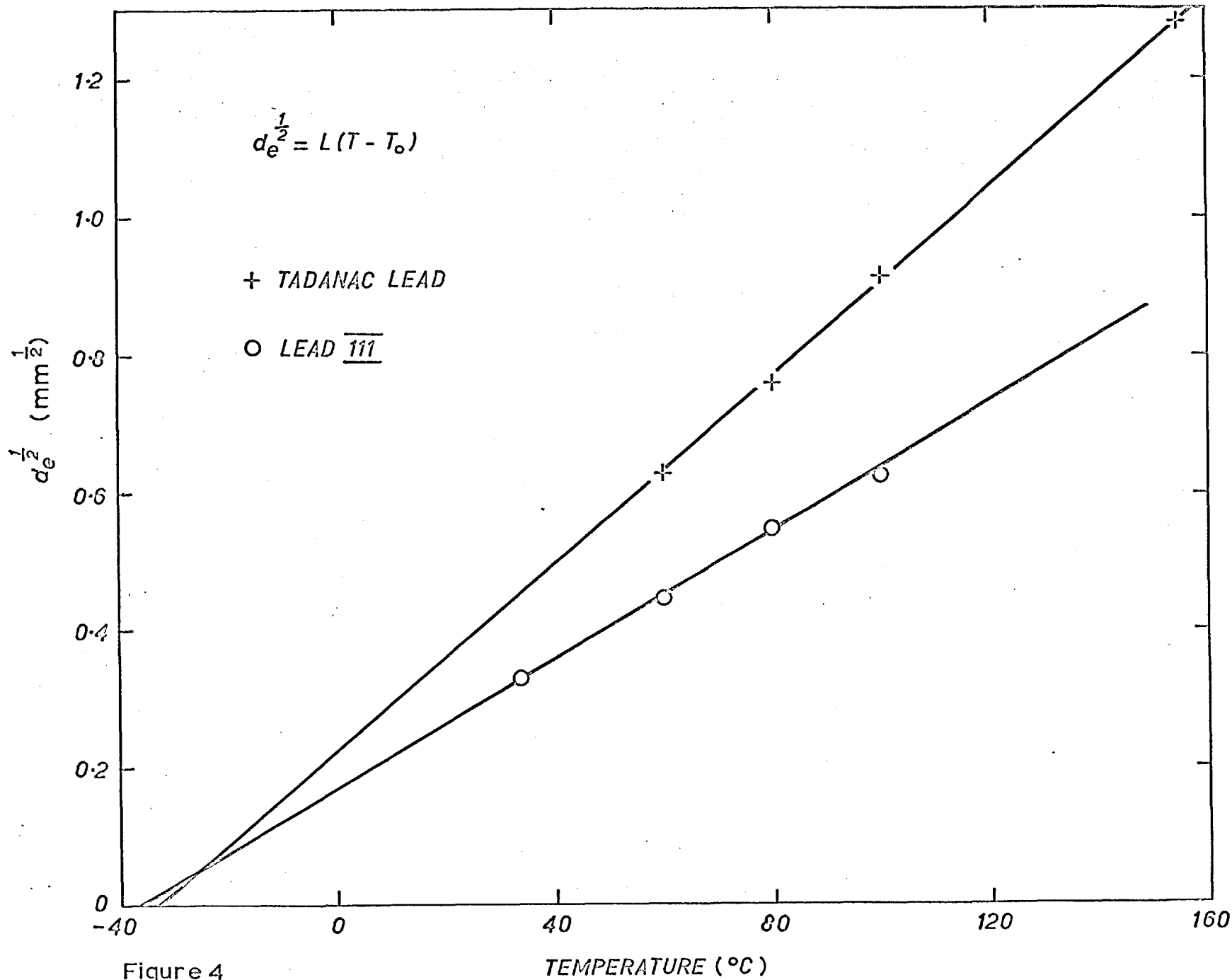


Figure 4

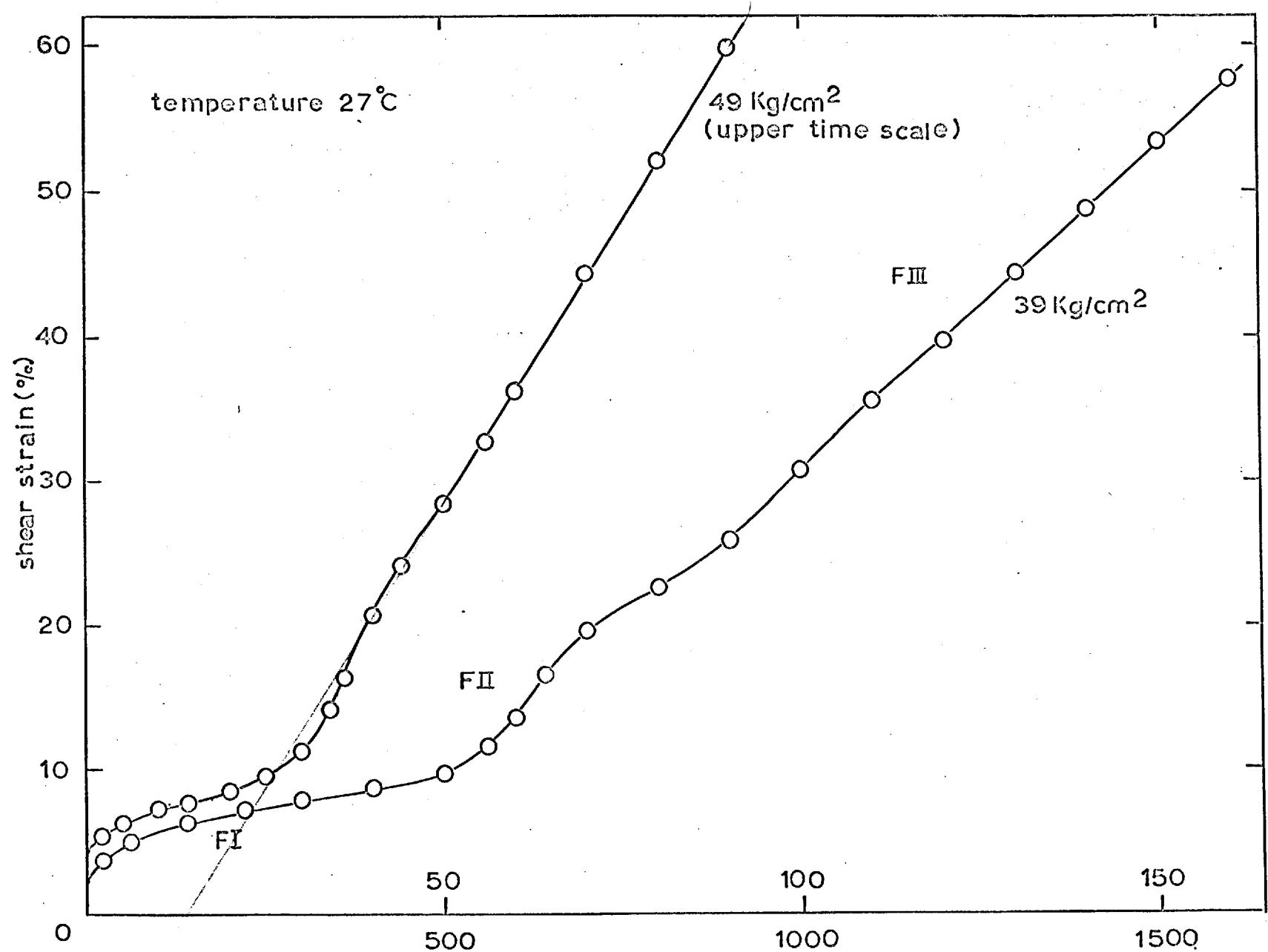
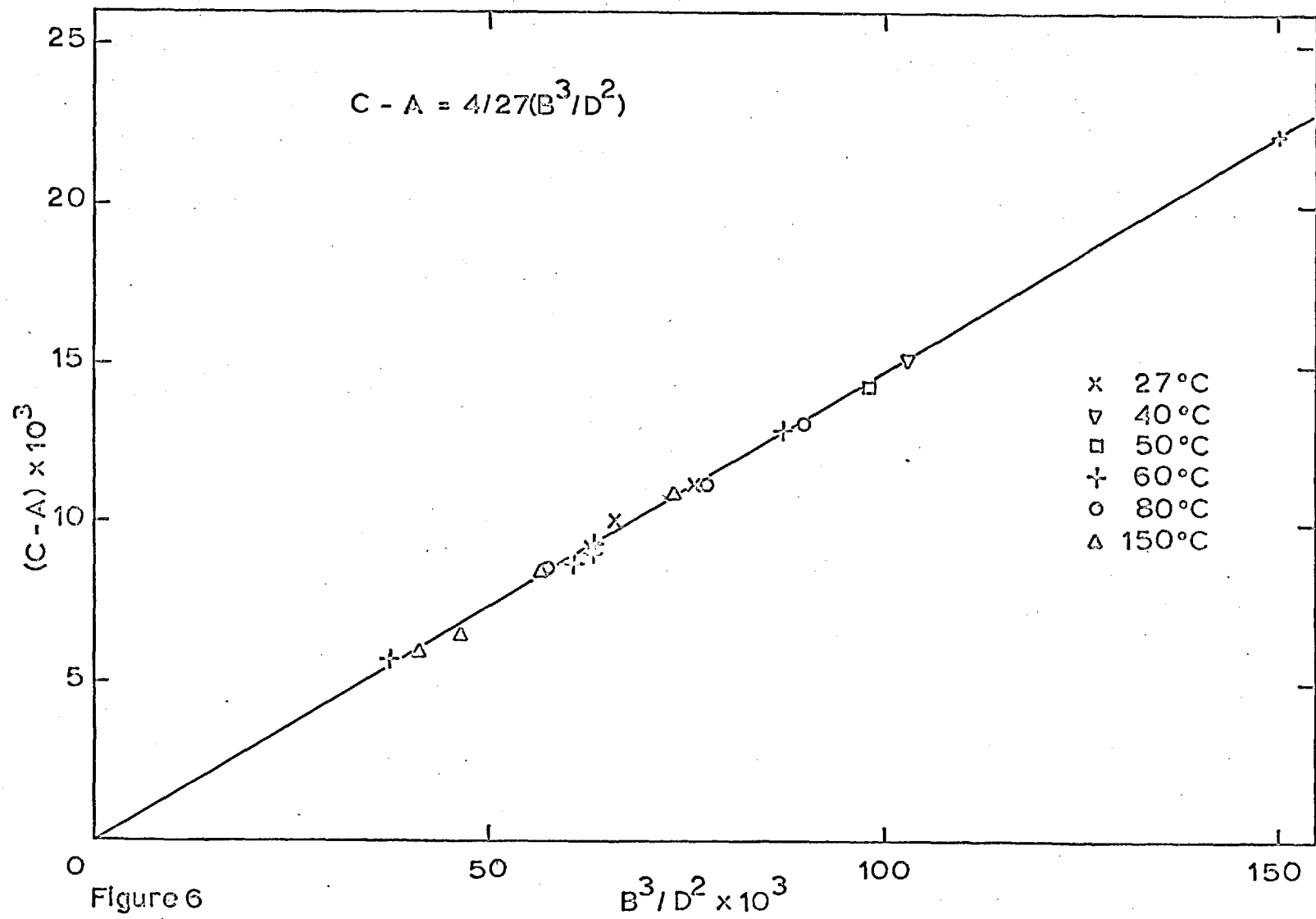


