

CREEP BEHAVIOUR

OF

~~REPER~~ COMPOSITE MATERIALS

A Thesis submitted
for the degree
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by

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ABSTRACT

The creep and rupture characteristics of the Al-Al₃Ni continuous fibre composite were controlled to a large extent by macroscopic inhomogeneities in the form of matrix grain boundaries. Discontinuous grains were responsible for an enhanced composite creep rate, non-uniform deformation and premature composite rupture. The mechanism proposed was one of inadequate intergranular stress transfer.

At 400°C, the basic behaviour of Al-Al₃Ni displayed a low and a high stress sensitivity of creep rate depending on the applied stress. Furthermore, a creep strain or matrix stress constraint effect resulted in a diminished composite transient strain which was attributed to the small interfibre separation.

Contrary to previous findings, a pronounced reduction in the Al₃Ni whisker aspect ratio was induced by accelerated annealing of the as-grown composite. Evidence was obtained which suggested that a matrix/fibre interaction strongly influenced the nature of fibre rupture and hence composite rupture.

Pb-phosphor bronze discontinuous fibre composites were characterized by a reasonably well-defined steady-state behaviour. At moderate stresses, the fibres alone exhibited logarithmic creep. Composite deformation was intrinsically unstable due to the variable degree of reinforcement and the high stress sensitivity of the matrix.

A theory was developed for the steady-state behaviour of a matrix creeping around either rigid or non-rigid discontinuous fibres. For the latter, in the limit the theory degenerated to the continuous fibre case. Fibre stress distributions were dependent on the basic matrix characteristics. Relatively good agreement was found between the predictions and available experimental data.

A general tensile theory was obtained from the creep theory development and was found to simplify, in one limit, to the experimentally verified Kelly and Tyson theory.

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CHAPTER I

PRINCIPLES OF FIBRE STRENGTHENING

1.1 Introduction

Until recently, the basic approach to obtain creep resistance in metallic materials was to impede the movement of lattice dislocations. Various blocking or pinning mechanisms have been devised. However, as a result of the increasing temperature requirements of load bearing components, it is becoming more difficult to obtain such mechanisms of either sufficient strength or stability over long periods of time.

Fibre reinforced materials represent an entirely different approach to creep strengthening. The basic strength must be available in fibrous or whisker form, such as ceramic phases of high melting temperature. However, fibres cannot sustain useful shapes and must be incorporated in a controlled manner with a metal matrix. It may then be expected that the composite mixture would be characterized in general by intermediate properties relative to the constituents. The composite is designed so that the majority of the applied load is transferred to or supported by the fibre phase leaving the matrix relatively stress free. In order to establish the basic principles of fibre reinforcement, it is necessary to understand the various composite parameters which control this transfer of load.

Both the apparent potential of fibre composites and the present day need for materials of superior creep strength provoked Kelly and Davies (1965) and more recently Alexander et al (1969) in their major reviews

to stress the need for elucidation of composite creep characteristics. Krokosky (1967) concluded also that time dependent behaviour should certainly be a future area of intense study. Ironically, although the creep properties (and more so the rupture strength) of a number of metal matrix composites have been measured (see Alexander et al, 1969 for a review), very few attempts have been made to determine the basic principles. The infancy of the problem is reflected in the number of works - two to three for continuous and the same for discontinuous fibre composites. These will be described below and the straightforward experimental works will be referred to later in the thesis.

Before considering creep behaviour, the basic principles of fibre tensile strengthening will be outlined as they constitute a necessary foundation. In line with the creep study, attention is focussed on metal matrices stressed beyond their yield point. Unless otherwise stated, the thesis is concerned only with those components of stress, strain and creep rate which are parallel to the uniaxially aligned fibres.

1.2 Tensile Behaviour

1.2.1 Continuous Fibre Composites

Continuous fibres within a composite are generally assumed to be gripped directly at their ends along with the matrix. Consequently, if no interaction occurs, both phases behave as they would in the pure state. It has been found experimentally (McDanel's et al, 1963) that when the matrix and fibres are elastic, the stage I or primary composite modulus E_{CI} is given by a so-called rule-of-mixtures formulation,

$$E_{cI} = E_f V_f + E_m (1-V_f) \quad \dots (1.1)$$

where the subscripts m , f , c refer to matrix, fibres and composite respectively, V_f is the fibre volume fraction, and E is Young's modulus. Theoretically, Hill (1963) has shown that (1.1) is a lower limit and is applicable to the case where Poisson's ratios of the phases are equal. When the matrix becomes plastic, the stage II or secondary modulus E_{cII} is

$$E_{cII} = E_f V_f + \left(\frac{d\sigma_m}{de} \right)_e (1-V_f) \quad \dots (1.2)$$

where $\left(\frac{d\sigma_m}{de} \right)$ is the strain-hardening rate at strain e . (1.2) requires that the fibres remain elastic.

At any value of applied stress σ_c , the fibres and matrix support a proportion according to their volume fractions, namely

$$\sigma_c = \sigma_f V_f + \sigma_m (1-V_f) \quad \dots (1.3)$$

In order to calculate the individual stresses, strain equality must be assumed,

$$e_c = e_f = e_m \quad \dots (1.4)$$

Thus, for stage I we have

$$\sigma_c = e_c E_f V_f + e_c E_m (1-V_f) \quad \dots (1.5)$$

and for stage II

$$\sigma_c = e_c E_f V_f + \sigma_m' (1 - V_f) \quad \dots (1.5a)$$

where σ_m' is taken from the stress-strain curve at e_c . For the case where the matrix stress is either very small or disappears, (1.3) and (1.5) involve only the fibre terms.

The composite ultimate tensile strength σ_c^* is given by (1.3) when $\sigma_f = \sigma_f^*$ the fracture stress of the fibres. Invoking (1.4), the matrix stress σ_m'' would be that at the fracture strain of the fibres e_f^* . Hence, we have

$$\sigma_c^* = \sigma_f^* V_f + \sigma_m'' (1 - V_f), \quad V_f > V_{fmin} \dots (1.6)$$

If the matrix can work-harden to its ultimate strength σ_m^* , the composite will fail immediately as the result of fibre fracture only if the matrix is unable to support the total stress,

$$\sigma_m^* (1 - V_f) < \sigma_c^* \quad \dots (1.7)$$

Combining (1.7) and (1.6) defines a minimum V_f ,

$$V_{fmin} = \frac{\sigma_m^* - \sigma_m''}{\sigma_f^* + \sigma_m^* - \sigma_m''} \quad \dots (1.8)$$

Similarly, if σ_c^* is to exceed the unreinforced matrix strength σ_m^* , a critical V_f must be surpassed,

$$V_{f \text{ crit}} = \frac{\sigma_m^* - \sigma_m''}{\sigma_f^* - \sigma_m''} \quad \dots (1.9)$$

1.2.2 Discontinuous Fibre Composites

The simplifying assumption of strain equality cannot be extended to discontinuous fibre systems as the fibres are not gripped directly at their ends. Fibre stress and strain must originate via a transfer mechanism from the continuous matrix phase. When the fibres are more rigid than the matrix, extension of the latter will produce a shear stress τ parallel to the fibres. The action of τ induces a tensile stress in the fibres which can be determined from a force balance on an element of cylindrical fibre of diameter d . We find

$$d\sigma_f = \frac{4}{d} \tau dz \quad \dots (1.10)$$

where the z axis is parallel to the fibre axis. For the case of a fully plastic matrix, the Kelly and Tyson (1965) model assumes a constant τ . Hence, integration of (1.10) from $z = 0$ at the fibre end to z gives

$$\sigma_{fz} = \frac{4}{d} \tau z \quad \dots (1.11)$$

That is, σ_f builds up linearly from the fibre end to some maximum value which could be obtained if the assumption of (1.4) is made. Load transfer across the fibre ends directly is neglected.

Considering only the situation at σ_c^* , it is apparent that a minimum value $z = z_c'$ must be present if σ_f is to reach σ_f^* . From (1.11) we find

$$\frac{z_c'}{d} = \frac{\sigma_f^*}{4\tau} \quad \dots (1.12)$$

For a fibre within a composite, the stress increases from both ends and hence we can write (1.12) as

$$\frac{l_c}{d} = \frac{\sigma_f^*}{2\tau} \quad \dots (1.13)$$

where l_c/d is termed the critical aspect ratio for fibre fracture. At fracture, the average fibre stress $\bar{\sigma}_f'$ in the region where the distribution is described by (1.11), is found from (1.11) and (1.13),

$$\bar{\sigma}_f' = \frac{1}{l_c/2} \int_0^{l_c/2} \sigma_f dz = \frac{\tau l_c}{d} = \frac{\sigma_f^*}{2} \quad \dots (1.14)$$

when τ is constant. The total average stress is

$$\bar{\sigma}_f = \left(1 - \frac{l_c}{2l}\right) \sigma_f^* \quad \dots (1.15)$$

If τ is not constant, (1.14) becomes $\bar{\sigma}_f' = \beta \sigma_f^*$ and (1.15) becomes

$$\bar{\sigma}_f = \left[1 - (1 - \beta) \frac{l_c}{l}\right] \sigma_f^* \quad \dots (1.16)$$

The parameter β is an unknown averaging coefficient which must have a value $0 < \beta < 1$. Combining (1.16) and (1.3), σ_c^* is obtained,

$$\sigma_c^* = \left[1 - (1 - \beta) \frac{l_c}{l} \right] \sigma_f^* V_f + \sigma_m'' (1 - V_f) \quad \dots (1.17)$$

Likewise, (1.15) could be combined with (1.3) to give a similar expression to (1.17) for the case of constant τ . It is apparent from (1.17) that when l is sufficiently large compared to l_c , σ_c^* approaches that for a continuous fibre composite, (1.6). In the same manner as utilized for the continuous fibre development, V_{fmin} and V_{fcrit} could be calculated.

When $l/d < l_c/d$, fibres will not be fractured and failure will take place entirely within the matrix. For a constant τ we find

$$\sigma_c^* = \tau \frac{l}{d} V_f + \sigma_m^* (1 - V_f) \quad \dots (1.18)$$

The strength σ_c^* can be calculated if $\beta = \frac{1}{2}$ (assume constant τ) and l_c is known. At this time there is no basis for selecting any other value of β . The ratio l_c/d can be determined from (1.13) if $\tau = \tau_y$ the yield stress of the ideal plastic matrix. For work hardening matrices, Kelly and Tyson (1965) have shown that τ must be equated to the ultimate shear strength of the matrix τ^* . An implicit assumption here is that the shear strength of the interfacial bond itself is greater than that of the matrix.

A value of l_c/d can be obtained also from single fibre pullout tests (Kelly 1966) or from fracture surface observations. For the latter,

Kelly and Tyson (1965a) give the relation

$$\frac{n_f}{n_p} = \frac{l}{l_c} - 1 \quad \dots (1.19)$$

where n_f = number of fractured ends and n_p = number of pulled out ends. Fibres with ends within a distance $l_c/2$ of the fracture surface will not be fractured.

1.3 Creep Behaviour

1.3.1 Continuous Fibre Composites

The proportioned composite stress σ_c of (1.3) must be valid also for a matrix and fibres which are creeping. However, during transient creep, the stresses in the fibres and matrix are not constant but undergo redistribution due to their different creep strengths and the presence of a sufficiently strong bond. Such a mechanism has been known for some time to occur in reinforced concrete (Washa and Fluck, 1956). Even when the fibres are elastic as in glass fibre/resin composites, transient creep strain can take place as the result of redistribution (Carswell et al, 1969). It follows that predictions of composite behaviour involve complex calculations.

One of the earliest attempts to calculate composite behaviour was that of Ellison and Landes (1967). They utilized a numerical procedure in conjunction with a computer to aid calculation. Based on basic time dependent strain and stress equations for the phases and the assumption of total strain equality at all times, they calculated the stress changes required during a creep increment for compatibility to be maintained.

The total composite strain was obtained by a summation of the incremental strains. Although a direct comparison with experiment was not obtained, calculated creep curves were qualitatively in agreement with expected behaviour. They also invoked a simple statistical model for fibre rupture which allowed a prediction of composite creep rupture behaviour.

A significant simplification found by Ellison and Landes consisted of a linear rule-of-mixtures relation for composite creep strength at various times and strains,

$$\sigma_{cet} = \sigma_{fet} V_f + \sigma_{met} (1 - V_f) \quad \dots (1.20)$$

where the stresses are those required to give a specified strain ϵ in a time t . Such a relation has been found experimentally for creep rupture (McDanel's et al, 1967) but it has not been proved for transient creep. Other analytical treatments of composite creep include deformation under combined axial and bending loads (Ellison and Webster, 1967) and creep buckling of composite members (Alexander and Webster, 1960).

The more simple steady-state problem was first considered by McDanel's et al (1967). For a constant equal creep rate in both phases, $\dot{\epsilon}_c$, the component stresses remain constant with time. From (1.3) they obtain directly

$$\sigma_c = \sigma_f \dot{\epsilon}_c V_f + \sigma_m \dot{\epsilon}_c (1 - V_f) \quad \dots (1.21)$$

Given the stress dependence of creep rate for fibres and matrix, (1.21) can be evaluated. Anthony and Chang (1968) calculated $\dot{\epsilon}_c$ from (1.21) for Al-B composites and found a value approximately 50% less than their experimental values. Their conclusion of fair agreement with theory is misleading, as it represents considerable disagreement in terms of composite creep rate due to the non-linear dependence of rate on stress. Pinnel and Lawley (1970) claimed to verify the form of (1.21) for Al-Stainless Steel but as they did not determine the basic characteristics of the fibres, such a claim is invalid. Alexander et al (1969) reported that Baskey (1967) found creep rates for Ni-W at 1100°C which were considerably greater than those predicted.

1.3.2 Discontinuous Fibre Composites

Transient creep behaviour was studied by de Silva (1968). He examined separately the changes in fibre and matrix stresses due to creep extension and to relaxation of ϵ . Qualitatively the fibre stress distribution was envisaged to change in an identical fashion to that described by Kelly and Tyson (1966). As the various time dependent events must be interrelated within a composite, consideration of their coupled behaviour is a necessity. For this reason, de Silva did not obtain a quantitative prediction and, secondly, the applicability of the isolated considerations to the creep problem must be questionable.

Kelly and Tyson Model and Simple Extension

A qualitative model for the steady-state creep of a matrix around rigid fibres was proposed by Kelly and Tyson (1966). They postulated that the steady-state coincided with a constant fibre stress distribution where σ_f increased linearly to a maximum at the midpoint of the fibre

length, independent of the fibre length. This followed from their assumption of a constant τ and the requirement of a stable stress distribution. Assuming that all the stress was carried by the fibres, they obtained the expression,

$$\sigma_c = \tau \frac{\ell}{d} V_f \quad \dots (1.22)$$

This is analogous to (1.18) with $\sigma_m = 0$. Thus the steady-state strength creep for rigid fibres is similar to the tensile strength σ_c^* when $\ell/d < \ell_c/d$. Experimentally, for their composites of Ag/W at 400°C and 600°C, they showed that from a load carrying standpoint, $\sigma_m = 0$ was a valid approximation.

As it stands, (1.22) cannot relate σ_c and creep rate. However, a very simple extrapolation of their tensile model to the creep model will result in the necessary prediction. For completeness, the matrix tensile stress will not be neglected at this point. Assume the matrix ^{behaviour} Λ is described by an empirical power law of the form

$$\sigma_m = \sigma_{m0} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{m0}} \right)^{1/m} \quad \dots (1.23)$$

where σ_{m0} , $\dot{\epsilon}_{m0}$ and m are known constants and $\dot{\epsilon}_m$ is the steady-state creep rate. Then if we make the same assumption that the equilibrium or steady-state shear stress acting on the fibre is $\tau = \frac{1}{2} \sigma_m$, we can write (1.18) as

$$\sigma_c = \frac{1}{2} \sigma_{m0} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{m0}} \right)^{1/m} \left[\left(\frac{\ell}{d} - 2 \right) V_f + 2 \right] \quad \dots (1.24)$$

For moderate values of ℓ/d and V_f , (1.24) simplifies to a good approximation to

$$\sigma_c \approx \frac{1}{2} \sigma_{m_0} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{m_0}} \right)^{1/m} \frac{\ell}{d} V_f \quad \dots (1.25)$$

which is equivalent to (1.22). For very small ℓ/d or V_f , it can be seen that (1.24) must be utilized and not (1.25).

Mileiko Model

Very recently, a more rigorous approach by Mileiko (1970) produced a solution for the steady-state creep of a composite containing short rigid fibres perfectly bonded to the matrix. In order to show the nature of the approach to the problem adopted by Mileiko, the relevant points are described. However, various mathematical manipulations which were introduced to obtain an average matrix creep strength and the effect of a random fibre distribution are not repeated in detail here as they do not alter the basic approach.

Mileiko interpreted the creep problem as one of simple shear creep of the matrix; the matrix tensile stress was neglected. He considered both plate (two-dimensional) and fibre (three-dimensional) reinforcement. The resulting expressions are of the same form except for geometrical factors.

Application of a load to the ends of two rigid plates separated by a distance h and overlapped by a distance ℓ' induces a uniform state of simple shear in the matrix between them. The constant axial shear stress in the matrix τ is given by

$$\tau = \frac{\text{load}}{\ell'} = \frac{d}{\ell'} \sigma' \quad \dots (1.26)$$

for plates of unit width.

where d is the plate thickness and σ' the tensile stress in terms of the cross-sectional area of the plates alone. Utilizing a power law for the shear creep of the matrix with constants and form as given by (1.23), he writes,

$$\dot{\gamma} = \dot{\epsilon}_{m0} \left(\frac{\tau}{\sigma_{m0}/z} \right)^m \quad \dots (1.27)$$

After combining (1.27) and (1.26), he obtains for the rate of relative axial motion v of the two plates,

$$v = \dot{\gamma} h'' = z^m \dot{\epsilon}_{m0} \left(\frac{\sigma_c}{\sigma_{m0}} \right)^m \left(\frac{d}{\ell'} \right)^m d \frac{\left(\frac{1}{V_f} - 1 \right)}{V_f^m} \quad \dots (1.28)$$

In (1.28), the geometry of the cell is described in terms of V_f and σ_c is the stress on the plates related to the total cross-section of the cell. Finally, from (1.28), the matrix or composite tensile creep rate $\dot{\epsilon}_m$ for an ideal distribution of plates (symmetrical overlap) is

$$\dot{\epsilon}_m = \dot{\epsilon}_c = \frac{zv}{\ell} = \dot{\epsilon}_{m0} \left(\frac{\sigma_c}{\sigma_{m0}} \right)^m \frac{z^{m+1}}{\left(\ell/d \right)^{m+1}} \frac{\left(\frac{1}{V_f} - 1 \right)}{V_f^m} \quad \dots (1.29)$$

where ℓ/d is the plate aspect ratio and ℓ the plate length. This basically is the form of the solution in general for plates and fibres with ideal and random distributions. It represents the creep rate of a longitudinal chain of two rows of plates and contained matrix. The composite is considered to consist of an array of such chains.

Before considering the composite behaviour, he refers to the problem of calculating the average creep behaviour of a non-homogeneous material made up of an infinite number of longitudinal elements which are loaded

in parallel. The basic creep strength (the constant σ_{m0} in (1.23) and (1.27)) of each element is variable although continuity requires that they creep at the same rate. From other considerations, he obtains an "expectation value" for σ_{m0} given by $J(m) \sigma_{m0}$ where $J(m)$ is a function of the parameter m . To simplify, $J(m) \approx 1/4$ for $m \gtrsim 4$.

Of interest to the present work, we write down his final solution for a hexagonal array of fibres with random overlap,

$$\dot{\epsilon}_c = \frac{\dot{\epsilon}_{m0}}{(m-1)} \left(\frac{\sigma_c}{J(m) \sigma_{m0}} \right)^m \frac{1 - V_f^{\frac{m-1}{2}}}{\left(\frac{l}{d}\right)^{m+1} V_f^m} \quad \dots (1.30)$$

It can be seen that the form of (1.30) is the same as (1.29). However, (1.30) is not valid for a Newtonian matrix, $m = 1$. Reasonably good agreement was found between (1.30) and the data of Kelly and Tyson (1966) for Ag/W.

Rearranging (1.30) and evaluating $J(m)$, $(m-1)^{1/m}$ and $\left(1 - V_f^{\frac{m-1}{2}}\right)^{1/m}$ for $m \gtrsim 4$ and $V_f \gtrsim 0.1$, we obtain

$$\sigma_c \cong 0.35 \sigma_{m0} \left(\frac{l}{d}\right)^{\frac{m+1}{m}} \left(\frac{\dot{\epsilon}_c}{\dot{\epsilon}_{m0}}\right)^{1/m} V_f \quad \dots (1.31)$$

Comparison with the extended Kelly and Tyson model, (1.25), reveals a surprisingly close similarity except for the power of l/d and the pre-expression constant.

It is implicit in the theory of Mileiko that σ_f is constant in any one fibre over its entire length. Such a fact must be important in his evaluation of the critical aspect ratio for fibre fracture, l_c/d . For brittle fibres, he obtains,

$$\ell_c/d = K \dot{\epsilon}_c^{-\left(\frac{1}{1+m}\right)} (\sigma_f^*)^{\frac{m}{m+1}} \quad \dots (1.32)$$

K contains the remaining constants of (1.30) and σ_f^* is the fibre fracture stress.

A minimum volume fraction V_{fmin} is obtained from (1.30) which corresponds to $\dot{\epsilon}_m'/\dot{\epsilon}_c = 1$ ($\dot{\epsilon}_m'$ is the creep rate of the unreinforced matrix). He defines V_{fmin} as the volume fraction below which $\dot{\epsilon}_m'$, or in effect σ_m , cannot be neglected. In reality, the carrying capacity of the matrix σ_m must continue to be significant until somewhat larger values of V_{fmin} which correspond to $\dot{\epsilon}_m'/\dot{\epsilon}_c \sim 0.1$.

1.4 Measurement of Composite Creep Behaviour

In addition to the analytical and theoretical complexities, it has become apparent that the measurement of the creep behaviour of fibre reinforced materials is far from straightforward. The complications which have been encountered and their origins are summarized briefly in this section in order to clarify the interpretation of various experimental results reported in later chapters. For completeness, some of the findings from the present work are included.

It is an intrinsic feature of composites that we rely on the relative shear between matrix and fibre to load the fibrous phase. In discontinuous fibre systems, this takes place throughout the entire specimen; in continuous fibre systems, it is a feature confined to the grips. For those forms of gripping that are generated by matrix shear alone, viz threaded or shouldered specimens, we can estimate the necessary geometry

to meet the requirements of the creep test. From a simple force balance in the grip of a tensile specimen, we obtain the familiar relation between the grip geometry L/d_s and σ_c ,

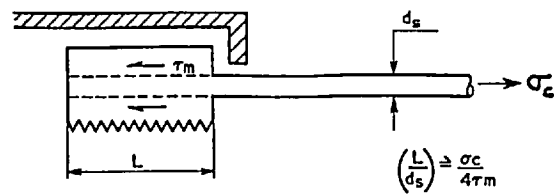
$$\frac{L}{d_s} = \frac{\sigma_c}{4\tau_m} \quad \dots (1.33)$$

The parameters are shown in the diagram of Fig. 1.1. For a given matrix shear creep strength τ_m , σ_c can be generated within the gauge length only if L/d_s from (1.33) is exceeded. Using typical values for τ_m ($= \sigma_m/2$) of pure metals at $T/T_m = 0.7$ and $\dot{\epsilon}_m = 10^{-5}/h$, and various values of σ_c , some examples of grip geometry calculated from (1.33) are given also in Fig. 1.1. It is apparent that under conditions of creep, when τ_m is very small, grip shear failures are likely to occur. Such an event is not uncommon.

To overcome the above problem, many workers have selected a gripping arrangement where lateral pressure can be exerted to effectively enhance τ_m . This is shown schematically in Fig. 1.2 along with examples of the fibre composites which have been tested with this arrangement.

The above considerations are most critical when composite creep rate is measured without the aid of gauge length extensometers: in many of the reported works, such has been the case. Hence, experimental results may be expected to include a grip shear creep contribution to the total composite strain unless very large gripping areas are used in conjunction with maximum lateral clamping forces. The contribution of grip shear can be further minimized by maximizing the total length of the specimen.

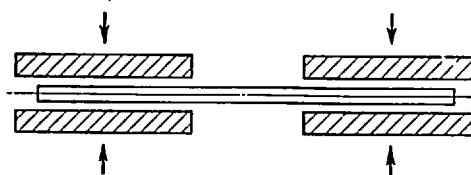
Three main problems that have been encountered with the attachment of extensometer limbs to composite specimens are depicted in Fig. 1.3. A low γ_m leads to slippage and hence erroneous extensions. Broken fibres at the clamping locations result in both non-uniform deformation and premature rupture. A variation of this was found in the present work where the Al_3Ni whiskers spherodized beneath the attachment points. Attachment problems can be overcome by observing directly the extension of specimens (optically) or by introducing shoulders in the gauge length at the clamping locations. The latter proved successful in the present study on $Al-Al_3Ni$ for temperatures up to $550^\circ C$. Unsuccessful specimens due to the reasons described above are shown in Fig. 1.4 along with a satisfactory shouldered specimen.



Matrix	T (°C)	τ_m (10^7 N/m ²)	σ_c (10^7 N/m ²)	L/ds	ds (mm)	L (mm)
Al	380	0.02	8. (Al-Al ₃ Ni)	100	3	300
			64. (Al-B)	800	3	2400
Cu	680	0.12	56. (Cu-W)	58	3	174
Ni	930	0.15	35 (Ni-W)	116	3	348

for $T/T_m = 0.7$
 $\tau_m \left(\frac{\sigma_m}{2} \right) \text{ at } \dot{\epsilon}_m = 10^{-5} / \text{h}$

Fig. 1.1 Required aspect ratios of grips L/ds if the composite stress σ_c is to be induced by the shear creep strength of the matrix τ_m in the grips.



Breinan and Kreider (1970) Al-B
 Ellison and Harris (1966) Ni-W
 Wilcox and Clauer (1969) LiMg-Fe
 Street - present - Pb - phosphor bronze

Fig. 1.2 Schematic diagram of the type of creep or rupture specimen utilized by various workers for fibre composite materials.

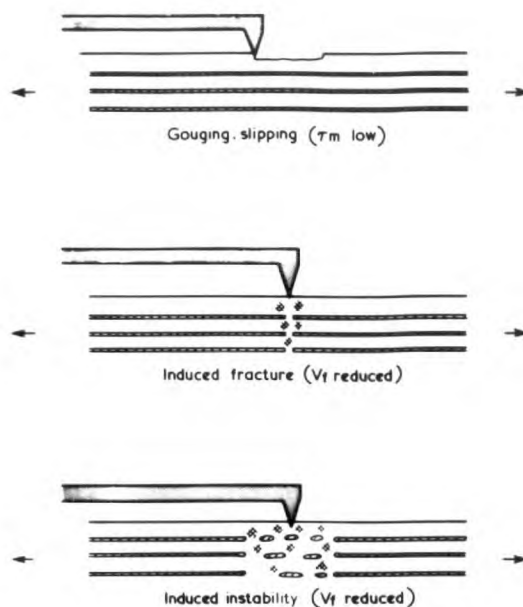


Fig. 1.3 Schematic of basic problem areas connected with attachment of creep extensometry to soft matrix reinforced with high strength fibres.



Fig. 1.4 Various designs of Al-Al₃Ni creep specimens in present work. Gauge length shoulders were required in order to prevent localized creep deformation and rupture (approx. full size).

CHAPTER II

Al-Al₃Ni CONTINUOUS FIBRE COMPOSITE

2.1 Introduction

This chapter is concerned primarily with establishing the factors responsible for the measured macroscopic creep characteristics of a metal matrix reinforced with continuous fibres. The creep regime of interest is one where both the fibres and matrix are likely to experience steady-state creep.

Studies of the creep behaviour of materials in general are often confined to the constant rate portion of the creep curve which is commonly denoted as secondary or steady-state creep. The basic reason for this is not only the much needed simplification but that most theories of creep behaviour based on various mechanisms have been developed for the steady-state (Mukherjee 1969). The application of empirical formulations to describe behaviour over a range of stress and creep rate is also simplified. This approach to the creep problem is not without practical justification as it is found that in many applications the constant rate period occupies most of the creep life and hence is the most important (Finnie and Heller 1959).

Regarding fibre composites, in addition to the above, two important reasons exist for adopting the same approach. First, the transient behaviour is unduly complex as the result of variable stress states in the fibres and matrix, apart from the usual variable creep rate (Ellison and Landes 1967). Secondly, as at this time fibre composite theories are phenomenological in general, the literature contains more

information on the steady-state behaviour of both matrices and fibres than on the transient characteristics.

A unidirectionally solidified binary eutectic alloy, Al-Al₃Ni, was selected for the experimental study. There were several reasons for choosing this particular system. The method of manufacture is simple, reproducible and the fibres possess an unusually regular arrangement within the matrix. Due to the relatively small inter-fibre spacings, the composite is amenable to metallographic studies and to electron microscopy, if desired, where it is perhaps the best suited system for examination of the fibres and matrix in the combined state. The Al matrix possesses an excellent oxidation resistance and is well characterized in the literature. The absolute melting temperature (peritectic transformation) of the fibres is not widely different from that of the matrix, inferring that steady-state creep should be obtainable at moderate stresses and temperatures. The phases are in chemical equilibrium and exhibit very good thermal stability. Finally, as Al-Al₃Ni has been the model eutectic composite of United Aircraft Research Laboratory workers since 1964, the wealth of available property data would provide valuable background for a time consuming creep programme. These data are summarized in the following section.

The creep behaviour at a temperature of 400°C was of primary interest. This represents a relatively high homologous temperature of 0.74, 0.72 and 0.60 for eutectic, matrix and fibres respectively. Supplementary tests were to be carried out at 550°C to ascertain the importance of thermomechanical stability, an often discussed but little studied topic. A brief comparison with some other work at the much lower temperature of 250°C was planned.

2.2 Basic Mechanical Behaviour of Al-Al₃Ni

Eutectic alloy microstructures and the principles of eutectic solidification were the subject of numerous review articles (Portevin 1923, Chadwick 1963, Kraft 1966 and Chadwick 1967). The growth of composite structures from the melt in general was considered by Hellawell (1967). The general mechanical behaviour of eutectic composites was reviewed by Salkind et al (1967a). Only those aspects pertinent to a creep study will be analyzed here.

The Al-Al₃Ni eutectic is characterized by a rod or fibre morphology of the Al₃Ni phase (Lemkey et al 1965) as distinct from the lamellar morphology common to many eutectics. Of importance to the following discussion, the interfibre spacing λ is inversely proportional to the square root of the solidification rate R . The absolute values are considered later in the chapter.

The obeyance of fibre composite principles by aligned eutectic alloys originated from the work of Hertzberg et al (1965). They revealed that rule-of-mixtures predictions for both composite strength σ_c^* and primary modulus E_{cI} were followed by Al-Al₃Ni. The strength decreased gradually with temperature but compared favourably with a dispersion hardened alloy SAP (sintered aluminium powder), Fig. 2.1. The high temperature strength retention was vastly superior to that of precipitation hardened aluminium.

In early work, an interfibre spacing λ dependence of σ_c^* was not found due to experimental scatter. Later, Salkind et al (1966) observed a small but definite increase in σ_c^* from 325 MN/m² at $\lambda = 2.3 \mu\text{m}$ ($R = 4 \text{ cm/h}$) to 365 MN/m² at $\lambda = 0.8 \mu\text{m}$ ($R = 38 \text{ cm/h}$), Fig. 2.2. This increase was for a rod morphology of the Al₃Ni phase

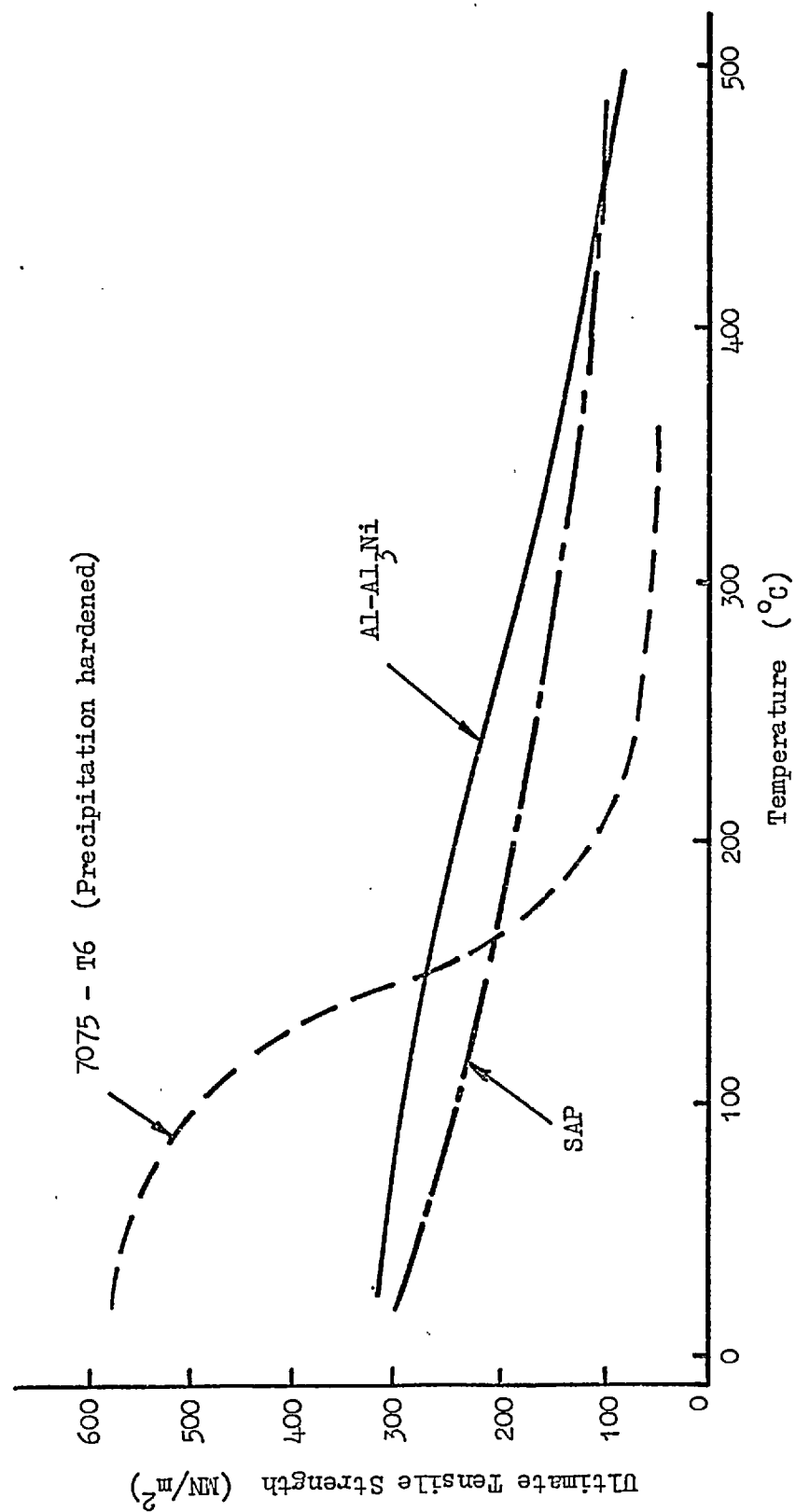


Fig. 2.1 Temperature dependence of the ultimate tensile strength (after Salkind et al 1967a).

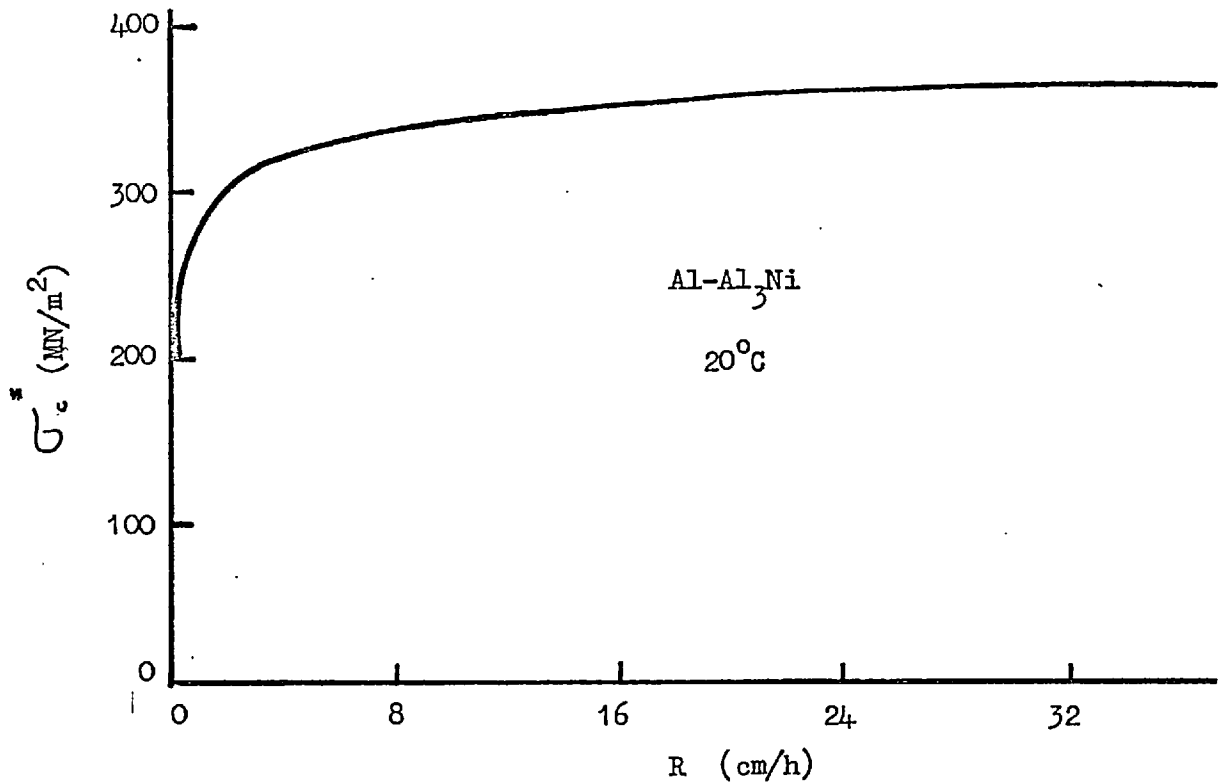


Fig. 2.2 Solidification rate dependence of the ultimate tensile strength of $\text{Al-Al}_3\text{Ni}$ (after Salkind et al 1966)

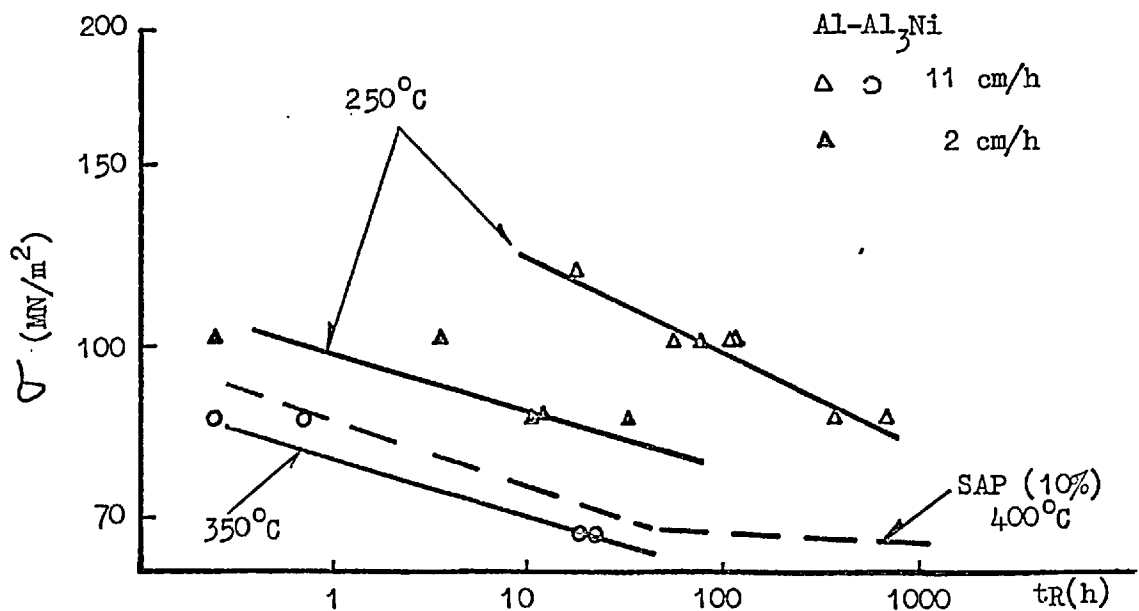


Fig. 2.3 Log stress vs log rupture time for $\text{Al-Al}_3\text{Ni}$ (after Salkind et al 1966) and SAP (after Boerman et al 1969)

as a platelet morphology was found previously to possess a greater number of surface defects which decreased the whisker strength (Hertzberg et al 1965).

Another reason for not observing a size effect may be that σ_c^* is relatively insensitive to changes in matrix behaviour. For example, taking a constant value of fibre fracture stress $\sigma_f^* = 2.75 \text{ GN/m}^2$ (as Hertzberg), it follows from a rule-of-mixtures formulation that the observed strength increase $\Delta \sigma_c^* = 40 \text{ MN/m}^2$ corresponds to an increase in σ_m'' (matrix stress at fibre fracture strain) of a factor of two, a fact which may not be insignificant in creep behaviour.

The composite yield stress σ_{cy} characteristics have not been studied for Al-Al₃Ni. Other eutectics have exhibited pronounced increases in σ_{cy} with decreasing lamellar spacing: Cd-Zn, Shaw (1967); Ag-Cu, Cline and Lee (1970). Shaw observed also that the degree of stress relaxation decreased considerably as λ decreased.

Lemkey et al (1965) found that Al-Al₃Ni possessed a unique crystallographic orientation of the phases such that the atomic matching at the interfaces was extremely good; the interface is semi-coherent. It has been postulated that a major outcome of such a feature is a relatively stable fibre structure at elevated temperatures as the low specific interfacial energy is insufficient to induce gross spheroidization (Salkind et al 1967, Bayles et al 1967). However, when the as-grown structure is heavily deformed (rolled, swaged), the low energy interfaces are destroyed and rapid spheroidization takes place. It follows that ^{good} creep strength requires an as-grown structure.

A question which arises is what structural changes are induced by highly localized deformation such as inhomogeneous shear in the single crystalline matrix?

The creep rupture behaviour of Al-Al₃Ni was studied by Salkind et al (1966). He observed that the rupture strength at 250°C was dependent upon spacing λ as shown in Fig. 2.3. The difference between the 11 cm/h and 2 cm/h specimens decreased with time, implying closer equality of behaviour at very large times. The rupture observations reflect the observed tensile strength dependence upon λ , Fig. 2.2.

An interesting observation is found when the rupture data are compared with that of SAP (Boerman et al, 1969) (see Fig. 2.3). Whereas the short time σ_c^* is comparable to that of SAP, the rupture strength of SAP (10%) at 400°C is greater than that of Al-Al₃Ni at 350°C, and likely even more so at longer times. It is surprising that this comparison has not been made before this time. It would appear that the long time properties of Al-Al₃Ni must be controlled by time dependent weakening of the continuous whisker phase. A question of interest is whether the weakening of Al₃Ni is intrinsic or due to some fibre/matrix interaction?

The initial experiments on the creep behaviour of Al-Al₃Ni were performed by Salkind et al (1966). No mechanisms were determined but the data revealed two distinct features. Nominally identical specimens subjected to a constant σ_c and temperature (250°C)

exhibited minimum creep rates $\dot{\epsilon}_{\min}$ which differed by a factor of 10^3 - 10^4 . This behaviour could not be explained. Secondly, $\dot{\epsilon}_{\min}$ apparently decreased with decreasing λ . These data are further analysed in the present work and a simple relation was found between $\dot{\epsilon}_{\min}$ and rupture time t_r ,

$$\dot{\epsilon}_{\min} = \dot{\epsilon}_{co} \left(\frac{t_r}{t_{ro}} \right)^{-r} \quad \dots (2.1)$$

where $\dot{\epsilon}_{co}$, t_{ro} and r are constants which depend slightly on composite stress but not on λ (see Fig. 1 of Appendix I). Consequently, it is suggested that the proposed creep rate dependence on λ is in reality a rupture dependence. If rupture had not occurred, identical values of $\dot{\epsilon}_{\min}$ would have been found in small and large λ specimens. The nature of the experimental results was confirmed very recently by Breinan et al (1970) who independently observed the same correlation between $\dot{\epsilon}_{\min}$ and t_r (see Appendix I).

Chemically extracted Al_3Ni whiskers were reported to creep at temperatures as low as 310°C , $T/T_m = 0.52$ (Breinan et al 1968). In contrast Cabrera and Price (1958) concluded that creep of Zn whiskers at $T/T_m = 0.42$ occurred only in defective whiskers. A similar conclusion was arrived at by Pearson et al (1957) for Si whiskers at $T/T_m = 0.63$. Brenner (1962) found delayed rupture but no creep in Al_2O_3 whiskers at $T/T_m = 0.99$. It would appear that the possibility exists that defects were induced into Al_3Ni during extraction and that in-situ whiskers are more creep resistant.

2.3 Composite Manufacture

2.3.1 Master Alloys

The Al-Al₅Ni eutectic composition given by Hansen (1958) as 5.7 wt % Ni was re-established by Lemkey et al (1965) as 6.2 wt %. The latter observed regular eutectic microstructures over the range 5.6 - 6.4 wt % Ni.

Master alloys of nominal composition 6.2 wt % Ni were produced from 99.99% Al (British Aluminium) and 99.995% Ni (Johnson Matthey). The constituents were melted in either graphite or alumina crucibles under a dynamic argon atmosphere utilizing a low frequency induction furnace to produce good mixing. A 1 - 2 hour equilibration at 950° - 1050°C was followed by gradual withdrawal of the crucible from the coil to produce a "pipe" free ingot. The ingots were surface machined to remove adhering matter and then hot extruded at 400°C into 12.7 mm diameter rods.

2.3.2 Unidirectional Growth

A length of extruded rod was placed in a cylindrical graphite crucible, remelted, and unidirectionally solidified in the apparatus shown in Fig. 2.4. The rate of solidification, taken to equal the rate of withdrawal of the crucible from the furnace, was maintained constant at selected values. A maximum thermal gradient in the melt was achieved by using a water spray heat sink. Such a design was considered advantageous from the standpoint of maintaining constant growth conditions. Crucible oxidation was deterred by a dynamic argon flow.

Chemical and spectrographic analyses of a specimen solidified at 7.2 cm/h are given in Tables 2.1 and 2.2. The analyses were performed by the Analyses Unit, NPL. The observed constancy of Ni concentration assured a constant volume fraction of intermetallic phase. However, some segregation of impurities Fe, Si and Cu was evident with their concentrations increasing along the growth direction. The level of impurities was essentially that of the raw materials inferring the manufacturing procedures had not contaminated the alloys.

2.4. Characterization of Composite Structure

2.4.1 Metallography

Metallographic studies were performed on alloy sections which were mechanically polished through to 6 μ m diamond paste and then electrochemically etched. The etching bath consisted of 13% perchloric acid (S.G. 1.54), 33% 2-butoxyethanol and 54% methanol by volume. The specimen was rendered anodic for about 4 seconds at a potential of 20 volts in the presence of an aluminium cathode.

2.4.2 Fibre Phase

It has been found elsewhere that the Al_3Ni phase particles are whiskers (single crystals) of orthorhombic structure with a $\langle 010 \rangle$ longitudinal growth axis (Lemkey et al 1965, Tice et al 1968).

The diameter, shape and spacing of the whiskers within the matrix is shown in Fig. 2.5 for sections transverse to the growth direction of the alloy. In order to resolve the true diameter and shape, it was necessary to use electron transmission microscopy. Standard electrochemical thinning techniques were used (Tice et al 1968).

Table 2.1 Chemical Analyses of Solidified Rod Al-Al₃Ni

<u>Slice No.</u>	<u>Distance from head ingot (±)</u>	<u>Nickel (%)</u> ^{wt.}	<u>Limit (%)</u> ^{wt.}
1	2.0 cm	6.18	± 0.020
2	4.0 cm	6.21	± 0.035
3	10.2 cm	6.29	± 0.026
4	18.5 cm	6.18	± 0.013
5	26.7 cm	6.25	± 0.020

Table 2.2 Spectrographic Analyses of Solidified Rod Al-Al₃Ni

<u>Element</u>	<u>Sweep Range (%)</u> ^{wt.}
Fe	0.002 - 0.007
Si	0.002 - 0.006
Cu	0.001 - 0.004
Mg	0.002 - 0.003

± Solidification direction from head to tail of ingot.

A solution of 18% perchloric acid (S.G. 1.54) in ethanol and pin-point stainless steel cathodes gave satisfactory results. The Al_3Ni phase possesses a distorted hexagonal cross-section.

Attempts were made to measure the length of the Al_3Ni particles. Numerous metallographic sections failed to reveal an unambiguous as-grown whisker end. Due to their small diameter, it was difficult to prepare longitudinal sections wherein the plane of observation coincided precisely with the whisker axis. In Fig. 2.6, chopped off ends are evident in the lower grain but not in the upper grain. Dissolution of the matrix in a solution of 15% HCl left a residue of a tangled mass of long whiskers which were susceptible to breakage during handling. From the observations, it became apparent that from a mechanical property standpoint, the as-grown whiskers can be assumed to be continuous in length.

A characteristic of the whiskers which has been observed also by other workers (Breinan et al 1970) is their variable diameter. Examples are shown in Fig. 2.6 and 2.7. The variability is by no means a consistent characteristic as it can be seen that some whiskers possess quite uniform diameters over a considerable length. The most likely origin of this feature is local perturbations in the melt during the growth process.

Beyond an initial growth zone of about 1 cm length, the alignment of the whiskers was found to be within a few degrees of the growth axis of the composite specimens, except in the immediate vicinity of some columnar matrix grain boundaries. Tensile specimens were always taken from locations well removed from the ends of an ingot.

The volume fraction V_f of Al_3Ni calculated from the phase diagram is 0.10 ± 0.01 . Studies of the solidification behaviour by various workers including Lemkey et al (1965) have shown that the measured spacing λ of whiskers obeys a relation of the form

$$\lambda = A R^{-1/2} \quad \dots (2.2)$$

where R = growth rate and A = material constant. Combining (2.2) with the relation between fibre diameter d and spacing for a hexagonal array of uniform cylinders, we obtain the relation between fibre diameter and growth rate

$$d = \left(\frac{2\sqrt{3}}{\pi} V_f \right)^{1/2} A R^{-1/2} \quad \dots (2.3)$$

Fig. 2.8 contains measured values of both spacing and whisker diameter which agree well with the calculated values from (2.3).

2.4.3 Matrix Phase

The aluminium matrix has been reported to contain approximately 0.006 wt % Ni in solid solution at 500°C (Hansen 1958). During solidification, the matrix forms an array of columnar grains with boundaries lying close to the growth direction. Substantial grain growth takes place along the solidification direction, Fig. 2.9, with the result that not all grains can be continuous. A Laue back-reflection technique was utilized to measure the orientation of the matrix phase at locations well removed from the start of solidification. The growth axis was found to be very near $Al \langle 110 \rangle$ ($< 6^\circ$) in accordance with the results of Lemkey et al (1965). The slight misorientation may be due to a non-vertical crucible or to a slightly misaligned metallographic section.

2.4.4 Grain Boundaries

One unique characteristic of eutectic alloy composites in general is the lack of intergranular fibre reinforcement. The fibres terminate at the columnar grain boundaries as shown in Fig. 2.10. The degree of fibre misorientation possible near a grain boundary is also apparent in Fig. 2.10 along with examples of branched whiskers. A more clear example of the nature of a eutectic boundary was obtained by deep etching and stereoscan observation by Walter and Cline (1970).

2.4.5 Defects

The most common defective structure from a mechanical property viewpoint was the transverse "growth band", Fig. 2.11. This defect is well understood to originate from fluctuations in the rate of movement of the solid/liquid interface during solidification (Chadwick 1963). A phenomenon associated with a band was the induced growth kinking ($10^\circ - 15^\circ$) of some fibres following the band. The kinked orientations were relatively stable as indicated by their lengths (see Fig. 2.11). As bands extend throughout the entire cross-section of an ingot, surface observations after macroetching are sufficient to reveal their presence. Creep specimens containing bands were generally unacceptable although a few experiments were performed for microstructural observations.

The well-known impurity cells or colonies (Weart and Mack 1958) were not found in the present work due to the high thermal gradients and low impurity level. Other minor defects such as fibre spacing perturbations and disrupted fibres due to the presence of inclusions

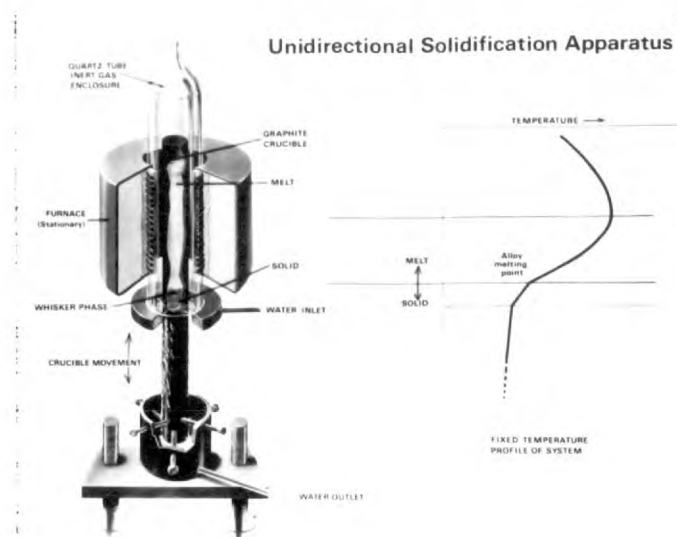


Fig. 2.4 Drawing of Unidirectional Solidification Apparatus

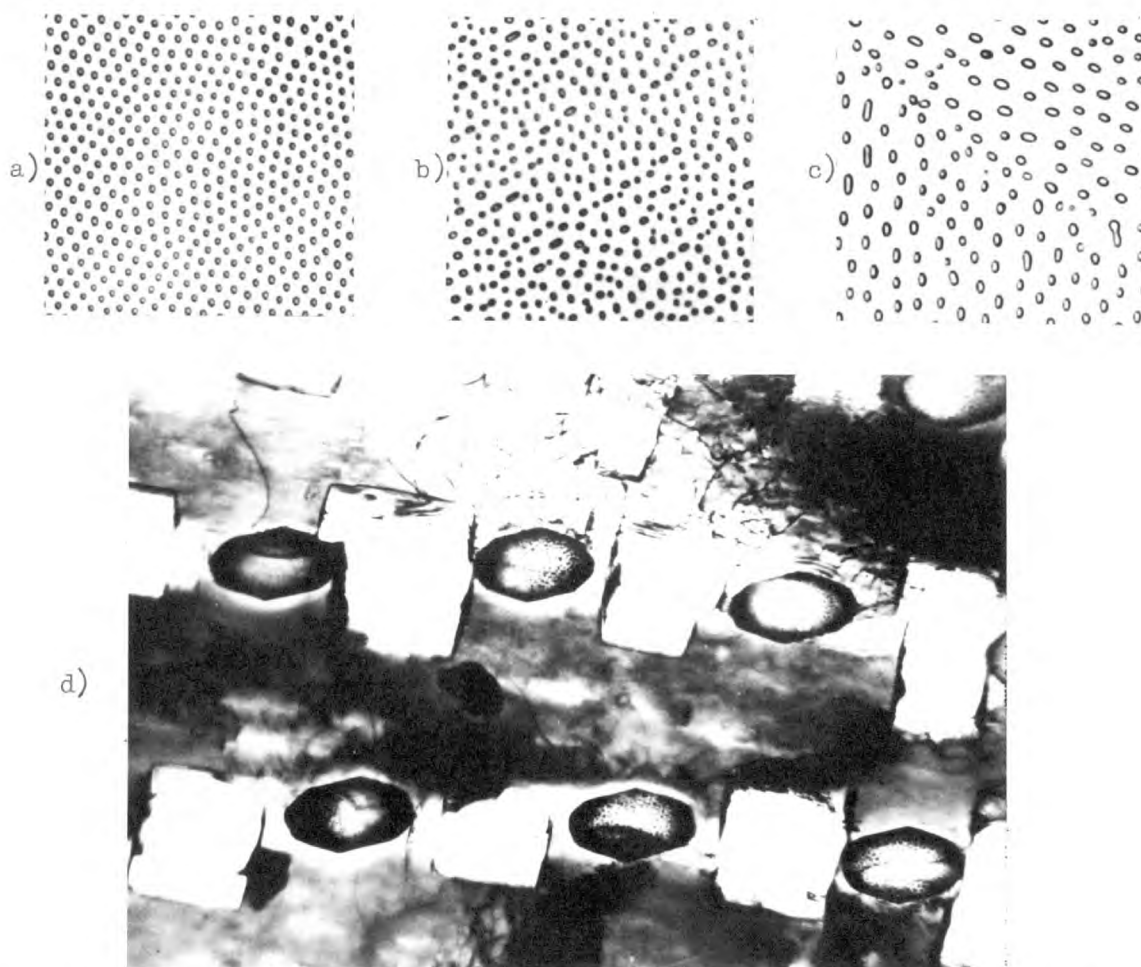


Fig. 2.5 Transverse sections showing Al_3Ni morphology a) 9.0 cm/h b) 7.2 cm/h c) 1.8 cm/h (Magn. 500X) d) 9.0 cm/h (Magn. 21,000X)



Fig. 2.6 Longitudinal section parallel to solidification axis (horizontal). $R = 7.2 \text{ cm/h}$ (Magn. 500 X)

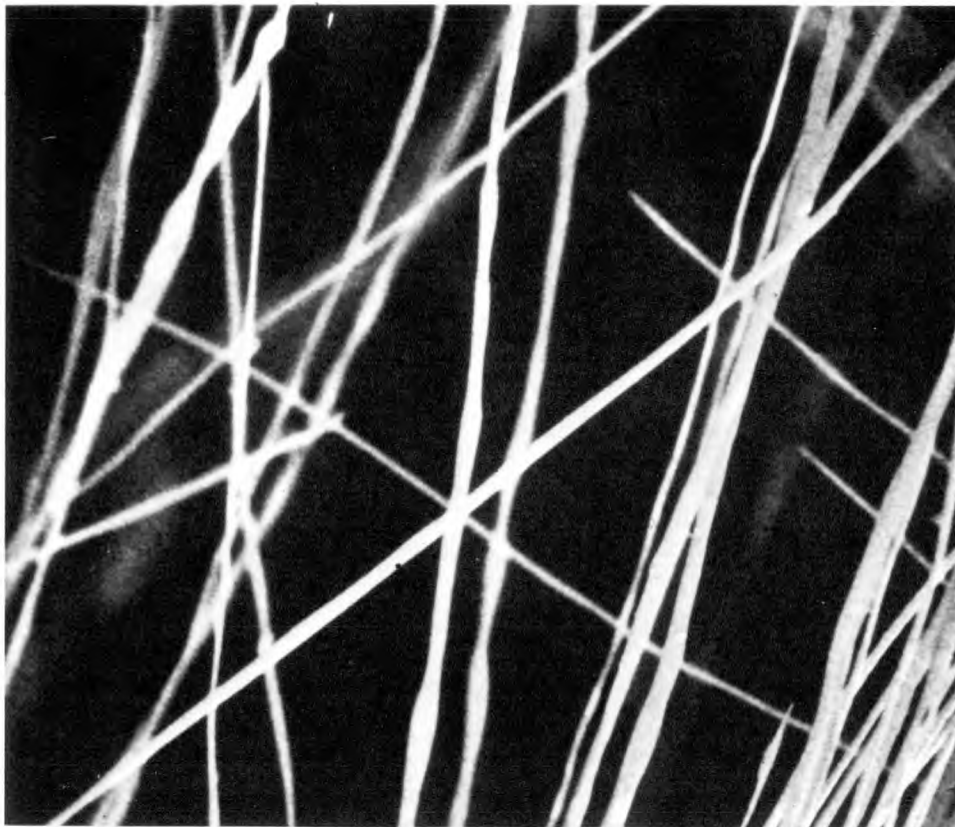


Fig. 2.7 Al_3Ni whiskers obtained by dissolution of the matrix (Magn. 2200 X)

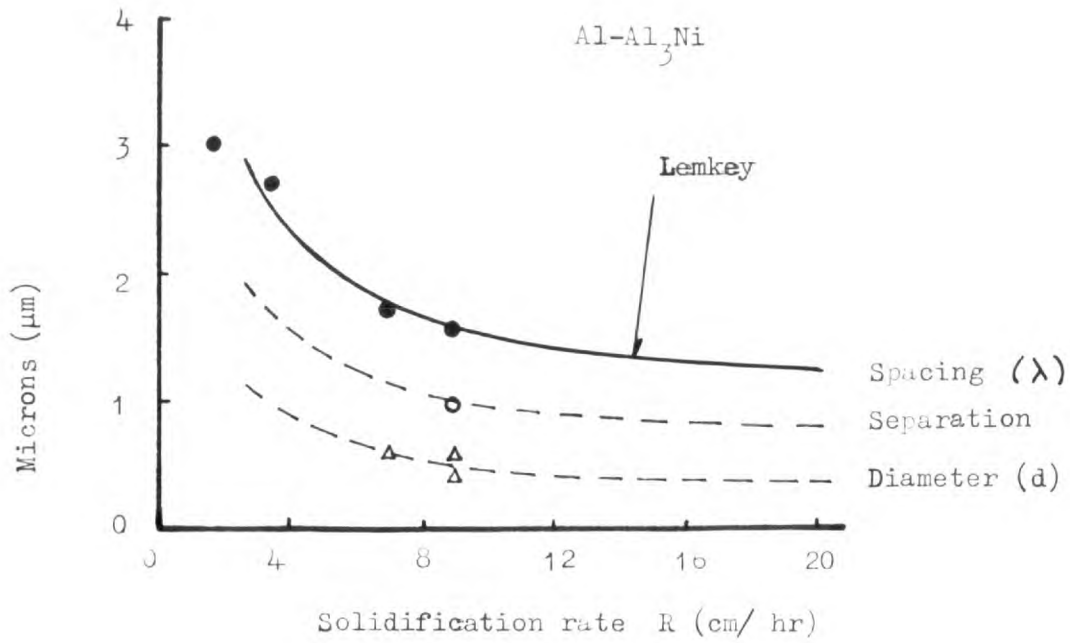


Fig. 2.8 Al₃Ni whisker spacing, separation and diameter as a function of solidification rate.

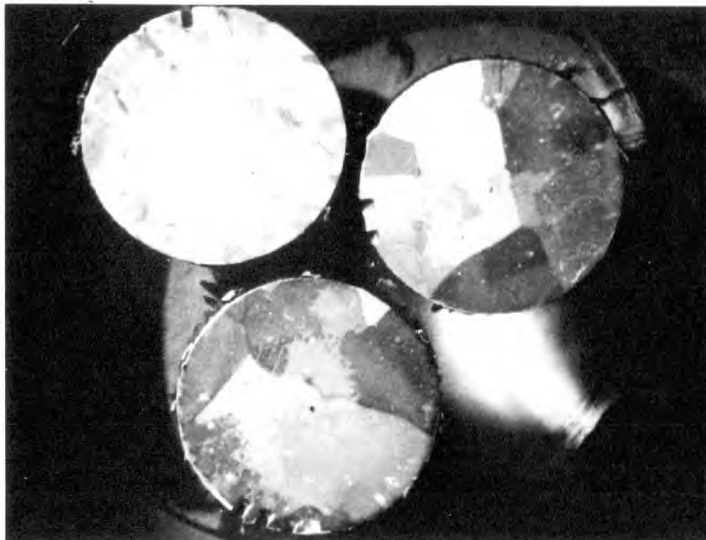


Fig. 2.9 Matrix grain size at various locations along composite ingot. Transverse sections at 2, 12 and 19.5 cm (one, two and three notches respectively) from start of growth. $R = 7.2$ cm/h (Magn. 2.9 X)



Fig. 2.10 Termination and degree of misorientation of whiskers at matrix grain boundary. (Magn. 500 X)

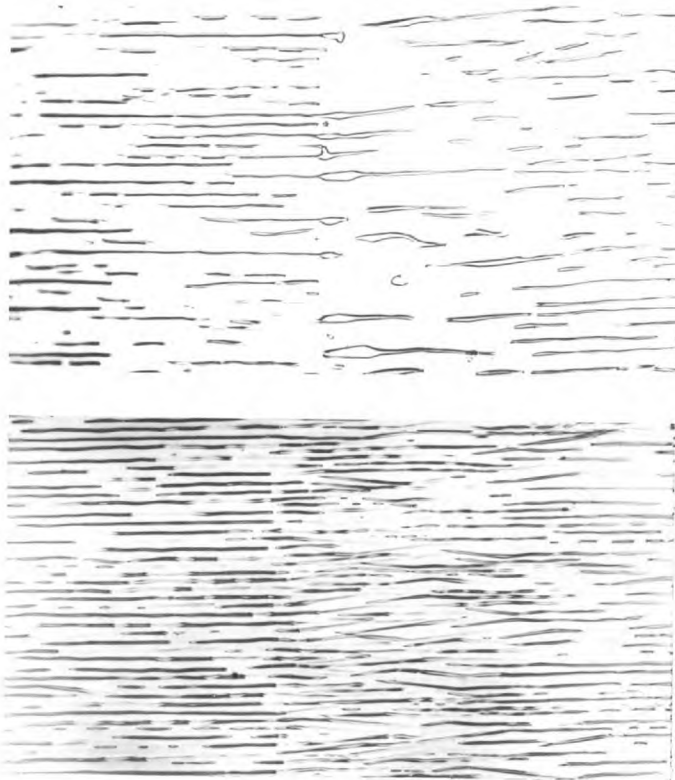


Fig. 2.11 Growth 'bands' transverse to solidification axis.
(Top) 0.9 cm/h, Magn. 350 X. (Bottom) 7.2 cm/h, Magn. 250 X

were detected on rare occasions. Specimens containing other than isolated inclusions were discarded.

Another type of structure observed in some solidified ingots was that of a regular lengthwise wave pattern of whiskers within the matrix grains. The waves appeared to be confined to one specific plane as shown by the three orthogonal plane sections, Fig. 2.12. Sections parallel to the wave planes reveal inclined rows of chopped off fibre ends whereas sections more orthogonal to the wave planes show the wave amplitude but chopped ends are less frequent. One important conclusion drawn from close observation was that no as-grown fibre ends were produced within the wave pattern.

2.5 Mechanical Testing

2.5.1 Creep Apparatus

Creep experiments were performed on two machines constructed at NPL. Load was applied via a lever of 5/1 magnification ratio, to the bottom of a 200 cm long loading train which contained universal ball joints 30 cm above and below the specimen. Load verification by the Force Section, Metrology Centre, NPL, revealed for both machines a maximum deviation of 0.6% and a much lower average deviation. The machines were equipped with moveable resistance furnaces which operated from EUROTHERM thyristor controllers. Both the temperature stability and the gradient over the specimen gauge length was maintained to within $\pm 2^{\circ}\text{C}$. Pt-13% Rh thermocouples were utilized throughout for stability.

Creep extension of a specimen was translated beyond the furnace by Nimonic alloy extensometer limbs, Fig. 2.13, and was measured either

optically (rhomb and mirror optical lever technique) or with transducers. Both techniques were checked against each other and gave consistent results. However, the optical method was found to exhibit superior stability and hence was favoured. A strain sensitivity of 5×10^{-6} was achievable.

2.5.2 Specimen Design

Cylindrical waisted specimens with gauge length shoulders and threaded ends were machined from the as-grown ingots utilizing a lathe former or copy specimen. The dimensions are shown in Fig. 2.14. A maximum cut depth of 0.25 mm per pass of the lathe tool was specified to minimize deformation; this became 0.12 mm for pure aluminium specimens.

Stresses were calculated on the diameter of the parallel portion. As some deformation must take place within the shoulders and their radii between which extensions were measured, an effective gauge length l_0 of 21.0 mm was utilized to calculate creep strain. Strains were sufficiently small to allow engineering strain to be used ($\epsilon = \Delta l / l_0$). The presence of shoulders and hence the inaccuracy in gauge length was necessitated by the need to overcome the problem of concentrated deformation and rupture at the extensometer clamping points. The problems in creep testing fibre composites, including those of the present work, were described in Chapter I.

2.5.3 Electrochemical Polishing

Subsequent to machining, the entire creep specimen was electrochemically polished in a solution of 15 ml perchloric acid (S.G. 1.54),

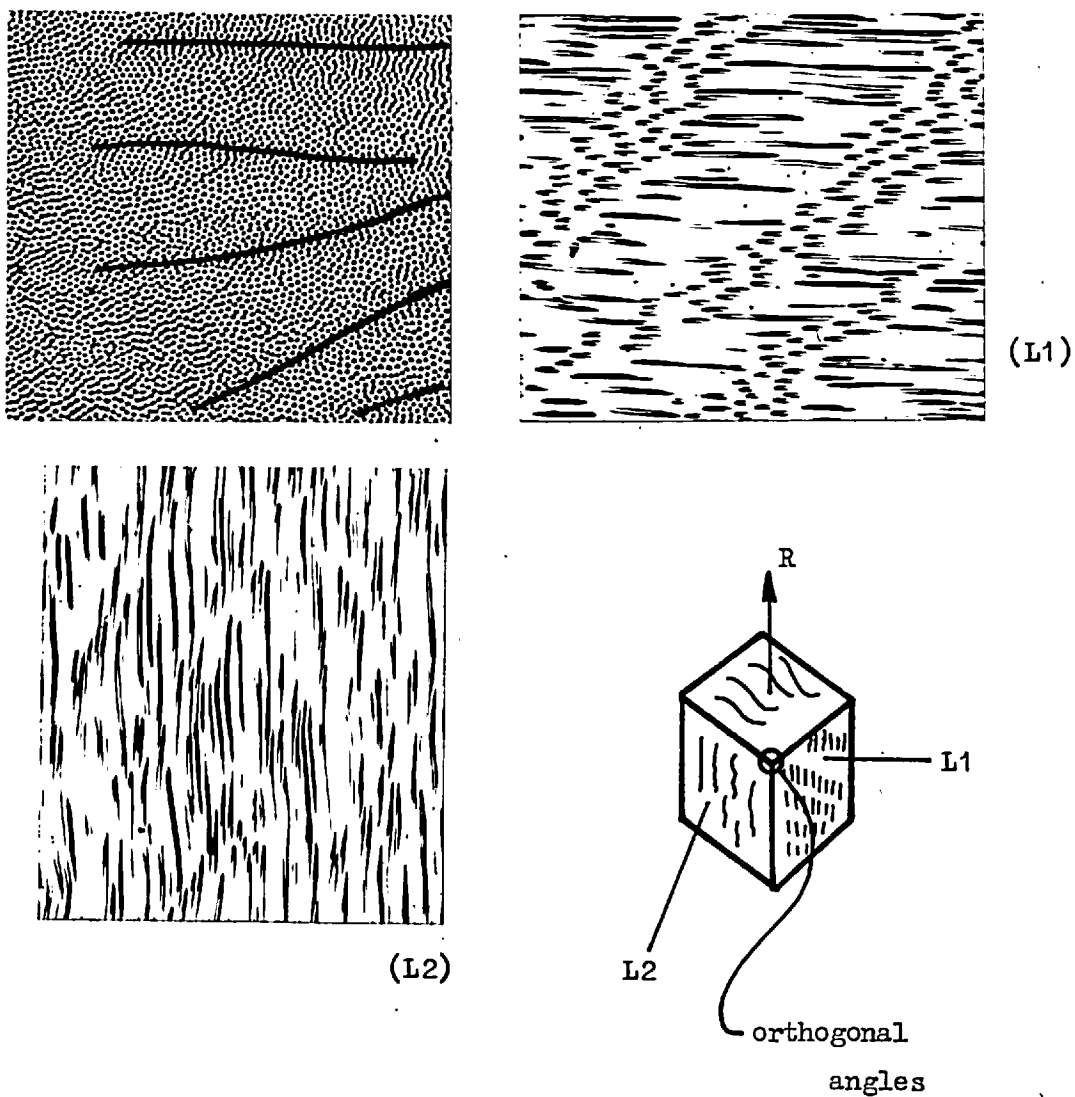


Fig. 2.12 Wave pattern of Al_3Ni whiskers within a single matrix grain.
 Area density variations in transverse section are marked. $R = 9.0 \text{ cm/h}$
 (Magn. 500 X)

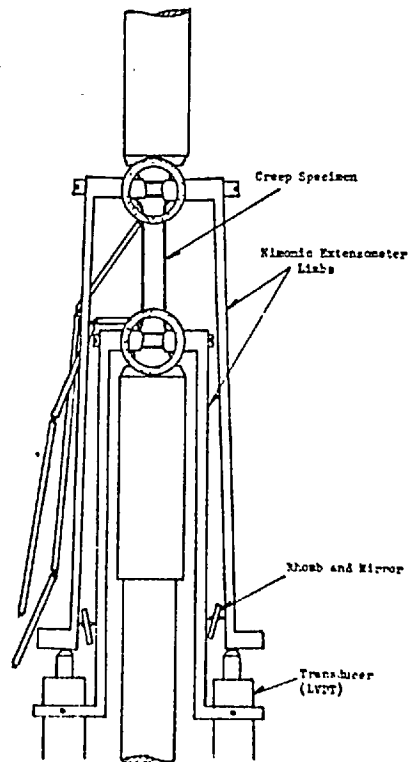


Fig. 2.13 Extensometer attachments to creep specimen.

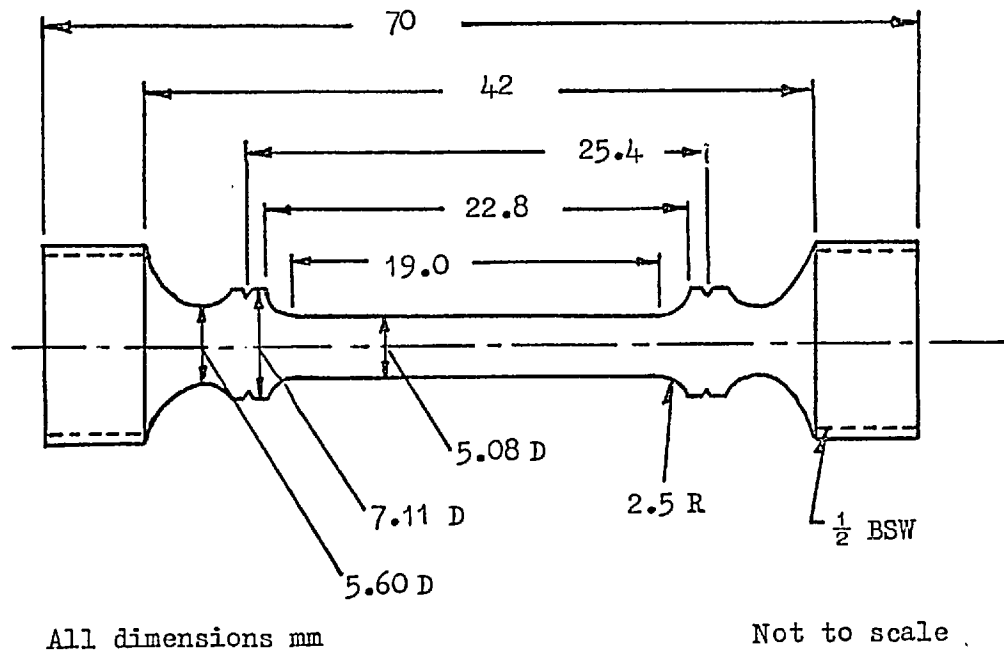


Fig. 2.14 Dimensions of cylindrical creep specimens.

88 ml 2-butoxyethanol and 148 ml methanol. A tubular aluminium cathode longer than the specimen was placed inside a cooling coil within a beaker containing the solution. This arrangement was situated in an ice bath. The specimen was rotated at approximately 150 rpm. An applied voltage of 10 V (current $\sim$ 2-3 amps) resulted in a polishing rate of 3 - 5 μ m per minute. If the bath temperature increased beyond 20°C, etch pitting of the surface occurred. A polishing time of 15 - 18 minutes gave a satisfactory single crystal-line Laue pattern of the surface of the specimen.

The polishing operation was found to be most important as it allowed complete pretest characterization of macrostructure and microstructure and, secondly, it eliminated layers susceptible to spheroidization during creep testing.

2.6 Results and Discussions

Many of the general characteristics of the Al-Al₃Ni composite have been described in the literature and hence duplication was not required. However, data were lacking in two areas which were of potential interest to the creep program - elevated temperature moduli and thermal expansion characteristics. The results of determinations of these characteristics are given in Appendices II and III.

2.6.1 Steady-State

Results

The steady-state behaviour at various stresses was determined primarily from incremental constant-load experiments. If the specimen

was unbroken after 120 hours and a well-defined constant creep rate existed, the load was increased at repeated intervals until failure. The results at the temperature of major interest, 400°C , are shown in Fig. 2.15 for specimens solidified at rates between 7 - 9 cm/h (variations in structure were considered to be insignificant, see Figs. 2.5 and 2.8). An unexpected result was the apparent scatter, the variation in stress σ_c being about 2.5 times for a given creep rate.

It would appear that the results can be approximated by a power law of the form

$$\dot{\epsilon}_c = \dot{\epsilon}_{co} \left(\frac{\sigma_c}{\sigma_{co}} \right)^{n_c} \quad \dots (2.4)$$

where the constants $\dot{\epsilon}_{co}$, σ_{co} and n_c are not well defined at this time. The stress sensitivity appears to be a relatively large value of ~ 12 .

If the creep results are extrapolated to a tensile rate of $\dot{\epsilon}_c = 6 \text{ h}^{-1}$, the stress σ_c is found to be 60 - 150 MN/m². This compares with $\sigma_c^* = 125 \text{ MN/m}^2$ from the tensile data of Salkind et al (1967a) (see Fig. 2.1).

The results of a few experiments which were carried out at 250°C and 550°C are given in Fig. 2.16. Some of the strain-time plots are given in the next section. The observed creep rates at 250°C are within the scatter band of rates obtained by Breinan et al (1970) (see also Appendix I). The 550°C data are consistent with a single measurement by Salkind et al (1966) at 572°C .

The stress sensitivity at 250°C displayed an increasing value reaching ~ 12 at larger creep rates. A slightly lower value of $n_c \sim 6.5$ was found at 550°C.

For comparison, the behaviour of the unidirectionally solidified pure matrix (Al + 0.005 wt % Ni) was determined at 400°C, Fig. 2.17. When extrapolated, the results were in accordance with those of Dushman et al (1944) for larger creep rates.

A power law can adequately describe the behaviour,

$$\dot{\epsilon}_m = \dot{\epsilon}_{m0} \left(\frac{\sigma_m}{\sigma_{m0}} \right)^m \quad \dots (2.5)$$

where $\dot{\epsilon}_{m0} = 10^{-3} \text{h}^{-1}$, $\sigma_{m0} = 1 \text{ MN/m}^2$ and the stress sensitivity $m = 4.1$. These constants are applicable to a temperature of 400°C. A generalized creep law for pure Al has been given by McLean (1962) in the form,

(the stress and rate units are those of Fig. 2.17),

$$\dot{\epsilon}_m = A' \left(\frac{\sigma_m}{G_m} \right)^m \exp \left(-\frac{Q}{RT} \right) \quad \dots (2.6)$$

where $A' = 10^{25}$, $m = 4$, and the activation energy $Q = 142 \text{ kJ/mole}$. The parameters have their usual meaning. Taking a value of $G_m \sim E/3$ from the temperature dependent matrix moduli given in Appendix II, excellent agreement was found between (2.6) and the present results, as shown by the dashed line of Fig. 2.17. Thus (2.6) can be utilized to provide an approximation of the matrix behaviour at other temperatures providing Q and m do not vary.

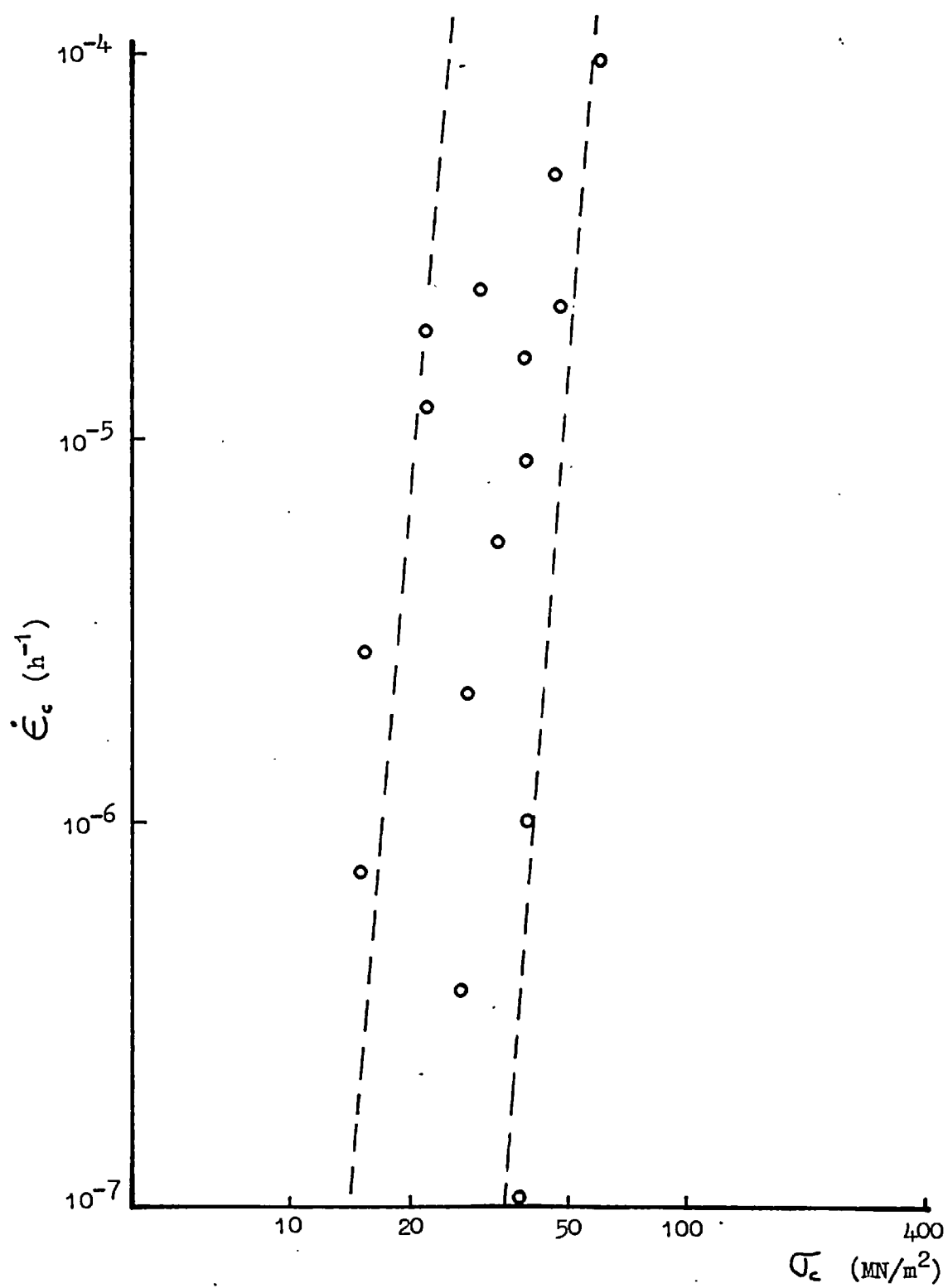


Fig. 2.15 Stress dependence of steady-state creep rate of Al-Al₇Ni at 400°C.