Figure 5



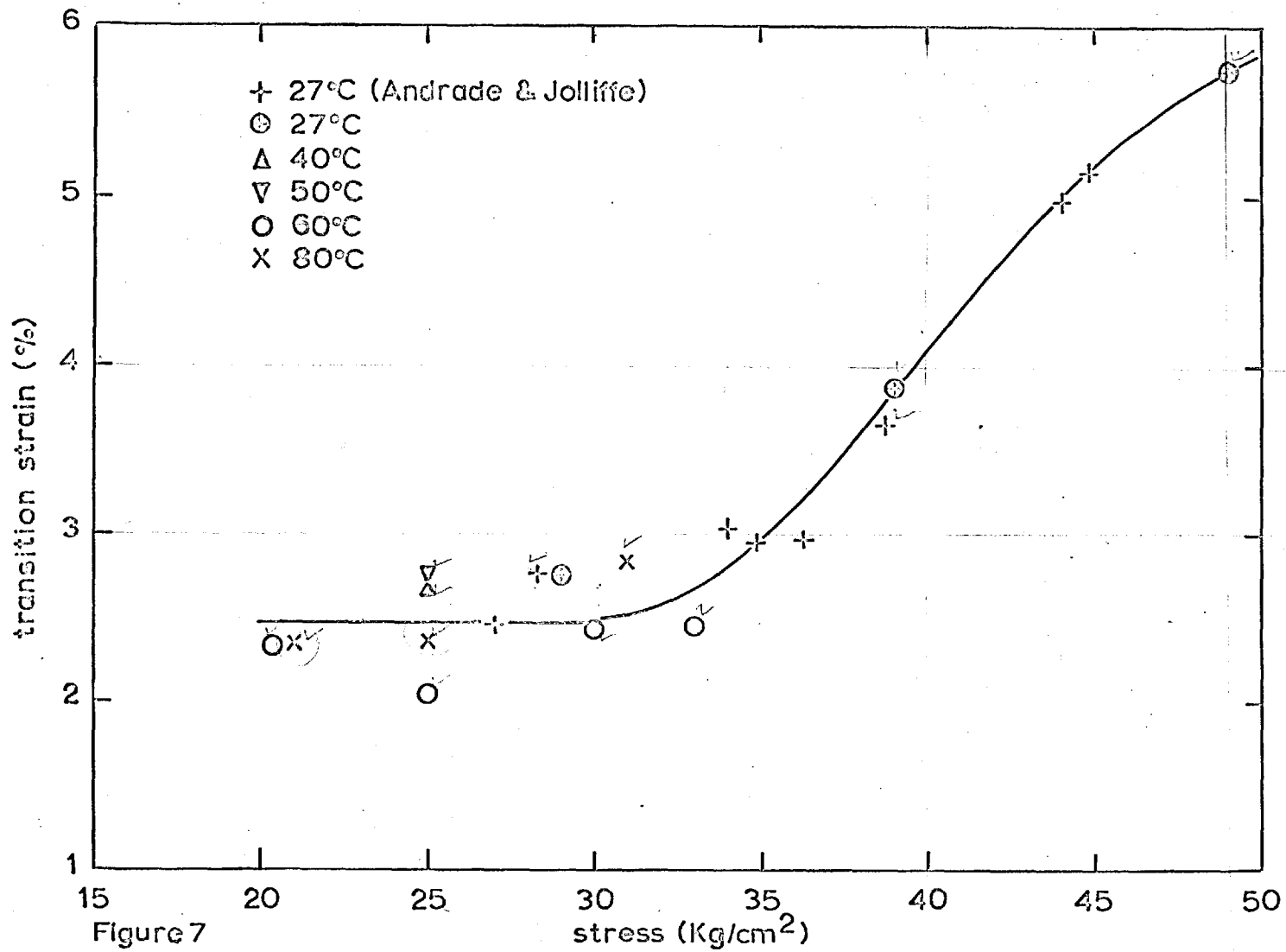


Figure 7



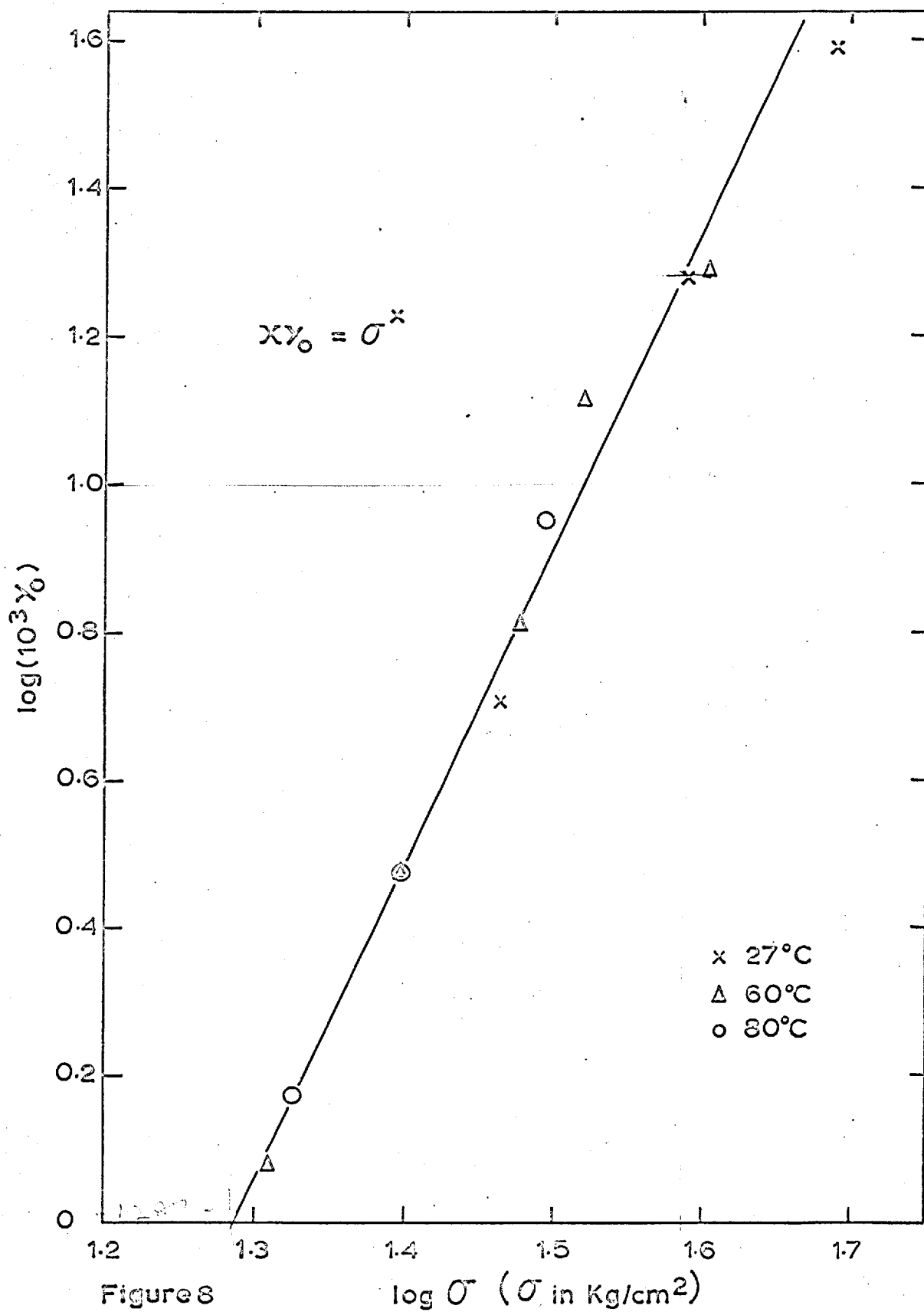


Figure 8

 $\log \sigma$  ( $\sigma$  in  $\text{Kg/cm}^2$ )