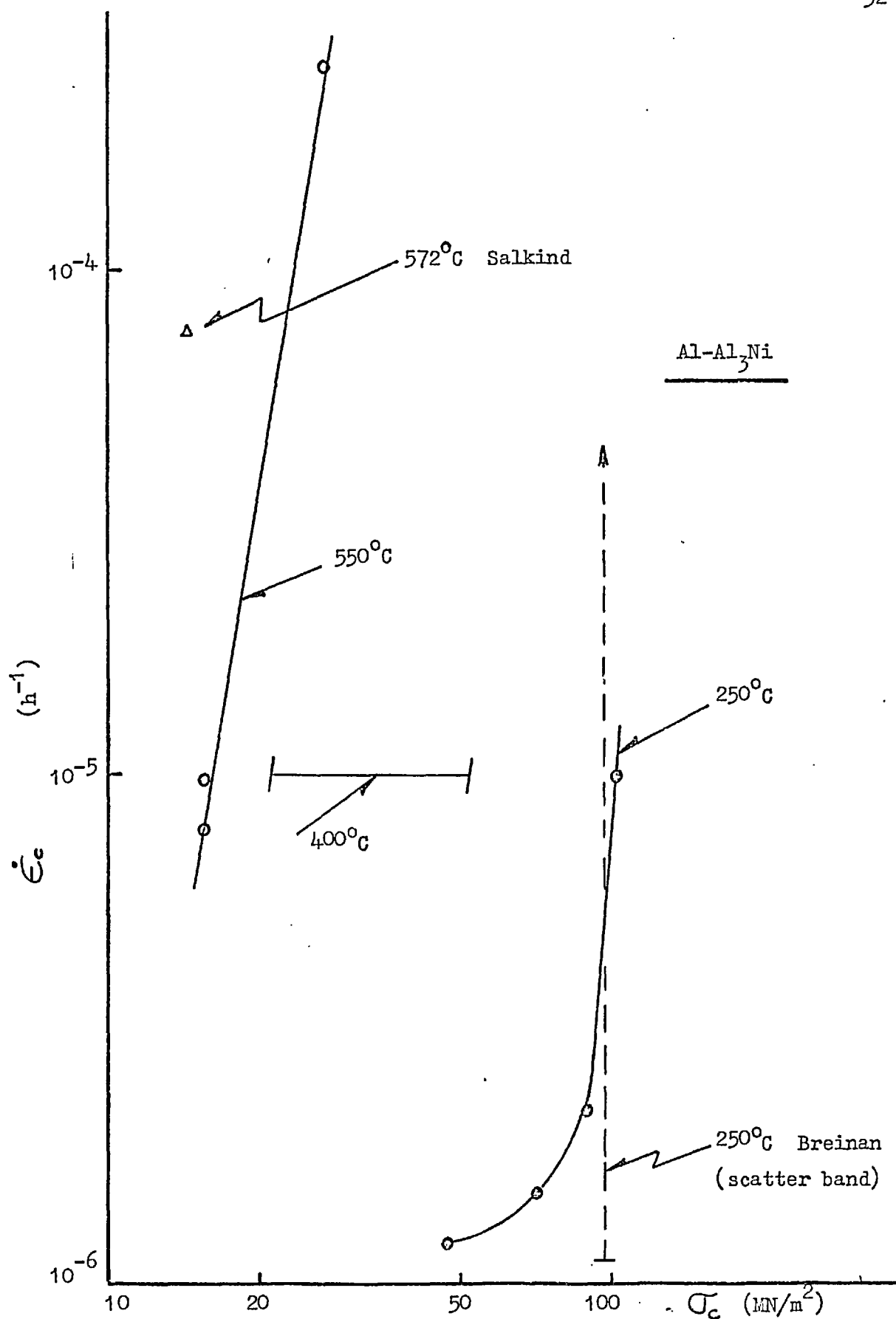


Fig. 2.16 Stress dependence of steady-state creep rate of Al-Al₃Ni at 250°C and 550°C.

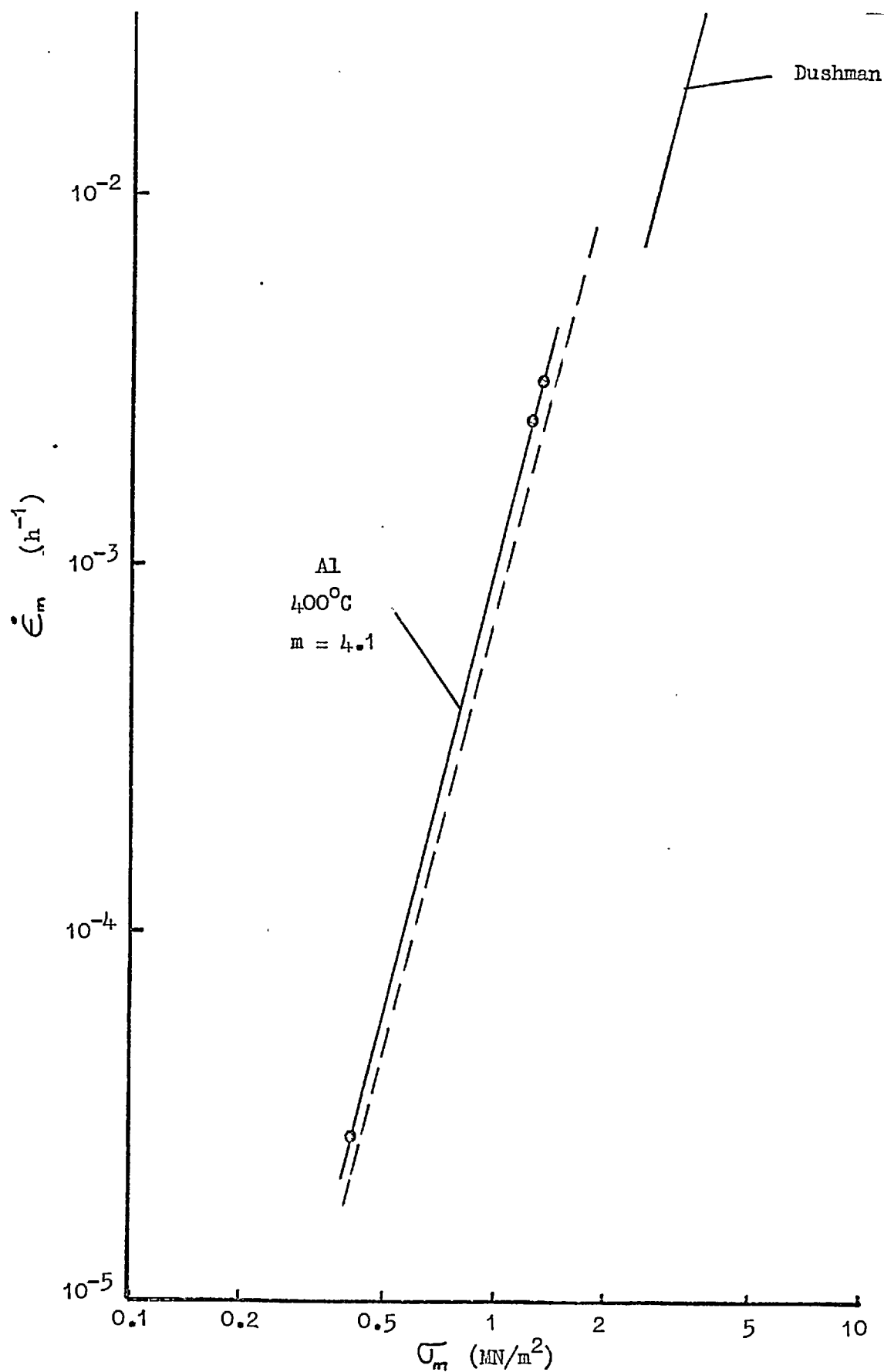


Fig. 2.17 Stress dependence of steady-state creep rate of Al matrix at 400°C.

Discussion

Steady-state creep of the composite implies that both matrix and fibres are experiencing creep under the stresses present in the composite. This was verified for the pure matrix. Qualitative evidence in support of the apparent Al_3Ni whisker creep was obtained from stress relaxation tests on extracted Al_3Ni by Breinan et al (1968). Whiskers were bent into loops and heated to elevated temperature for various times, then cooled to ambient temperature and the permanent set measured. Qualitatively, plastic deformation was observed at the lowest temperature examined, 310°C , but only data for 510°C were reported. Minimum creep rates measured from the calculated strain-time plots given by Breinan (ibid) are shown in Table 2.3.

Table 2.3 Measured Al_3Ni Creep Rates from Data of Breinan et al (1968)

<u>$\sigma_f (20^\circ\text{C})$</u>	<u>Temp ($^\circ\text{C}$)</u>	<u>$\dot{\epsilon}_f (\text{h}^{-1})$</u>
1100 MN/m^2	510	7.2×10^{-4}
1680 MN/m^2	510	1.1×10^{-3}

At values of composite creep rate similar to those of the Breinan results, $\dot{\epsilon}_c \approx 10^{-4} \text{ h}^{-1}$, the present experiments indicate maximum fibre stresses ($\sigma_{f_{\max}} = \sigma_c / V_f$) of

$$\sigma_{f_{\max}} \sim 700 \text{ MN/m}^2 \text{ at } 400^\circ\text{C}$$

and

$$\sigma_{f_{\max}} \sim 270 \text{ MN/m}^2 \text{ at } 550^\circ\text{C}$$

Both of these values are considerably lower than the σ_f values of Breinan. However, the latter values were the stresses present in the bent whisker loops at ambient temperature as the elastic modulus E_f is known only at this temperature. At elevated temperature, σ_f would be reduced if the fibre modulus was reduced.

The data of Table 2.3 indicate a fibre stress sensitivity n of approximately unity. This may explain the observation of Breinan that the variable diameter of the whiskers appeared not to concentrate the plastic deformation as it can be shown when $n = 1$, creep does not accentuate differences in cross-sectional area (McLean 1968). However, such a low stress sensitivity was not apparent in the present composite data at 400°C or 550°C.

2.6.2 Role of Grain Boundaries

The magnitude of the variability in creep behaviour suggested the existence of an uncontrolled macroscopic composite parameter. Based on the characterization studies of Section 2.4, isolated inclusions and fibre diameter or spacing perturbations were considered likely of secondary importance. Growth bands were discounted because rupture was not confined to transverse planes. It was decided to examine the role of the matrix grain boundaries. At the same time, closer quality control and pretest examination of specimens was introduced.

Creep Behaviour

Creep specimens were characterized for number and nature of grain boundaries. The boundaries were either continuous over the entire gauge length or discontinuous, that is, terminating within the gauge length. It was not possible to measure the angle between grain

boundary and tensile axis, but qualitatively the discontinuous boundaries form somewhat larger angles than a continuous boundary. In general, maximum angles of some $5^\circ - 10^\circ$ are of concern here. Specimens from a single solidified ingot were tested sequentially in the same creep machine to minimize test variations. An incremental stress procedure was adopted with the creep behaviour being measured over time periods of 120 - 160 hours to ensure a stable state.

The results of tests at 400°C are given in the form of total extension-time curves, Fig. 2.18, and creep rate $\dot{\epsilon}_c$ - stress σ_c plots, Fig. 2.19. The specimens were designated mono - , bi - , and multicrystalline; the latter always inferred that discontinuous grains were present. The findings show that the total strain and creep rate at any one stress level was largest in the multicrystal. However, less creep strain occurred in the bicrystal than the monocrystal due to differences in transient creep. A similar trend existed between the multi- and monocrystals at low stresses.

For the monocrystal at the lower stresses, the creep rate was essentially zero whereas at $\sigma_c = 38.4 \text{ MN/m}^2$, $\dot{\epsilon}_c \sim 10^{-7} \text{ h}^{-1}$, a creep rate which is less than the majority of the test results of Fig. 2.15. The bicrystal exhibited a small but measurable $\dot{\epsilon}_c$ at all stress levels.

The stress sensitivity n_c (slope of the curves of Fig. 2.19) increased from near zero to a much larger value at large σ_c . This trend was similar to that of the 250°C data shown in Fig. 2.16. The unusually low value of n_c could not be dissociated from the presence of grain boundaries in general. However, the possibility

that the monocrystal may be characterized by a similar behaviour at much lower creep rates cannot be dispelled. The results suggest a more uniform composite behaviour at large σ_c (or $\dot{\epsilon}_c$) where the role of grain boundaries is apparently much reduced.

The integrated total strain (elastic and plastic) to rupture (taken as the beginning of tertiary creep) was found to be virtually independent of grain boundary characteristics, being $0.26 \pm 0.02\%$, Fig. 2.20. About $1/3$ of this value was instantaneous strain. Breinan et al (1970) reported plastic strains at creep rupture of 0.2% maximum at 250°C for Al-Al₃Ni. Also from Fig. 2.20, the cumulative rupture times were consistent with the observed relative creep rates shown in Fig. 2.19.

The influence of grain boundaries on the behaviour at 550°C was much less pronounced for the single stress level examined, Fig. 2.21. Lower stresses were not achieved easily with the present creep machines. The creep curves were confined for the most part to the tertiary stage. Nevertheless, the tricrystal displayed slightly smaller creep rates and a longer rupture time than the multicrystal. The uniformity of behaviour is in accordance with the existence of a high stress sensitivity as shown in Fig. 2.16.

At 250°C , a multicrystalline specimen tested over a range of stresses displayed pronounced creep rates, Fig. 2.22. The final stress level achieved was fractionally larger than that used by both Salkind et al (1966) and Breinan et al (1970) at this temperature. At rupture, the total strain was 0.7% and the creep strain was 0.3%. The creep rate results are given in Fig. 2.16.

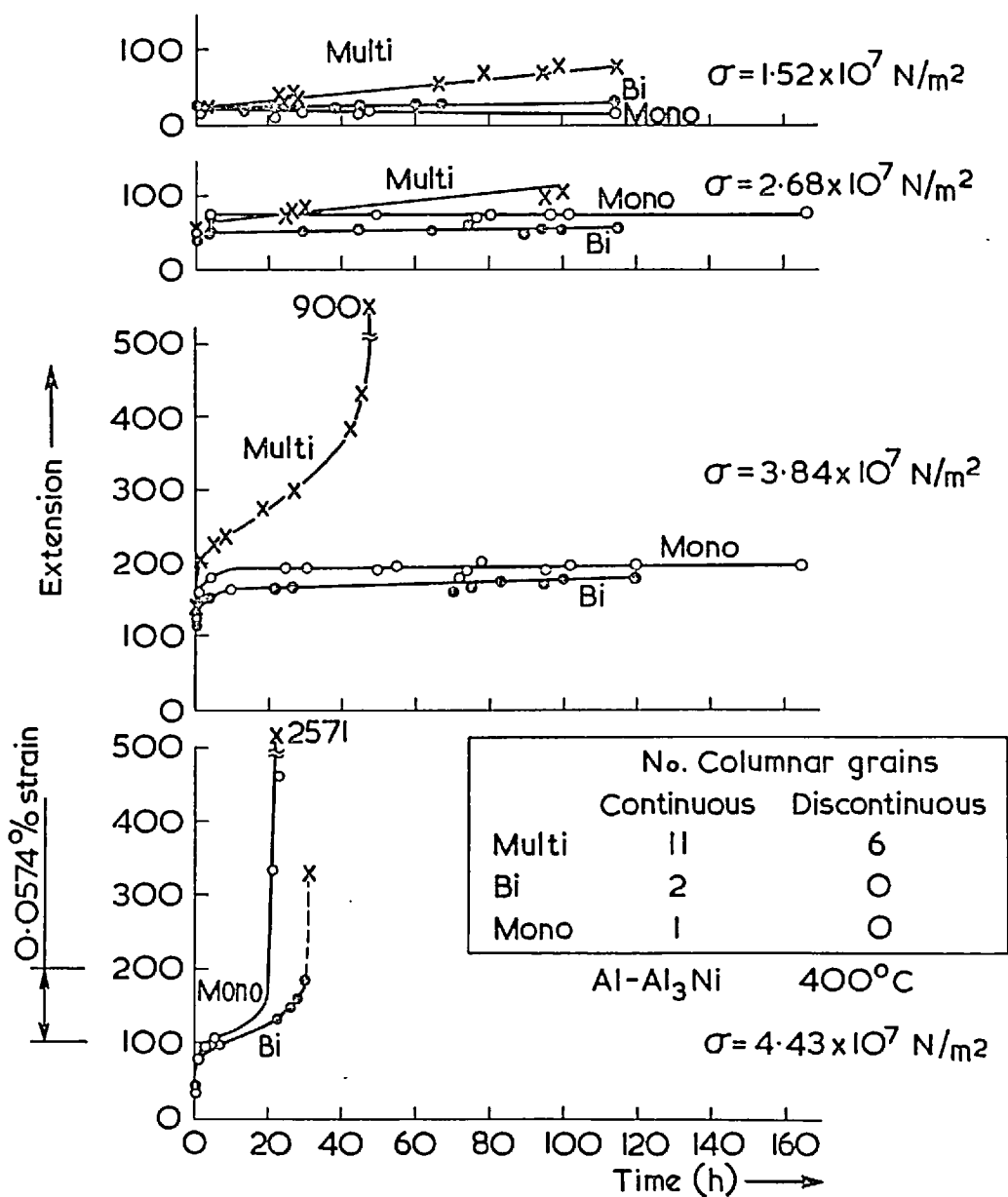


Fig. 2.18 Total extension vs time for Al-Al₃Ni at 400°C.

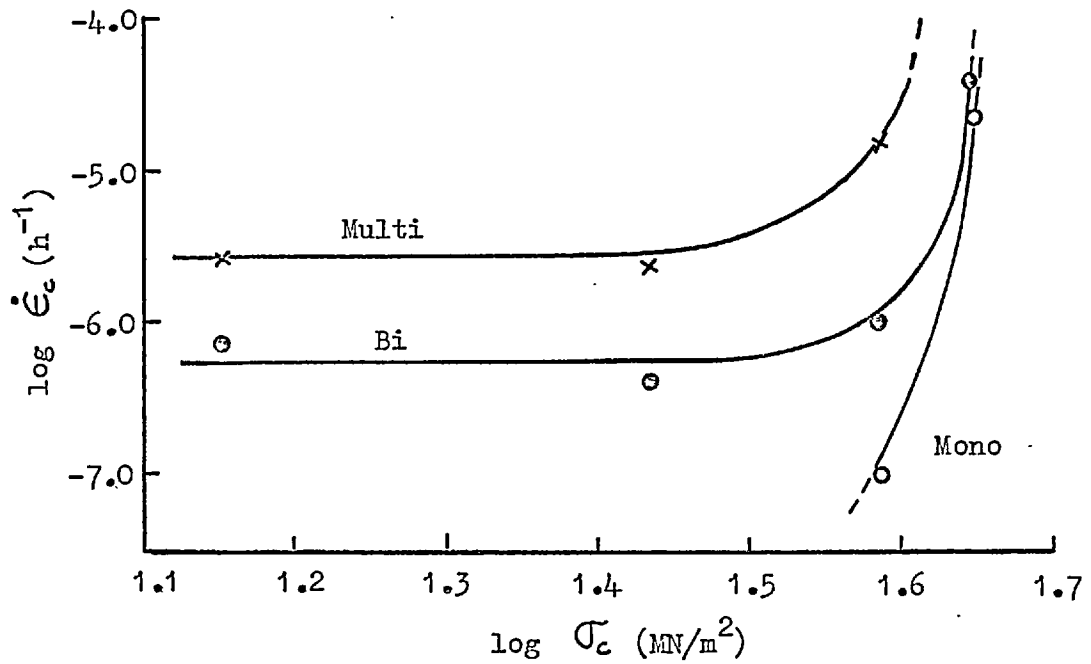


Fig. 2.19 Stress dependence of steady-state creep rate (from Fig. 2.18) at 400°C for Al-Al₃Ni.

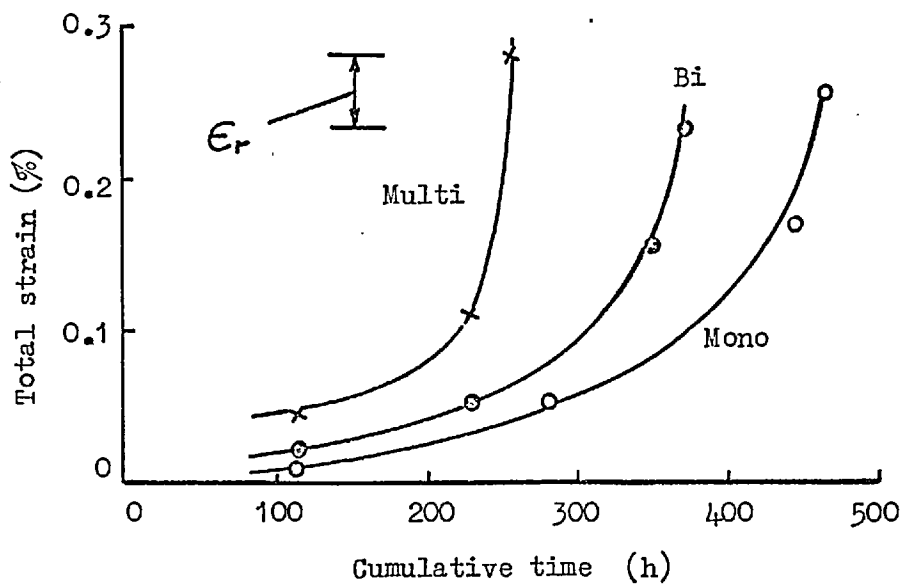
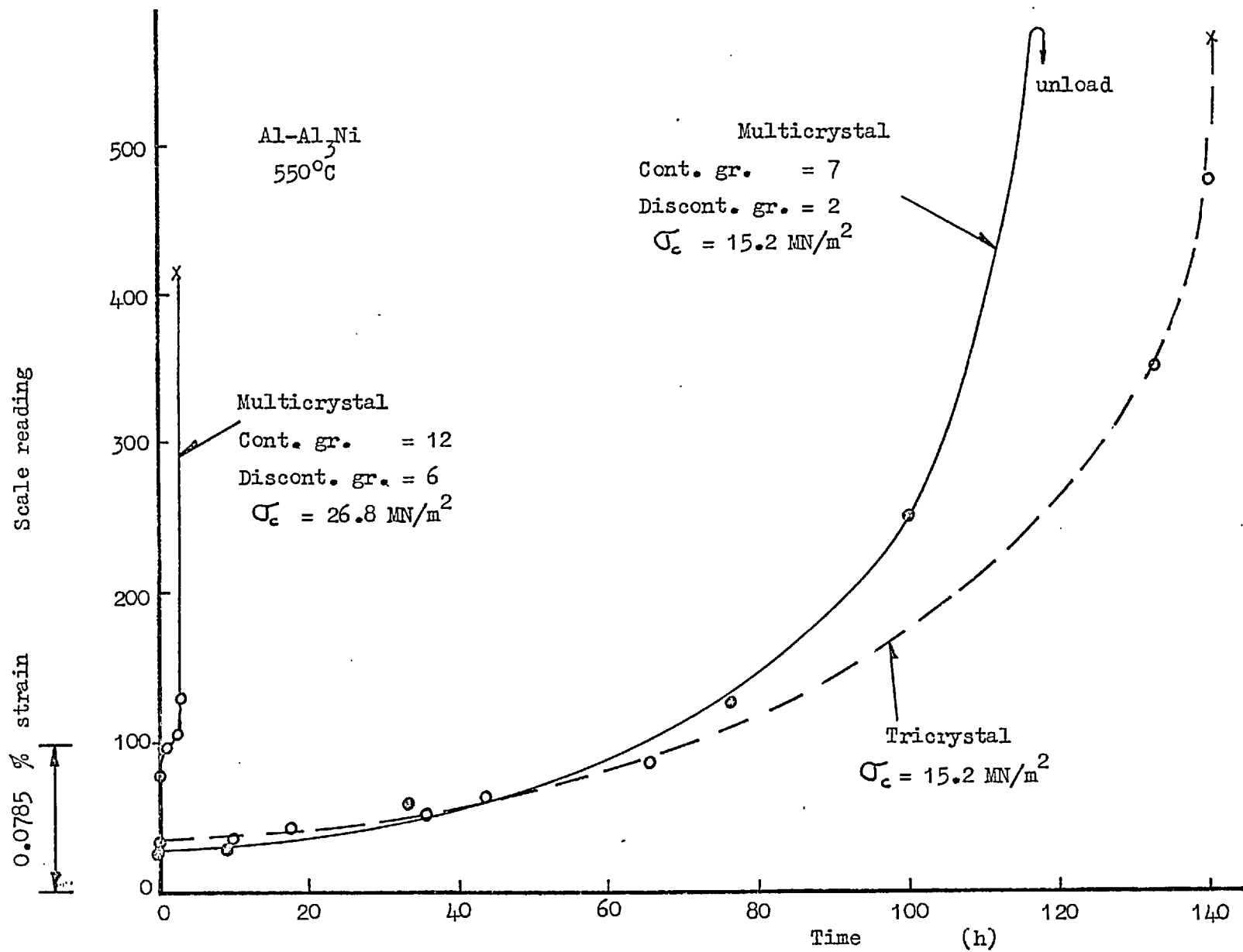


Fig. 2.20 Total strain as a function of cumulative time to rupture (from Fig. 2.18) at 400°C for Al-Al₃Ni.

Fig. 2.21 Total strain vs time for Al-Al₃Ni at 550°C. R = 9.0 cm/h



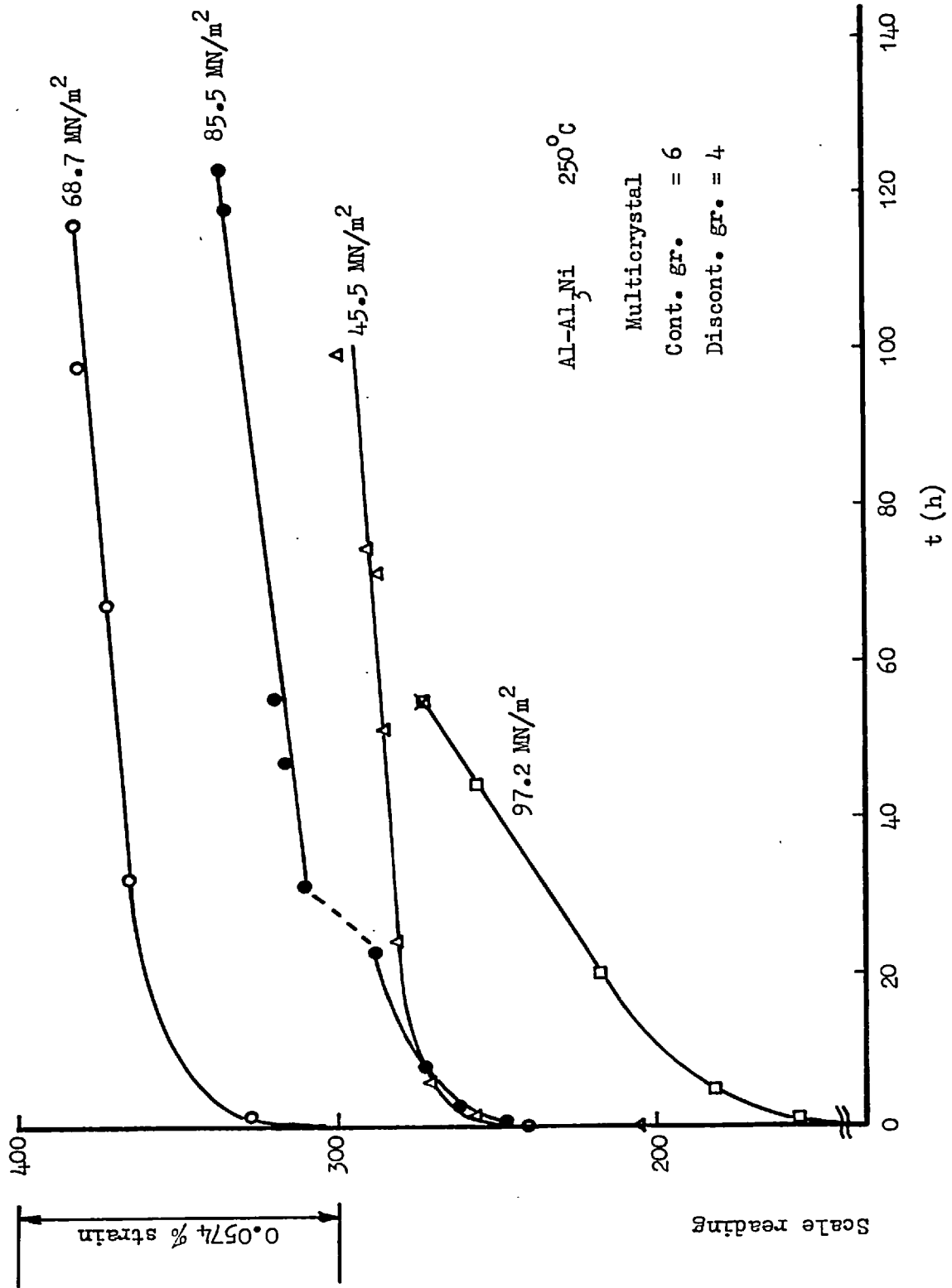


Fig. 2.22 Time dependence of total composite strain of multicrystal at 250°C. $R = 9.0 \text{ cm/h.}$

Boundary Displacements

Prior to creep testing, marker lines were lightly scribed with a razor blade across the grain boundaries of some specimens. Observations were made at the termination of a test for relative displacement between grains.

At 400°C, displacements were observed along discontinuous grain boundaries providing the adjacent grain was a continuous one. The character was that of simple sliding at the interface as shown in Fig. 2.23. The displacement in this figure corresponds to $\sim 0.043\%$ tensile strain ϵ_c over the gauge length, a value not widely different from the cumulative composite creep strain at the end of the second stress level, $\sim 0.055\%$ (Fig. 2.18, $\sigma_c = 26.8 \text{ MN/m}^2$, multicrystal). Distinct cracks or cavities were not found along the displaced boundaries.

At 550°C, only the boundaries of the tricrystal were marked for displacement observations. One case of simple sliding was found, Fig. 2.24(a). However, as it was relatively close to the rupture surface (few mm either side of the rupture) it may have been induced in the final stages of failure. The relative displacement between grains corresponds to $\epsilon_c \sim 0.025\%$ over the gauge length. Another feature of the same grain boundary was irregular cracks in the superficial oxide layer, Fig. 2.24(b). The profiles of the cracks suggest differential creep or differential contraction on cooling in the adjacent grains. The nature of the cracks also imply the presence of a shearing effect.

At 250°C, the multicrystalline composite revealed also boundary displacement in association with discontinuous grains only. Coupled with simple sliding, considerable axial shear occurred within the adjacent continuous grain, Fig. 2.25. Both the total marker displacement (sliding plus shear) and the sliding displacement increased towards the radius of the specimen shoulder, Fig. 2.26. As this particular specimen ruptured outside the gauge length, there is no question that the observations reflect creep behaviour. No other boundary effects such as concentrations of broken fibres, voids or microcracks were detected after considerable examination. However, the boundary was very disturbed, indicating a considerable surface roughness resistance to sliding. This source of boundary strength may be the main reason for most of the relative displacement occurring by shear in the matrix parallel to the fibres.

Rupture

A careful control of the orientation of the creep specimens with respect to the solidification direction resulted in the observation that > 80% of all creep ruptures occurred at that end of the specimen formed first during solidification. This end coincides with either the smaller grain size or the larger number of discontinuous grains. Exceptions to the observation were found to be specimens which contained only continuous boundaries. Furthermore, the effect was independent of testing procedures.

Composite rupture was found to originate within a continuous grain adjacent to a discontinuous one when the boundary between them had experienced sliding. This is shown in Fig. 2.27(a). Microscopically, the rupture of fibres occurred along regular inclined planes which were

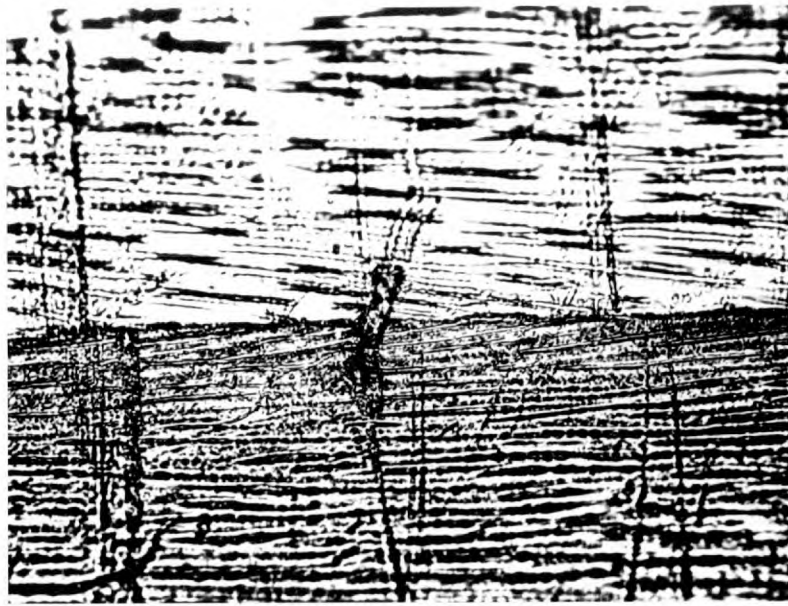


Fig. 2.23 Grain boundary displacement between discontinuous grain (top) and continuous grain (bottom) after creep at 400°C . (Magn. 750 X)

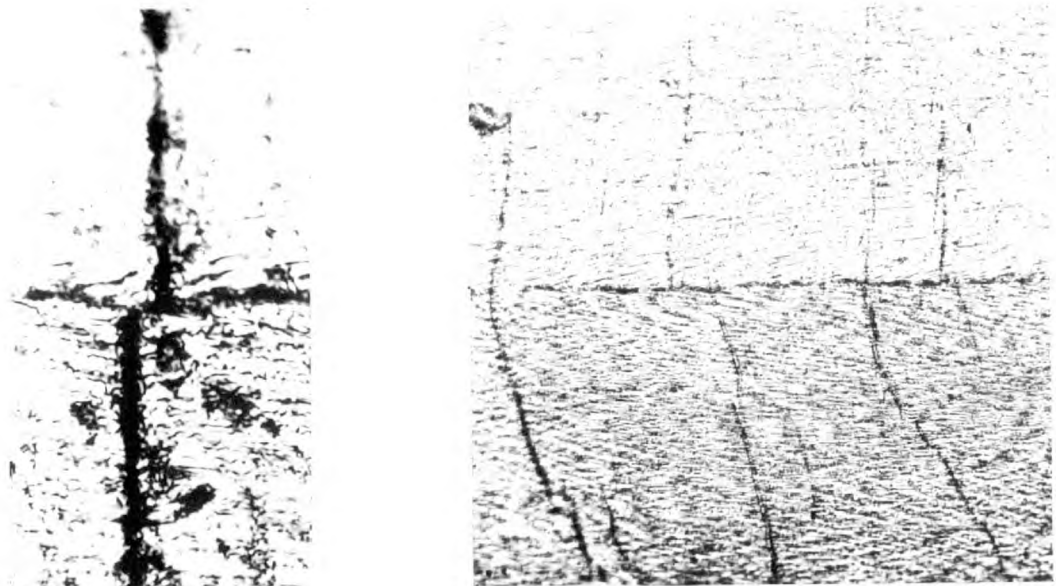


Fig. 2.24 Left: Displacement between two continuous grains after creep at 550°C . (Magn. 1000 X).

Right: Cracks in superficial oxide film adjacent to boundary of left photo. (Magn. 250 X).

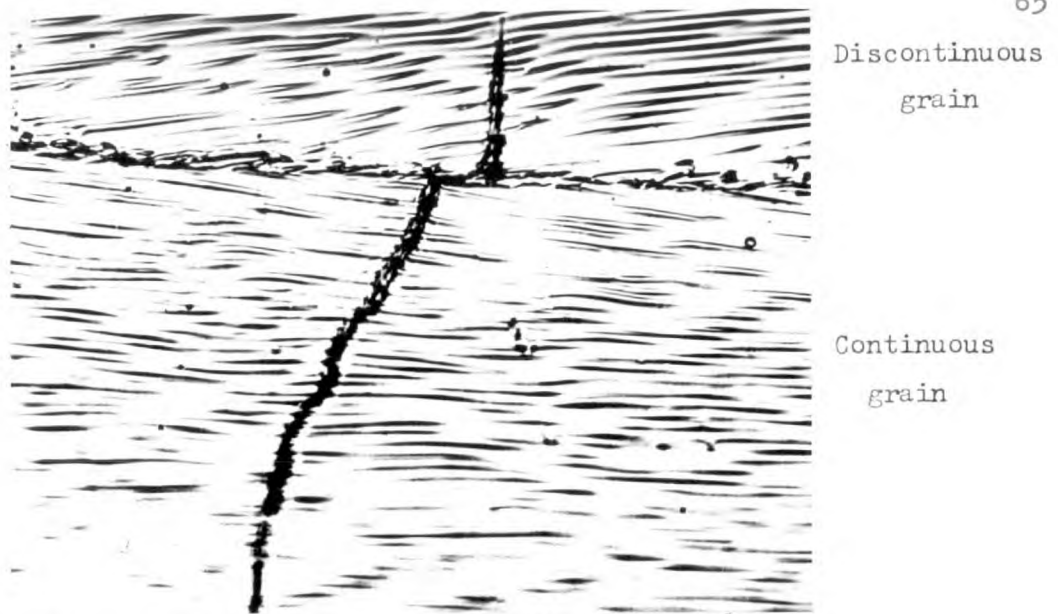


Fig. 2.25 Marker line after creep at 250°C (Magn. 750X)

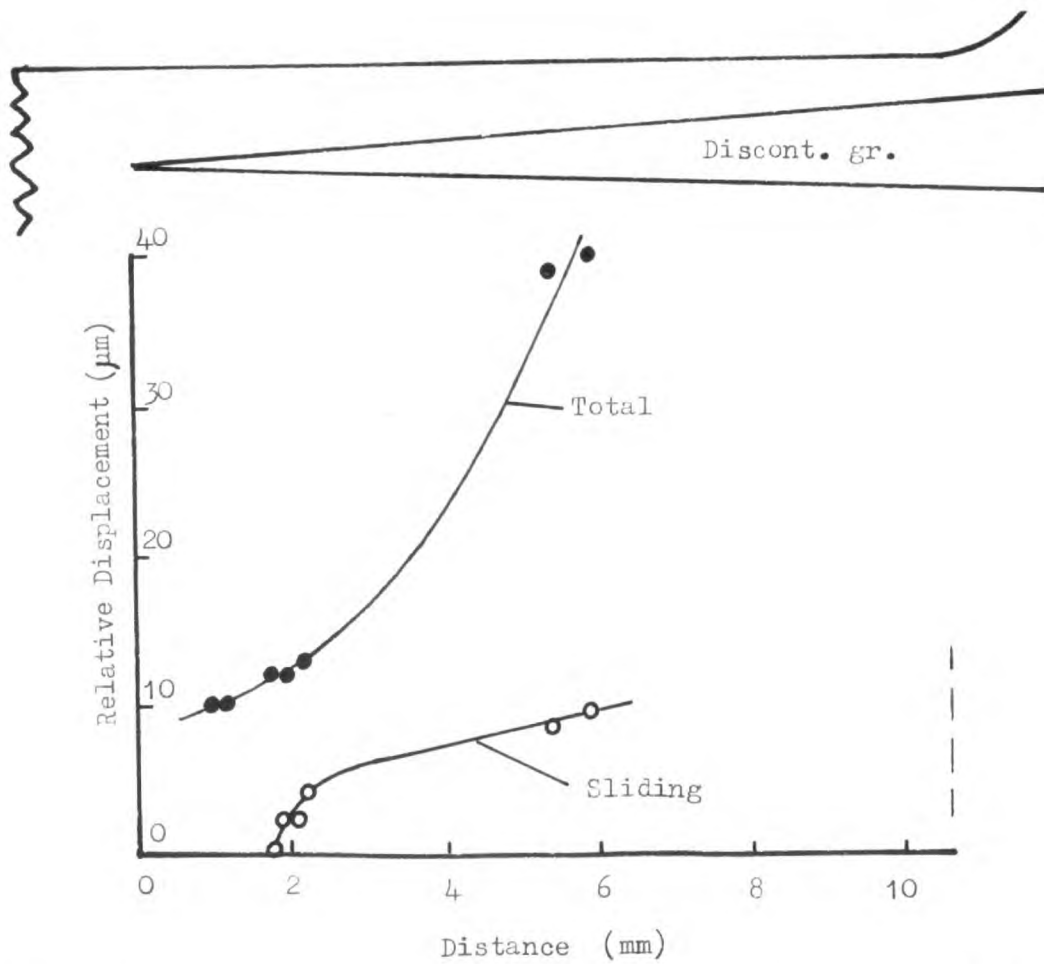
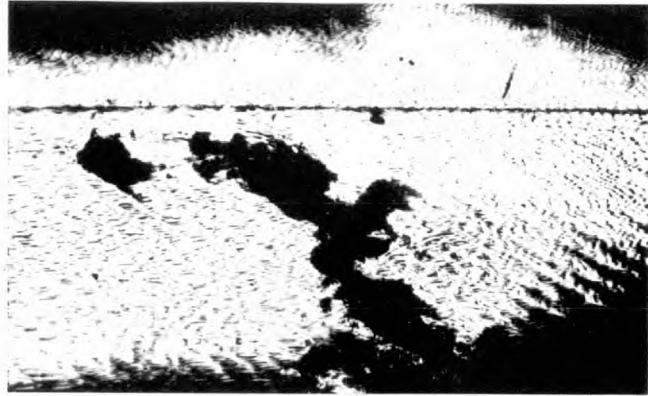
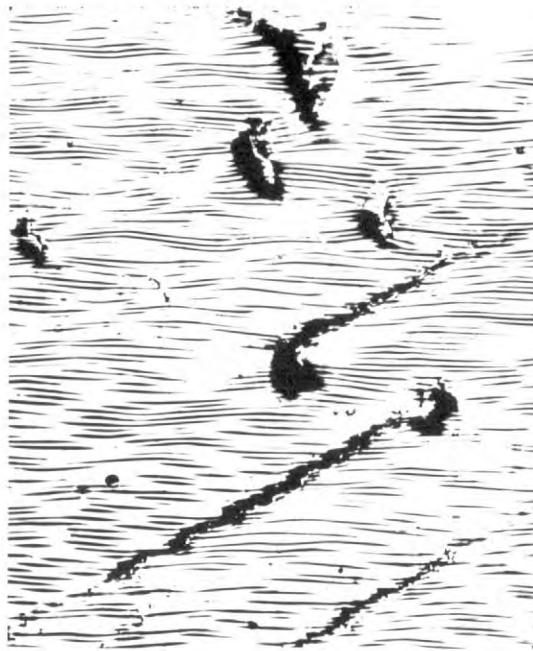


Fig. 2.26 Measured relative displacement along boundary of Fig. 2.25 (to scale).



(a)



(b)

Fig. 2.27 (a) Rupture cracks within a continuous grain (bottom) which was adjacent to a discontinuous grain (top). (Magn. 40 X)

(b) Whisker rupture profiles within the continuous grain. 250°C. (Magn. 400 X).



(a)



(b)

Fig. 2.28 Correlation of creep rupture surface details and matrix grain boundaries (a) 250°C, (b) 400°C. (Magn. 4-5 X).

associated with a whisker wave pattern in general, Fig. 2.27(b).

It is recalled that as-grown wave patterns were observed in the composite (see Fig. 2.12). In addition, fractographic examination revealed a correlation between topographical discontinuities and grain boundaries, Fig. 2.28.

Discussion

Under conditions of creep deformation, it is apparent that the grain boundaries of the Al-Al₃Ni composite are inherently weaker than the reinforced grains. The boundary strength is derived from the matrix plus any surface irregularities. Conclusive evidence showed that both the composite creep rate and the rupture characteristics are adversely affected.

Walter and Cline (1970) associated fracture surface observations with both colony or cell boundaries and grain boundaries in the NiAl-Cr eutectic which was tensile tested at 1200°C ($T/T_m = 0.86$). It was concluded that the shear strength of the cell walls limited the ultimate tensile strength of the composite. The present work suggests that the boundaries play more of an indirect role. Whereas fracture surface observations inferred boundary shear failure, microscopic studies revealed that rupture originated away from the boundaries within a continuous grain. De Silva (1969) observed boundary sliding in the grip of a Fe-Fe₂B eutectic specimen tensile tested at 810°C, but this is a more expected result.

The extensive creep rupture results on Al-Al₃Ni at 250°C by both Salkind et al (1966) and Breinan et al (1970) do not indicate the location of rupture relative to growth direction but most of their

examples show failure at one end of the gauge length adjacent to a radius. It is also noted that the early work on the ambient temperature tensile behaviour of Al-Al₅Ni by Hertzberg et al (1965) revealed radius or shoulder failures in all but one specimen, neglecting banded specimens.

The creep rate dependence upon stress found at 250°C and 400°C is similar to the effect of grain boundaries in pure polycrystalline Al (Servi et al 1951). At low rates (or stresses), boundary strength is a controlling factor of the deformation and the stress sensitivity is relatively low. At larger rates, grain strength determines the behaviour and a larger stress sensitivity exists. The transition rate, $\dot{\epsilon}_e$, coupled with the particular temperature defines the "equicohesion temperature", T_e . Servi found values of $\dot{\epsilon}_e \sim 10^{-3} \text{h}^{-1}$ at 250°C and 10^{-1}h^{-1} at 400°C. It is noted that the composites exhibited much lower transition rates of $\sim 10^{-5} \text{h}^{-1}$.

The role of grain boundaries, especially discontinuous ones is expected to be related to their number, orientation relative to the tensile axis and the misorientation with respect to adjacent grains. This follows from Tung and Maddin (1957) who observed that both the shear rate and the shear activation energy in Al bicrystals (common $\langle 110 \rangle$ axis) were orientation dependent.

Model - Influence of Discontinuous Boundaries

From the experimental findings and discussions of the previous section, a model is proposed to qualitatively account for the influence of discontinuous grain boundaries. Consider a simple eutectic composite composed of one continuous grain plus two discontinuous grains, subjected

to an applied load, Fig. 2.29(a). During extension a shearing stress τ is necessarily induced along the discontinuous boundaries, as only the continuous grain is loaded directly. When the overall rate of extension is low (grains themselves are strong), the low strength of the composite boundaries is insufficient to prevent sliding. Such sliding per se does not constitute a direct contribution to the total extension of the composite as it does in polycrystalline materials. The measured extension and hence creep rate is that of the continuous grain. Hence a simple low stress sensitivity mechanism associated with, for example, grain boundary sliding (Alden 1969) is not feasible. A more basic mechanism which involves creep of the continuous Al_3Ni whiskers is required. At larger values of σ_c , the continuous grain itself possesses a greater $\dot{\epsilon}_c$ and the boundaries become less significant. The composite is now characterized by a higher stress sensitivity. Schematically, the overall creep rate dependence is shown in Fig. 2.29(b).

Regarding the rupture behaviour, the model is more clear. In effect, the discontinuous grains cannot be made to support their share of the load and the adjacent continuous grain experiences an increased stress. This results in an even greater localized creep rate due to the non-linear dependence upon stress and, ultimately, rupture within the continuous grain at the overstrained end of the specimen. It is obvious that creep strain cannot be uniform along the gauge length.

The simplified model can qualitatively account for the majority of the results reported in this section except for the role of continuous boundaries which is less clear. A differential extension (elastic or creep) between two continuous grains would induce a boundary shear stress.

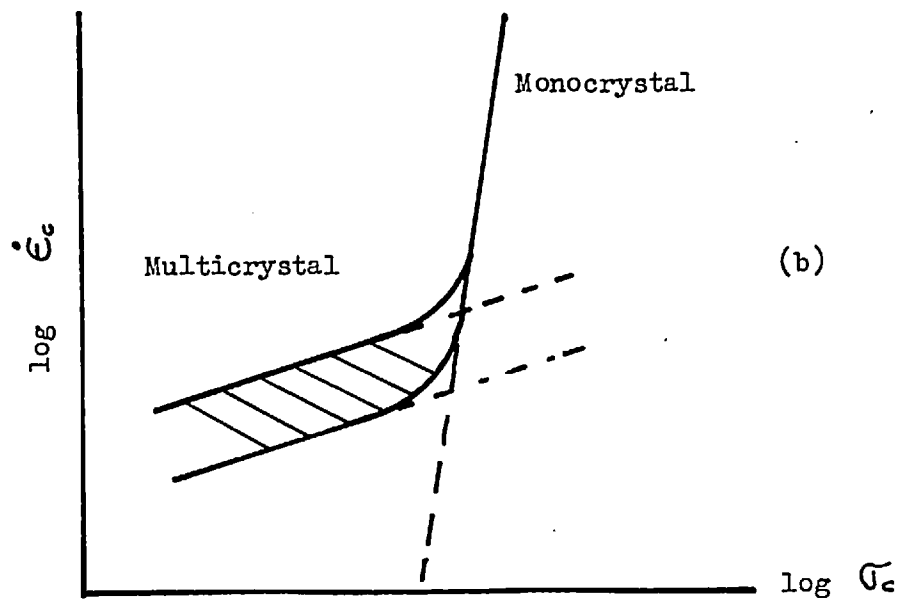
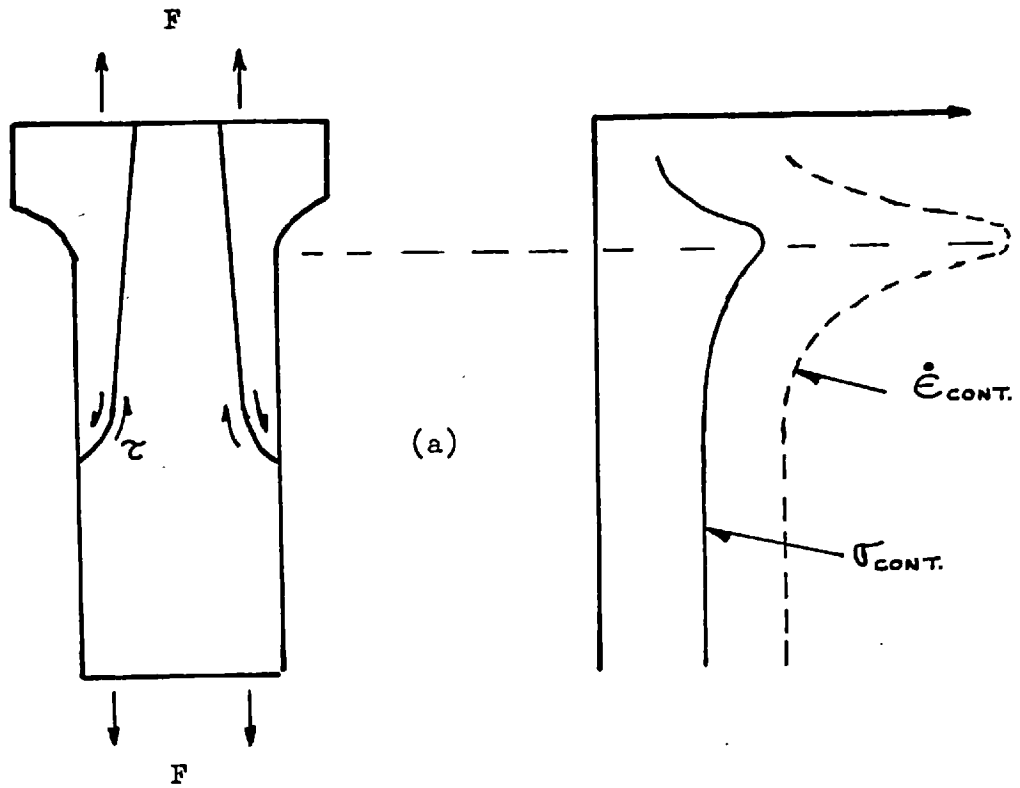


Fig. 2.29 Model of discontinuous grain boundary effects in Al-Al₃Ni
 (a) stress and creep rate profiles along continuous grain
 (b) stress σ_c dependence of composite creep rate $\dot{\epsilon}_c$.

However, such boundaries were not obvious sources of weakness. In fact, the opposite appears to be true - the boundaries effectively strengthened the composite by reducing the transient creep strain. This finding is similar to the observation of Chalmers (1937)(1937a) that the creep yield strength of Sn bicrystals increased with the misorientation between grains and not simply the number of grains.

2.6.3 Microstructures and Thermomechanical Stability

The apparent creep behaviour of Al-Al₃Ni may have been influenced by several other factors such as fibres rupturing due to either over-stress or to thermal instability effects. To separate these factors from any localized effects caused by composite rupture itself, microstructural observations were made at locations remote from the composite fracture surface.

Fibre Integrity Within Crept Specimens

The stress history of the test at 250°C shown earlier in Fig. 2.22 resulted in only isolated breaks over the gauge length even though a well-defined wave pattern of whiskers was present. Similarly, at 400°C, no cases were detected of more than a few broken whiskers. It was concluded that the overall macroscopic creep rates were not significantly influenced by rupturing fibres. Such was not the case in B/Al composites where it was found that breakages in the B fibres induced an apparent creep strain (Breinan and Kreider, 1970).

At 550°C, a distinctly modified microstructure was found. Numerous regular inclined rows of whisker breaks such as shown in Fig. 2.30 existed along the gauge length. The area density of these "fibre rupture bands" increased near the fracture location and decreased in

the low stress regions of the specimen as shown by the plot of Fig. 2.31. As a result of this finding, it was concluded that the measured creep behaviour (as distinct from the rupture characteristics) at this temperature was likely influenced by rupturing whiskers.

Thermomechanical Stability

As a preliminary experiment, the structural modifications in as-grown material due to temperature alone were determined in accelerated anneals at 610°C (T/T_m eutectic 0.96). Temperature was recorded by a thermocouple contacting the specimen. Metallographic examination of the specimen interior after various times revealed that the whiskers had experienced considerable coarsening or ripening as evident in the transverse sections of Fig. 2.32. This characteristic has been observed before by Bayles et al (1967) for anneals at the same temperature. He measured average fibre densities, an analysis which unfortunately is not amenable for comparison with theories of coarsening. Hence, at this time, a basic mechanism has not been found.

Although sectioning in a plane parallel to the whiskers is very difficult, all of the longitudinal sections of Fig. 2.32 show quite clearly true fibre ends after the anneals, a feature never observed in as-grown ingots. Similar results were obtained from three separate solidified ingots. These observations are in contradiction to Bayles (ibid) who reported no foreshortening of the whiskers after 500 h at 608°C . The difference may be due to either metallographic luck or to some unknown variation in the as-grown microstructures.

The importance of this phenomenon on creep behaviour at lower temperatures must depend upon the responsible diffusion rates and the

effect of stress (or strain). In the Bayles work, no effect on the ambient temperature tensile strength of the composite was found. However, tensile strength may not be as sensitive to fibre aspect ratio changes as creep strength.

The only distinguishing feature of the broken whiskers in creep tested specimens is the enlargement or bulbing of the ends at 550°C (see Fig. 2.30). This characteristic was not associated with the thermal induced ends. Cratchley (1965) and Sulkind et al (1967) observed that mechanical deformation, which destroys the as-grown well-matched interfaces, resulted in extensive spheroidization of Al_3Ni at temperatures as low as 350°C. Similarly, the bulbing could then be due to the deleterious effect of localized strain which occurred on fibre rupture. Spheroidization was even more pronounced adjacent to the main rupture of the composite as shown by Fig. 2.33. If the ruptures actually originated from thermal instability, coarsening phenomena as observed in transverse sections are irrelevant to creep and rupture characteristics of fibre composites. A completely different mechanism may exist locally within the composite.

A creep specimen was subjected to a diminished stress $\sigma_c = 13 \text{ MN/m}^2$ and a temperature of 425°C for a relatively long time of 1400 hours. The resulting microstructure contained only isolated whisker breaks and no ripening was detected in the transverse plane.

It would appear justifiable to conclude that the measured creep characteristics at 250°C and 400°C are representative of combined matrix and fibre creep behaviour and are not the product of either mechanical or combined thermal and mechanical instability effects. As the composite

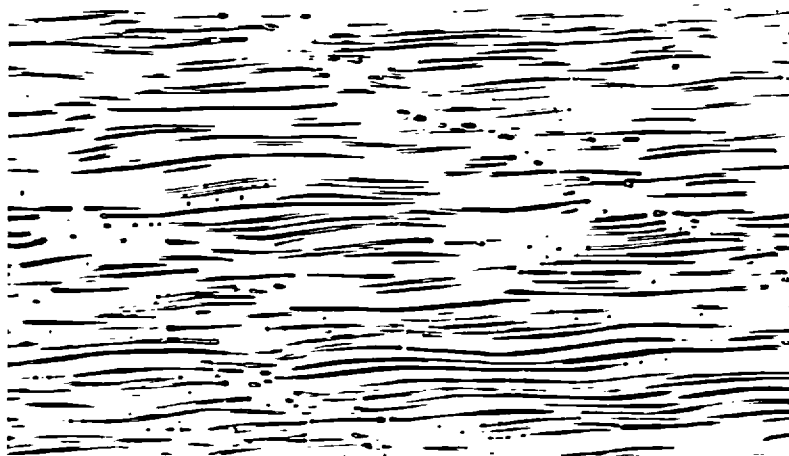


Fig. 2.30 Nature of broken whiskers remote from incipient composite fracture after creep test at 550°C. (Magn. 500 X).

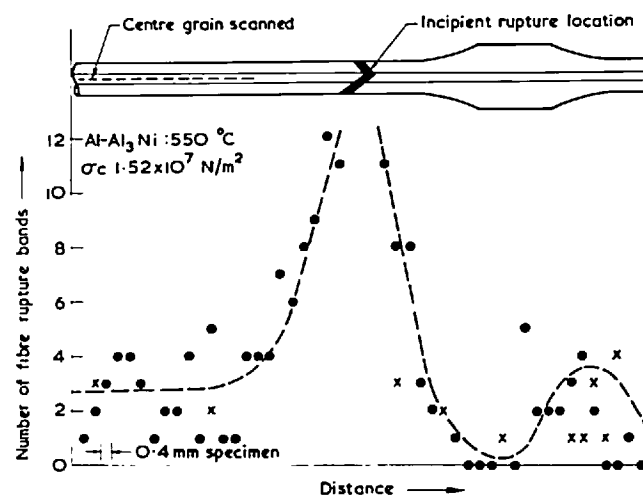
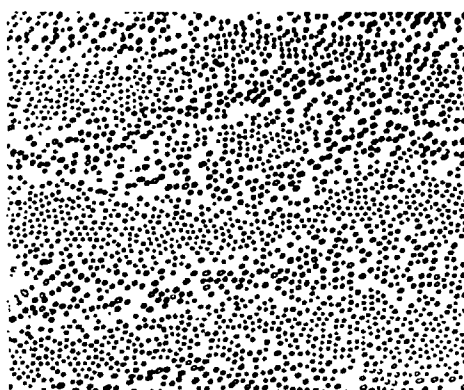
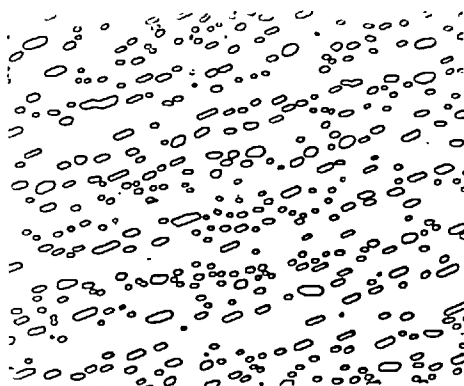
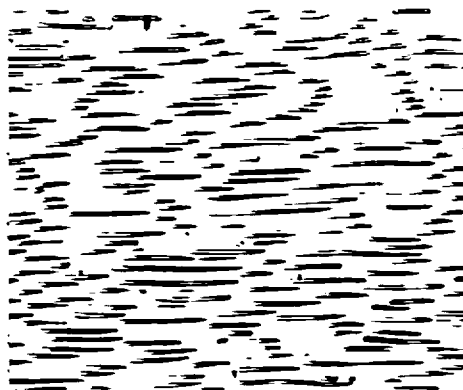


Fig. 2.31 Density of "fibre rupture bands" (as shown in Fig. 2.30) along length of specimen after creep test at 550°C.



27.
h.



190.
h.



455.
h.



Fig. 2.32 Transverse and longitudinal section microstructures after zero stress anneal for various times at 610°C . $R = 7.2 \text{ cm/h}$. (Magn. 500 X).

has been found to be substantially less stable at 610°C than previously reported, the creep and rupture characteristics at 550°C could well have been controlled by coarsening phenomena but on a localized scale.

2.6.4 Rupture Characteristics

Although the programme was concerned with the factors which control the apparent creep behaviour of the composite, a few observations on the characteristics of rupture are intrinsic in the creep study. Such observations may be of interest to a separate programme on composite rupture characteristics.

Results

Macroscopically, composite ruptures displayed knife-edge characteristics varying from single shear to complex multiple shear surfaces, for all creep temperatures, Fig. 2.34. With few exceptions, the shear surfaces formed angles of 30° - 40° with the tensile axis. Pure Al single crystals rupture in a similar fashion (Johnson et al 1956). The extensive macroshearing which occurred on separation of the specimen obliterated the surfaces of detail.

Microscopically, the majority of whisker breaks were confined to regular matrix planes (see Fig. 2.30). The observed angles between the rupture bands and tensile axis depended on the plane of the section, but a minimum angle (true angle if one set of planes exist) of $\sim 25^{\circ}$ was measured.

Exceptions to knife-edge ruptures occurred in specimens containing transverse growth defect bands. Rupture, as well as creep deformation, was confined to the transverse band profile, Fig. 2.35(a). Such a

structure must render the composite unamenable to quantitative analyses such as attempted by Butcher et al (1969) for banded Al-Al₂Cu. Two additional unique features found in banded specimens were numerous voids or cavities adjacent to the rupture surface, Fig. 2.35(b), and pulled-out whisker ends, Fig. 2.35(c). The voids may have been the result of triaxial stresses induced during concentrated deformation within the band as they were never observed in unbanded specimens.

Occasionally etch pit rows terminating at unbroken fibres in creep tested specimens were observed, Fig. 2.36. The etching was accidental and may have occurred only when the appropriate etching plane coincided with the observation plane. The nature of the pits suggests dislocation pile-ups.

Discussion

The tendency for Al₃Ni to rupture along well-defined matrix planes implies an interdependence between fibre rupture and matrix behaviour. The observations suggest that composite rupture follows from fibres progressively rupturing along well-defined matrix planes, leading ultimately to failure of such planes in shear. The significant feature is, of course, the origin of the preferential ruptures. The association of fibre breaks and the fibre wave patterns shown earlier in Fig. 2.12 infers a similar origin for these two phenomena.

Breinan et al (1970) proposed that the creep rupture of Al-Al₃Ni at 250°C was initiated by isolated fracture of whiskers due to local stresses associated with dense dislocation tangles in the matrix. However, as they observed that such tangles did not build up during steady-state creep (total plastic strains were $\sim 0.2\%$) and were

present only in ruptured specimens, the tangles may be a composite rupture induced effect only.



Fig. 2.33 Microstructure adjacent to composite rupture surface after creep test at 550°C.

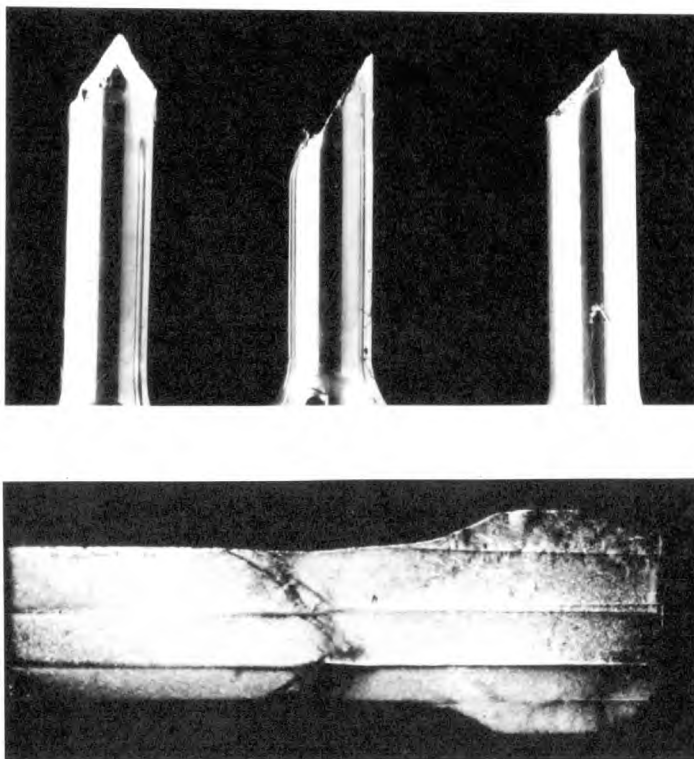


Fig. 2.34 External and internal macroscopic characteristics of composite rupture.

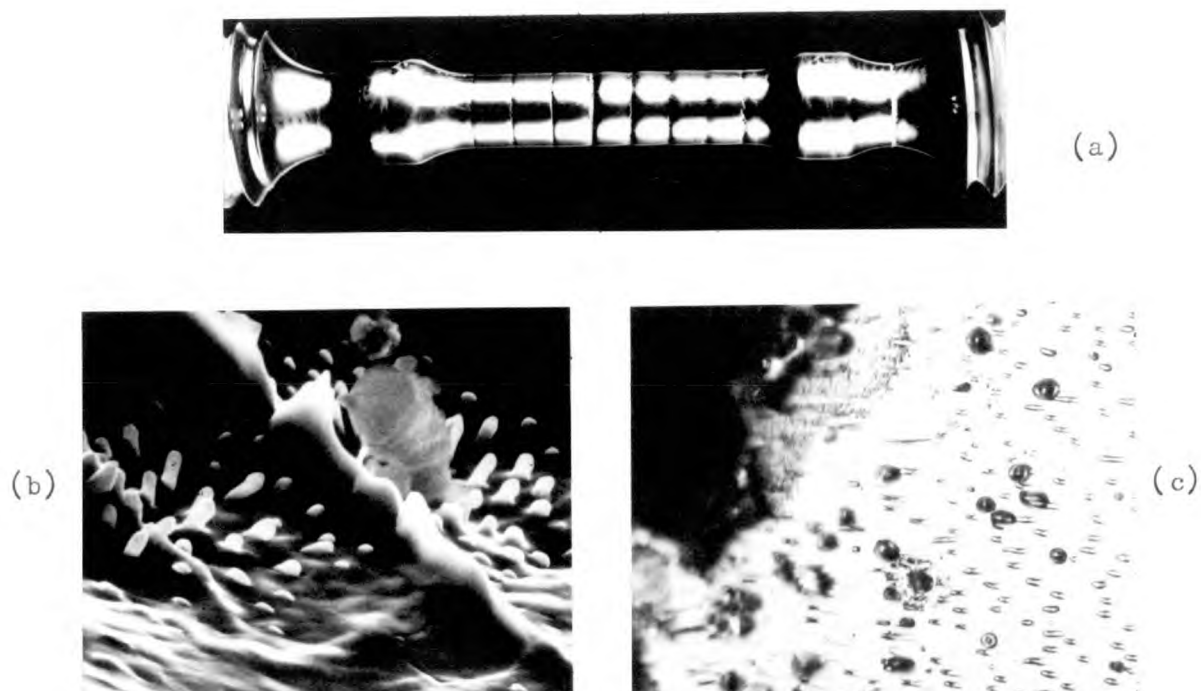


Fig. 2.35 Characteristics of "banded" specimens.

- (a) Concentrated creep deformation within the bands (250°C)
- (b) Whisker ends at rupture surface (450°C) (Magn. 2000 X)
- (c) Cavities adjacent rupture surface (450°C) (Magn. 500 X)



Fig. 2.36 Etch pit rows at unbroken locations along whiskers in creep tested specimen.

CHAPTER III

THEORY OF STEADY-STATE CREEP OF CONTINUOUS FIBRE COMPOSITES

In this chapter the established experimental data of Chapter II are further utilized in conjunction with a creep theory for continuous fibre composites. The nature of transient creep is described as a basis for determining the existence of a creep strain constraint effect.

3.1 Theory of Steady-State Creep

3.1.1 Formulation

Application of a constant load to a continuous fibre composite establishes instantaneous stresses in the fibres and the matrix which can be calculated from the stress-strain characteristics of the constituents by assuming equal strains (Kelly 1966). If the matrix possesses a lower creep resistance than the fibres, differential creep in the phases produces stress redistribution. The fibre stress increases and the matrix stress decreases until a state of equilibrium is reached, namely a steady-state creep rate $\dot{\epsilon}_c$ in both phases. The stress changes which take place during this transient period $t < t_e$ are shown schematically in Fig. 3.1. If the fibres themselves do not creep, $\dot{\epsilon}_c = 0$ at the end of the transient period.

Providing the fibres are unbroken when the steady-state is achieved, maintenance of strain equality in the phases requires a rate equality,

$$\dot{\epsilon}_f = \dot{\epsilon}_m = \dot{\epsilon}_c \quad \dots (3.1)$$

where the rates now involve creep strains only. Subscripts c, f, m refer to composite fibre and matrix respectively. A true steady-state in a composite is possible only for fibres and matrix possessing themselves such a creep characteristic. Utilizing the often found power law dependence between stress and creep rate, we have for both phases at any one temperature,

$$\sigma_f = \sigma_{f0} \left(\frac{\dot{\epsilon}_f}{\dot{\epsilon}_{f0}} \right)^{1/n} \quad \dots (3.2)$$

and

$$\sigma_m = \sigma_{m0} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{m0}} \right)^{1/m} \quad \dots (3.3)$$

where σ_{f0} , $\dot{\epsilon}_{f0}$, n , and σ_{m0} , $\dot{\epsilon}_{m0}$ and m are known constants. The applied composite stress σ_c is partitioned between the phases according to their volume fractions,

$$\sigma_c = \sigma_f V_f + \sigma_m (1 - V_f) \quad \dots (3.4)$$

Combining (3.4) with (3.2)(3.3) and (3.1) we obtain the simple relation

$$\sigma_c = \sigma_{f0} V_f \left(\frac{\dot{\epsilon}_c}{\dot{\epsilon}_{f0}} \right)^{1/n} + \sigma_{m0} (1 - V_f) \left(\frac{\dot{\epsilon}_c}{\dot{\epsilon}_{m0}} \right)^{1/m} \quad \dots (3.5)$$

where V_f is the volume fraction of fibres. In (3.5) σ_c is defined as the creep strength of the composite, that is the stress to produce a specified steady-state creep rate $\dot{\epsilon}_c$. It is implicit in (3.5) that no interaction between phases exists which might modify their behaviour as determined separately. This is the same form of equation formulated

first by McDanel's et al (1967). Its absolute prediction has never been verified although various workers have claimed to verify the 'form' of the equation (McDanel's et al 1967, Pinnel and Lawley 1970). It is apparent that (3.5) is analogous to the tensile strength equation for continuous fibre composites (Kelly 1966). Similarly a creep rupture strength formulation would involve the rupture strength of the fibres and the matrix stress at the time of fibre rupture.

Perhaps due to its apparent simplicity, the nature of (3.5) has not been considered in any detail.

3.1.2 Stress Sensitivity

It is evident from (3.5) that basically the composite does not possess a unique stress sensitivity n_c of creep rate (slope of the curve of log rate vs log stress) although the fibres and matrix do. This is shown in Fig. 3.2 where composite curves ($V_f = 0.1$ and $V_f = 0.5$) have been calculated from the given matrix and fibre curves. The exponent n_c is dependent on both V_f and $\dot{\epsilon}_c$, Fig. 3.3. Any change in either matrix or fibre stress sensitivity at high stresses would produce a similar change in the composite stress exponent.

The effect of neglecting the matrix in creep rate calculations is shown in Fig. 3.2 by the lines $\sigma_c = \sigma_f V_f$ for $V_f = 0.1$ and 0.5 . At low volume fractions, the omission could over-estimate the composite creep rate by a significant amount.

3.1.3 Activation Energy

The temperature dependence of creep rate is generally described

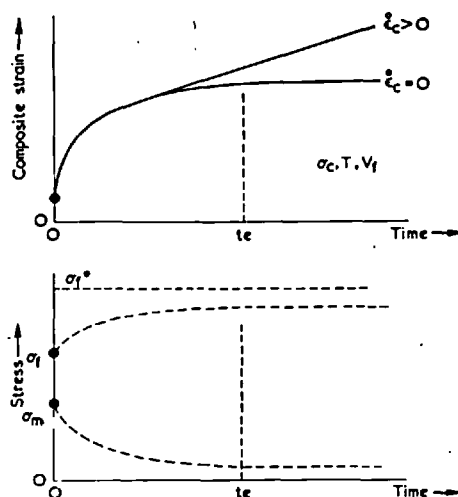


Fig. 3.1 Time dependence of composite strain and constituent stresses

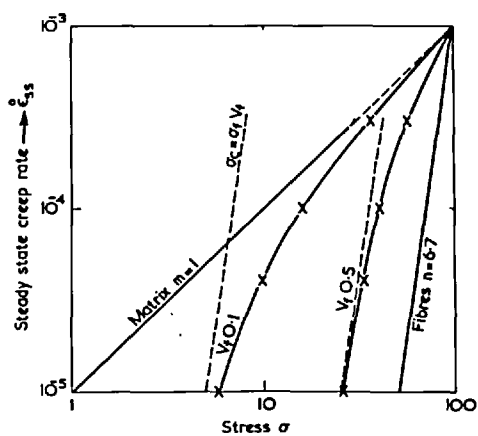


Fig. 3.2 Stress dependence of creep rate for matrix, fibres and calculated composites.

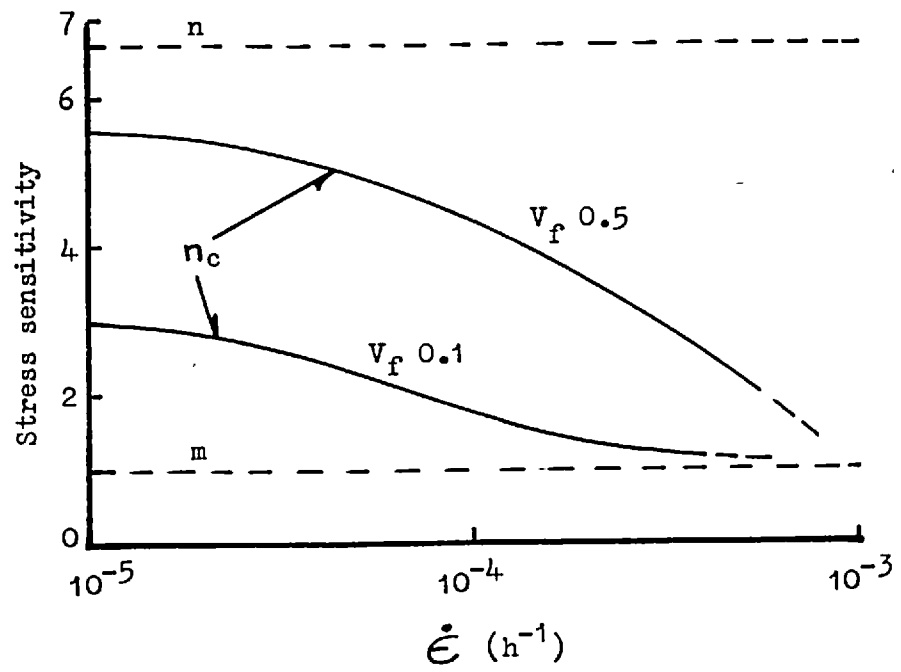


Fig. 3.3 Measured composite stress sensitivity (from Fig. 3.2) vs creep rate.

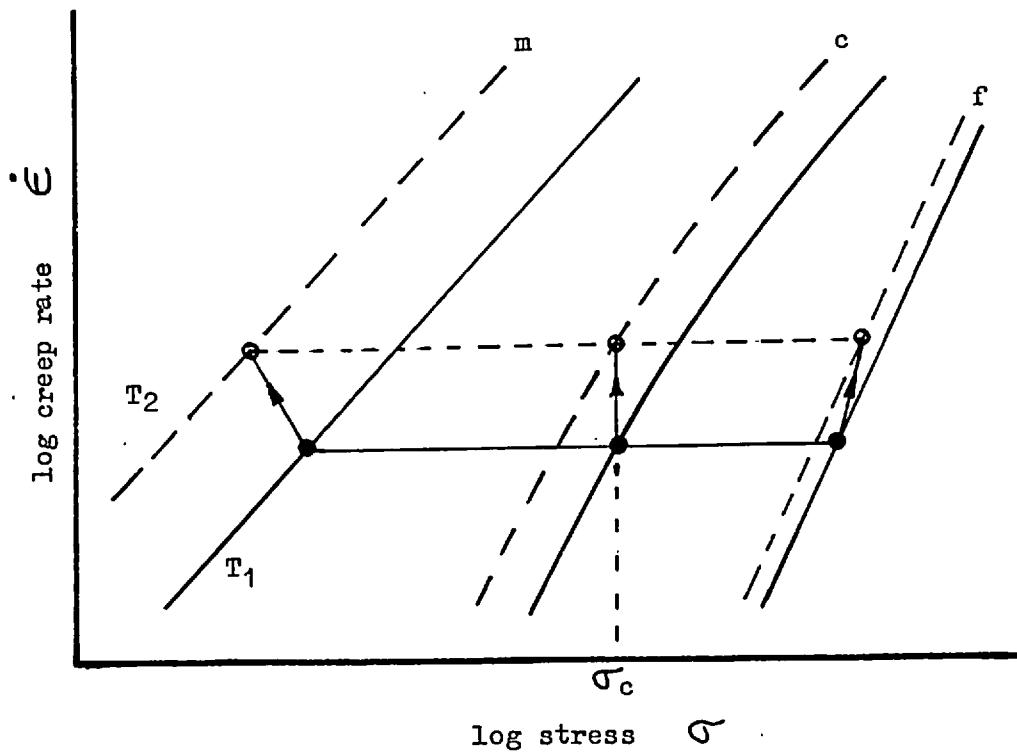


Fig. 3.4 Schematic of temperature dependence of creep rate. $T_2 > T_1$.

by an activation energy Q which is often utilized to determine the rate controlling creep mechanism (Mukherjee et al 1969, Garofalo 1966). To isolate the effect of stress, classical experiments involve temperature cycling under constant stress. It has been assumed that such methods can be applied directly to fibre composites (see Kossowsky 1970, Sheffler et al 1969, Thompson et al 1970).

From the previous considerations, the creep characteristics of the composite involve a balance or equilibrium rate/stress state between the constituents. From (3.5), this balance is a function of the material constants which depend on temperature. It follows that unless the temperature sensitivity of the constants is identical in fibres and matrix, a redistribution of stresses must occur when the temperature is changed even though σ_c is constant. A schematic illustration of this, such as Fig. 3.4, will suffice here but the appropriate equations could be obtained. The activation energy is related to the vertical change in $\dot{\epsilon}$ between the curves for temperatures T_1 and T_2 . Thus, the apparent composite activation energy will, in general, differ from those of the constituents but more important, an interpretation of the former in terms of the latter must account for the stress changes.

3.2 Comparison with Experiment

3.2.1 Al-Al₃Ni

The creep characteristics of Al₃Ni whiskers were not measured in the present programme. Hence (3.5) was utilized to provide a predicted whisker behaviour which could be compared with other available data. The result is shown in Fig. 3.5 for the 400°C data. The matrix curve is from Fig. 2.17.

The composite curve representing the high stress sensitivity regime where grain boundary effects are minimized is marked C1 (maximum creep strength data from Fig. 2.15). The predicted whisker properties from these data are considerably less than the values obtained experimentally at the higher temperature of 510°C by Breinan et al (1968). The discrepancy suggests several possibilities including the one of a lower in-situ whisker creep strength. Initially, it was considered that the opposite would be more likely as extracted whiskers are susceptible to handling defects. A potential in-situ weakening mechanism could be associated with penetration of the semi-coherent interface by matrix dislocations (Cline and Lee 1970, Shaw 1967). The presence of time-dependent weakening is supported by the observation of an inferior creep rupture strength of $\text{Al-Al}_3\text{Ni}$ compared to SAP in contrast to a superior tensile strength (see Figs. 2.1 and 2.3). However, the rupture behaviour may be more closely connected with the local thermal stability of the whiskers.

Although the Al_3Ni data of Breinan are not without considerable uncertainty, it does suggest a low stress sensitivity value of near unity. The composite behaviour displayed a low stress sensitivity regime but the apparent exponent n_c was less than unity. It is not unreasonable to propose that other factors such as grain boundary effects may have distorted the true value of n_c . Thus, an approximated composite behaviour for $n_c = 1$ is shown also in Fig. 3.5 by the dashed line C2. The values of $\dot{\epsilon}_c$ are close to those of the bicrystal data of Fig. 2.19. It is now apparent that a calculated whisker behaviour from C2 is qualitatively in better agreement with the isolated Al_3Ni data.