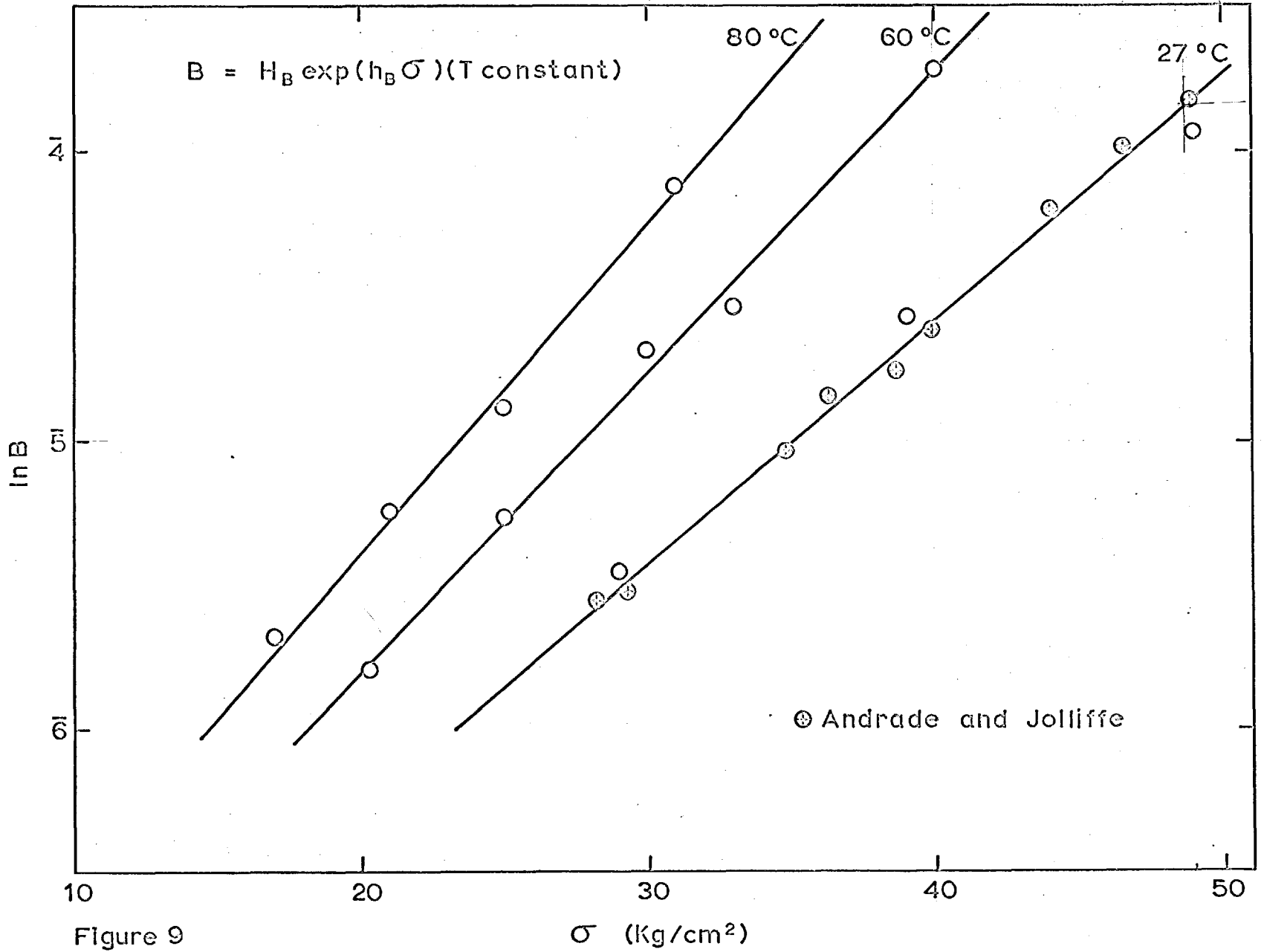


Figure 9

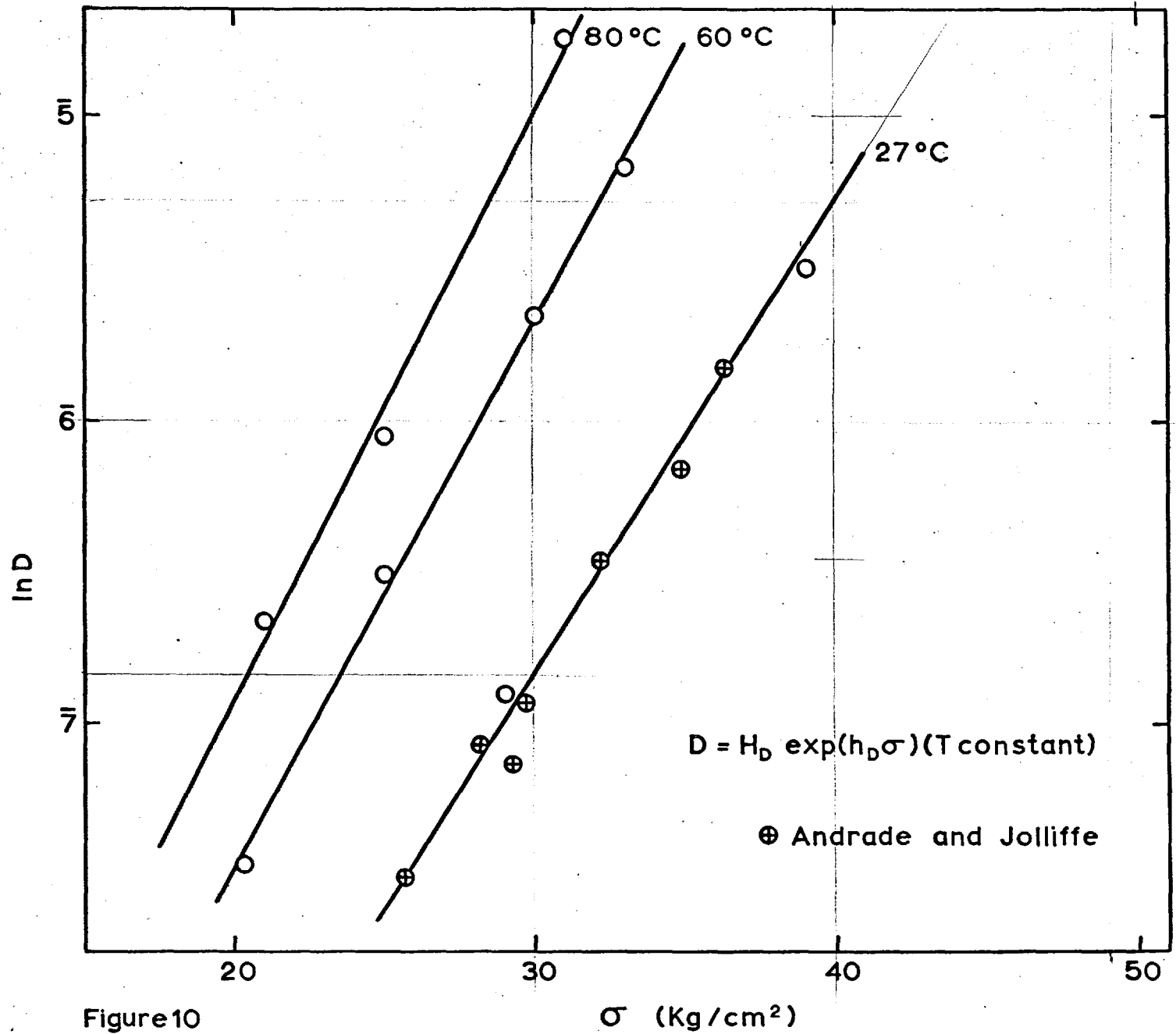


Figure 10

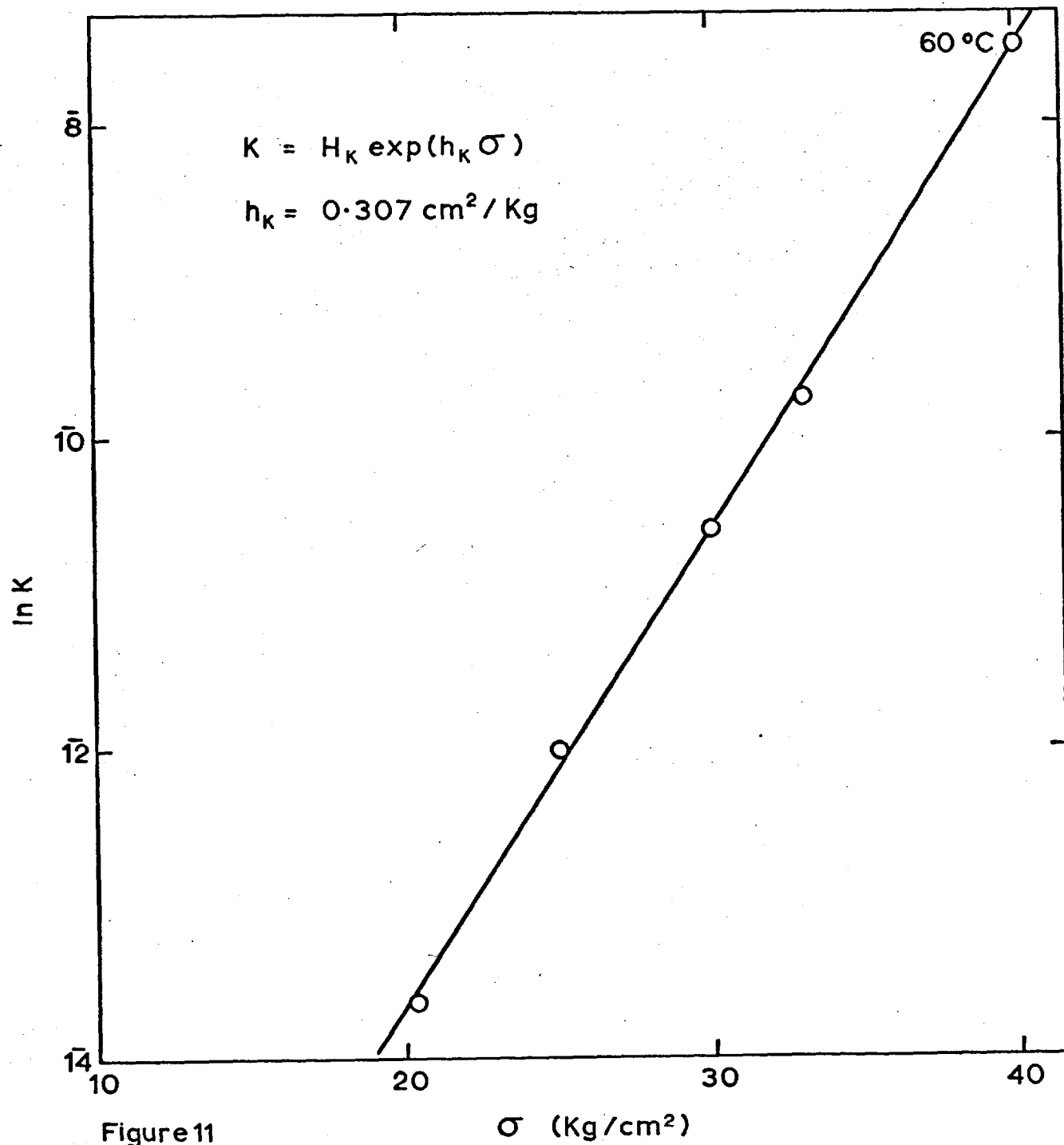


Figure 11

$\sigma$  (Kg/cm<sup>2</sup>)