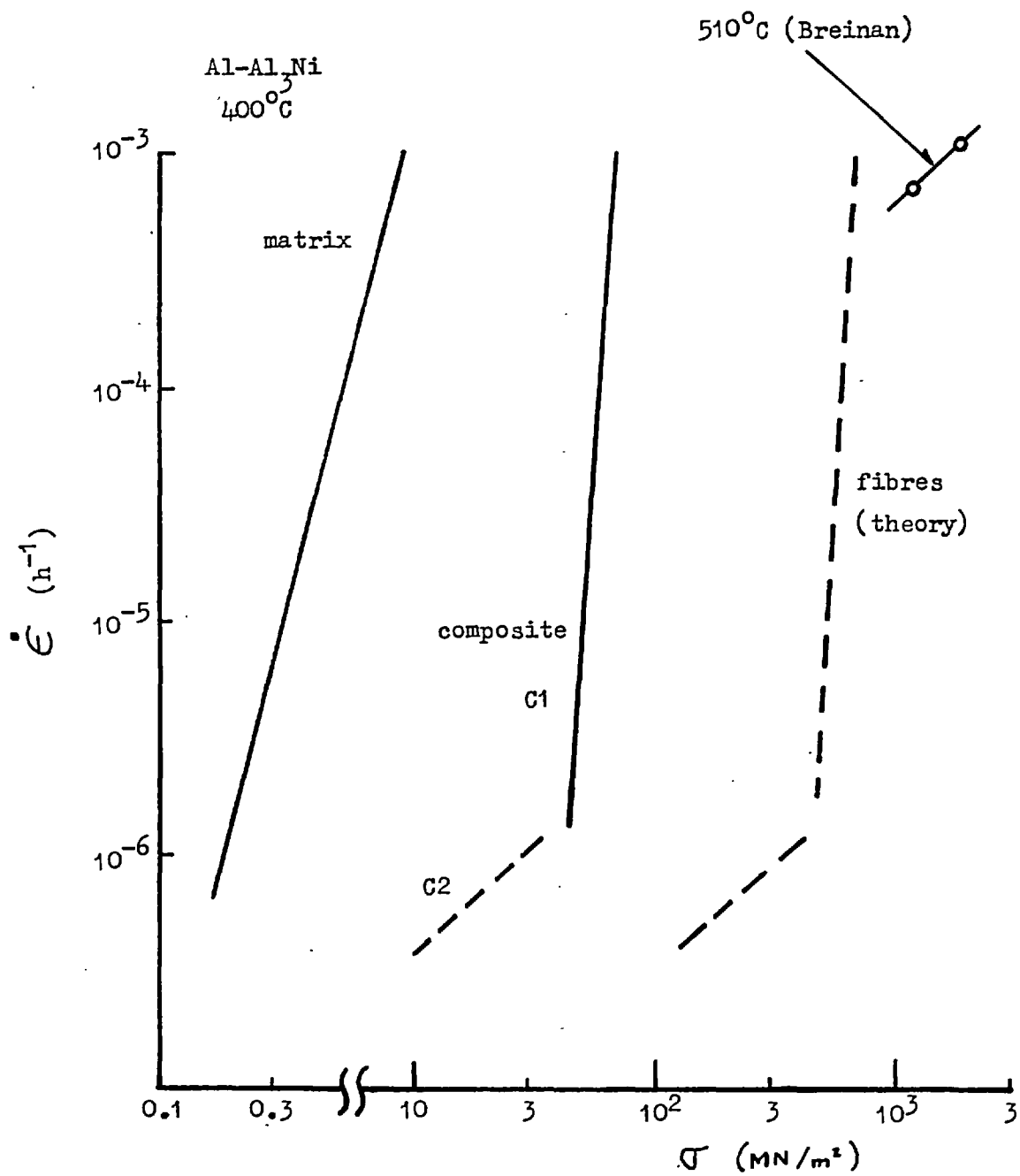


Fig. 3.5 Stress dependence of creep rate for Al, Al-Al₃Ni and predicted Al₃Ni at 400°C.

3.2.2 Cu/W and Inconel 600/W

As a rigorous comparison between theory and experiment was not obtained above, literature data on other composite systems were combined with separate fibre or matrix data in an attempt to verify (3.5).

The only well-documented creep rate data for continuous fibre composites showing an apparent steady-state are those for Cu/W by McDanel's et al (1967). The data for W fibres of Harris and Ellison (1966) are used to calculate theoretical lines for $V_f = 0.10$ and $V_f = 0.74$ as shown in Fig. 3.6. The disagreement between experiment and theory is considerable with the theory predicting lower creep rates.

The behaviour of Inconel 600/W fibre composites at 650°C as measured by Ellison and Harris (1966) is compared with the prediction of (3.5) in Fig. 3.7. Once again, the theory predicts a lower creep rate than experiment. The data for W fibre of the same diameter are those of later work by Harris and Ellison (1966). As the stress exponent of the pure matrix is unknown, a value of 4.5 for pure nickel was assumed (Evans 1968, Mitra 1967).

Although the comparison between theory and experiment is hindered by incomplete data, it is noted that in the two examples the theoretical $\dot{\epsilon}_c$ was substantially less than experiment. Also, η_c was less than expected. These features suggest the possibility that the creep measurements may have included grip shearing or that the observed rates were fibre rupture induced inflections in the creep curves. The experimental difficulties inherent in creep measurements on composites have been discussed by Breinan and Kreider (1970) and by Street (1970).

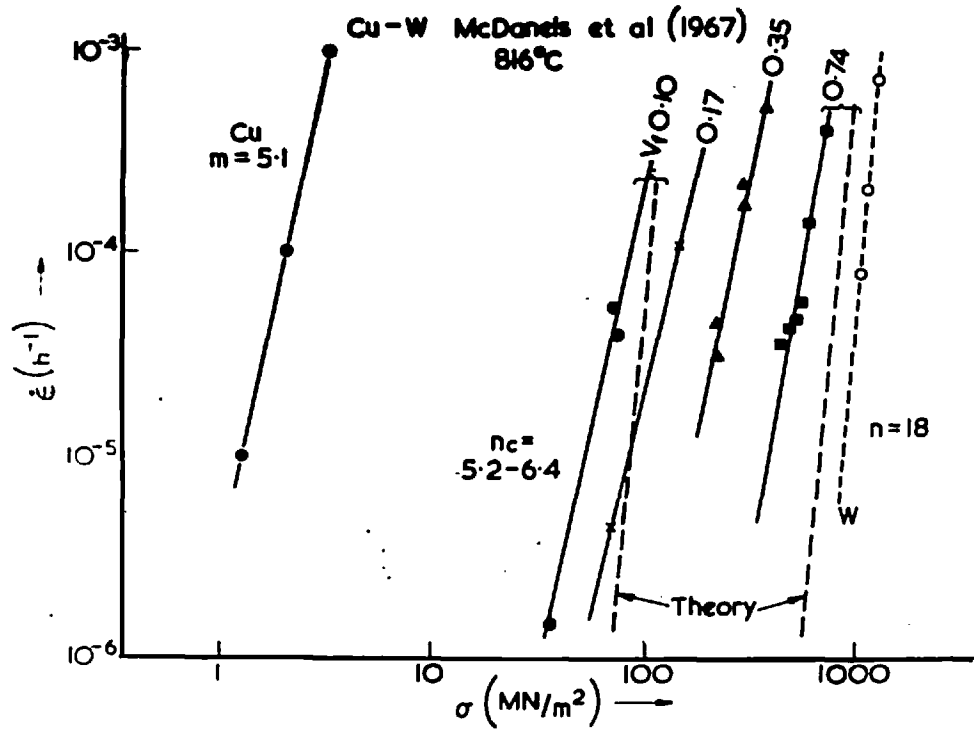


Fig. 3.6 Comparison of creep theory with Cu/W data.

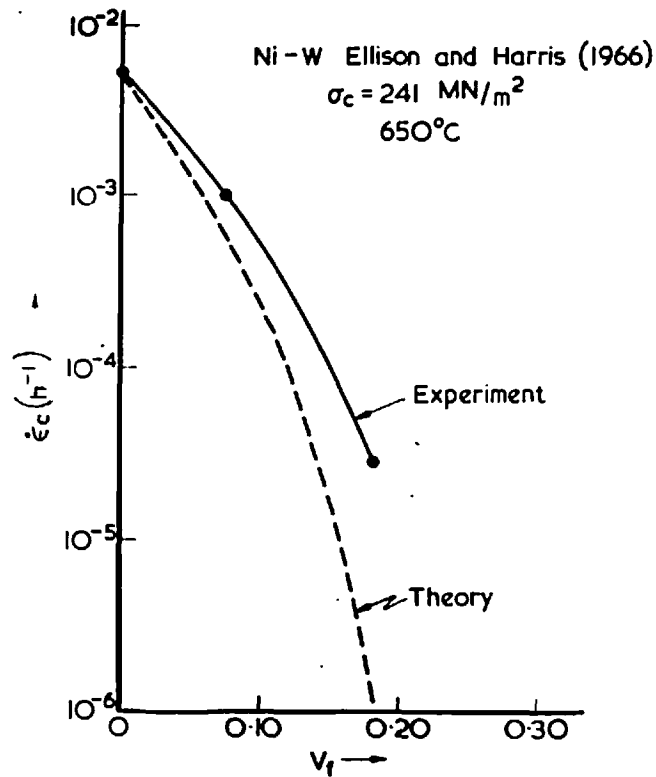


Fig. 3.7 Comparison of creep theory with Ni/W data.

It may be concluded that at this time, the theory is perhaps more help in understanding the experimental findings than the experiments in testing the theory. The need for fibre and matrix data coupled with post-test examination is obvious.

3.3 Creep Strain Constraint

3.3.1 Model Behaviour

The transient effects to be expected in a composite containing a low creep strength matrix have been shown in Fig. 3.1. In the limit when the matrix is extremely weak, we may expect $\sigma_m \rightarrow 0$ after a sufficiently long time and if the fibres do not creep, $\dot{\epsilon}_c \rightarrow 0$. At this stage, assuming strain equality, it follows that $\sigma_c = \sigma_f V_f$ or

$$\epsilon_{c \max} = \frac{\sigma_c}{E_f V_f} \quad \dots (3.6)$$

where $\epsilon_{c \max}$ is the total axial strain experienced by the composite and E_f the elastic modulus of the fibres. This expression is a first order approximation only as it assumes no lateral interaction between the phases during the stress redistribution.

Second order effects have not been considered in composite creep behaviour. However, Hill (1964) derived a more precise prediction of the axial composite stress-strain behaviour taking into account the lateral stresses which originate from differences in Poisson's ratio. When both phases are elastic, the lateral stresses are almost negligible. However, when the matrix becomes plastic, the lateral stresses may exert considerable influence on the axial composite stress. For the case of

interest to the present creep constraint model, an ideal plastic matrix of zero yield strength ($\sigma_m = 0$), the Hill development would transform (3.4) into

$$\sigma_c = \sigma_f V_f + \sigma_i (1 - V_f) \quad \dots (3.7)$$

$$\text{where } \sigma_i = E_i e_c = \frac{V_f (1 - 2 V_f)^2 e_c}{\frac{V_f}{K_m} + \frac{(1 - V_f)}{k_{pf}} + \frac{1}{G_f}} \quad \dots (3.7a)$$

The parameters are defined in Table 3.1.

As shown in a similar analysis by Kelly and Lilholt (1969), (3.7) is the rule-of-mixtures formulation for a fluid reinforced with continuous fibres. The lateral or hydrostatic stresses induce the interaction stress σ_i or interaction modulus $E_i = \sigma_i / e_c$ in the formulation.

Writing (3.7) in the form of (3.6) gives a second order approximation,

$$e'_{cmax} = \frac{\sigma_c}{E_f V_f + E_i (1 - V_f)} \quad \dots (3.8)$$

Consequently, (3.6) and (3.8) are expressions for the maximum total strain in a continuous fibre composite of elastic fibres when the matrix axial stress has relaxed to zero.

3.3.2 Comparison with Experiment

At certain stresses, the creep behaviour of Al-Al₃Ni followed that of the above model, namely $\dot{\epsilon}_c \rightarrow 0$ after a transient period. This was observed for a monocrystal and approximately for a bicrystal at

Table 3.1

Values of Parameters of Equation (3.7a) for 400°C

<u>Symbol</u>	<u>Value</u>	<u>Parameter</u>	<u>Ref</u>
V_f	0.1	Volume fraction	
$\sqrt{v}_f$	0.33	Poisson's ratio (assumed) ($\sqrt{v}_{Cu_3Au}$, 300°C, ~ 0.36)	Simmons (1965)
$\sqrt{v}_m$	0.5	Poisson's ratio	
K_m	65 GN/m ²	Bulk modulus $(\frac{c_{11} + 2c_{12}}{3})$	Simmons (1965)
E_f	118 GN/m ²	Young's modulus (assumed)	
G_f	44 GN/m ²	Shear modulus $(E_f/2(1 + \sqrt{v}_f))$	
k_{pf}	129 GN/m ²	Plane strain bulk modulus $\frac{E}{2(1 + \sqrt{v}_f)(1 - 2\sqrt{v}_f)}$	

400°C as shown earlier in Fig. 2.18. It may be expected then that $\sigma_m \approx 0$ otherwise composite creep would continue to occur. The total strain-time data for the monocrystal is replotted in Fig. 3.8 along with the calculated values of $\epsilon_{c\max}$ from (3.6) and $\epsilon'_{c\max}$ from (3.8) for each stress level. The values of the parameters used in determining $\epsilon'_{c\max}$ are given in Table 3.1. Due to lack of data, the whisker properties were calculated assuming an isotropic material. The considerable uncertainty in the calculations is fully appreciated. However, it is noted that the term $(1/G_f)$ in (3.7a) constitutes about 70% of the denominator in the calculations.

The results show that the measured strains after redistribution (transient creep) are considerably less than the calculated upper limits. This was true also when the singularly known ambient temperature fibre modulus ($G_f \sim \frac{E_f}{3}$) was utilized in the calculation of (3.6). At the higher stress levels, the measured strains surpass the calculated values only when tertiary creep exists.

The findings infer the existence of an unrelaxed matrix stress σ_{mr} which is determined from the relation

$$\sigma_{mr} = \frac{\sigma_c - \epsilon_{ct} E_f V_f}{(1 - V_f)} \quad \dots (3.9)$$

where ϵ_{ct} is the total strain. After redistribution of stress, the contribution supported by the fibres is $\epsilon_{ct} E_f V_f$ and the remainder is assumed to be carried by the matrix. Due to the apparent smallness of σ_i from (3.7a), it has been excluded from (3.9). The calculated values of σ_{mr} shown in Fig. 3.9 reveal a maximum of $\sim 20 \text{ MN/m}^2$.

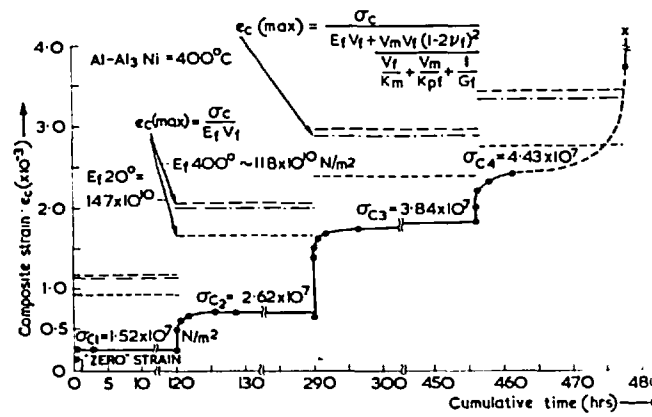


Fig. 3.8 Total composite strain vs cumulative time for Al-Al₃Ni monocrystal at 400°C.

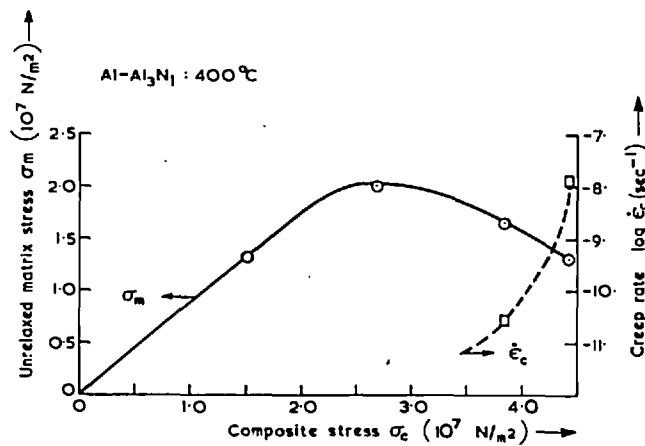


Fig. 3.9 Unrelaxed matrix stress vs composite stress.

This is considerably greater than the creep strength of the unreinforced Al matrix at 400°C, $\sigma_m \sim 0.1 \text{ MN/m}^2$ (Fig. 2.17 for $\dot{\epsilon}_m \sim 10^{-7} \text{ h}^{-1}$).

Thus, although $\dot{\epsilon}_c = \dot{\epsilon}_m = 0$ and hence the effective matrix stress responsible for creep is zero, an axial component of matrix stress has not vanished. This suggests a creep strain or creep strength constraint effect.

The creep data at 350°C of Miura and Honma (1967) for Al/W fibres of diameter 500 μm revealed that the creep rate $\dot{\epsilon}_c \rightarrow 0$ after a transient of about 35 hours. Comparison of the measured and calculated strains according to (3.6) gave the result:

$$\text{Measured: } \epsilon_c = 0.27\%$$

$$\text{Calculated: } \epsilon_{c\text{max}} = 0.11\%$$

The values of the parameters in (3.6) were $E_f = 380 \text{ GN/m}^2$, $V_f = 0.10$, and $\sigma_c = 41 \text{ MN/m}^2$. Certainly a constraint effect does not exist. The origin of the apparent 'overstrain' is not known but the creep strain measurement must be suspect. The average interfibre spacing in this composite is $\sim 1000 \mu\text{m}$ compared to only $1.5 \mu\text{m}$ in the Al-Al₃Ni composite.

3.3.3 Discussion

The Hill type constraint is concerned only with a modified stress system as the intrinsic characteristics of the matrix are assumed to be unmodified. Such a constraint must exist, but it constitutes only a lower limit to a constrained matrix behaviour. It was unable to account for the magnitude of the present findings. A similar conclusion was

arrived at by Kelly and Lilholt (1969) for the tensile strain hardening behaviour of Cu/W composites with average interfibre separations of 6 - 30 μm . The origin of the observed behaviour was not determined. These data were reanalyzed (Appendix IV) and evidence was found which indicated that the basic strain hardening characteristics of the matrix had been enhanced by the small interfibre separations. This type of behaviour may be termed a "modified constraint effect" to distinguish it from the Hill type effect.

The apparent increase in creep strength (or yield strength) of the matrix in $\text{Al-Al}_3\text{Ni}$ could be associated with an Orowan type hardening mechanism due to the whiskers themselves and their small spacing. The Orowan stress to force a dislocation between two rigid particles (see Kelly and Nicholson, 1963) is given to a good approximation as

$$\tau_o = \frac{G_m b}{(\lambda - d)'} \quad \dots (3.10)$$

where G_m = shear modulus, $(\lambda - d)'$ = fibre separation or mean free path in the plane of dislocation motion and b = Burgers vector. Taking $G_m = 19 \text{ GN/m}^2$, $(\lambda - d)' = 1.43 \mu\text{m}$ (Fig. 2.8, $R = 9 \text{ cm/h}$, climb or glide plane at 45° with tensile axis) and $b = 28.2 \text{ nm}$, gives the tensile stress $\sigma_o = 2 \tau_o = 7.5 \text{ MN/m}^2$ at 400°C . The estimated Orowan contribution is about one third of the calculated unrelaxed matrix stress, a moderately high value. It could then account for most of the strengthening if a more precise calculation was possible. The lack of data for the Al_3Ni phase precludes such a calculation based on (3.8).

CHAPTER IV

Pb - PHOSPHOR BRONZE DISCONTINUOUS FIBRE COMPOSITE

4.1 Introduction and Previous Experimental Work

Whereas in the continuous fibre development, steady-state creep was a required condition of both phases if the composite was to creep, intuitively one can see that discontinuous rigid fibres may restrict but need not prevent creep flow of a matrix around them. If some frictional resistance to matrix flow occurs at the fibre/matrix interface through surface roughness or good bonding, then the created shear stress could lead to an indirect load in the fibres. Consequently, the load transfer phenomenon is fundamentally the same as that envisaged in the tensile theory of discontinuous composites (Kelly and Davies 1965).

The only experimental work of any content upon which our ideas can be more formalized is that of Kelly and Tyson (1966). Ag/W composites with fibre aspect ratios of 30 and 60 were observed to creep with a well-defined steady-state regime. Assuming all the load was carried by the fibres, comparison of calculated values of $\sigma_{f \max}$ and literature data for W lead to the conclusion that the fibres themselves were not creeping. The additional findings of the activation energy and the effect of temperature on the creep behaviour being similar to that expected of the pure matrix supported the conclusion that composite creep originated within the matrix phase only. However, an apparent increase in the stress sensitivity of creep rate m could not be explained, partly because of insufficient matrix data.

Regarding composite parameters, Kelly and Tyson (ibid) investigated the effect of l/d for fixed d and V_f . They observed that at a constant stress σ_c , a decrease in l/d increased $\dot{\epsilon}_c$ only if $\dot{\epsilon}_c$ itself was large. At smaller rates (low m region), composite behaviour was independent of l/d . For the l/d used, a few broken fibres were found at the fracture surface of some specimens.

Several other isolated experiments have not enlarged upon the Kelly and Tyson work. Miura and Honma (1967), Ellison and Harris (1966) and Mehan (1970) found that discontinuous fibres are capable of enhancing the creep strength of a pure matrix.

In summary, for the simplest case of rigid fibres, steady-state composite behaviour would appear to be controlled by the time dependent characteristics of the matrix both away from and adjacent to the fibre/matrix interface. The interface has a major role as it must possess the ability to create the required τ for load transfer. The complexities at the ends of fibres and creeping fibres would further complicate the situation.

As for continuous fibre composites, the primary need at this time is considered to be some sound experimental data for a composite system and both of its constituents. Furthermore, an independent examination of the time dependence of interfacial shear strength may be illuminating. These are the aims of this chapter. The experimental data could then constitute a basis for development and verification of a creep theory, the subject of the following chapter.

4.2 Composite Manufacture

To offset some of the experimental complexities of creep testing, pure lead was selected as the matrix as it would allow testing at ambient temperature ($T/T_m = 0.5$). Also, considerable data existed in the literature on the creep and relaxation of lead. The material utilized was Almex lead sheet (BSS 1176, > 99.9%) from Associated Lead Manufacturers Limited.

Moreen (1960) examined the wetting characteristics of molten lead/metal combinations and found that only copper alloys, such as bronze and brass, were adequately wetted by lead. With this information, preliminary experiments of dipping wires into molten lead revealed that phosphor bronze wire was partially wetted at a bath temperature $\sim 550^\circ\text{C}$. With a proprietary fluxing agent, Bakers Soldering Fluid, complete wetting was attained. Similar tests on various steel wires resulted in no wetting. Hence phosphor bronze was selected as the fibre material. The wire, BSS 384 (Sn = 4-7%, P = 0.02-0.40%, impurities < 0.20%, Cu remainder) was obtained from J. Smith and Sons; the diameter was 450 μm .

Composites were prepared by liquid infiltration of the matrix around fibres which had been prepacked into a split-section stainless steel mould with a 'dog-bone' shaped interior. The mould was carefully filled (while horizontal) with chopped wires of constant aspect ratio. This gave an average $V_f \sim 0.40$. After clamping the lid in place, it was raised to the vertical position and tapped lightly to settle the fibres. The lid was removed and the fibres checked visually for regions of low volume fraction (due to jamming on settling). After

reclamping and inserting a plug of matrix, the mould was placed in an inert atmosphere (A-5% H_2) container and the assembly was lowered at 38 cm/h through a preheated resistance furnace, Fig. 4.1. A maximum temperature of 550-600°C was achieved.

Pure matrix specimens produced in the same manner were extremely difficult to handle without introducing some deformation. Consequently they were annealed in air at either 100°C for 24 hours or 190°C for 6 hours, immediately before creep testing. Lengths of phosphor bronze wire were subjected to the same manufacturing (annealing) cycle as the composite specimens, without the addition of lead. This was done to provide single fibres with basic properties similar to those contained within the creep specimens.

Some composites contained chopped wires which were precoated with the lead matrix. Coating was accomplished by drawing a continuous wire through a proprietary flux (Bakers Soldering Fluid) and then into a lead bath held at 475° - 500°C. A rapid drawing rate of 11-15 m/min was found to produce a good coating.

4.3 Mechanical Testing

The type of composite specimens utilized in the creep experiments are shown in Fig. 4.2. No machining was required. The as-cast dimensions were 1.59 x 3.18 mm in cross-section by ~ 270 mm parallel portion gauge length. The major advantage in using specimens of such large aspect ratio is that any non-axial loading or grip effects are minimized. Additional features introduced to overcome these problems were: universal joint alignment and serrated face friction grips with relatively large surface areas of gripping. Careful post test examination of the gripped regions did not reveal any shearing.

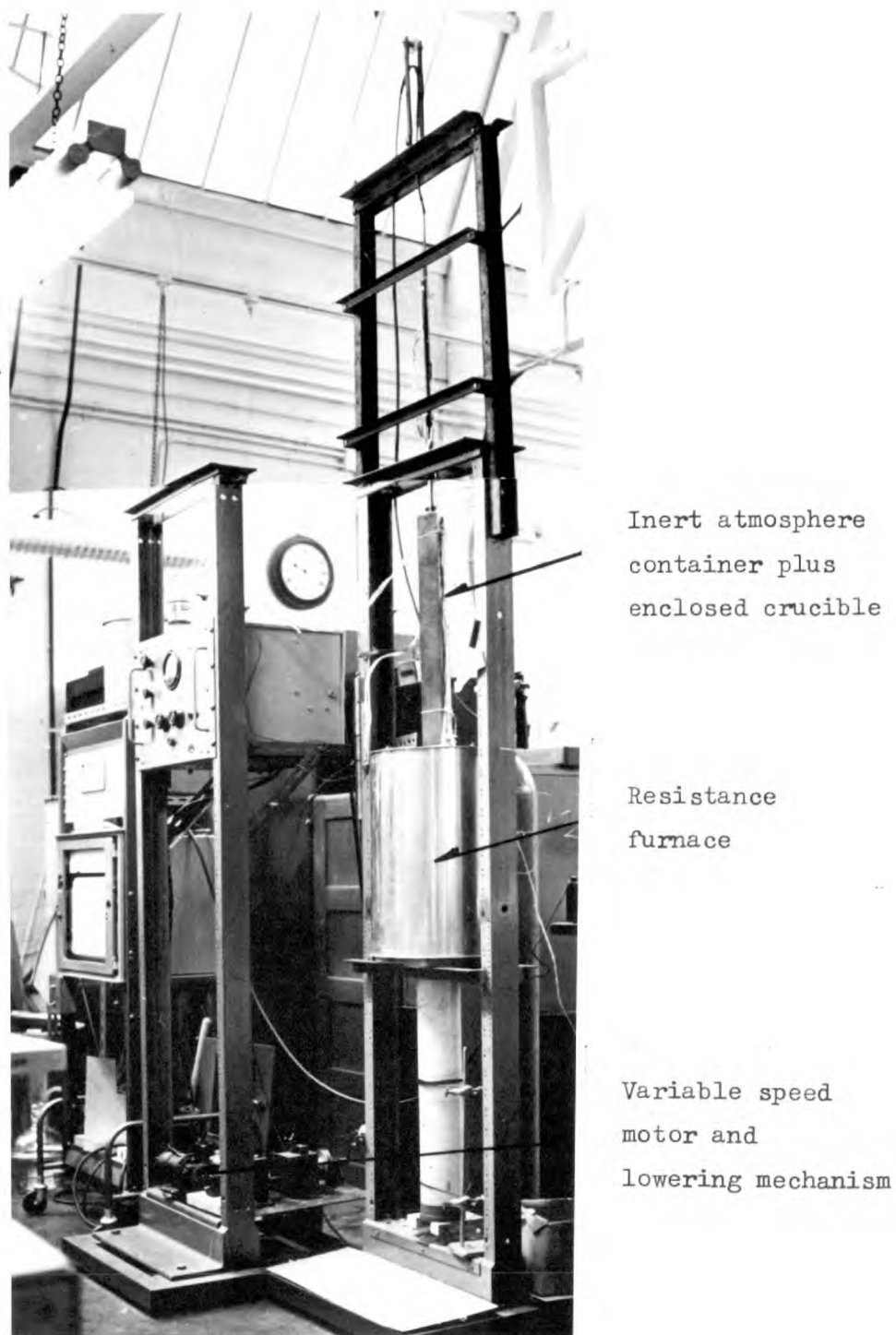


Fig. 4.1 Dynamic liquid infiltration apparatus for preparation of Pb/phosphor bronze composites.

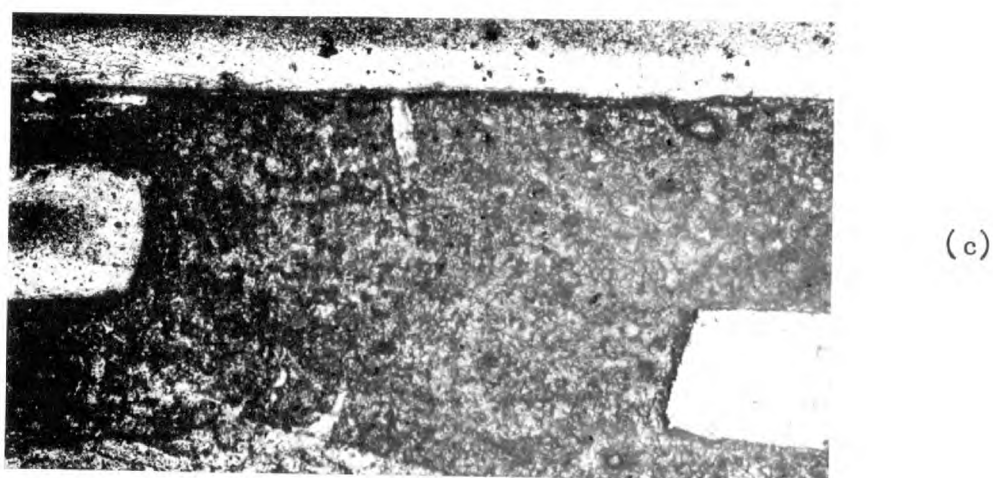
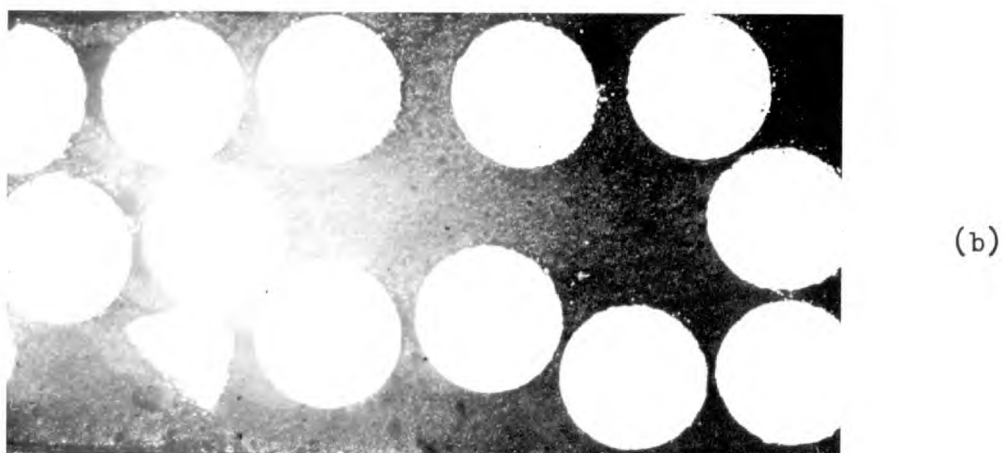
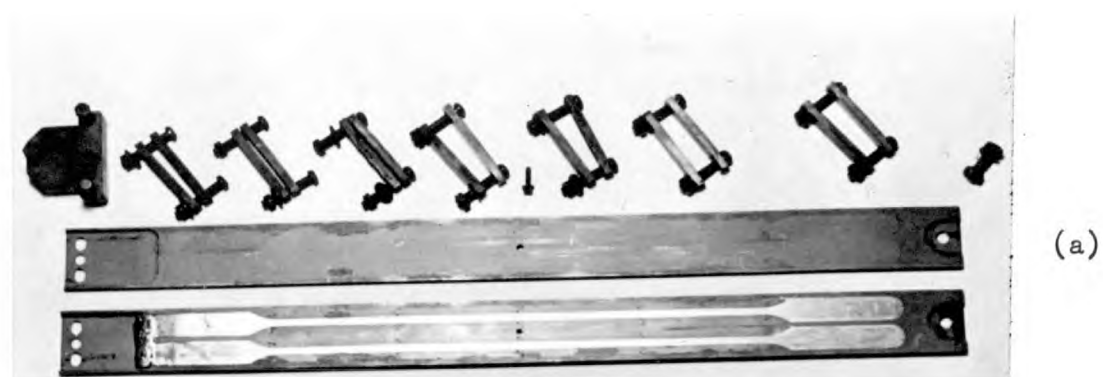


Fig. 4.2 Pb/phosphor bronze wire composite specimens
(a) as-cast creep specimens (Magn. $\frac{1}{4}$ X)
(b) transverse section (Magn. 40 X)
(c) longitudinal section (Magn. 40 X)

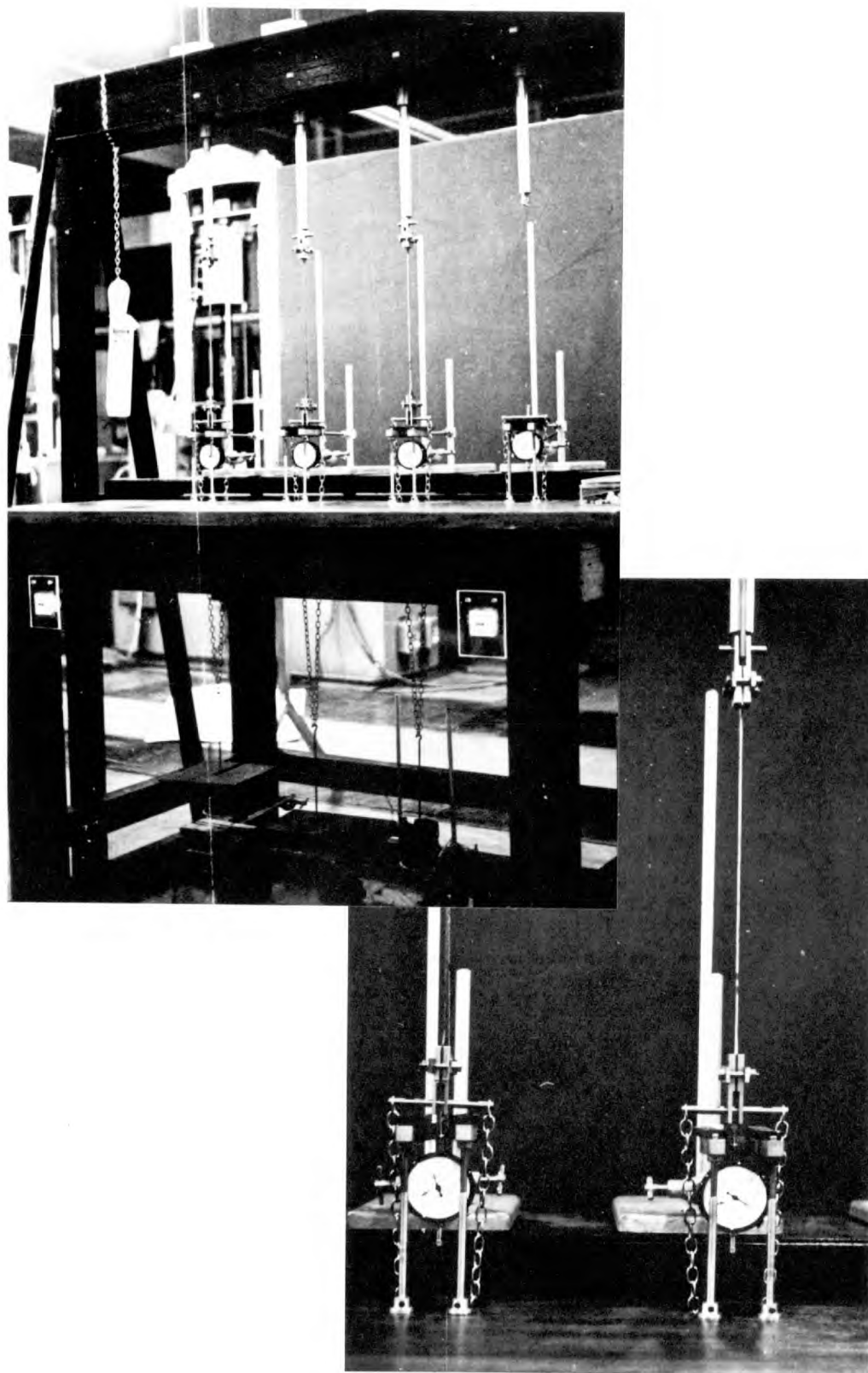


Fig. 4.3 Direct loading creep apparatus for pure matrix and single fibre tests.

Due to the small loads required, a direct loading creep apparatus was constructed to determine the behaviour of the pure matrix and the free fibres. Creep extension was measured by a rigidly mounted dial gauge which contacted the lower grip of the specimen. Four specimens could be tested at one time. The general nature of the apparatus is apparent from Fig. 4.3.

Composite specimens were tested in standard Dennison 10:1 ratio lever arm machines. Extensions were measured by dial gauges mounted at the 5:1 magnification location on the lever arm. All tests (uniaxial tension) were carried out under constant load in the temperature controlled creep laboratory (NPL) which was maintained at $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

4.4 Results and Discussions

4.4.1 Creep Behaviour

Strain-Time Plots

Collective creep strain-time plots for single fibres, pure matrix, discontinuous and continuous fibre composites are shown in Figs. 4.4, 4.5, 4.6 and 4.7 respectively. Appropriate data extracted from more sensitive plots are given in Tables 4.1, 4.2, 4.3 and 4.4.

The single fibres displayed an approximate logarithmic creep behaviour which can be described by the relation

$$\epsilon_f = \epsilon_{f_0} + \alpha \log t \quad \dots (4.1)$$

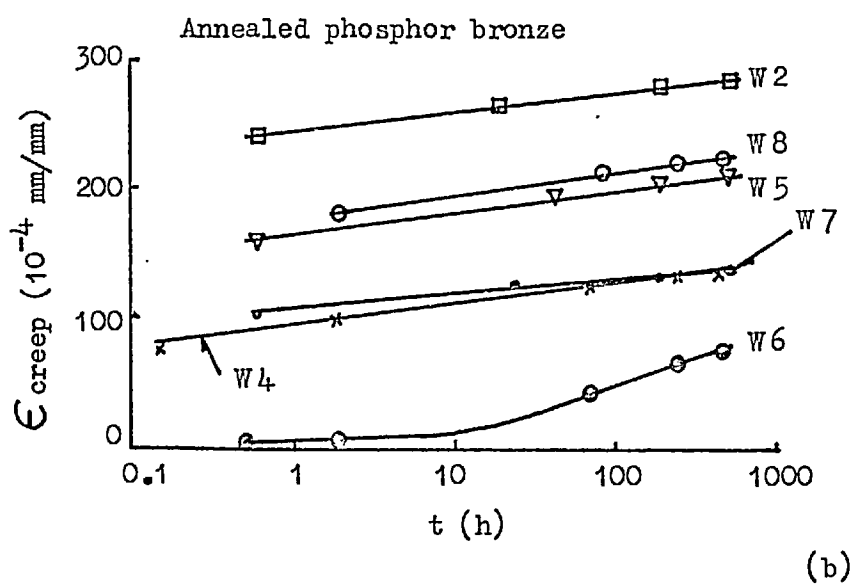
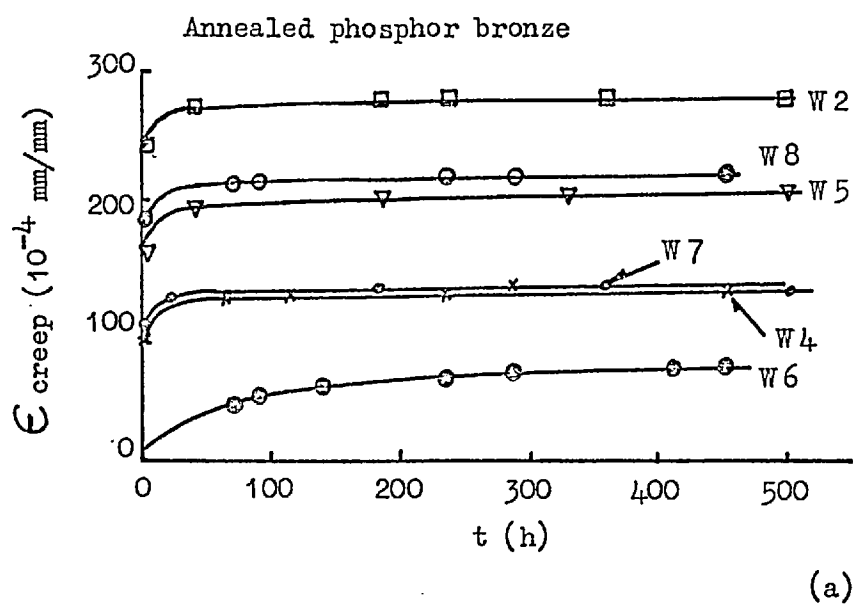


Fig. 4.4 Creep behaviour of annealed phosphor bronze wires at 20°C ($d = 450 \mu\text{m}$). (a) creep strain vs time (b) creep strain vs log time.