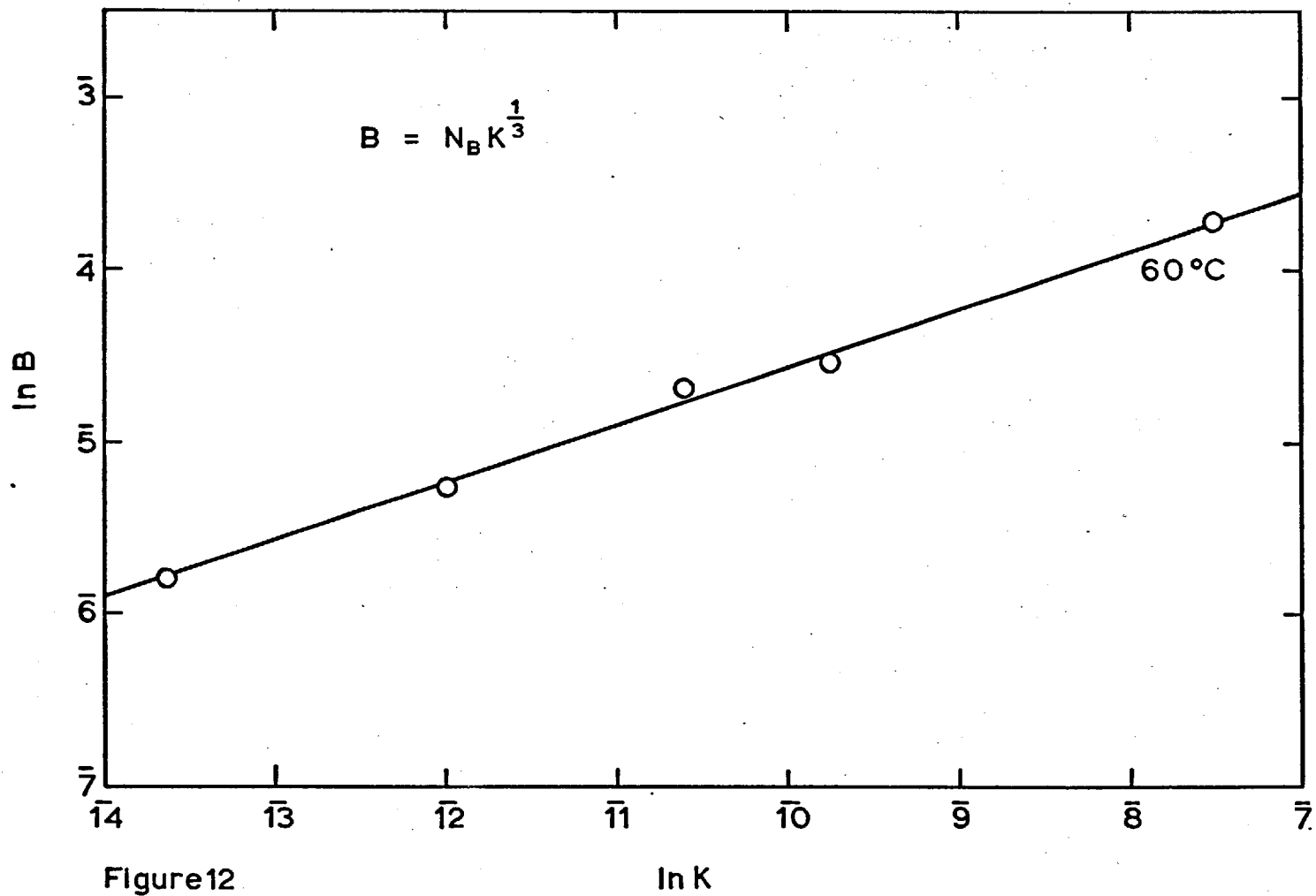


Figure 12

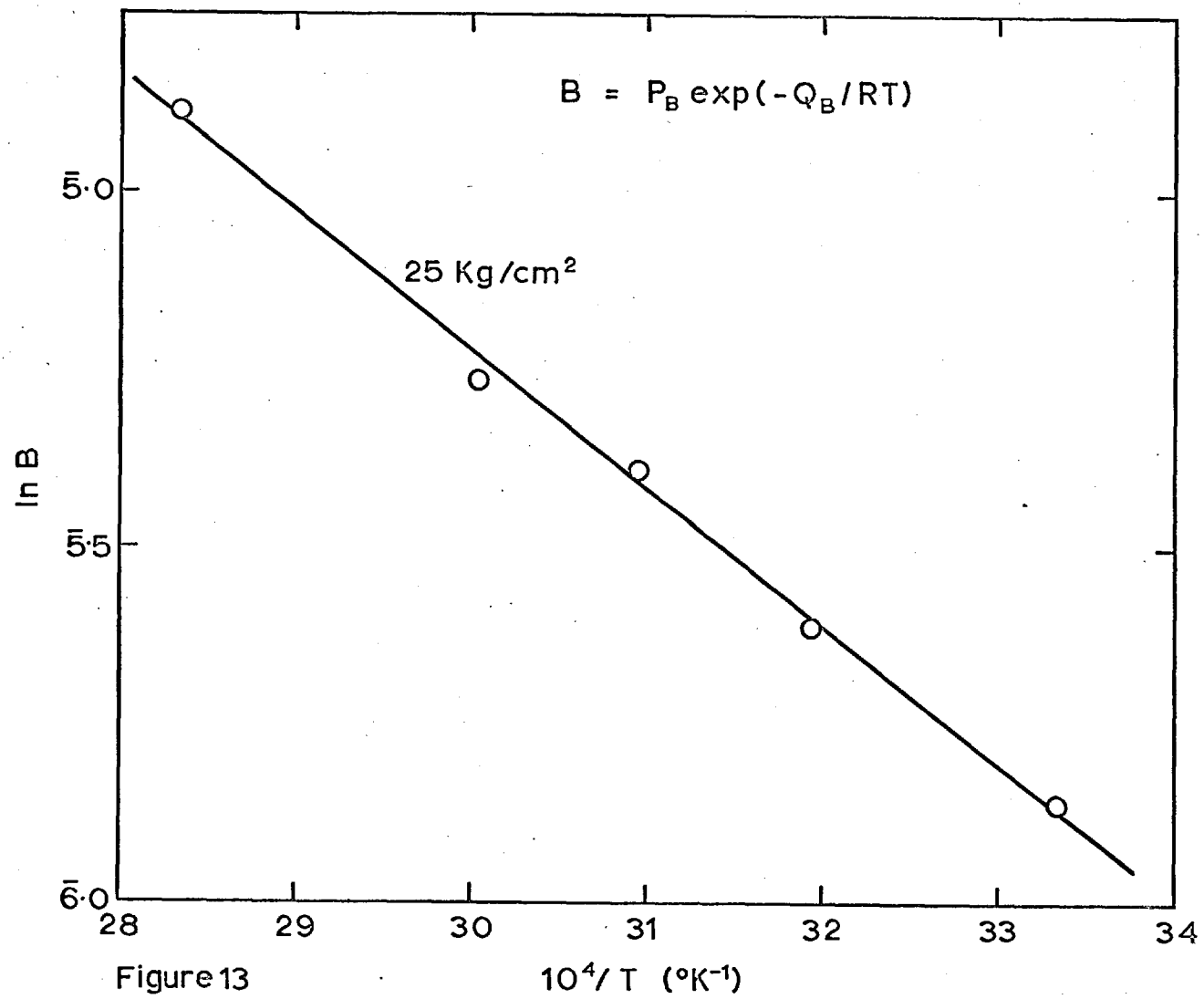


Figure 13

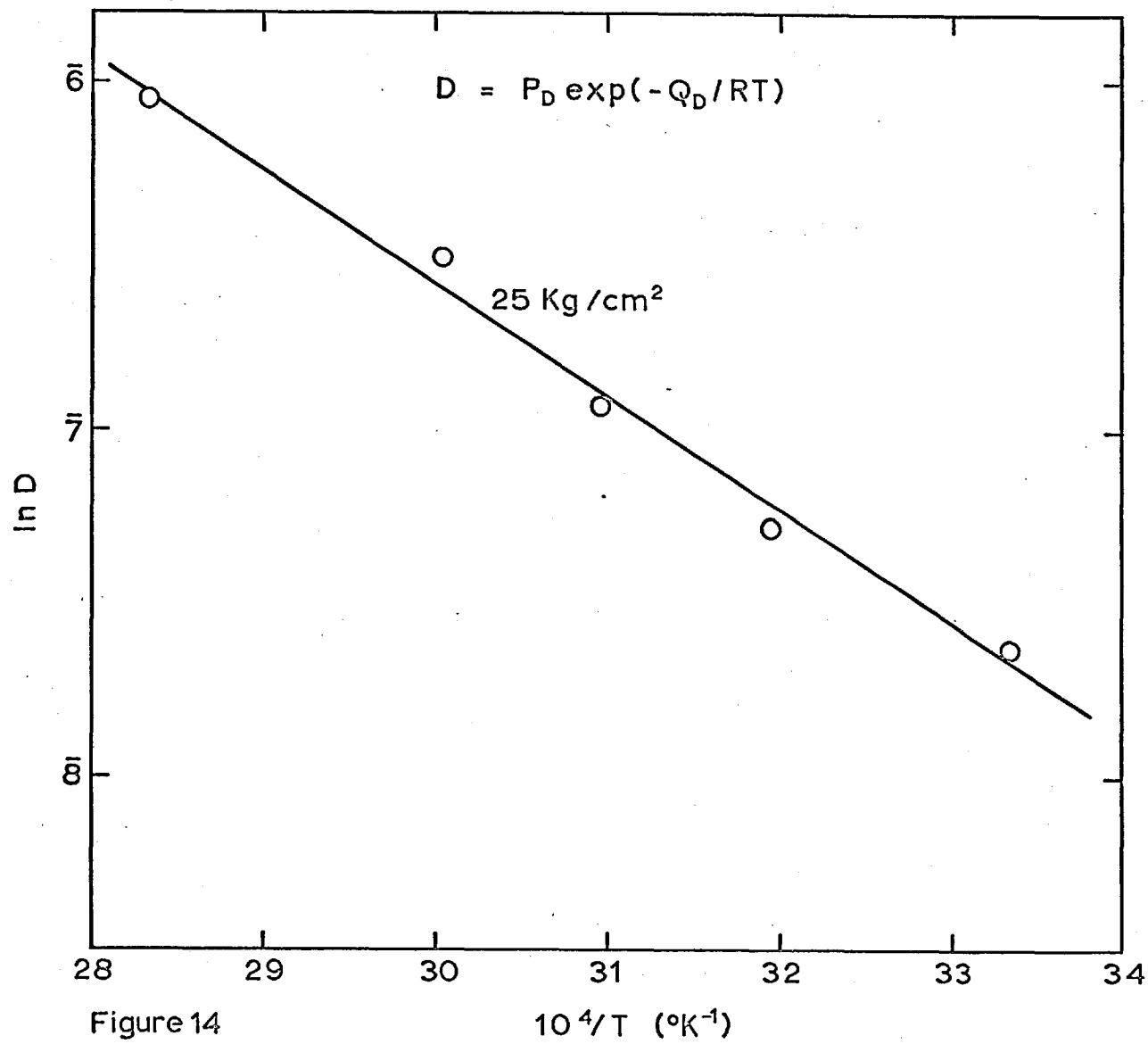


Figure 14

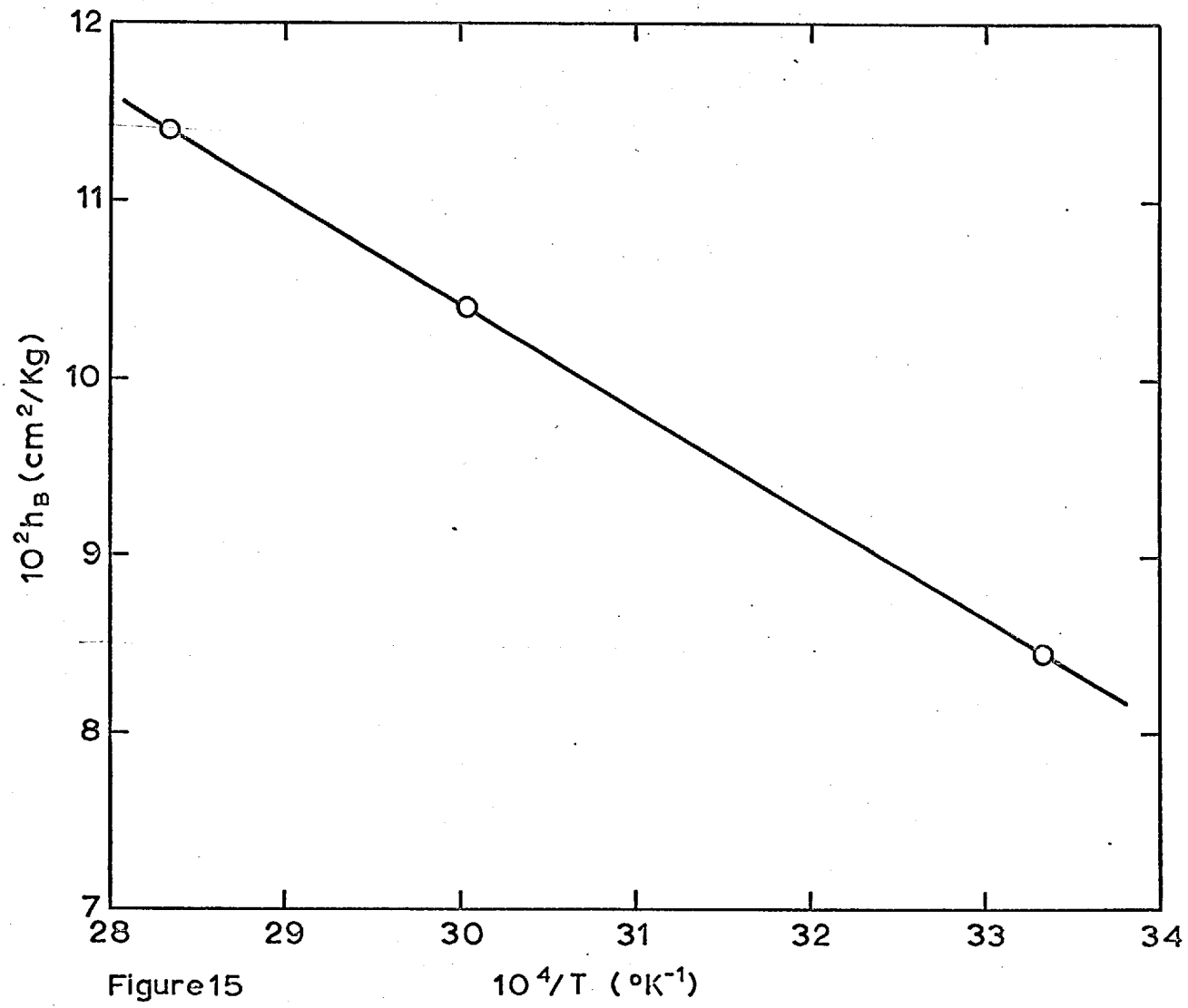


Figure15



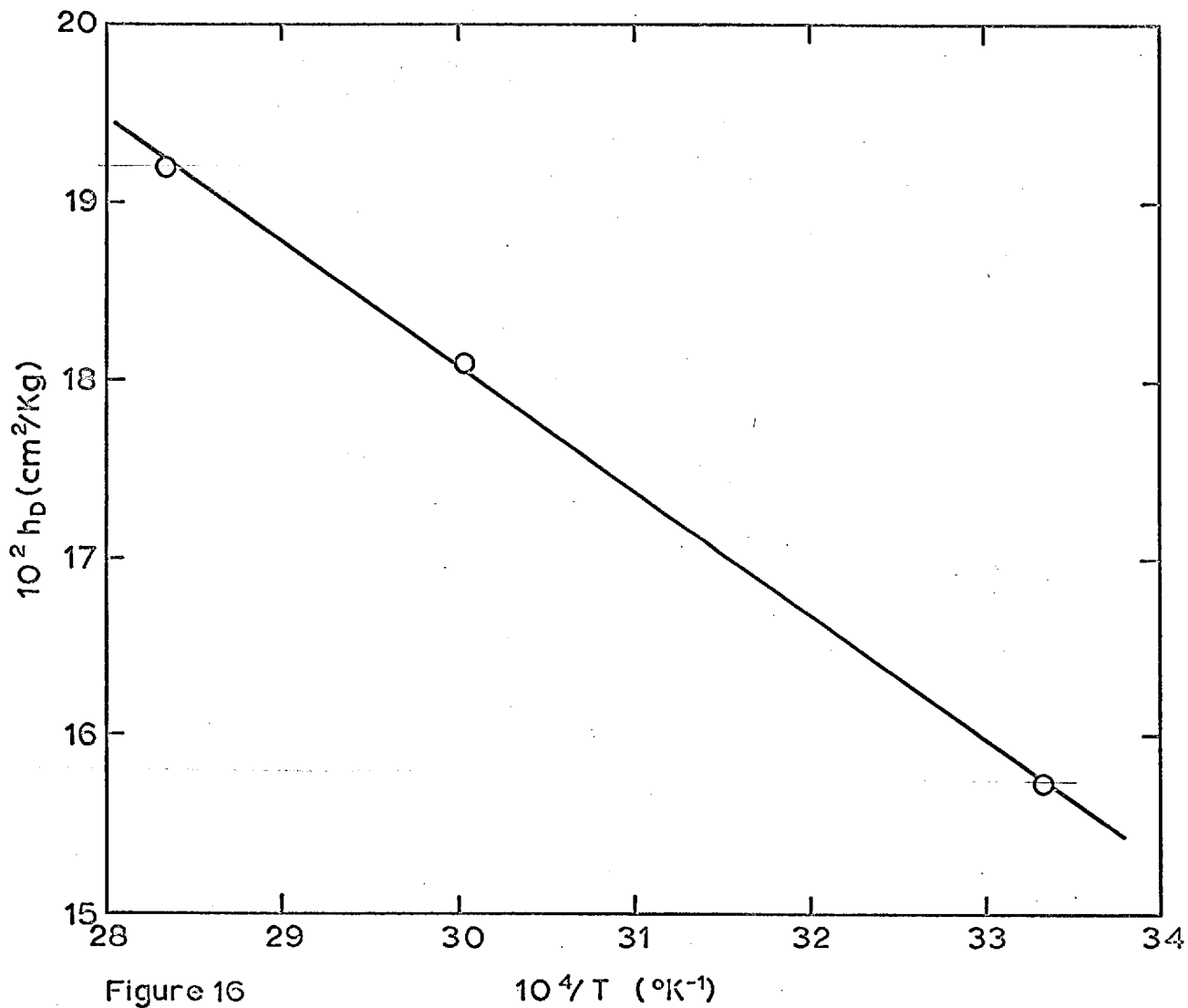


Figure 16