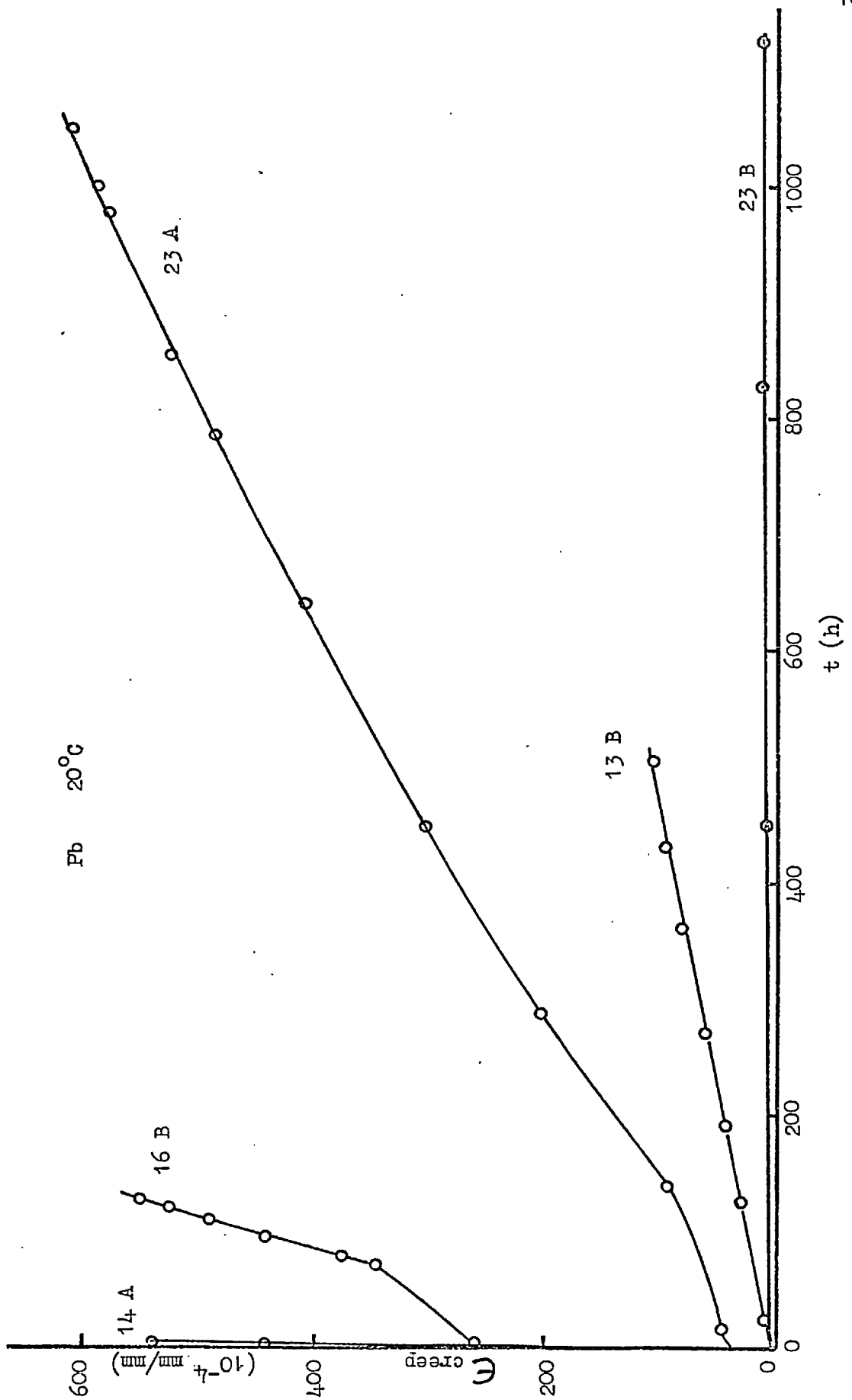
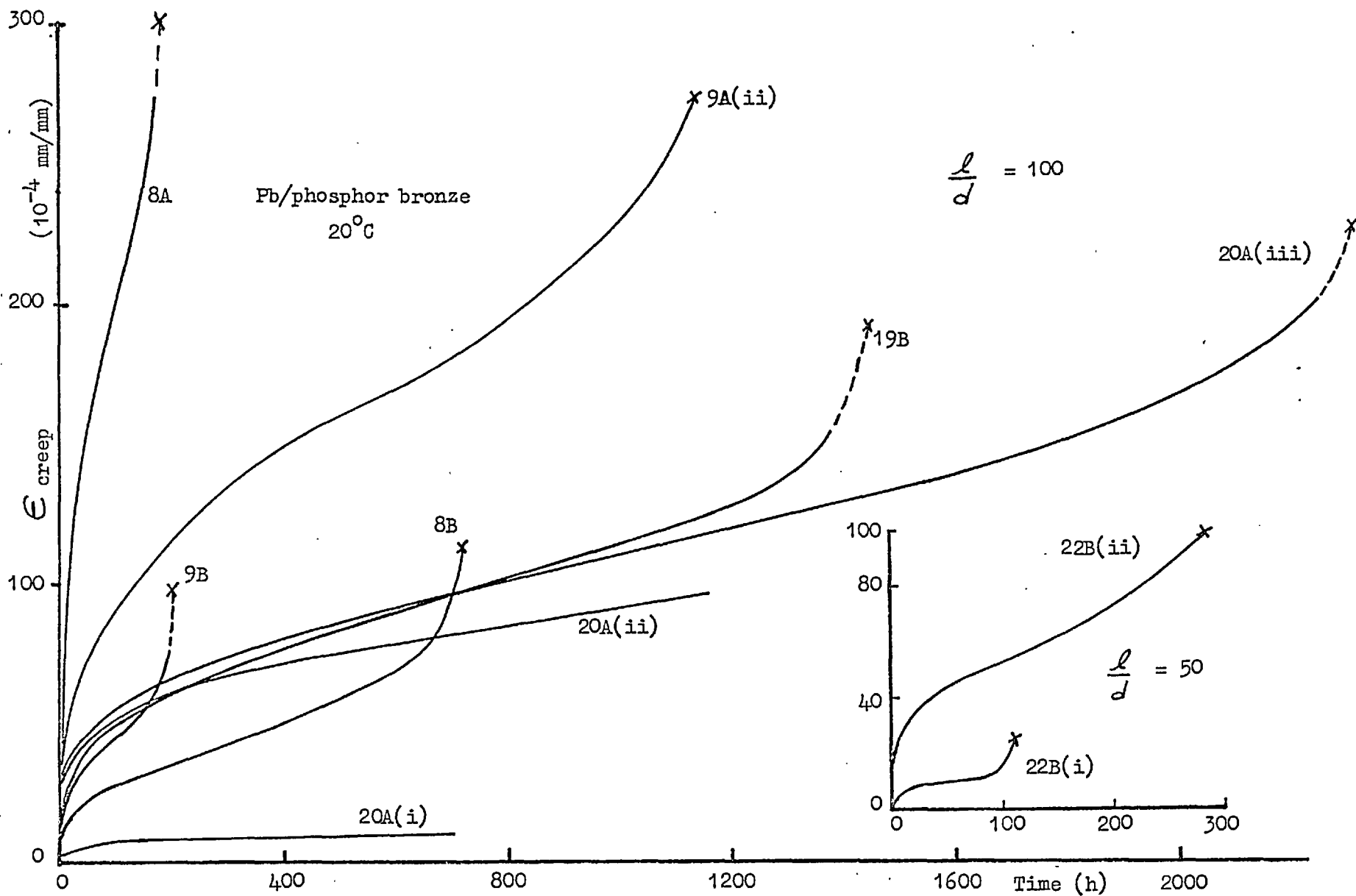
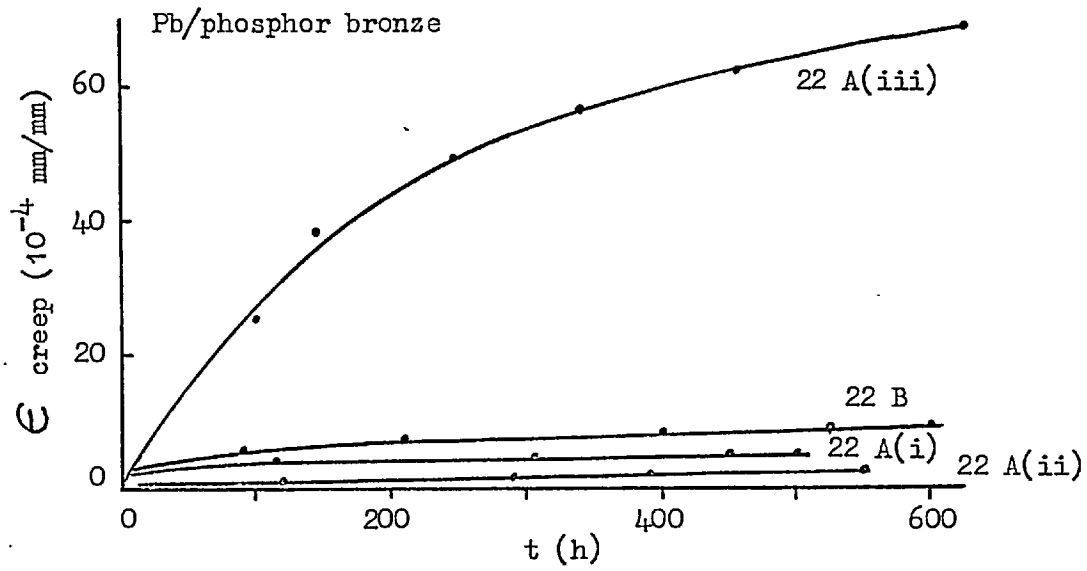


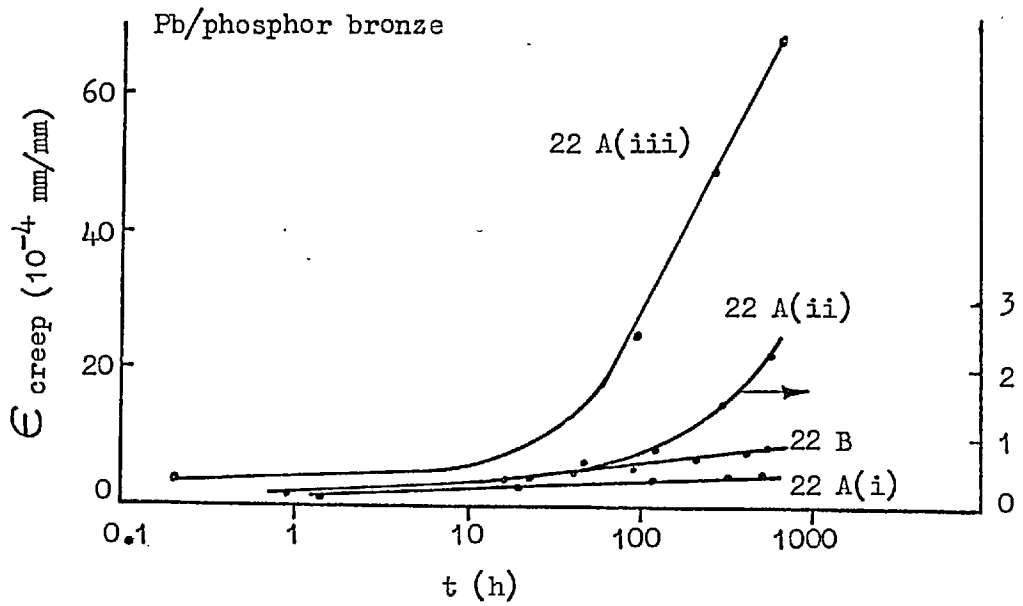
Fig. 4.5 Creep strain vs time for Pb.

Fig. 4.6 Creep strain vs time for discontinuous fibre composites.





(a)



(b)

Fig. 4.7 Creep behaviour of continuous fibre composites at 20°C ($d = 450\mu\text{m}$)
 $V_f = 0.425$. (a) creep strain vs time (b) creep strain vs log time.

Table 4.1Phosphor Bronze Wire Creep Data

<u>Spec. No.</u>	<u>σ_f (MN/m²)</u>	<u>α (10⁻⁴)</u>	<u>ϵ_{f_0} (10⁻⁴)</u>	<u>$\dot{\epsilon}_{\min}$ (400-500 h) (10⁻⁷ h⁻¹)</u>
W4	183	11	107	4.5
W7	170	15	97	2.2
W8	157	17	180	4.0
W5	157	13	166	10.0
W6	142	-	-	34.0
W2	142	14	245	5.0

Table 4.2Pure Lead Matrix Creep Data

<u>Spec. No.</u>	<u>σ_m (MN/m²)</u>	<u>$\dot{\epsilon}_{\min}$ (h⁻¹)</u>	<u>Specimen History</u>
16 B	4.20	3.5×10^{-4}	Ann. 100°C/24 h
23 A	3.35	4.5×10^{-5} (‡)	Ann. 190°C/6 h
13 B	4.20	1.8×10^{-5}	As cast
23 B	2.45	8.6×10^{-7}	Ann. 190°C/6 h
14 A	5.10	2.2×10^{-3}	As cast

(‡) apparently presteady-state

Table 4.3

Pb/Phosphor Bronze Discontinuous Fibre Composite

Creep Data ($d = 450 \mu\text{m}$, $V_f \sim 0.40$)

Spec.	Stress σ_c (MN/m ²)	Min. creep rate $\dot{\epsilon}_c$ (h ⁻¹)	Rupture time t_r (h)	Rupture strain ϵ_r (10 ⁻⁴)	No. fibre fractures n_f	Wire Precoated
<u>$\frac{l}{d} = 100$</u>						
8 A	53.5	8.5×10^{-5}	174	260	-	Yes
8 B	44.3	7.8×10^{-6}	730	80	3	Yes
9 A	57.5	1.0×10^{-6}	1150	250	3	Yes
9 B	44.3	1.5×10^{-5}	175	56	4	Yes
19 B	44.3	5.8×10^{-6}	1440	145	2	No
20 A(i)	44.3	3.2×10^{-7}	700+	9	-	No
(ii)	53.5	3.2×10^{-6}	1150+	95	-	No
(iii)	57.5	4.2×10^{-6}	2370	170	3	No
<u>$\frac{l}{d} = 50$</u>						
21 B(i)	44.3	$\sim 9 \times 10^{-6}$	110	~ 14	2	No
(ii)	53.5	1.9×10^{-5}	~ 300	90	-	No

Table 4.4 Pb/Phosphor Bronze Continuous Fibre
Composite Creep Data ($d = 450 \mu\text{m}$, $V_f = 0.425$)

<u>Spec.</u>	<u>Stress</u> <u>σ_c (MN/m²)</u>	<u>Min. creep rate</u> <u>at time</u>		<u>Max. strain</u> <u>ϵ (10^{-4})</u>
		<u>$\dot{\epsilon}_c$ (h⁻¹)</u>	<u>t (h)</u>	
22 A(i)	44.3	1.4×10^{-7}	500	4.7
(ii)	53.5	2.5×10^{-7}	550	2.2
(iii)	61.5	4.1×10^{-6}	625	68.0
22 B	53.5	1.7×10^{-7}	600	8.7

where ϵ_f = creep strain, and ϵ_{f_0} and α are constants as given in Table 4.1. The logarithmic behaviour is not strictly followed as the measured average creep rates when the tests were terminated ($t = 400 - 500$ h) are slightly less than those calculated from the above relation. Logarithmic creep is in accordance with expected creep behaviour at low homologous temperatures ($T/T_m = 0.22$) and moderate stresses (McLean 1962).

The continuous composites showed a similar behaviour but were less well characterized due to the effect of strain-hardening in the incremental load tests. A logarithmic characteristic in both fibres and composite was found also by Wilcox and Clauer (1969) for stainless steel/Mg-Li alloy.

Steady-State

A double logarithmic plot of steady-state creep rate $\dot{\epsilon}$ (or minimum value) against applied tensile stress σ for the pure matrix and the discontinuous fibre composites is shown in Fig. 4.8. The results of Betty (1935) and McKeown (1937) for nominally pure lead are in reasonable agreement with the present data. Regarding the matrix stress sensitivity m , the present findings are in good agreement with the slope of Weertman's (1960) temperature compensated creep rate data. These data were arbitrarily placed along the ordinate to compare slopes only. The main source of variation between the results is likely purity and grain-size effects.

Over the limited ranges of $\dot{\epsilon}$ and σ , the pure matrix data can be described by a power law of the form

$$\dot{\epsilon}_m = \dot{\epsilon}_{m_0} \left(\frac{\sigma_m}{\sigma_{m_0}} \right)^m \quad \dots (4.2)$$

where the constants are $\sigma_{m0} = 3.35 \text{ MN/m}^2$, $\dot{\epsilon}_{m0} = 10^{-5} \text{ h}^{-1}$ and $m \approx 14$. The power law is utilized in lieu of an exponential law for convenience only.

The behaviour of the discontinuous fibre composites could also be approximated by a power law relation although considerable scatter prevents any precise characterization. The stress sensitivity appears to be distinctly larger than that of the pure matrix. As shown in the next section, the stronger specimens (minimum creep rate at any one stress) are considered to be more representative of average composite behaviour. This would explain the fact that the two $\ell/d = 50$ data points fall within the scatter band of $\ell/d = 100$ specimens.

The maximum average stress on the discontinuous fibres can be estimated by assuming that the composite stress is supported by the fibres alone, that is

$$\bar{\sigma}_{f \max} = \sigma_c / V_f = 110. - 150. \text{ MN/m}^2$$

The maximum stress $\sigma_{f \max}$ would be even larger. Comparison with the stresses utilized in the single fibre tests (145 - 185 MN/m^2) and in the continuous fibre composites,

$$\sigma_{f \max} = \sigma_c / V_f = 105. - 150. \text{ MN/m}^2$$

leads to the conclusion that the discontinuous fibres must have experienced creep. Further evidence for this appears later.

As shown by the data of Table 4.3, the effect of precoating the fibres with the Pb matrix had no apparent affect on the creep behaviour.

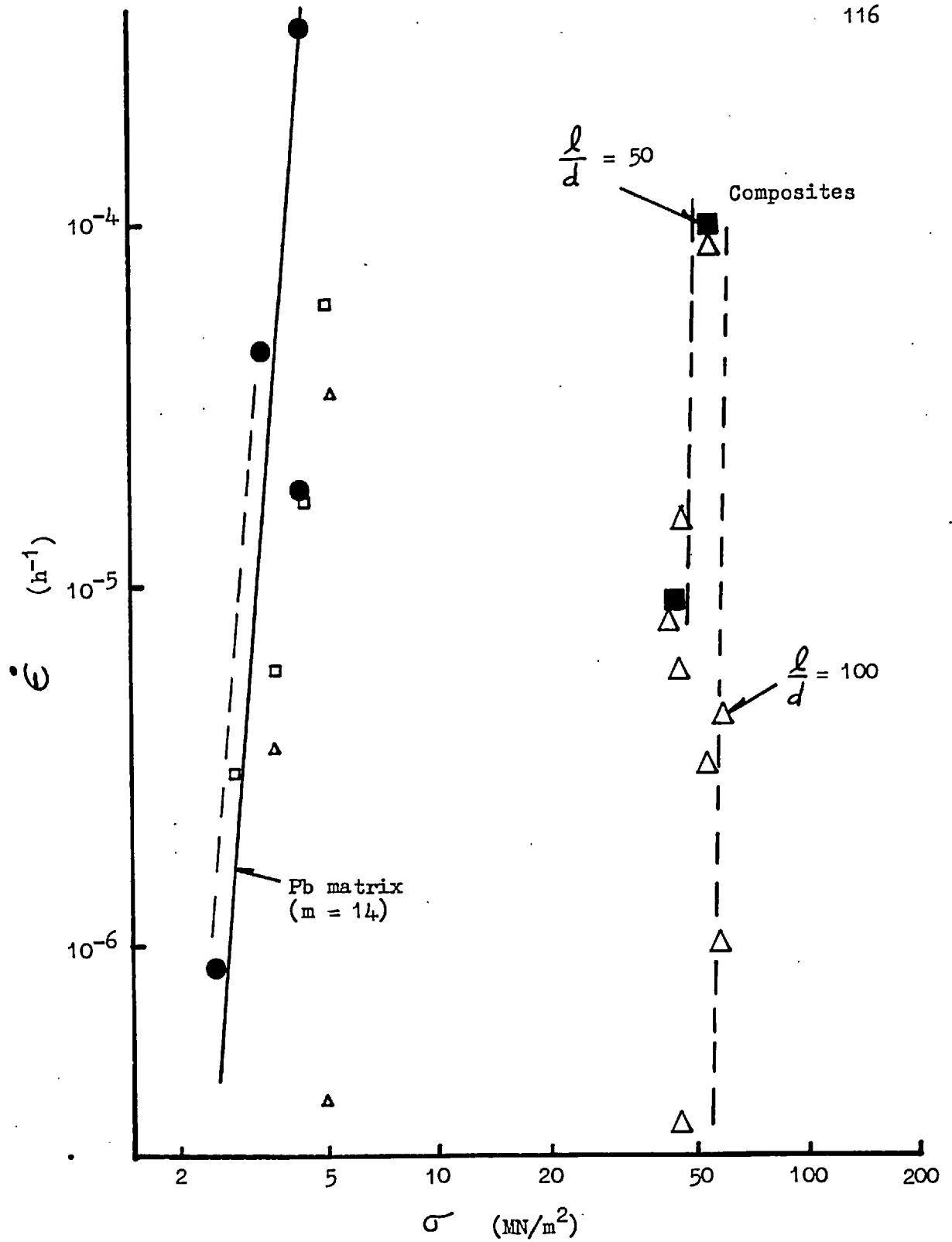


Fig. 4.8 Stress dependence of steady-state creep rate for pure Pb and Pb/phosphor bronze composites. Additional Pb matrix data are after Betty (1935)(Δ), McKeown (1937)($\square$) and Weertman (1960)(dashed line).

The incorporation of fibres of $\ell/d = 100$ has produced a strengthening $\sigma_c/\sigma_m \approx 17$. This compares to $\sigma_c/\sigma_m = 7-9$ for Ag/W composites, $\ell/d = 30$ (Kelly and Tyson 1966). In the latter work, it was concluded the fibres themselves were not creeping.

Surface Characteristics and V_f Distribution

After creep testing, the external surface of some discontinuous fibre specimens exhibited numerous dimples or cracks associated with the ends of the fibres along the gauge length. In other specimens, very few of these effects were present. Based on these observations, a simple criterion was invoked in an attempt to reduce the scatter in the measured creep strengths. A specimen exhibiting many dimples over its entire gauge length must have experienced relatively uniform creep deformation and hence may be expected to possess a relatively good creep strength. Likewise, few fibre end effects would infer that the specimen must have contained a relatively weak section where the deformation was concentrated. For the same stress, the creep strength likely would be reduced.

The surface characteristics of specimens after test are described in Table 4.5. Using the above criterion, it is seen that the correlation between the frequency of dimples, the predicted relative creep strengths and the measured strengths is good. It would appear then that the main source of scatter in the results of Fig. 4.8 is the variable volume fraction of fibres. The stronger composite data points appear to be more valid.

As a further check on the above, the distribution in V_f was measured along the length of a discontinuous fibre ($\ell/d = 100$) composite

specimen by counting fibres in sections taken at equally spaced intervals. The results, Fig. 4.9, show conclusively that a wide range of V_f was possible in the present composites.

4.4.2 Creep Rupture

Rupture Strain and Time

Composite rupture strain ϵ_r as a function of rupture time t_r for the discontinuous fibre composites is shown in Fig. 4.10. No obvious trends exist, likely because rupture is controlled by the minimum V_f cross-section in the composite. The two $l/d = 50$ specimens exhibited ϵ_r values less than those of the $l/d = 100$ specimens for constant σ_c . The maximum plastic strain found for the discontinuous fibre composites was $\sim 2.5\%$.

The rupture characteristics of the pure matrix and single fibres were not determined due to the large strains which would have resulted. Valid relationships would only follow from constant stress tests.

Rupture Mode

Discontinuous fibre composite rupture was characterized by extensive matrix shear between fibres over large distances adjacent to the fracture surface, Fig. 4.11. Consequently, fractures were extremely fibrous in appearance, Fig. 4.12(a). The few fractured fibres which were found at the rupture surface, (see Table 4.3) exhibited ductile cup-cone fractures, Fig. 4.12(b). Counts of pulled out fibres were not made as they were not clearly defined.

Table 4.5 Post-test Creep Specimen Surface Characteristics

<u>Spec.</u>	<u>No. dimples/cracks on surface</u>	<u>Apparent Deformation</u>	<u>Exp. Rel. Creep Strength</u>
19 B	few	nonuniform	weak
20 A	many	uniform	strong
9 B	-	-	weak
8 B	few	nonuniform	weak
9 A	many	uniform	strong
8 A	moderate	~ uniform	weak

Table 4.6 Characteristics of Residual Broken Fibres After
Matrix Dissolution

<u>Spec. No.</u>	<u>Total no. breaks in each segment</u>	<u>ℓ/d of broken fibres</u>	<u>(ℓ/d) min</u>
9 A	3	44, 79, 57 34, 29.5, 57	29.5
20 A	3 (larger segment only)	70, 81, 43	(100-81) = 19

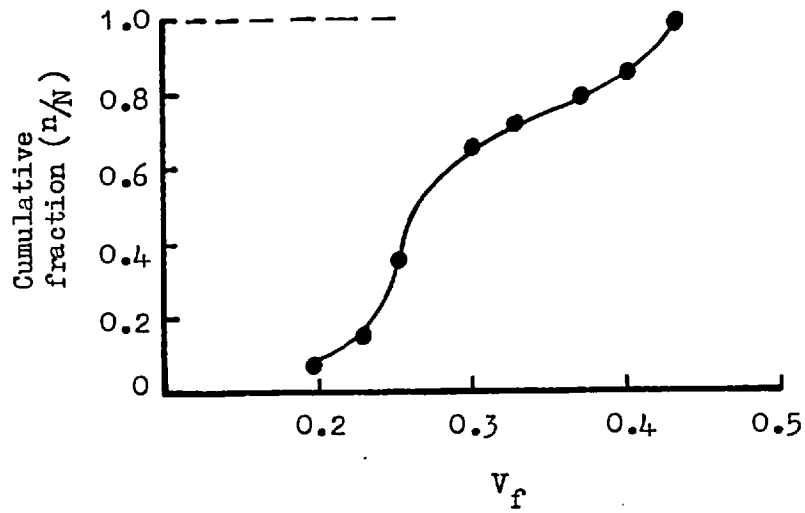


Fig. 4.9 Longitudinal distribution of volume fraction in $\ell/d = 100$ composite specimen.

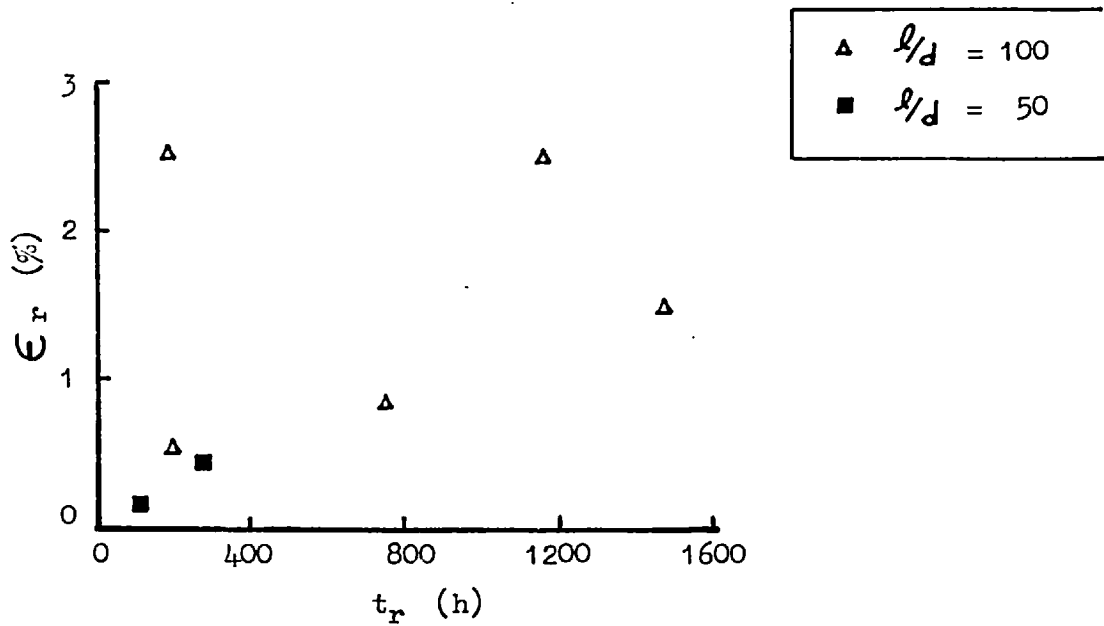


Fig. 4.10 Rupture strain vs rupture time for discontinuous fibre composites.

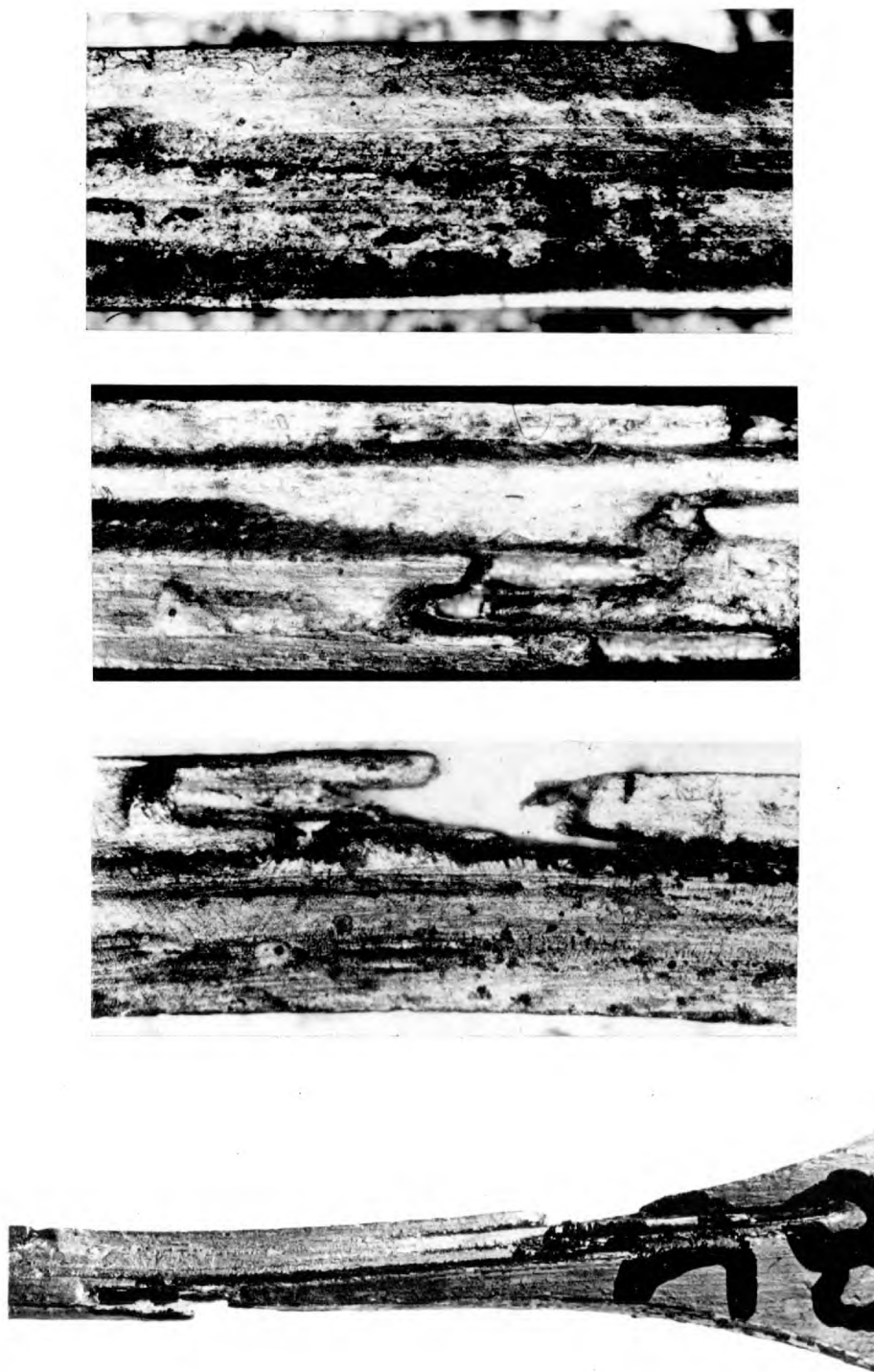
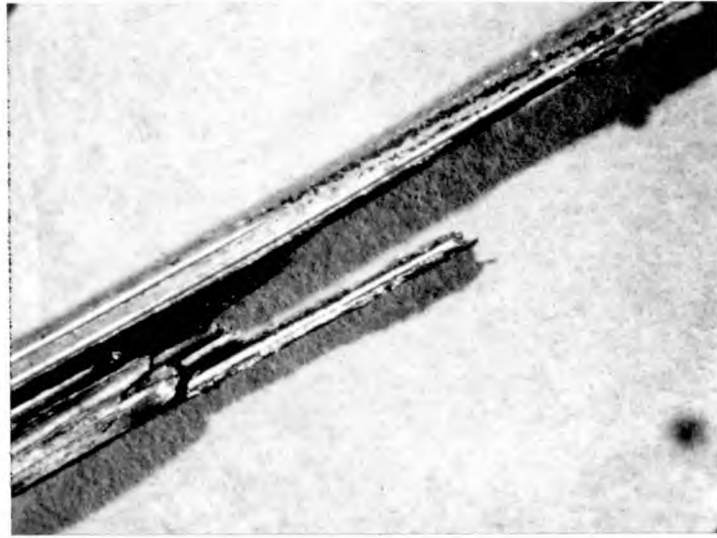


Fig. 4.11 Various stages of composite rupture, $l/d = 100$.



(a)



(b)

Fig. 4.12 (a) Fibrous rupture, $l/d = 100$
(b) Cup-cone ductile failures of fibres removed from composite rupture surface.

Table 4.7 Uncorrected Tensile Data (20°C)

<u>Spec.</u>	<u>Engineering yield stress σ_y (MN/m²)</u>	<u>Maximum load (N)</u>	<u>Nominal strain-rate (h⁻¹)</u>	<u>Uniform strain (%)</u>	<u>No. of breaks</u>
<u>Ann. phosphor bronze</u>					
W 9	193.	58.	$\sim 2 \times 10^{-2}$	34.	-
W 10	120.	58.	$\sim 8 \times 10^{-2}$	34.	-
<u>Composite $l/d = 100$</u>					
9 AT	100.	157.	5.3	16.	6
6 AT	30.5	82.	1.2×10^{-1}	11.	6
6 BT	28.5	53.	1.2×10^{-2}	2.3	3

Frequency of Broken Fibres

To ascertain whether fibres had been fractured at locations remote from the rupture surface, the matrix was dissolved from two specimens using a solution of 50% hydrogen peroxide (30 vols) in glacial acetic acid. The characteristics of the broken segments are given in Table 4.6. It was found that the total number of breaks coincided with the number observed at the rupture surface itself. Hence, fibres were not broken elsewhere in the composites. Both Kelly and Tyson (1966) and Mehan (1970) reported fractured fibres at the rupture surface but no assessments were carried out at remote locations.

4.4.3 Tensile Behaviour

The ambient temperature tensile characteristics of both annealed phosphor bronze wires and discontinuous fibre composites are given in Table 4.7. The fracture strain of the single fibres was considerably larger than that of the discontinuous fibre composites (both tensile and creep tests). One other distinct feature was that the number of broken fibres at the fracture surface was slightly greater in the tensile tests than in the creep tests.

4.4.4 Single Fibre Pullout Tests

Due to their simplicity, single fibre pullout experiments have been utilized to provide an estimate of the critical fibre aspect ratio l_c/d and the interfacial shear stress τ (Kelly and Tyson 1965a, Jech and Signorelli 1967). However, it must be stated that the precise relation between these two parameters determined by this test and those applicable to discontinuous fibre composite behaviour has not been firmly established.

No attempts are known where strain-rate or time dependent effects have been evaluated. As the creep problem was envisaged to incorporate such effects, two types of experiment were performed with the aim of investigating the relation between composite creep behaviour and pullout-type data. The first was the normal one, a constant velocity experiment utilizing a nominally 'hard' tensile machine. The second was a constant load or creep pullout test. All experiments were performed on $450\text{ }\mu\text{m}$ diameter phosphor bronze wires embedded in pure lead blocks. Specimens were produced by casting Pb at $\sim 500^{\circ}\text{C}$ around as-received wires (partially wetted) or around as-received wires coated with a soldering flux (BICC Coraline Paste Flux) (wetted fibre specimen). The embedded length of fibre was measured with a Pye Universal Measuring Microscope after pullout. In cases where fibres fractured inside the block, the nominal embedded length measured before casting the matrix was utilized.

Constant Velocity

Using an Instron machine, the dependence of maximum pullout load F on embedded length ℓ_e for various cross-head speeds is shown in Fig. 4.13. The behaviour of the partially wetted fibres was very erratic inferring surface irregularities played an important role. Only isolated patches of adhering matrix were found on the pulled out surfaces. The main resistance to pullout appeared to be friction at the interface and this decreased as the cross-head speed increased.

The wetted fibres were more self consistent. The resistance to pullout α' (slope of the solid lines of Fig. 4.13) increased slightly with a cross-head speed increase of 100/1. In all cases, a complete layer of adhering matrix was found on the embedded fibre surface after

pullout. Consequently, ultimate pullout involved shear through the matrix near the fibre. One other difference from the partially wetted fibres was that the extrapolated linear relation between F and l_e did not pass through the origin (see Fig. 4.13).

The results can be described by an equation of the form

$$F = F_0 + \alpha' l_e \quad \dots (4.3)$$

where F_0 is the fibre end load. The approximate linearity of the slope α' implies the existence of a constant τ along the interface of the embedded length at the moment of pullout. A force balance on an element of fibre gives

$$\tau \pi d \, dz = dF \quad \dots (4.4)$$

or
$$\tau = \frac{1}{\pi d} \left(\frac{dF}{dz} \right) = \frac{\alpha'}{\pi d} \quad \dots (4.4a)$$

Calculated values of τ from (4.4a) for the wetted fibres only are given in Table 4.8.

At the fibre fracture load, $F = F^*$, the embedded length reaches a critical value $l_e = l_{c/2}$. The factor $1/2$ relates to a fibre within a composite specimen which is necessarily loaded from both ends via matrix shear. From (4.3) we then obtain

$$\frac{l_c}{d} = \frac{(F^* - F_0)}{\tau \frac{\pi d^2}{2}} = \frac{\sigma_f^* - \sigma_{f_0}}{2 \tau} \quad \dots (4.5)$$

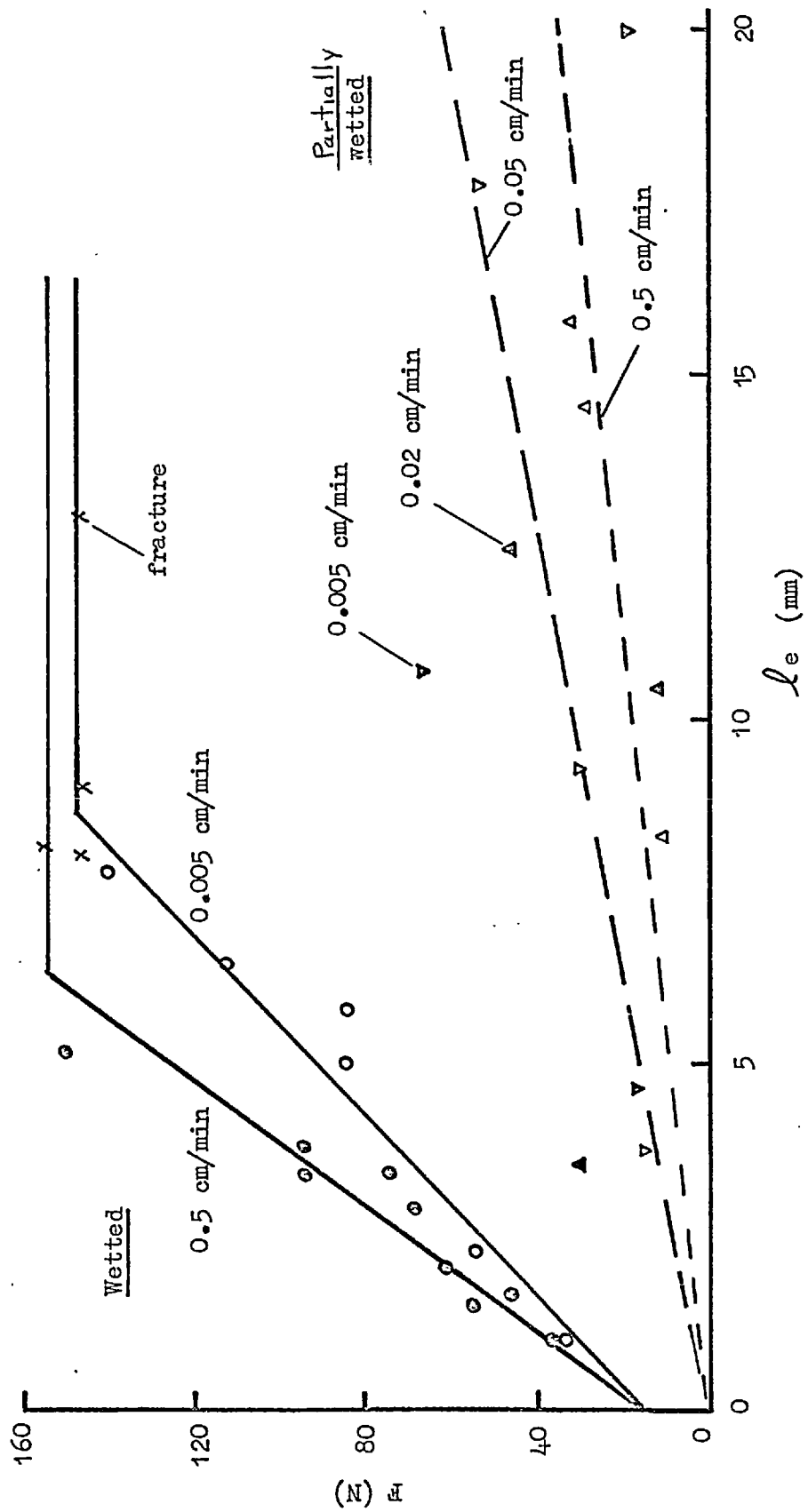


Fig. 4.13 Maximum load vs embedded fibre length from pullout tests.
20°C. (Constant velocity).

Table 4.8 Pullout Data for Phosphor Bronze Wires in Pb.