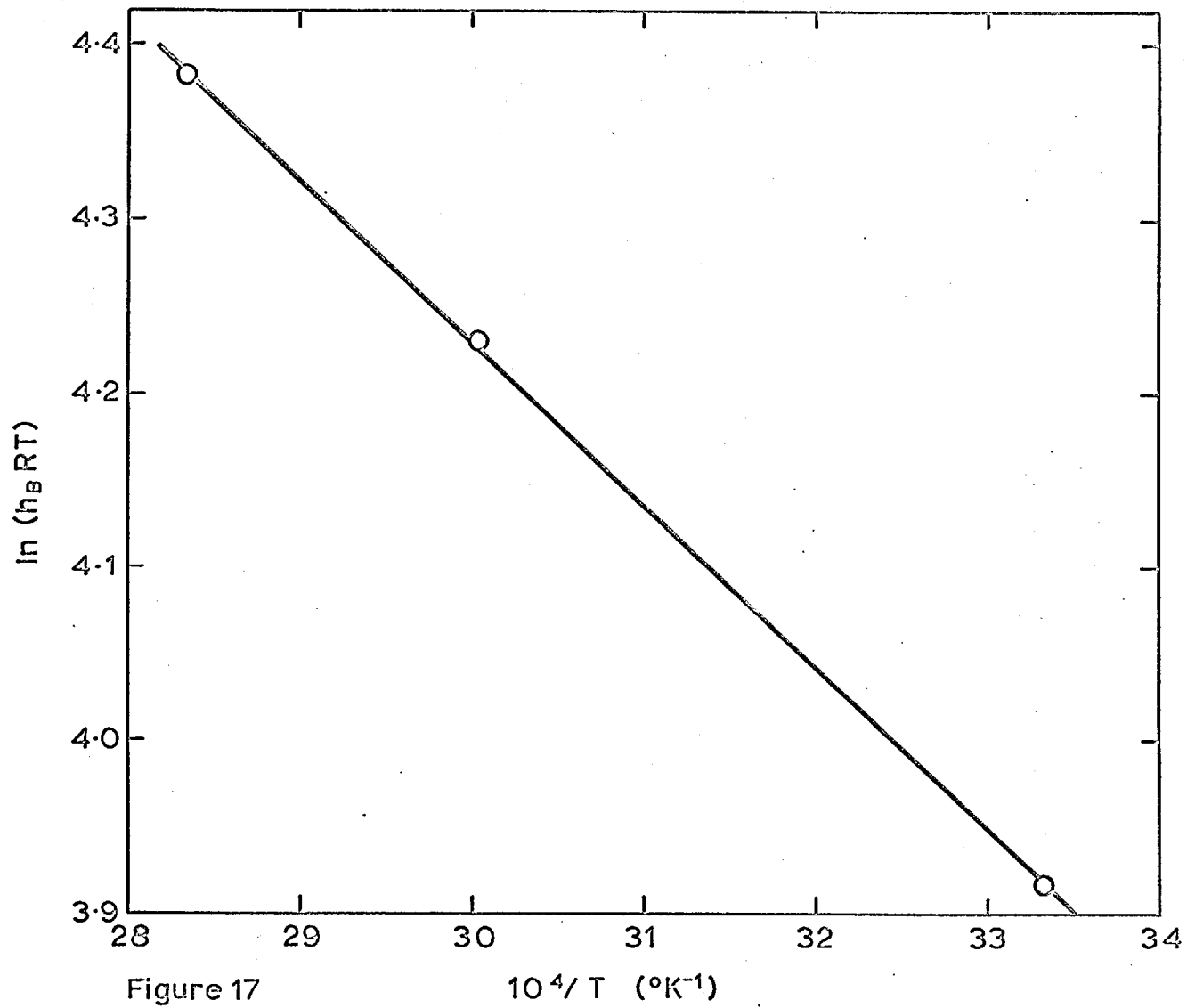


Figure 17

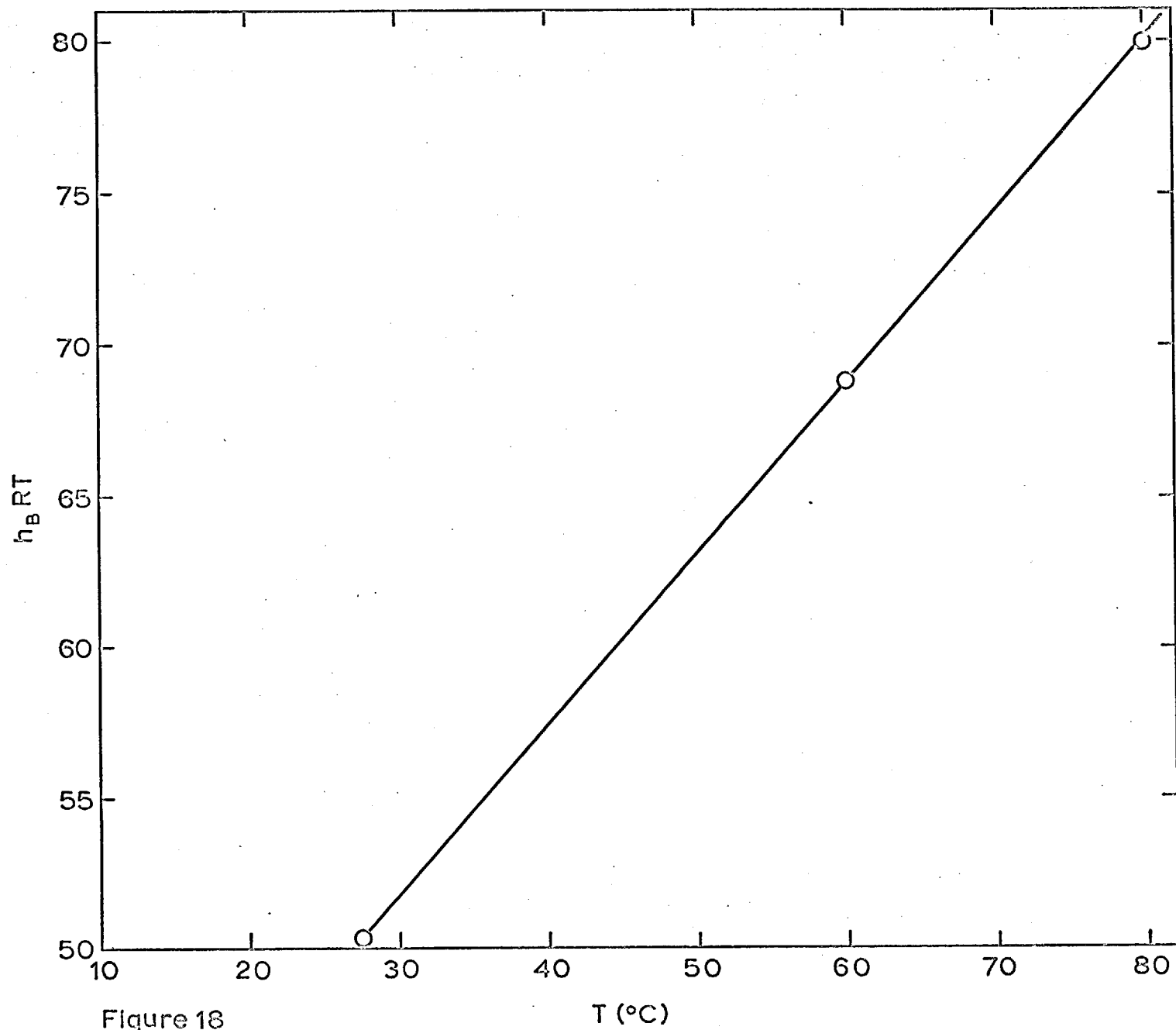
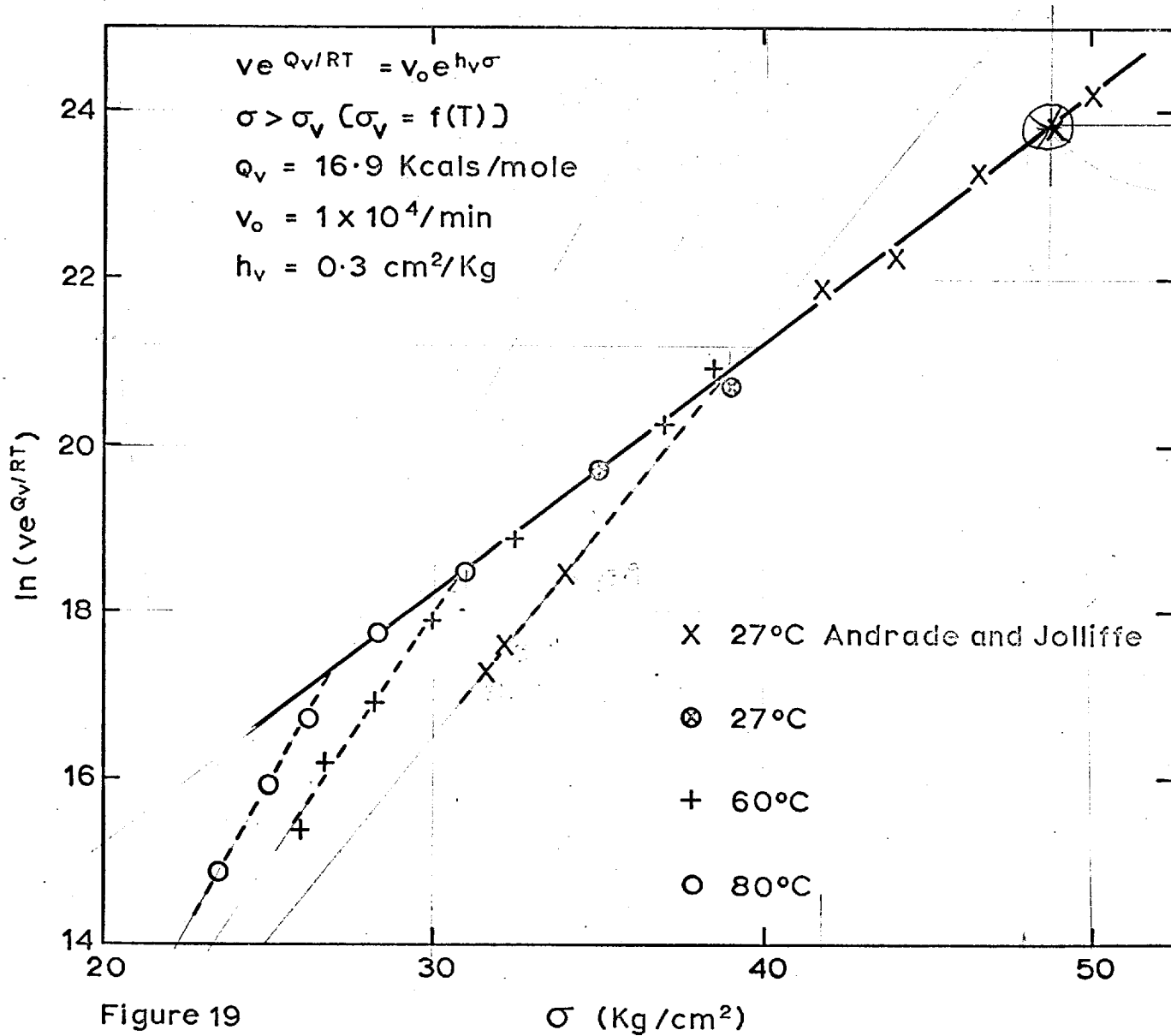


Figure 18

T (°C)



Handwritten notes on the right side of the graph:  
 -  $\sigma_v = 2.5$   
 -  $h_v = 0.3$

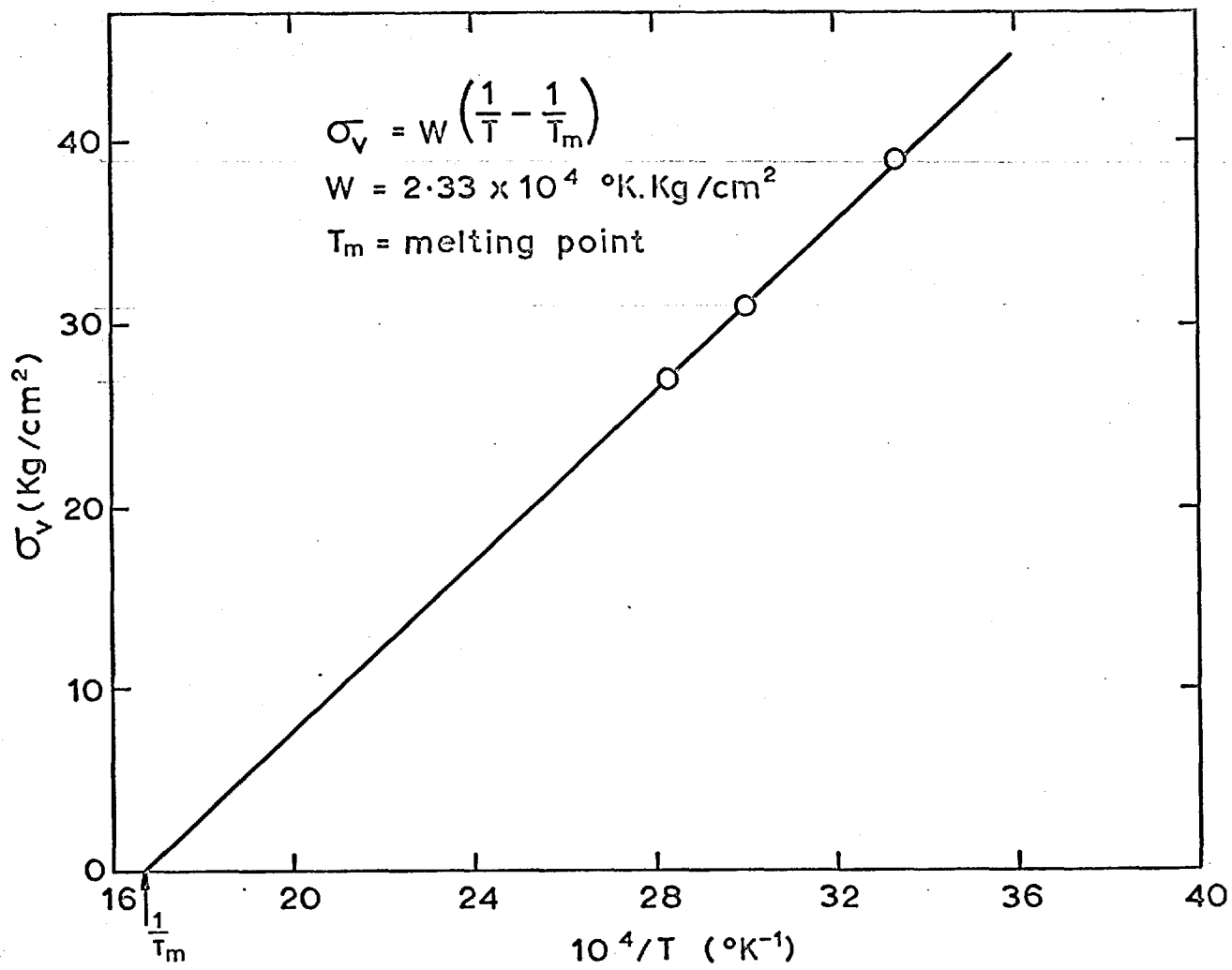


Figure 20

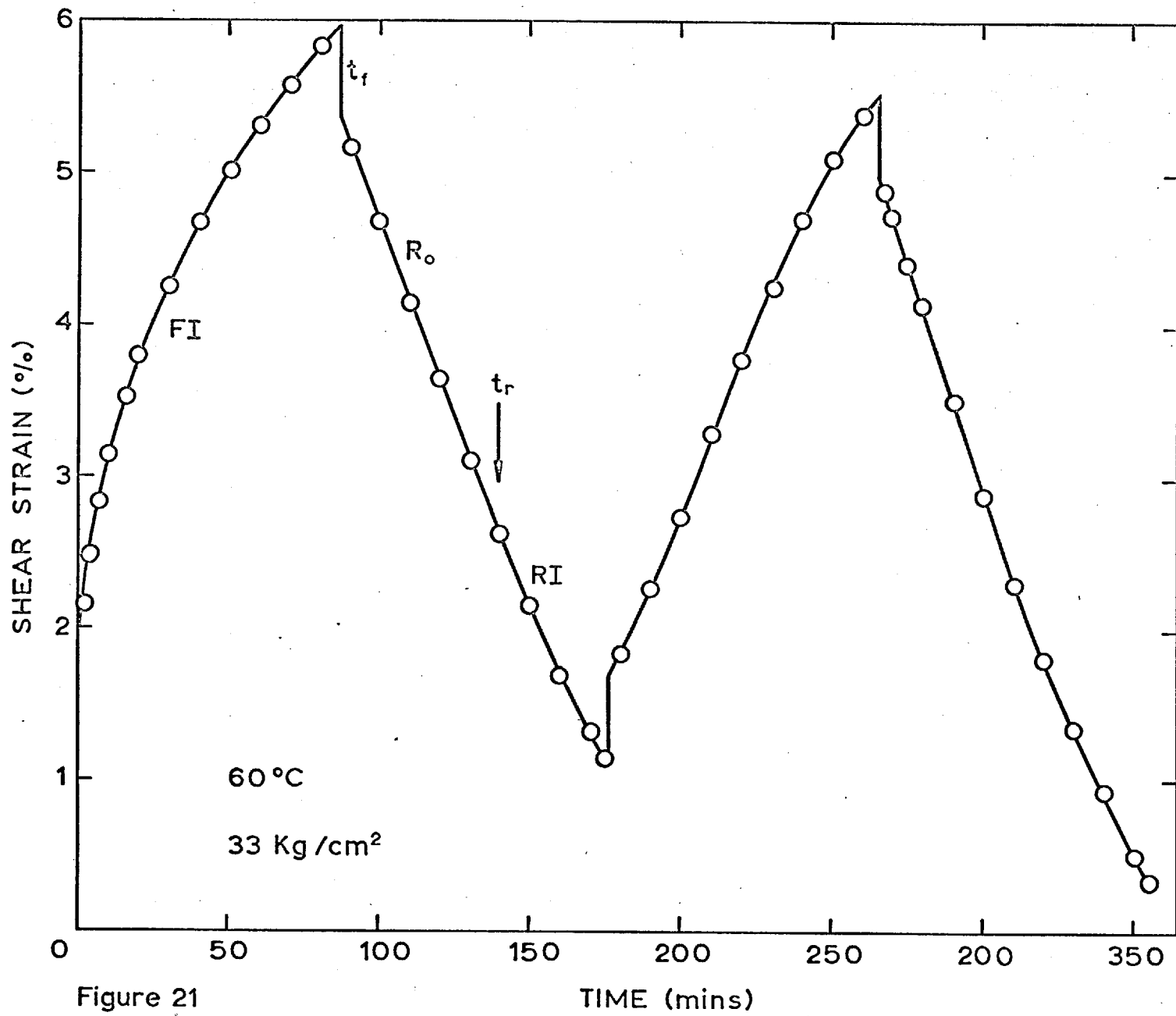


Figure 21

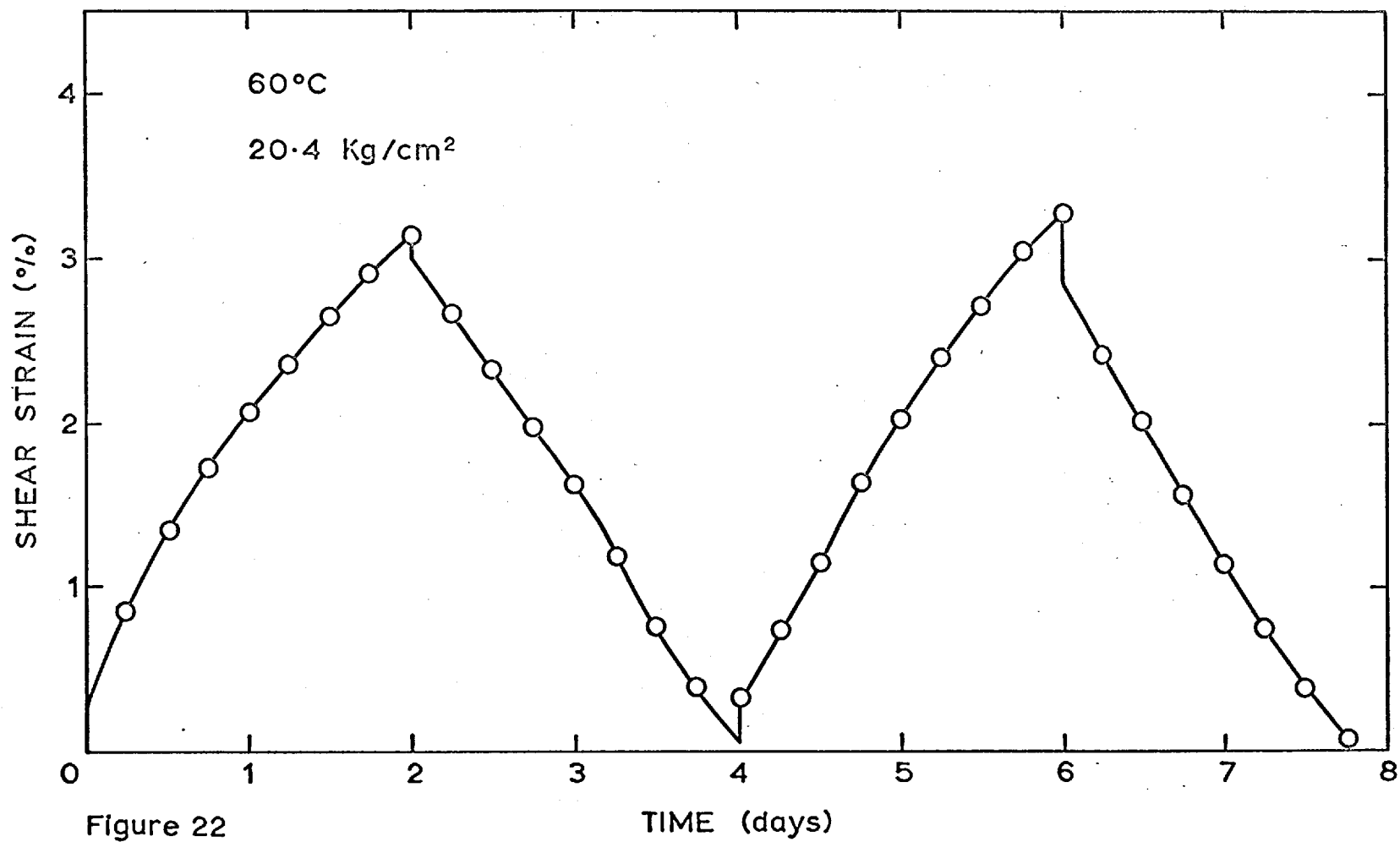


Figure 22

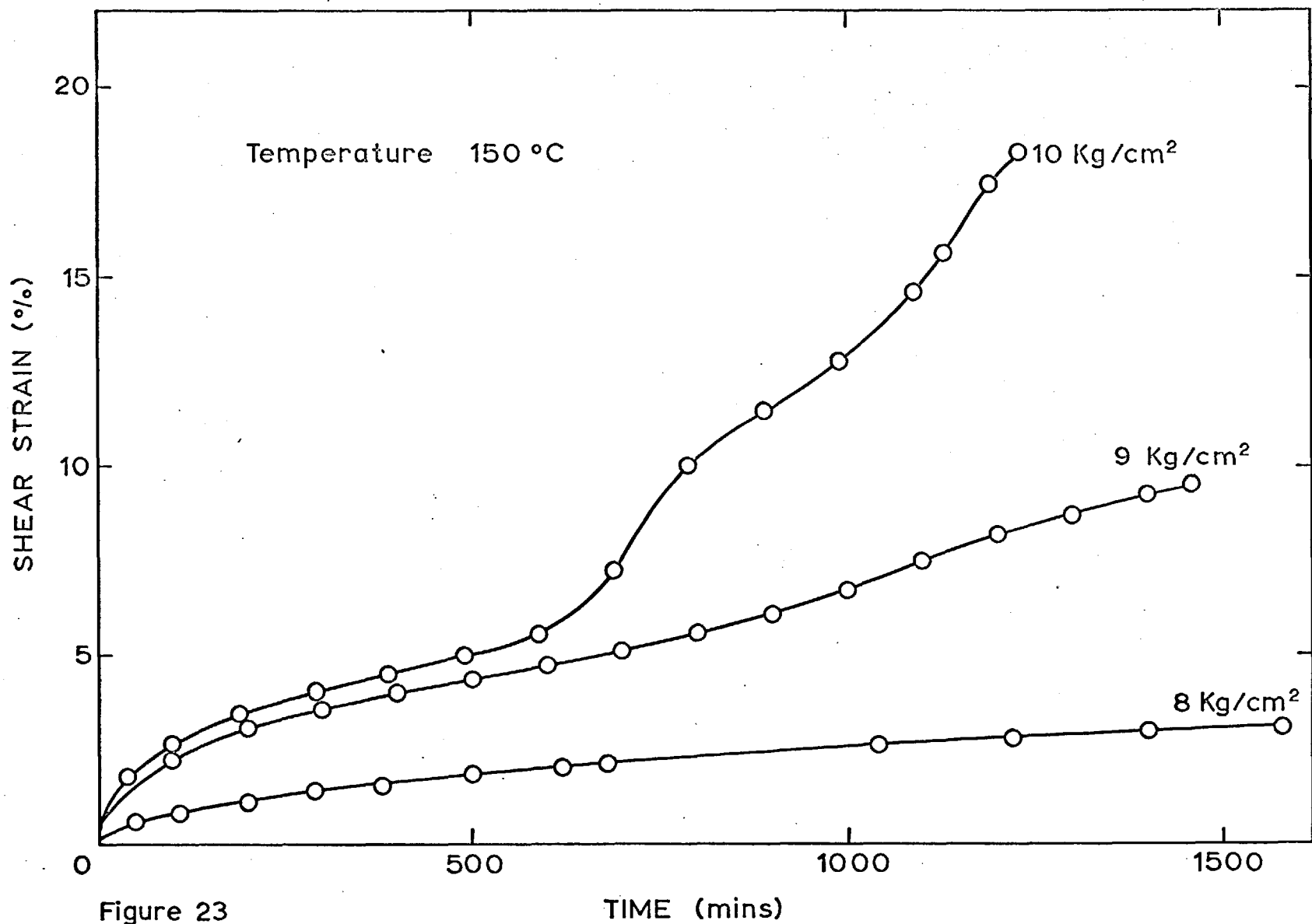


Figure 23





Figure 24

(magn. x 900).

 $d=1\text{mm}$ ,  $\sigma=9\text{Kg/cm}^2$ ,  $T=150^\circ\text{C}$ ,  $\gamma=0.0952$ .



Figure 25

(magn. x 200).

 $d=1/3\text{mm}$ ,  $\sigma=30\text{Kg/cm}^2$ ,  $T=60^\circ\text{C}$ ,  $\gamma=0.0666$ .



Figure 26



Figure 27

(magn. x210).

 $d=1/3\text{mm}, \sigma=25\text{Kg/cm}^2, T=80^\circ\text{C}, \gamma=0.0911.$