(d = 450 μ m)

<u>Cross-head speed (cm/min)</u>	τ <u>(MN/m²)</u>	l_c/d ($\frac{\dagger}{\ddagger}$)	
		<u>(F* = 150 N)</u>	<u>(F* = 58 N)</u>
0.5	14.7 ($\frac{\dagger}{\ddagger}$)	27	9
0.005	10.8 ($\frac{\dagger}{\ddagger}$)	37	12
creep	2.8 -		90 -
	4.1 ($\frac{\dagger}{\ddagger}$)		130

($\frac{\dagger}{\ddagger}$) calculated from (4.4a) and slope of Fig. 4.13

($\frac{\dagger}{\ddagger}$) measured creep strength (σ_m/τ) from Fig. 4.8

($\frac{\dagger}{\ddagger}$) from (4.5) with F* = 150 N from Fig. 4.13

and F* = 58 N from Table 4.7.

This is the expression for ℓ_c/d obtained by Kelly and Tyson (1965). Utilizing the determined values of τ , ℓ_c/d values are given in Table 4.8 for F^* as indicated in Fig. 4.13 and for F^* of annealed wires (composite wires from Table 4.7). The calculated ℓ_c/d are much less than the composite fibres, $\ell/d = 100$, and hence fibre fracture was to be expected in the tensile tested specimens at least.

Whereas the above data are of use at tensile test rates, at creep rates the shear strength of the matrix would be much less and hence ℓ_c/d much greater. Using the measured creep strengths of the pure matrix (Fig. 4.8, $\sigma_m = 2\tau$) ℓ_c/d was found to be substantially larger, 90 - 130 over the range of creep rates experienced by the composites. Thus it would appear that some fibre fracture due to creep deformation should occur based on this simple model. However, broken fibres were found only at the composite rupture surface. This suggests that locally tensile rate test conditions may have prevailed during final rupture of the specimens.

Constant Load (Creep)

A series of experiments were performed on wetted fibres only which gave the time to pullout failure t_{po} for various constant loads F and embedded lengths ℓ_e . An Instron tensile machine was rendered 'soft' by utilizing specimens with a large total length of protruding fibre ($\ell_t/\ell_e = 30 - 120$). On attainment of the desired load, the cross-head motion was terminated and t_{po} measured from the chart record. A slight relaxation took place immediately after the cross-head was stopped, but a near constant load existed thereafter. A recorder tracing is shown in Fig. 4.14.

In addition, a few experiments were performed in the direct loading creep apparatus where a true constant load was maintained until pullout failure.

The results from the two modifications of a creep test are given in Fig. 4.15. The values of t_{po} increased much more rapidly with l_e at small loads than at large loads. The pulled out ends exhibited matrix adhering to both the sides and the ends of the fibres.

An analysis similar to that for the tensile pullout tests can be invoked. At any time $0 < t < t_{po}$, a stable balance of forces along the embedded length must exist if pullout is not to take place,

$$\pi d \int_0^{l_e} \tau_t dz = \int_{F_0}^F dF \quad \dots (4.6)$$

However, now both τ_t and F_0 are variable as the matrix experiences creep deformation,

$$\tau_t = \tau_t(t, z)$$

and

$$F_0 = F_0(t)$$

As a consequence, evaluation of (4.6) at all times is not possible without additional information.

Considering only the stress state at the moment of pullout failure, an arbitrary assumption is made that when $t = t_{po}$, $\tau_t = \tau_t^*$, a constant critical value. Then from (4.6) we have

$$\tau_t^* = \frac{F - F_0^*}{\pi d l_e} \quad \dots (4.7)$$

where F_0^* is the end loading force at the start of pullout failure.

τ_t^* calculated from (4.7) and Fig. 4.15 is plotted against t_{po} in double logarithmic co-ordinates in Fig. 4.16. For consistency and lack of a better value F_0^* was taken equal to the end load force of the tensile test data (Fig. 4.13) although the true value must be somewhat less due to stress relaxation.

The results approximate a linear relation which can be described by the equation,

$$\tau_t^* = \tau_o \left(\frac{t_{po}}{t_o} \right)^{-p} = 7. \left(\frac{t_{po}}{400} \right)^{0.10} \dots (4.8)$$

where the units are MN/m^2 and minutes. The unified behaviour of Fig. 4.16 for various applied loads suggests that a unique relation between τ_t^* and t_{po} exists.

There would appear to be no basis for associating the attainment of τ_t^* itself with the onset of failure. Under a stable shear stress distribution, a slow pulling of the fibre from the matrix is possible. If so, then sudden pullout must be dictated by some instability such as cavitation at the fibre end, shear failure of the matrix (not interface in present work) or perhaps simple geometrical instability due to the decreasing embedded length. Ultimate failure involved shear of the matrix but this does not necessitate a similar origin.

The failure origin is unknown but it is noted here that it may be related to the origin of creep rupture of the discontinuous fibre composites. Due to the inherent scatter in composite rupture data resulting from V_f variations, single fibre pullout tests may be a valuable approach to the rupture problem.

The composite specimens exhibited rupture times of 200 - 2000 hours (Table 4.3). From (4.8) a similar range of t_{po} corresponds to $\tau_t^* = 5 - 4 \text{ MN/m}^2$. Comparing with the shear strength of the matrix τ at creep rates comparable to those of the composites, we find $\tau \sim \frac{1}{2} \tau_t^*$. If there is any validity in associating these two quantities, it may be expected that composite rupture times would be considerably larger than found. However, as rupture was due to localized behaviour in regions of minimum V_f , local creep rates and hence matrix stresses could conceivably have been much larger.

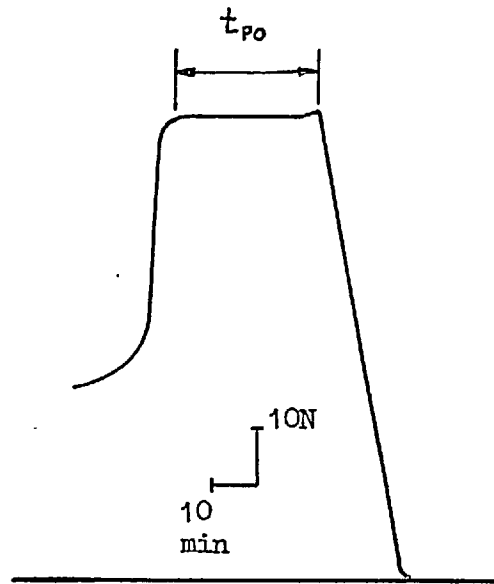


Fig. 4.14 Reduced size recorder tracing of applied load vs time to pull-out a single phosphor bronze wire from pure Pb at 20°C (constant load test).

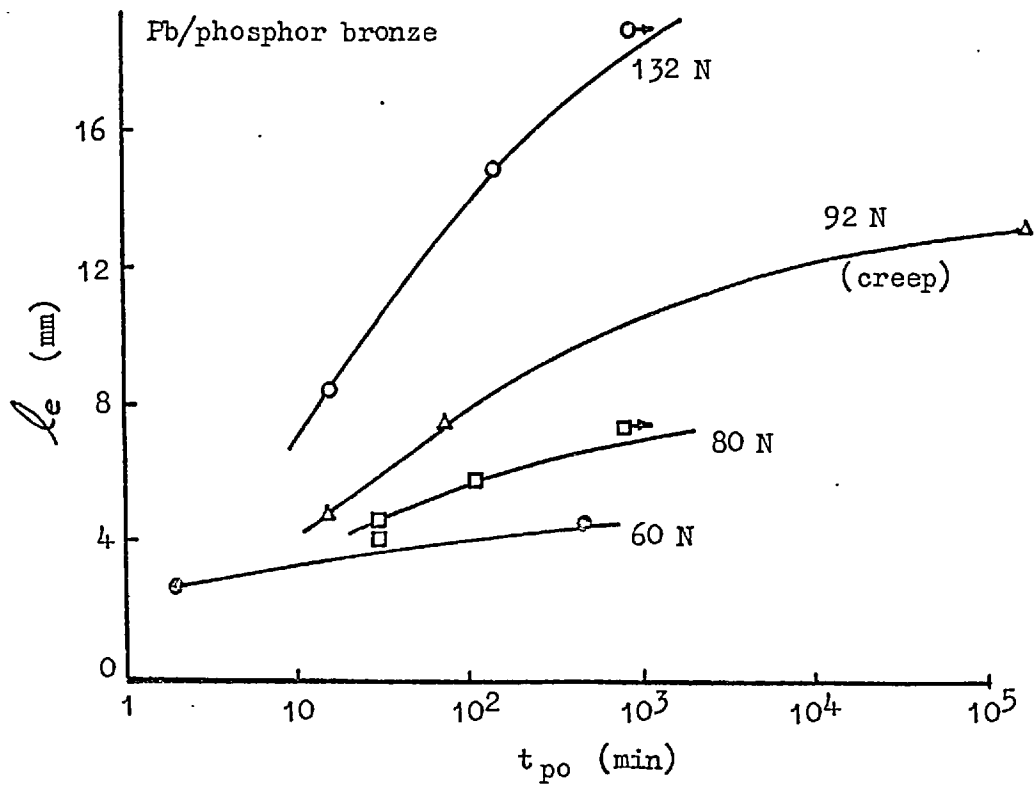


Fig. 4.15 Embedded length vs log time to pull-out failure for various loads (20°C).

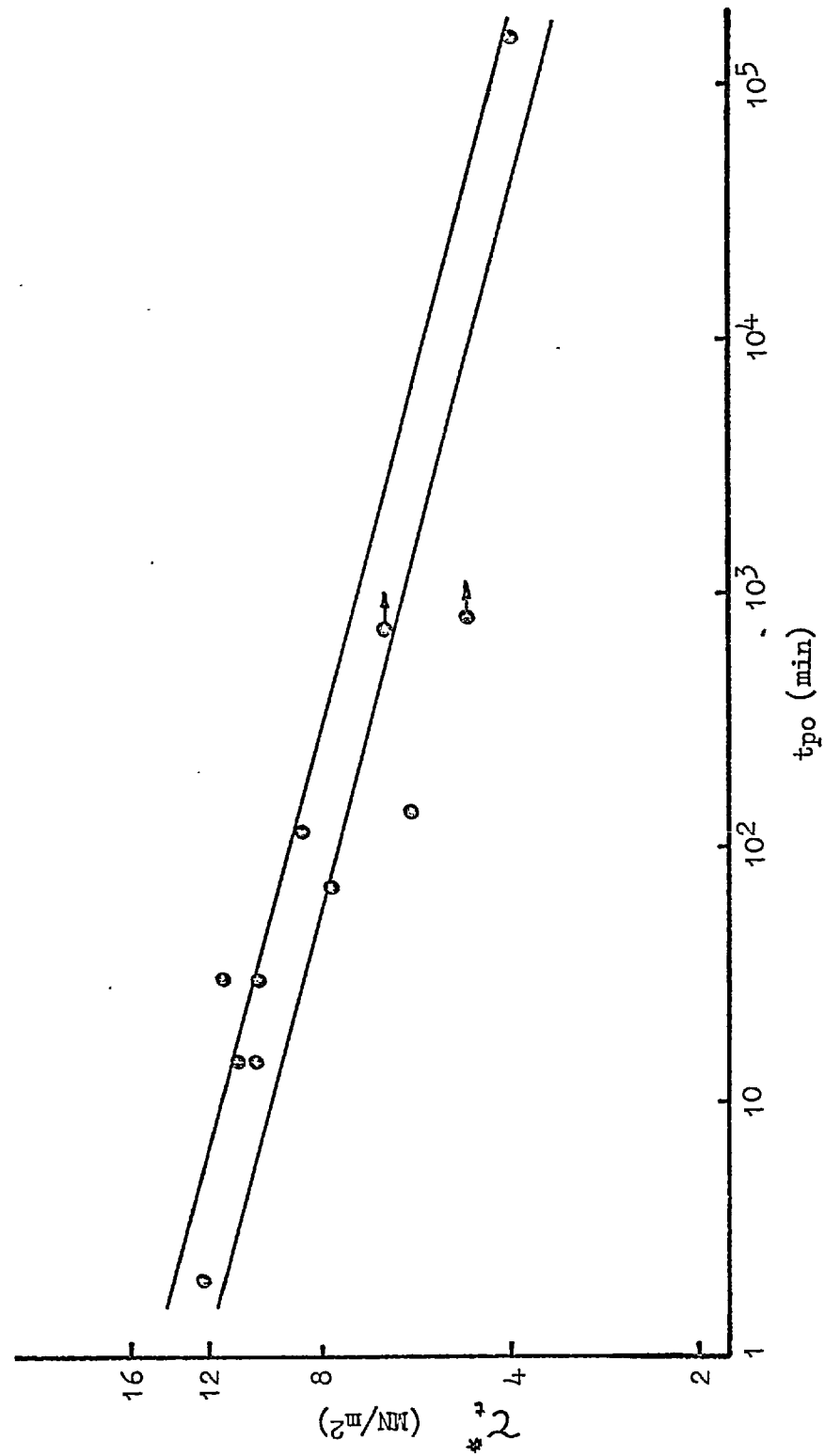


Fig. 4.16 Log critical shear stress vs log time to pullout failure.
 20°C .

CHAPTER V

THEORY OF STEADY-STATE CREEP OF

DISCONTINUOUS FIBRE COMPOSITES

5.1 Introduction

Mileiko (1970) developed a theory for the steady-state creep of discontinuous fibre composites based on a model wherein the composite stress σ_c was applied directly to the fibres alone (matrix strength equal zero). This implies a constant fibre stress over the fibre length. The calculated creep rate was that of the matrix under conditions of simple shear. The model was based on rigid fibres and a perfectly bonded interface.

This chapter describes a theory which is based on a different approach. As distinct from Mileiko, both matrix and fibres support a tensile stress. The former induces a matrix creep extension which is opposed in the vicinity of more rigid fibres by some form of bonding between the phases. This opposition manifests itself in a shearing force which generates a non-uniform stress in the fibre. The theoretical model allows for both rigid and creeping fibres and also for sliding at the interface. Comparison is made between various theoretical predictions and the experimental data of Chapter IV.

5.2 Behaviour of Model Constituents

Matrix

It is assumed that a constant tensile creep rate $\dot{\epsilon}_m$ exists at all locations within the matrix. This is in accordance with

experimentally observed steady-state composite behaviour. Complexities at the ends of fibres are ignored. The basic behaviour is that of the pure matrix.

Fibres

When the fibre stress exceeds some yield value, creep deformation will occur in the vicinity of the central portion of the fibre length where the stress is largest. This will become more apparent later from calculated fibre stress distributions. Within a composite material, the fibre creep rate $\dot{\epsilon}_f$ is limited by that of the surrounding matrix. The adopted model neglects the fibre creep rate transient up to the point where $\dot{\epsilon}_f = \dot{\epsilon}_m$. The fibre length is then characterized by two regions as shown in Fig. 5.1, (i) $\dot{\epsilon}_f = \dot{\epsilon}_m$ near the centre ($z \leq z_c$) and (ii) $\dot{\epsilon}_f = 0$ near the ends ($z \geq z_c$). This is considered to be a good approximation if the stress sensitivity of the fibre is relatively large which is likely if it is operating at a low homologous temperature. If $\dot{\epsilon}_f < \dot{\epsilon}_m$ everywhere, the fibres are assumed to be rigid.

Interface Continuity

The interfacial bond between fibres and matrix must resist matrix extension if load is to be transferred efficiently to the more rigid discontinuous fibres. Neglecting lateral effects, it is apparent that over the region $\dot{\epsilon}_f = \dot{\epsilon}_m$, interface continuity is not taxed. However, when $\dot{\epsilon}_f = 0$, sliding will occur unless a perfect bond exists. Bearing in mind that grain boundary sliding is a well-known creep phenomenon, it is conceivable that sliding at the fibre/matrix interface will take place.

5.3 Composite Unit Cell

The composite considered contains only aligned fibres of constant diameter d and aspect ratio ℓ/d which are randomly distributed in the direction of their axes. The applied composite stress is static and parallel to the fibres. Load transfer to the fibres via direct stresses across their ends is assumed to be negligible. This implies ℓ/d cannot be exceedingly small.

From the random array, a single fibre unit cell is extracted as shown in Fig. 5.2. It is assumed there exists a state of dynamic equilibrium between the constant tensile creep rate $\dot{\epsilon}_m$ of the matrix away from the fibre and a uniform laminar creep rate $\dot{\gamma}$ within a distance h of the fibre surface. For the laminar zone, at any value of h and at location z relative to the fibre centre, we have

$$\dot{\gamma}_z = \frac{1}{h} \dot{\Delta}_z = \frac{1}{h} (\dot{u}_m - \dot{u}_f - \dot{u}_i)_z \quad \dots (5.1)$$

where $\dot{\Delta}_z$ is the relative velocity between matrix and fibres and $\dot{u}_m$, $\dot{u}_f$, $\dot{u}_i$ are the velocities of matrix, fibre and interface.

The nature of the interfacial sliding velocity will be a specific characteristic of any one composite mixture and hence must be experimentally determined. In the present model, for simplicity, $\dot{u}_i$ is allowed to be directly proportional to the velocity difference $(\dot{u}_m - \dot{u}_f)$,

$$\dot{u}_i = \eta (\dot{u}_m - \dot{u}_f) \quad \dots (5.2)$$

where η is the proportional factor. Depending on whether the bond is perfect or non-existent, $0 \leq \eta \leq 1$. Thus the unit cell possesses two distinct regimes which are characterized by

$$\begin{aligned} z \leq z_c & : \quad \dot{u}_m = \dot{u}_f = \dot{\epsilon}_m z \\ \dot{u}_i & = 0 \end{aligned} \quad \dots (5.3)$$

$$\begin{aligned} z \geq z_c & : \quad \dot{u}_m = \dot{\epsilon}_m z, \quad \dot{u}_f = \dot{\epsilon}_m z_c \\ \dot{u}_i & = \dot{\epsilon}_m \eta (z - z_c) \end{aligned} \quad \dots (5.3a)$$

Because of the nature of the creep phenomenon, there must be a strong tendency for the shear zone to propagate far into the tensile zone away from the interface. The overlapping of shear and tensile zones represents a complex stress state and any assumed behaviour must be an oversimplification. To obtain a definite result, the assumption is made that at any z , $\dot{\gamma}$ is uniform and acts over the entire matrix region of the cell ($h = \frac{D-d}{2}$). The validity of this assumption is best judged by comparison between the theory to be developed and experiment.

Utilizing the power law expression (3.3) for the tensile creep behaviour of the matrix,

$$\sigma_m = \sigma_{m0} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{m0}} \right)^{1/m} \quad \dots (5.4)$$

in conjunction with the approximation that $\sigma_m = 2\tau$, the matrix shear stress, and $\dot{\epsilon}_m = \frac{2}{3}\dot{\gamma}$, we have for the basic characteristics of the laminar shear zone,

$$\dot{\gamma} = \frac{3}{2} \dot{\epsilon}_{m0} \left(\frac{2\tau}{\sigma_{m0}} \right)^m \quad \dots (5.5)$$

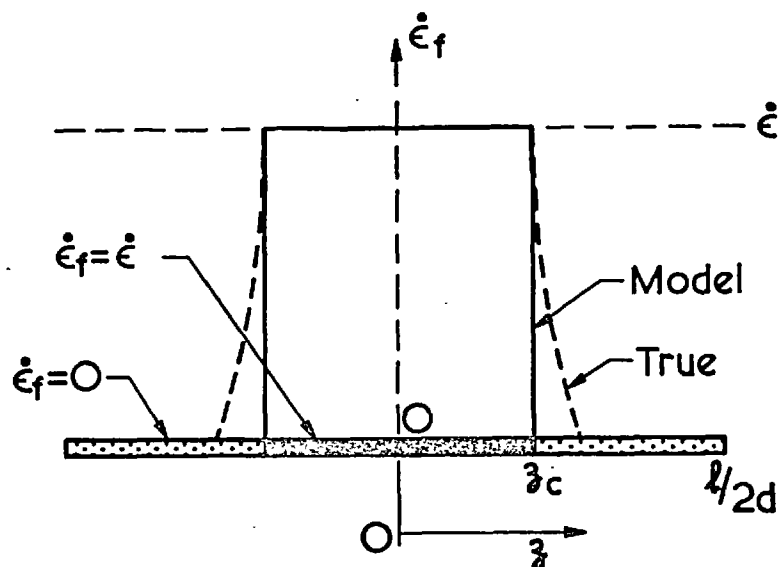


Fig. 5.1 Fibre creep rate distribution over fibre length for the creep model compared to the approximate true distribution.

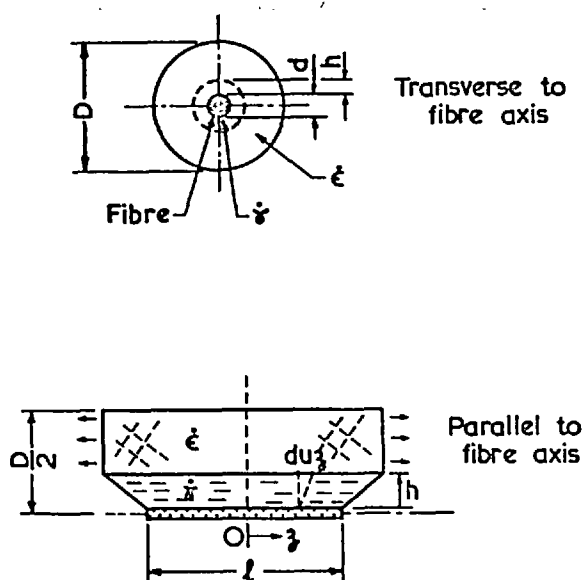


Fig. 5.2 The geometry of the single fibre unit cell of the creep model. Top - the fibre encased in cylindrical volume of matrix. Bottom - the tensile creep zone $\dot{\epsilon}$ and the laminar creep zone $\dot{\gamma}$.

For a regular hexagonal array of unit cells in a void free composite volume we have,

$$\frac{d}{D} = \left(\frac{2\sqrt{3}}{\pi} V_f \right)^{1/2} \quad \dots (5.6)$$

Combining (5.1), (5.3a), (5.5) and (5.6) we find the shear stress acting on the fibre for $\bar{\gamma} > \bar{\gamma}_c$,

$$\tau_{\bar{\gamma} > \bar{\gamma}_c} = \beta' \sigma_{mo} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{mo}} \right)^{1/m} \left(\frac{\bar{\gamma} - \bar{\gamma}_c}{d} \right)^{1/m} (1 - \eta)^{1/m} \quad \dots (5.7)$$

where

$$\beta' = \frac{1}{2} \left(\frac{4}{3} \right)^{1/m} \left[\left(\frac{2\sqrt{3}}{\pi} V_f \right)^{-1/2} - 1 \right]^{-1/m} \quad \dots (5.7a)$$

For $\bar{\gamma} < \bar{\gamma}_c$, $\tau = 0$. Utilizing this expression for τ , it is now possible to calculate the characteristics of the composite mixture.

This will be done for three specific cases, (i) rigid fibres, perfect bond, (ii) creeping fibres, perfect bond, (iii) rigid fibres, imperfect bond.

5.4 Rigid Fibres, Perfect Bond

For this case, τ is given by (5.7) with $\bar{\gamma}_c = 0$ and $\eta = 0$. The value of τ depends on m as shown in Fig. 5.3 where for illustration we have taken

$$\beta' \sigma_{mo} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{mo}} \right)^{1/m} = 1$$

It is evident that τ is essentially constant over the fibre length at large m values but is far from constant at small m values. A maximum τ occurs at minimum m and maximum V_f (through the parameter β').

5.4.1 Fibre Stress

From a force balance on an element of fibre, we obtain for the fibre stress distribution

$$\sigma_f = - \int_0^z \frac{1}{d} \tau dz = 4\beta' \left(\frac{m}{m+1} \right) \bar{\sigma}_{mo} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{mo}} \right)^{1/m} \left(\frac{l}{2d} - \frac{z}{d} \right)^{\frac{m+1}{m}} \dots (5.8)$$

where l/d is the aspect ratio of the fibre. The average stress in the fibre is then

$$\bar{\sigma}_f = \frac{2}{l} \int_0^{l/2} \sigma_f dz = \phi \bar{\sigma}_{mo} \left(\frac{l}{d} \right)^{\frac{m+1}{m}} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{mo}} \right)^{1/m} \dots (5.9)$$

where

$$\phi = \left(\frac{2}{3} \right)^{1/m} \left(\frac{m}{2m+1} \right) \left[\left(\frac{2\sqrt{3}}{\pi} V_f \right)^{-1/2} - 1 \right]^{-1/m} \dots (5.9a)$$

The quantity ϕ is called the 'load transfer parameter.' It is apparent that a maximum $\bar{\sigma}_f$ requires a maximum ϕ .

5.4.2 Composite Creep Strength

Substitution of (5.9) and (5.4) into the normal relation which partitions the composite stress σ_c between the components according to their volume fractions, (3.4), we obtain

$$\sigma_c = \phi \sigma_{m0} \left(\frac{l}{d} \right)^{\frac{m+1}{m}} \left(\frac{\dot{\epsilon}_c}{\dot{\epsilon}_{m0}} \right)^{1/m} V_f + \sigma_{m0} \left(\frac{\dot{\epsilon}_c}{\dot{\epsilon}_{m0}} \right)^{1/m} (1-V_f) \dots (5.10)$$

The quantity ϕ depends strongly on the matrix characteristic m and V_f as shown in Fig. 5.4. The value of $V_f = 0.44$ represents a volume fraction above which a decreasing m always increases ϕ and hence the fibre carrying capacity. This feature is coupled to the additional strengthening by the $\left(\frac{l}{d} \right)^{\frac{m+1}{m}}$ term when m is decreased.

To compare (5.10) with Mileiko (1970), we rewrite his equation (26) in the same form, namely

$$\sigma_c'' = \left[\frac{J(m) (m-1)^{1/m}}{(1-V_f^{\frac{m-1}{2}})^{1/m}} \right] \sigma_{m0} \left(\frac{l}{d} \right)^{\frac{m+1}{m}} \left(\frac{\dot{\epsilon}_c}{\dot{\epsilon}_{m0}} \right)^{1/m} V_f \dots (5.11)$$

For $m > 4$ and for normal values of V_f , the terms grouped together in the square brackets reduce to a value of 0.3 - 0.4. Equations (5.10) and (5.11) are quite similar except for the initial constant and the matrix contribution in (5.10).

It is interesting to compare superficially the form of the present theory with various theoretical expressions for the viscosity of Newtonian fluids containing rigid particles. First, (5.10) can be rewritten for $m = 1$ in the form

$$\frac{\eta_c}{\eta_m} = (1-V_f) + \phi \left(\frac{l}{d} \right)^2 V_f \dots (5.12)$$

where η_c/η_m is the ratio of viscosity of the composite mixture to the

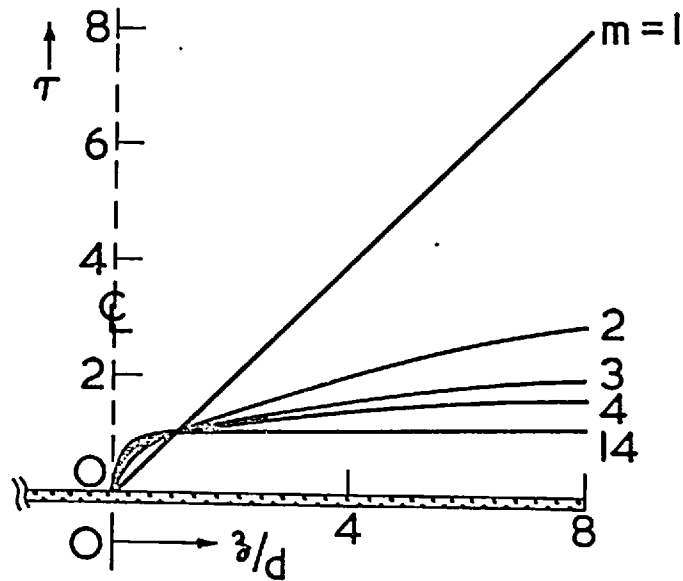


Fig. 5.3 Profiles of calculated shear stress acting on fibre over one-half of its length, for various values of the matrix stress sensitivity m .

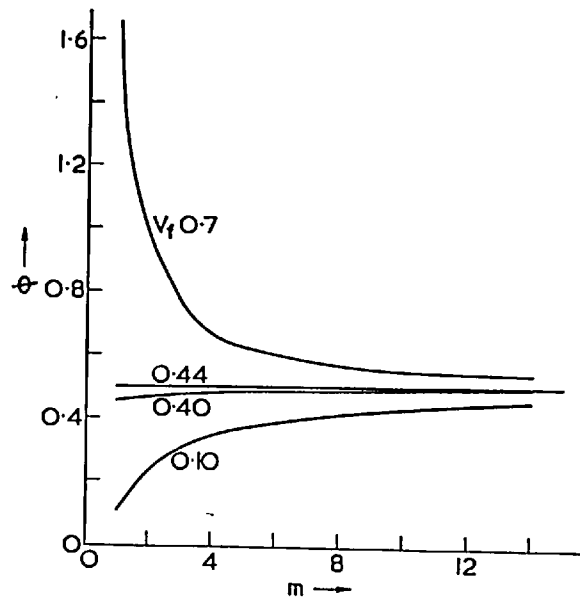


Fig. 5.4 Values of the load transfer parameter ϕ as a function of m and V_f .

pure matrix. For fluid flow, typical equations for viscosity are (Frisch and Simha, 1956),

$$\text{spheres: } \frac{\eta_c}{\eta_m} = 1 + \frac{5}{2} V_f + 14.1 V_f^2 + \dots \quad \dots (5.13)$$

$$\text{dumb-bells: } \frac{\eta_c}{\eta_m} = 1 + \frac{3}{2} \left(\frac{\ell}{d}\right)^2 V_f + \left(\frac{87}{50}\right) \left(\frac{\ell}{d}\right)^4 V_f^2 \quad \dots (5.13a)$$

$$\text{or rods}$$

In these expressions, the constants depend in general on the rod size, shape and orientation and on the particular theory. The V_f^2 terms enter at high volume fractions (≥ 0.1). Needless to say, similarities do exist between the present theory (5.12) and (5.13a). However, the fluid flow developments for $m = 1$ only will not be applicable in general to the creep problem where usually $m > 1$.

5.4.3 Limiting Parameters

The present theory has virtually no lower limit of V_f associated with its application. At $V_f = 0$, (5.10) degenerates to that of the pure matrix.

Although an upper limit of V_f must be that corresponding to a maximum packing density of fibres, in real composites fibre-fibre contact will likely exist at somewhat smaller V_f values. The relative motion of contacting fibres would be determined by some sliding coefficient of friction and deviations from the theory would occur.

If the creep strength of the composite (5.10) is to exceed that of the pure matrix (5.4), we must have a minimum fibre aspect ratio given by

$$\sigma_c > \sigma_m ,$$

$$\phi\left(\frac{l}{d}\right)^{\frac{m+1}{m}} > 1 \quad \dots (5.14)$$

For m large, $l/d > 2$ and for $m = 1$ ($V_f = 0.44$), $l/d > 2^{1/2}$.

The values are typically very small and generally will be easily achieved in manufacture of composites.

Related to the above is the effect of neglecting the contribution of the matrix to the carrying capacity of the composite. It is worthwhile examining the conditions where this simplification can be judged valid. If we neglect the matrix contribution, we cancel the second term in (5.10). Forming the ratio of the neglected matrix composite strength σ_c' to the included matrix composite strength σ_c we find

$$\frac{\sigma_c'}{\sigma_c} = \frac{\phi\left(\frac{l}{d}\right)^{\frac{m+1}{m}} V_f}{\left[\phi\left(\frac{l}{d}\right)^{\frac{m+1}{m}} - 1\right] V_f + 1} \quad \dots (5.15)$$

As seen in Fig. 5.5 for the case of m large, considerable error can result unless both V_f and l/d are quite large.

At the other extreme, if a creeping matrix (or deforming matrix in general) is to fracture a fibre, a minimum aspect ratio of fibre must be present in the composite. This is termed the critical aspect ratio l_c/d and is defined from (5.8) when $\sigma_f = \sigma_f^*$, the breaking strength of the fibres, and $\beta/d = 0$ (for a maximum). Thus,

$$\frac{l_c}{d} = 2 \left[\frac{1}{4\beta'} \left(\frac{m+1}{m} \right) \left(\frac{\dot{\epsilon}_{mo}}{\dot{\epsilon}_m} \right)^{1/m} \frac{\sigma_f^*}{\sigma_{mo}} \right]^{\frac{m}{m+1}} \quad \dots (5.16)$$

It follows from (5.16) that the dependence of ℓ_c/d on $\dot{\epsilon}_m$ is considerably greater for small m than for large m matrices.

Definition of ℓ_c/d similarly defines both σ_c^* and $\dot{\epsilon}_c^*$, critical stress and creep rate. For $\sigma_c \geq \sigma_c^*$, rigid fibres will be fractured and the composite rupture mode will most likely change from a matrix shear fracture origin to a broken fibre origin. The value of σ_c^* is found from (5.16), (5.9a), (5.7a) and (5.10) to be

$$\sigma_c^* = \sqrt{1 - \left(1 - \frac{m+1}{2m+1}\right) \frac{\ell_c}{d}} \sigma_f^* \sqrt{V_f} + \sigma_m (1 - V_f) \dots (5.17)$$

For the purposes of predicting $\dot{\epsilon}_c$ from the creep theory, we must have $\sigma_c < \sigma_c^*$. The only creep rate prediction that is possible for a composite containing rigid fibres which are fracturing is an estimate of the increased rate produced by the new ℓ/d value, for example equal to $\frac{1}{2}$ the original ℓ/d value. However, as it is unlikely that all fibres would fracture at the same time, such a prediction would have little meaning.

5.4.4 Longitudinal Distribution of V_f

It is assumed in the model that the fibre distribution is a random one throughout and that at any cross-section there exists a constant V_f . In a real composite, such will not be the case. Due to the non-linear dependence of $\dot{\epsilon}_c$ on V_f , it is necessary to take account of a variable V_f if the theoretical prediction is to be made more accurate.

At any V_{fi} , we have from (5.10)

$$\dot{\epsilon}_{ci} = \left(1 - 2 V_{fi}\right)^{-m} \dot{\epsilon}_{m0} \left(\frac{\sigma_c}{\sigma_{m0}}\right)^m \dots (5.18)$$

where
$$\Omega = \phi \left(\frac{l}{d} \right)^{\frac{m+1}{m}} - 1 \quad \dots (5.18a)$$

If we take N equal lengths along a composite specimen, it follows that the measured mean creep rate, $\bar{\dot{\epsilon}}_c$, over the total length is given simply by

$$\bar{\dot{\epsilon}}_c = \sum \frac{n_i}{N} \dot{\epsilon}_{ci} \quad \dots (5.19)$$

where n_i/N = fraction of segments of V_{fi} . Ideally N should be as large as possible. Combining (5.18) and (5.19) we obtain

$$\bar{\dot{\epsilon}}_c = \sum \frac{n_i}{N} (1 + \Omega V_{fi})^{-m} \dot{\epsilon}_{mo} \left(\frac{\sigma_c}{\sigma_{mo}} \right)^m \quad \dots (5.20)$$

Thus, a knowledge of the longitudinal V_{fi} distribution would allow (5.20) to be used for the theoretical prediction of composite creep rate. This involves a rather laborious calculation which may or may not be justified depending on the circumstances. A simple determination of an average V_f will not produce as accurate a prediction as the above.

A more serious outcome of a variation in V_f is that the matrix will be overstressed in regions of low V_f . The effect is analogous to a variable cross-section in a pure matrix specimen. Because of the non-linear relation between stress and creep rate, it is found that if $m > 1$, creep accentuates the differences in cross-sectional area and hence V_f (McLean 1968). Thus, composite deformation is inherently unstable.

Related to the same problem is the theoretical finding that the load transfer parameter ϕ increases with V_f , particularly at low m values, Fig. 5.4. Thus, the greater efficiency of load transfer in the high V_f regions tends to magnify the weak sections. However, in opposition to the cross-sectional stability above, a large m is now favoured to minimize this effect.

The main conclusion that may be drawn here is that composite rupture will most likely occur at the weakest cross-section (minimum V_f or fibre-fibre overlap).

5.5 Creeping Fibres, Perfect Bond

τ is obtained from (5.7) with $\eta = 0$. The shear stress profiles are identical to those for the rigid fibre development Fig. 5.3, except that they originate at $z = z_c$ instead of the fibre centre. The value τ_{max} is reduced from being proportional to $\left(\frac{l}{2d}\right)^{1/m}$ to $\left(\frac{l}{2d} - \frac{z_c}{d}\right)^{1/m}$.

5.5.1 Fibre Stress Distribution

The fibre stress distribution becomes

$$\sigma_f = 4\beta' \left(\frac{m}{m+1}\right) \tau_{m0} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{m0}}\right)^{1/m} \left[\left(\frac{l}{2d} - \frac{z_c}{d}\right)^{\frac{m+1}{m}} - \left(\frac{z}{d} - \frac{z_c}{d}\right)^{\frac{m+1}{m}} \right] \dots \quad (5.21)$$

$z > z_c$

The maximum fibre stress occurs at $z = z_c$ and is given from (5.21) by

$$\sigma_{fmax} = 4\beta' \left(\frac{m}{m+1}\right) \tau_{m0} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{m0}}\right)^{1/m} \left[\frac{l}{2d} - \frac{z_c}{d} \right]^{\frac{m+1}{m}} \dots \quad (5.22)$$

The rate of stress build-up from the fibre end is strongly dependent on the matrix stress sensitivity m . This is shown in Fig. 5.6 for $m = 1$

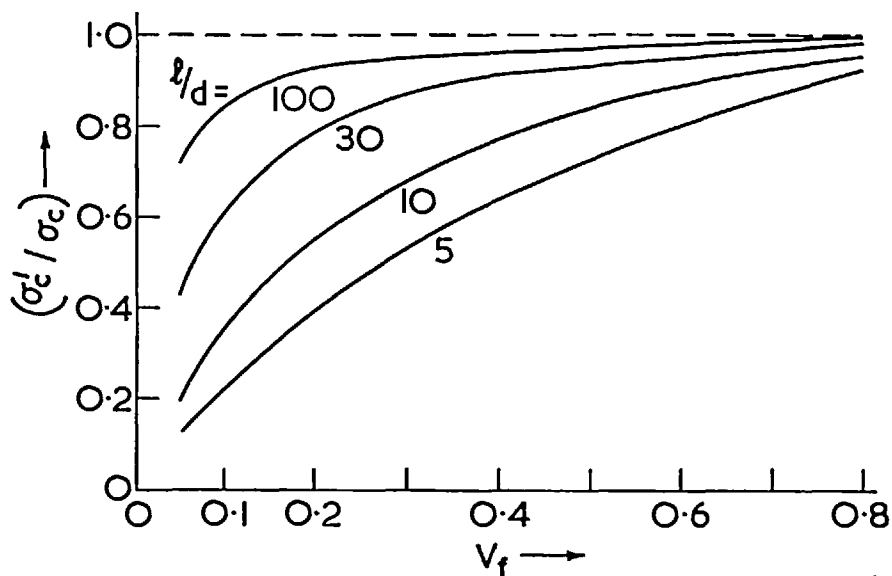


Fig. 5.5 Ratio of composite strength when matrix tensile stress neglected σ'_c to that when it is included σ_c for various l/d and V_f .

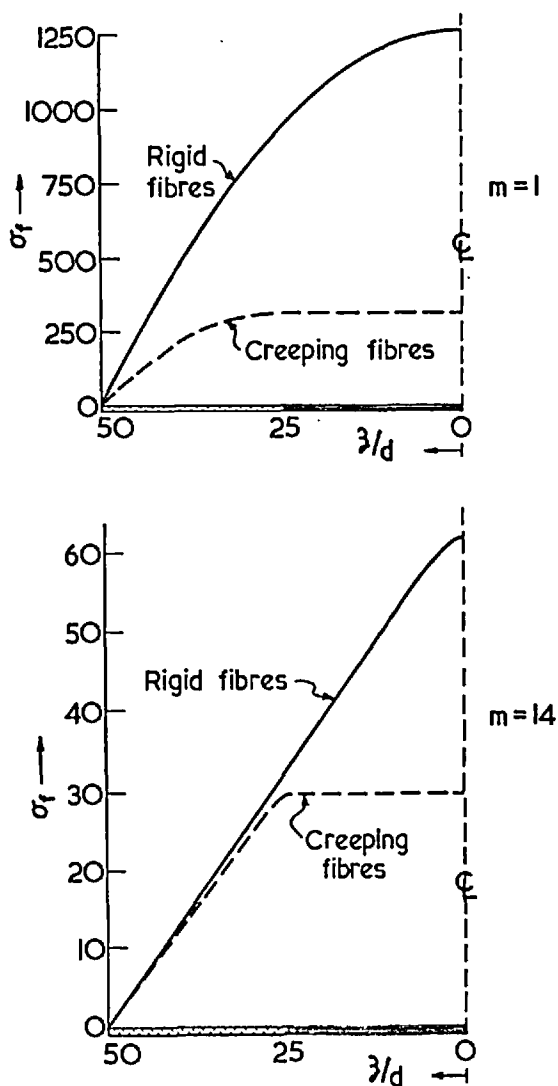


Fig. 5.6 Calculated stress distributions in rigid and creeping fibres for two extreme values of stress sensitivity m .

and $m = 14$ for both rigid fibres and creeping fibres (50% of fibre length creeping). To simplify, we have taken $4\beta' \sigma_{m0} (\dot{\epsilon}_m / \dot{\epsilon}_{m0})^{1/m} = 1$. The very strong dependence of σ_{fmax} on m is also apparent in this figure.

It is evident that at large m values, the distribution in a creeping fibre is essentially that of the rigid fibre distribution with some σ_{fmax} restriction. This would allow a simplified approach to be utilized in determining the effect of creeping fibres. However, at low m values, an exact solution is required in order to determine a more precise value for such quantities as the average fibre stress.

5.5.2 Stress Transfer Aspect Ratio (STAR)

In expressions (5.21) and (5.22) the quantity $\left(\frac{l}{2d} - \frac{z_c}{d}\right)$ is termed the 'stress transfer aspect ratio' STAR. It represents the zone at each end of the fibre over which the shearing traction is non-zero and hence the region where stress transfer is taking place. From (5.22) we have

$$\text{STAR} = \left(\frac{l}{2d} - \frac{z_c}{d}\right) = \left[\frac{\sigma_{fmax}}{4\beta'} \left(\frac{m+1}{m}\right) \frac{1}{\sigma_{m0}} \left(\frac{\dot{\epsilon}_{m0}}{\dot{\epsilon}_m}\right)^{1/m} \right]^{m/(m+1)} \dots (5.23)$$

The dependence of STAR on $\dot{\epsilon}_m$ requires some knowledge of the variability of σ_{fmax} with creep rate. If a power law is applicable also to the fibres, (3.2), then substitution of such a relation into (5.23) with $\sigma_f = \sigma_{fmax}$ and $\dot{\epsilon}_f = \dot{\epsilon}_m$ yields,

$$\left(\frac{l}{2d} - \frac{z_c}{d}\right) = \theta (\dot{\epsilon}_m^{1/n - 1/m})^{m/(m+1)} \dots (5.24)$$

with

$$\theta = \left[\frac{\frac{m+1}{m}}{4\beta'} \cdot \frac{\sigma_{fo}}{\sigma_{mo}} \cdot \frac{\dot{\epsilon}_{mo}^{1/m}}{\dot{\epsilon}_{fo}^{1/n}} \right]^{m/m+1} \dots (5.24a)$$

From (5.24), it follows that STAR decreases with increasing $\dot{\epsilon}_m$ if $n > m$, remains constant if $n = m$, and increases if $n < m$. This is shown schematically in Fig. 5.7. For the latter case, the fibre creep rate may eventually become zero along its entire length even though $\dot{\epsilon}_m$ and hence σ_f has been increased. For rigid fibres, STAR is a constant value $l/2d$.

5.5.3 Average Fibre Stress and Composite Creep Strength

In the portion of the fibre where the stress is increasing to some constant maximum value, we have from (5.21) for the average stress,

$$\overline{\sigma_f'} = \phi \sigma_{mo} \left(\frac{l}{d} - \frac{2z_c}{d} \right)^{\frac{m+1}{m}} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{mo}} \right)^{1/m} \dots (5.25)$$

The overall average stress along the entire length of the fibre is then given in general form by

$$\overline{\sigma_f} = \left[\overline{\sigma_f'} \left(\frac{l}{2d} - \frac{z_c}{d} \right) + \sigma_{fmax} \left(\frac{z_c}{d} \right) \right] \frac{2d}{l} \dots (5.26)$$

Combining (5.22) and (5.25) with (5.26) we have

$$\overline{\sigma_f} = \sigma_{mo} \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{mo}} \right)^{1/m} \frac{2d}{l} \left[\phi 2^{\frac{m+1}{m}} \left(\frac{l}{2d} - \frac{z_c}{d} \right)^{\frac{2m+1}{m}} + 4\beta'^{1/m} \left(\frac{l}{2d} - \frac{z_c}{d} \right)^{\frac{m+1}{m}} \frac{z_c}{d} \right] \dots (5.27)$$

To evaluate (5.27), a value for $\frac{z_c}{d}$ from (5.23) or (5.24) must be determined.

The composite creep strength is then given by the combined stresses in the matrix and fibre,

$$\sigma_c = \bar{\sigma}_f V_f + \sigma_{m0} (1 - V_f) \left(\frac{\dot{\epsilon}_m}{\dot{\epsilon}_{m0}} \right)^{1/m} \quad \dots (5.28)$$

where $\bar{\sigma}_f$ is obtained from (5.27).

It follows from the above development that the average stress generated in a creeping fibre will depend upon how much of the fibre length is creeping and the matrix stress sensitivity m . The relative loss in $\bar{\sigma}_f$ within a creeping fibre compared to that in a rigid fibre under identical conditions is shown in Fig. 5.8. Even when the central 25% of a fibre length is creeping, the loss in $\bar{\sigma}_f$ for common values of m (≈ 5) is a moderate 10-15%. Translated into a composite creep rate, this produces an increase in rate by a factor of only ~ 2 times.

5.5.4 Limiting Parameters

For creeping fibres, l_c/d cannot be defined as σ_{fmax} is determined by the intrinsic properties of the fibres and is limited by a given $\dot{\epsilon}_m$. Hence, σ_{fmax} is independent of l/d . However, a modified l_c/d may exist for creeping fibres which represents the transition from a regime where the matrix within the composite ruptures first (matrix control) to one where fibres rupture first (fibre control). Simply comparing the rupture times of pure matrix and fibres for a constant $\dot{\epsilon}_m$ is unlikely to define the two regimes as the effect of fibre ends on matrix rupture is not taken into account. The behaviour here is basically the creep rupture problem of a discontinuous fibre composite, an area which has not received attention to date.

5.5.5 Composite Stress Sensitivity

In the theory developed for rigid fibres, the stress sensitivity of the composite creep rate was simply equal to that of the pure matrix. For the case of creeping fibres, such a simplification does not exist. If the fibres either begin or cease to creep at some intermediate composite stress, a change in stress exponent would necessarily follow due to the change in the carrying capacity of the fibres. This is shown schematically in Fig. 5.9. As for rigid fibres, a change in the exponent of the matrix itself would induce a similar change in the composite at a comparable creep rate.

5.5.6 Degeneration at Large ℓ/d and Small STAR

Allow ℓ/d to become very large and the fibre to creep over most of its length, then STAR is very small, or $3\epsilon/d \approx \ell/2d$. It then follows that the first term in the brackets of (5.27) for $\bar{\sigma}_f$ is small relative to the second term. Combining the remainder of (5.27) with (5.24) and (5.28), we obtain the familiar result,

$$\sigma_c = \sigma_{f0} V_f \left(\frac{\dot{\epsilon}_c}{\dot{\epsilon}_{f0}} \right)^{1/n} + \sigma_{m0} (1 - V_f) \left(\frac{\dot{\epsilon}_c}{\dot{\epsilon}_{m0}} \right)^{1/m} \quad \dots (5.29)$$

This is precisely the expression for the creep strength of a continuous fibre composite as formulated in Chapter III, equation (3.5).

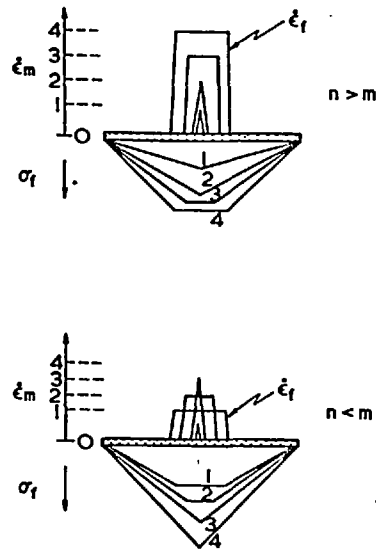


Fig. 5.7 Schematic illustration of changes in fibre stress and creep rate distributions with increasing matrix creep rate.

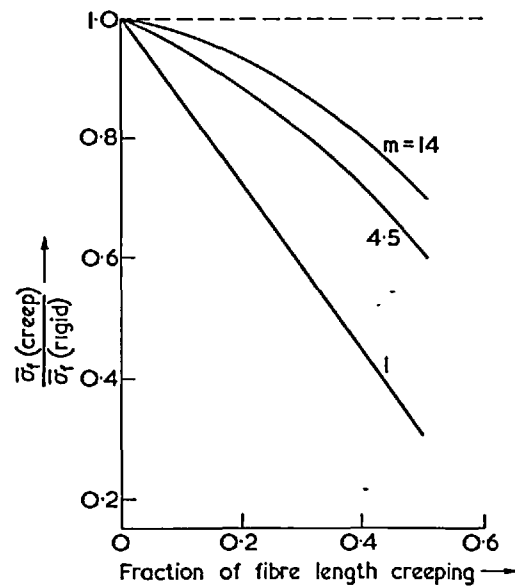


Fig. 5.8 Reduction in the average fibre stress due to an increasing fraction of fibre length undergoing creep.

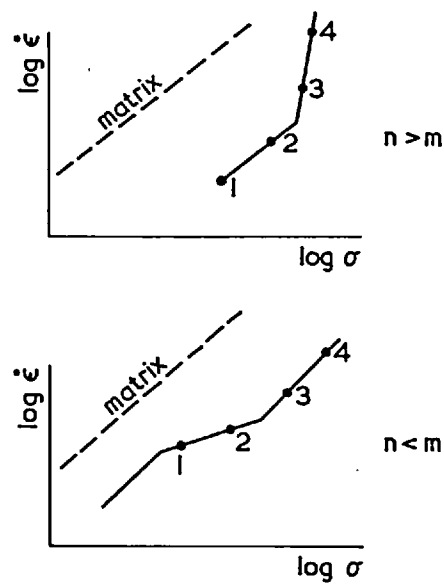


Fig. 5.9 Changes in composite stress sensitivity coinciding with Fig. 5.7.

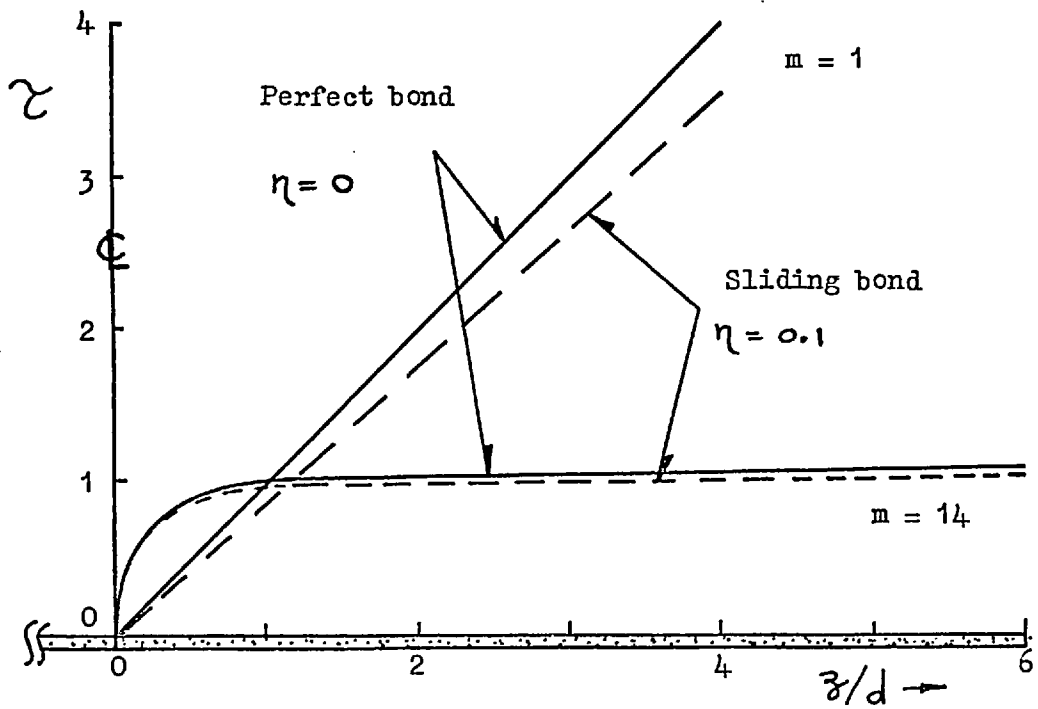


Fig. 5.10 Effect of a sliding interface on the shear stress profile along a fibre length.

5.6 Rigid Fibres, Sliding Interface

Once again, τ is obtained from (5.7) with $\beta c/d = 0$. However, a value for η is now required for a solution.

Shear stress profiles are plotted in Fig. 5.10 for $\eta = 0.1$ when $m = 1$ and $m = 14$. This value of η is considered to represent a moderate sliding velocity. It is seen that when m is large, sliding has a relatively small effect on the distribution and magnitude of τ . It follows that the σ_f distribution and hence σ_c will likewise be relatively insensitive to some sliding for large m values. If this is true, control of the interface strength (coatings, wettability, etc) is more important for low m matrices.

As sliding must be a specific characteristic of any one composite system, the development here is not carried any further. An experimentally measured value for η , possibly from a single fibre pullout test, would allow a creep strength prediction to be made using the present theory. Sliding in general may be more common to resin matrix composites than to metal matrix systems.

5.7 Comparison of Theory and Experiment

5.7.1 Pb/Phosphor Bronze System

Utilizing the data of Chapter IV, comparison between theory and experiment for composite creep rate as a function of stress is shown in Fig. 5.11. The rigid fibre prediction of (5.10) for $L/d = 100$ overestimates the creep strength by a considerable amount in qualitative agreement with the observation that the fibres themselves experienced creep. The data utilized are given in Table 5.1. However, the

prediction of minimum creep strength of Mileiko (1970) does not over-estimate the composite strength.

A precise calculation of the creep strength using the creeping fibre prediction (5.28) is not possible as the phosphor bronze fibres were characterized by logarithmic creep and not steady-state behaviour. Based on the measured creep rates and fibre stresses, the fibre characteristics are approximated by the dashed line shown in Fig. 5.11. With such an approximation, the prediction of (5.28) for creeping fibres is found to be in moderate agreement with experiment. The predicted composite stress sensitivity $n_c > m$ when $n > m$ appears to be in accordance with experiment.

Table 5.1

Pb/Phosphor Bronze Composite Data

$\sigma_{mo} = 3.24 \text{ MN/m}^2$	$\beta' = 0.55$
$\dot{\epsilon}_{mo} = 10^{-5} \text{ h}^{-1}$	$\phi = 0.5$
$m = 14$	$\ell/d = 100$
$\sigma_{fmax} \sim 170 \text{ MN/m}^2$	$V_f = (0.35-0.40)$

The approximated fibre behaviour is not unfounded. The strain-rate sensitivity of copper at ambient temperature is very low (Lubahn and Felgar, 1961) in accordance with a high stress sensitivity. Furthermore, the stress sensitivity of W fibres at low homologous temperatures was found to be quite large, $n = 43$ at $T/T_m = 0.25$ (Harris and Ellison, 1966).

The difference between the assumed value of σ_{fmax} in the creep fibre model and that calculated from the rigid fibre model, (5.8), for $\ell/d = 100$ is apparent from Fig. 5.12. However, for $\ell/d = 50$, the

difference was very small as shown below by the respective average fibre stress ratios.

$$\frac{\bar{l}}{\bar{d}} = 50 \quad \frac{\overline{\sigma_f}^{\text{creep}}}{\overline{\sigma_f}^{\text{rigid}}} = 0.98$$

$$\frac{\bar{l}}{\bar{d}} = 100 \quad \frac{\overline{\sigma_f}^{\text{creep}}}{\overline{\sigma_f}^{\text{rigid}}} = 0.61$$

Consequently, the rigid and creeping fibre predictions for $\bar{l}/\bar{d} = 50$ are almost superimposed in Fig. 5.11. Due to the lack of data points for $\bar{l}/\bar{d} = 50$, a rigorous comparison between theory and experiment is not possible. However, no major disagreement is apparent.

The calculated fibre stress distribution in the creeping fibres, Fig. 5.12, infers that any fracture of fibres should take place within the region of fibre undergoing creep ($\sigma_f = \sigma_{f_{max}}$ region). Taking the broken fibre aspect ratios $\bar{l}'/\bar{d}$ from creep specimens as measured earlier in Table 4.6 and marking them along the fibre length of Fig. 5.12, reasonably good agreement with this prediction exists. Although the $\bar{l}'/\bar{d}$ values are considered to be characteristic of the rupture zone alone, the above agreement is due to the fact that STAR from (5.24) is relatively insensitive to changes in $\dot{\epsilon}_m$ when m is large and $n > m$.

The apparent indifference in creep behaviour between specimens containing matrix precoated fibres and those with uncoated fibres is partially due to the scatter in the results. However, a more basic reason is believed to be associated with the finding of Section 5.6 that when m is large, the effect of interfacial sliding is minimized.

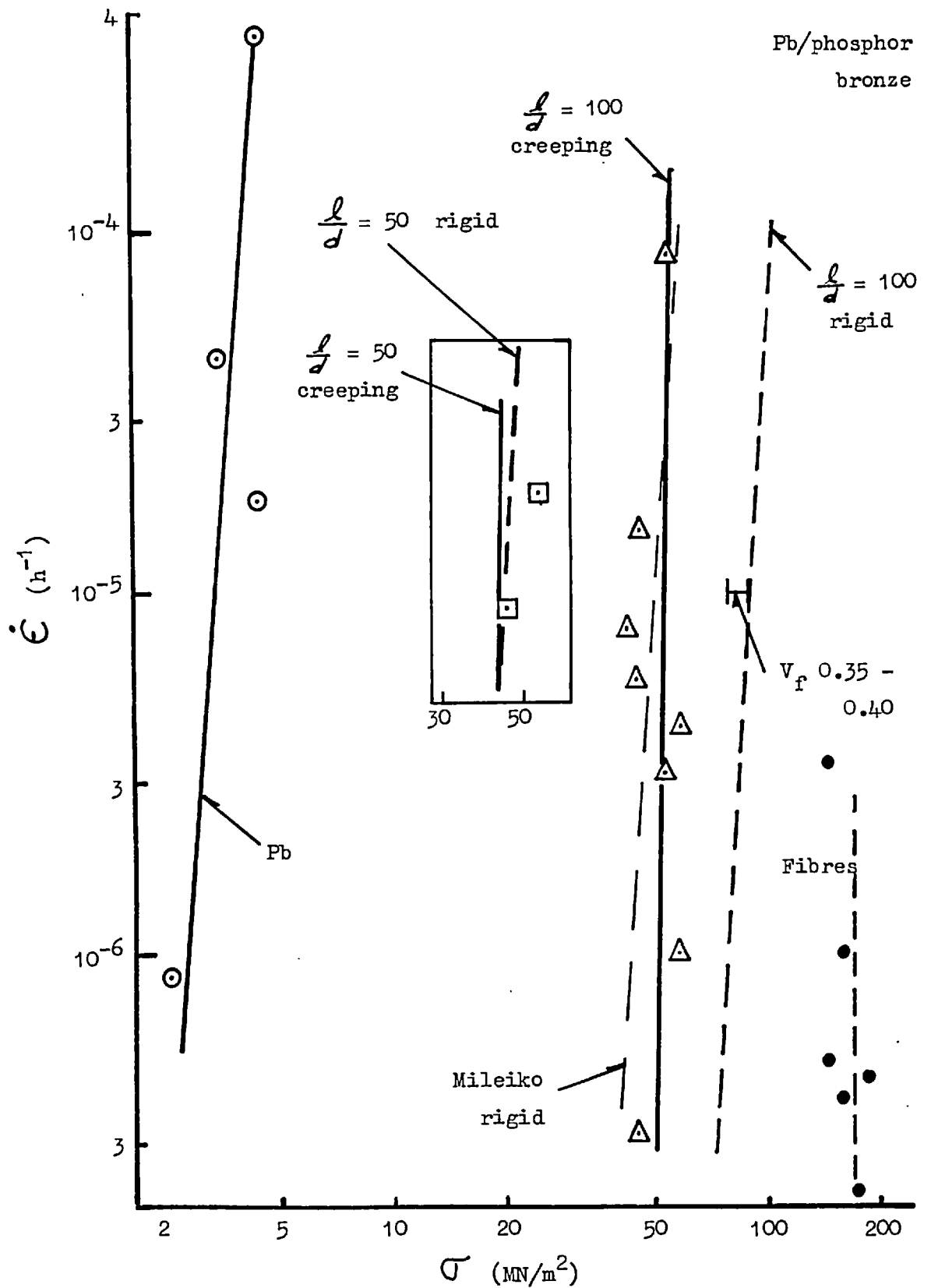


Fig. 5.11 Comparison of Pb/phosphor bronze steady-state creep rate data and theoretical predictions

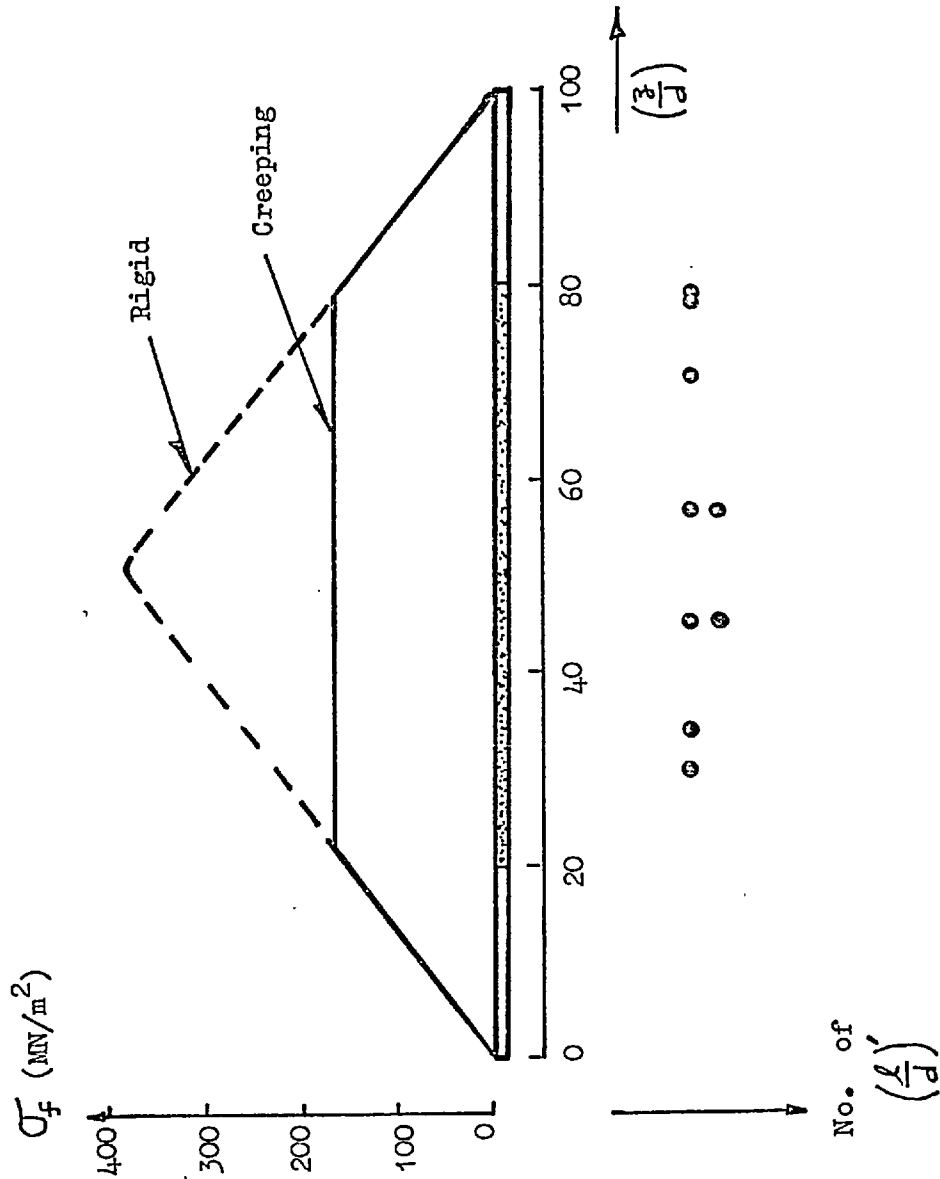


Fig. 5.12 Calculated stress distributions in phosphor bronze fibres, $l/d = 100$, from the rigid (Eq.(5.8)) and creeping (Eqs.(5.21 and (5.23)) fibre models. Appropriate data are given in Table 5.1. Also plotted along the abscissa are the experimentally measured broken fibre aspect ratios from Table 4.6.

5.7.2 Literature Data

An attempt was made to utilize as much of the available literature data as possible. Unfortunately, as all comparisons involved either data from other sources or unknown composite parameters, a detailed corroboration was not possible. Nevertheless, as described below, no major faults in the predictions became apparent.

Kelly and Tyson (1966) determined the behaviour of Ag/W composites (rigid fibres) of $l/d = 30$. The present theory provides a moderately good prediction of the 400°C data but over-estimates the creep strength at 600°C , Fig. 5.13. The latter may be due to either oxidation of the fibres as reported or to the lack of pure matrix data which necessitated using some pure Ag data of Price (1966). The trend between theory and experiment is in accordance with that expected from a grip shear contribution to the composite creep strain. Such a feature was reported to be the primary source of apparent creep strain in continuous fibre specimens. The theoretical prediction of Mileiko (1970) varies somewhat from that of the present theory.

A few experimental points from Miura and Honma (1967) for Al/W composites (rigid fibres) were compared with theory in Fig. 5.14. The authors report an approximate fibre aspect ratio of 15 - 25 (private communication) of which the minimum value was used in the theory. Although the matrix was not well characterized, the theoretical predictions are consistent with the few results.

A final comparison is made with the semi-quantative data of Mehan (1970) for Al/sapphire whisker composites (rigid fibres) creep tested at 500°C . Unfortunately, creep rates and l/d were not accurately

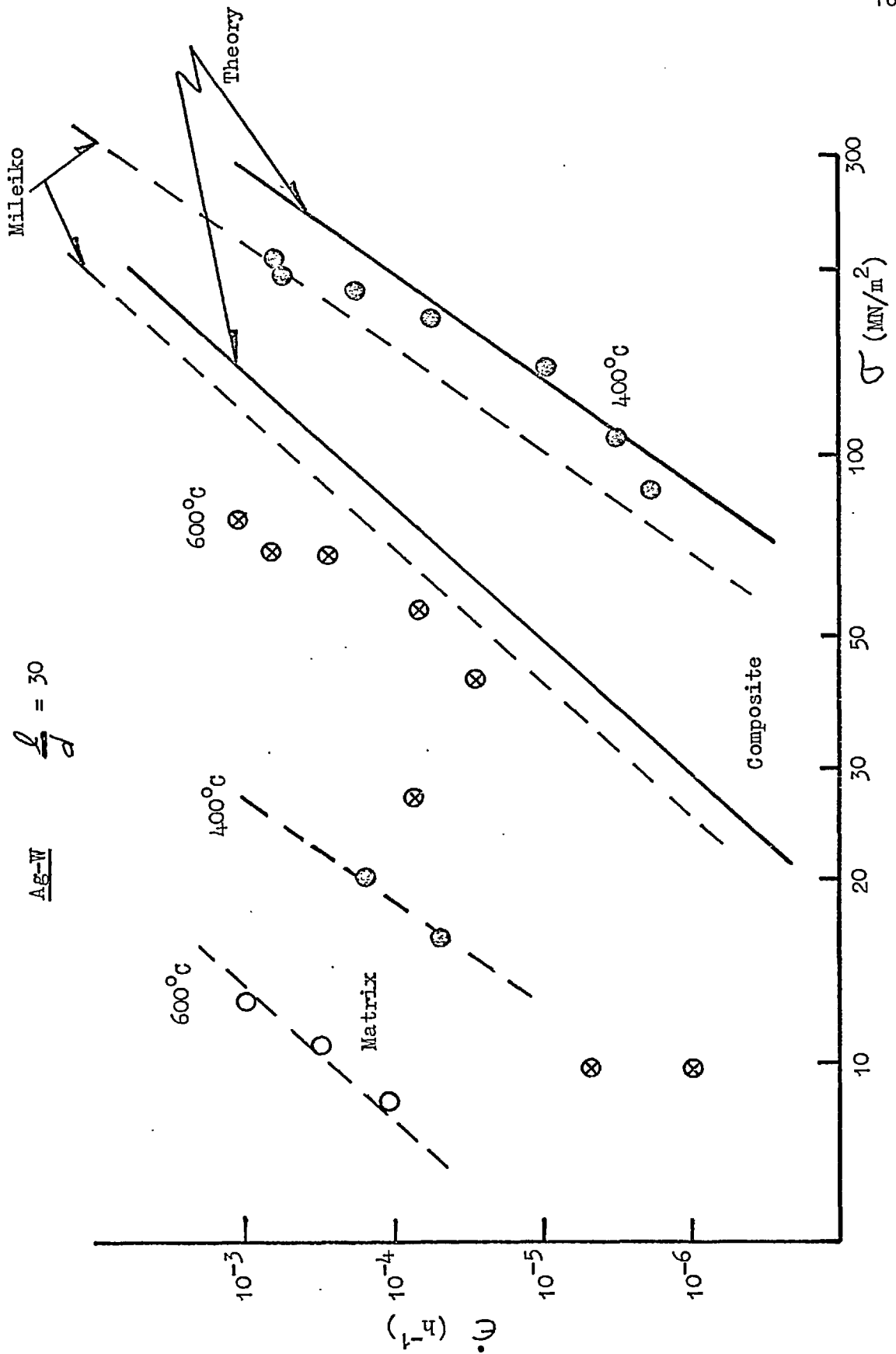


Fig. 5.13 Steady-state creep rate as a function of stress for Ag/W wire composites, Ag matrix and theoretical predictions for rigid fibre models.

measured, nor was the pure matrix characterized. The theoretical predictions are shown in Table 5.2. If the true whisker ℓ/d is 1500 (reported $\frac{\ell}{d} > 1000$), then theory and experiment are in agreement.

Table 5.2 : Al/Sapphire Whisker Composite Data

<u>Sapphire</u>	<u>Composite and Theory</u>
$\frac{\ell}{d} > 1000$	$\dot{\epsilon}_c \sim 5 \times 10^{-6} \text{ h}^{-1}$
	$V_f = 0.20$
<u>Al (McLean (1962))</u>	$\phi = 0.39$
$\sigma_{mo} = 0.3 \text{ MN/m}^2$	$\sigma_c \sim 83 \text{ MN/m}^2$
$\dot{\epsilon}_{mo} = 5 \times 10^{-6} \text{ h}^{-1}$	$\sigma_c(\text{theory})(\ell/d = 1000) = 47 \text{ MN/m}^2$
$m = 4$	$\sigma_c(\text{theory})(\ell/d = 1500) = 83 \text{ MN/m}^2$
$T = 500^\circ\text{C}$	

5.8 Extension to Short Time Tensile Behaviour

The purpose of this section is twofold. First, certain predictions of the creep theory will be compared with those of an existing tensile theory and also with tensile data. This is a logical development as basically a rate dependent strength theory should possess some similarities with a high strain-rate tensile theory. Consequently, indirect verification of the creep theory may be obtained.

Secondly, the creep theory will be extended to provide a prediction of some hitherto unpredicted tensile characteristics of discontinuous fibre composites. The extension and its predictions are not a requisite of the creep theory at all, but constitute an attempt to obtain additional useful predictions.

5.8.1 Tensile Strength

For $l/d > l_c/d$, the breaking stress of the composite for a given deformation rate can be obtained from (5.17). For the extremes we have,

$$\underline{m = 1} : \quad \sigma_c^* = \left(1 - \frac{l_c}{3l}\right) \sigma_f^* V_f + \sigma_m (1 - V_f) \quad \dots (5.30)$$

$$\underline{m \text{ (large)}} : \quad \sigma_c^* = \left(1 - \frac{l_c}{2l}\right) \sigma_f^* V_f + \sigma_m (1 - V_f) \quad \dots (5.30a)$$

In these expressions, l_c and σ_m are, of course, strain-rate dependent. It is immediately apparent that (5.30a) is precisely the expression for the composite ultimate tensile strength σ_c^* in the Kelly and Tyson (1965) tensile theory for the case of a linear build-up in fibre stress. As in the present theory, large m coincides with such a linear build-up, the agreement is not simply numerical. The present theory can now explicitly define their general parameter β ; from (5.17), $\beta = \frac{m+1}{2m+1}$.

The simplification of the creep theory to the tensile theory of Kelly and Tyson which itself was shown elsewhere to agree with numerous experimental results (typically m large) is supporting evidence for the present development. A duplication of comparison with experiment is not required here.

5.8.2 Critical Aspect Ratio

The creep theory predicts l_c/d from (5.16). At large m values, this equation becomes

$$\left(\frac{l_c}{d}\right)_{in\ large} = \frac{\sigma_f^*}{\sigma_{mo}} = \frac{\sigma_f^*}{2\tau_{mo}} \quad \dots (5.31)$$

This expression is again identical to that of Kelly and Tyson.

It is worth emphasizing another point which is associated with the dependence of l_c/d on $\dot{\epsilon}_m$ in (5.16). It can be expected that once tertiary creep or composite tensile fracture is initiated, with an ensuing increase in strain rate (especially in the localized fracture zone), l_c/d will decrease. Consequently, considerable fibre fracture may take place during this stage. If the method developed by Kelly and Tyson (1965) for determining l_c by observing the ratio of fractured to pulled-out fibres at the composite fracture surface is utilized, an underestimate of l_c would result. The importance of this point is clear in creep tests when it is realised that the local strain rates may increase by $\sim 10^6$ times from steady-state to rupture.

5.8.3 Secondary Composite Modulus

As virtually no attempt has been made to date to predict the secondary modulus E_{cII} (matrix fully plastic) of a discontinuous fibre composite, it seems worthwhile to investigate the possibility of placing the creeping fibre model on a strain (or time) dependent basis. To do so, we assume the following:-

- a) fibres elastic to fracture
- b) ideal plastic matrix at all strains.

The basis of the development is very simple. At any strain rate $\dot{\epsilon}_c$ (equal to $\dot{\epsilon}_m$), σ_{fmax} given by (5.22) is allowed to depend on the

accumulated strain ϵ_c (equal to $\dot{\epsilon}_c t$),

$$\sigma_{fmax} = E_f \epsilon_{fmax} = E_f (\dot{\epsilon}_c t) \quad \dots (5.32)$$

where strain equality $\epsilon_{fmax} = \epsilon_c$ is assumed; t is the time of application of the constant $\dot{\epsilon}_c$ and E_f the fibre modulus. Thus STAR also becomes strain dependent and by combining (5.23) with (5.32) we have

$$\left(\frac{l}{2d} - \frac{\sigma_c}{d} \right) \epsilon = \left[\frac{E_f \dot{\epsilon}_c t}{4 \beta' \left(\frac{m}{m+1} \right) \sigma_{m0}} \left(\frac{\dot{\epsilon}_{m0}}{\dot{\epsilon}_c} \right)^{1/m} \right]^{m/m+1} \quad \dots (5.33)$$

Therefore, STAR increases with increasing strain ($\dot{\epsilon}_c t$).

The expressions for $\bar{\sigma}_f$ and σ_c are now evaluated as in the creep theory, with the simple modification that at any strain (or time) (5.32) is valid.

The stress-strain relation for large m only is found to be

$$\sigma_c - \sigma_m (1 - V_f) = E_f V_f \epsilon_c - \frac{E_f^2 V_f \epsilon_c^2}{2 \sigma_{m0} l/d} \quad \dots (5.34)$$

It then follows that the secondary modulus is

$$E_{cII} = E_f V_f - \frac{E_f^2 V_f \epsilon_c}{2 \sigma_{m0} l/d} \quad \dots (5.35)$$

(m large)

The composite stress-strain curve in the secondary region is parabolic and E_{cII} falls off linearly with ϵ_c from a maximum value of $E_f V_f$. If the matrix tends to work-harden, the rate of fall off would decrease with increasing composite strain.

There does not appear to exist an accurate curve for a discontinuous fibre composite material. A comparison of E_{cII} is made between theory and a single measurement over a small strain range by McDanel's et al (1965), Fig. 5.15. If the given value for V_f is increased from 0.324 to 0.360, the theory bisects the experimental determination. Such an increase is not unfounded as V_f must vary slightly along the length of the specimen.

The relatively good agreement between theory and experiment was an unexpected result considering the simplifications involved in the calculation. Furthermore, consideration of the relation between $\sigma_{f \max}$ and $\bar{\sigma}_f$ and hence $\epsilon_{f \max}$ and $\bar{\epsilon}_f$ during elastic loading of a discontinuous fibre shows that a non-linear E_{cII} is not a surprising result.

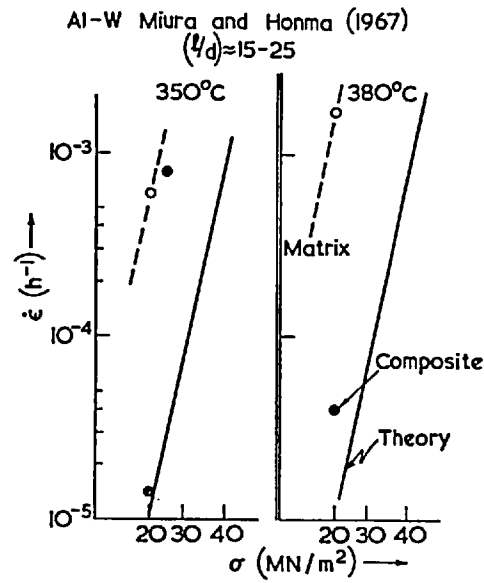


Fig. 5.14 Comparison of the rigid fibre theory with the data for Al-W composites.

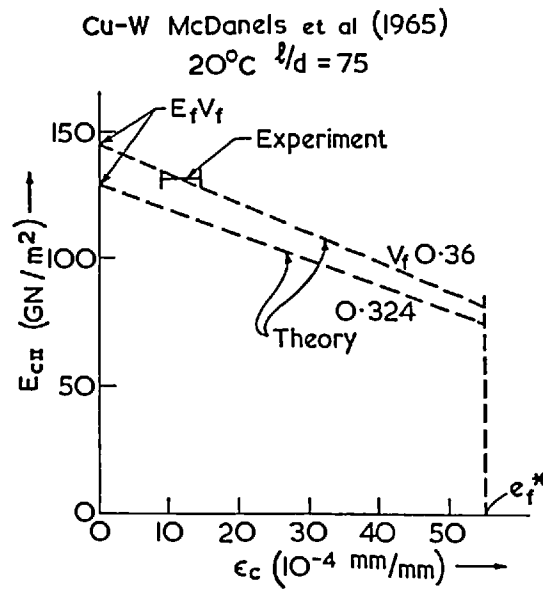


Fig. 5.15 Comparison of the theoretical secondary modulus with experimental data for Cu-W.

CHAPTER VI

SUMMARY AND CONCLUSIONS AND SUGGESTIONS

FOR FURTHER WORK

6.1 Al-Al₃Ni Continuous Fibre Composite

6.1.1 Summary and Conclusions

At 400°C, the basic creep behaviour of Al-Al₃Ni was characterized by a low and a high stress sensitivity of creep rate depending on the applied stress. The characteristics of the former were not accurately determined due to the masking effects of matrix grain boundaries and the smallness of the creep rates. As the whiskers are continuous, the composite behaviour must reflect the creep characteristics of the constituent phases with the stronger Al₃Ni phase more or less dictating the overall behaviour. Some confirmation for the presence of a low stress sensitivity regime in Al₃Ni was found in the basic behaviour of isolated whiskers at a higher temperature of 550°C.

At large stresses or creep rates, the composite stress sensitivity became relatively large. In this regime the effects of matrix grain boundaries were diminished. However, the discontinuous grains continued to induce inhomogeneous creep and ultimately lead to premature rupture of the composite.

The lack of control of the nature and number of matrix grains produced abnormally large scatter in both creep and rupture behaviour. It was proposed that such an origin was responsible for the findings of other workers.

Basically, aligned multicrystalline Al-Al₃Ni must function as a fibre composite on both a microscopic level (whisker-matrix) and a macroscopic level (matrix grain-matrix grain). The transfer of load from a continuous grain to a discontinuous grain necessitated a grain boundary shear stress of sufficient magnitude to facilitate the transfer. At elevated temperature under conditions of creep, the basic strength of the boundary was inadequate as evidenced by sliding. Much of the resistance to sliding at moderate temperatures appeared to be associated with the frictional resistance provided by protruding whisker ends.

The formulation developed for the creep strength of continuous fibre composites in terms of the known characteristics of the aligned phases was not rigorously proved. The assumption of no fibre/matrix interaction may prove to be less valid for creep behaviour due to the sensitivity of creep rate on stress. In particular, the small inter-fibre spacing in Al-Al₃Ni resulted in a creep strain or matrix stress constraint effect. Presumably, such a constraint would also modify the composite steady-state creep rate as the matrix would support a larger proportion of the axial composite stress than it would if the constraint was removed.

At and below 400°C, Al₃Ni whisker integrity did not appear to be influenced by thermomechanical instability effects for the times and stresses of interest in the present work. Accelerated annealing of the as-grown composite at 610°C induced substantial numbers of whisker ends, a feature which was thought not to occur in this composite. Qualitative evidence suggested that the rupture and possibly the creep strength of the composite at 550°C may have been influenced by instability.

Composite rupture was reflected in whisker rupture bands which coincided with regular matrix planes. Matrix grain boundaries in general tended to deter the formation of a single fibre rupture band throughout the entire cross-section of the composite. One may speculate that the rupture strength of a very fine columnar grained eutectic composite may in fact be superior to that of a monocrystalline composite. Hence, the simple extrapolation of the mechanical property improvements inherent in monocrystalline superalloys to eutectic fibre composites may be invalid.

6.1.2 Suggestions for Further Work

Various characteristics of isolated Al_3Ni whiskers such as creep, rupture and moduli must be obtained if quantitative composite theories are to be applied to the $\text{Al}-\text{Al}_3\text{Ni}$ eutectic. No doubt, this would be a formidable task. However, future work on whisker reinforced materials most certainly will require basic knowledge of the mechanisms controlling whisker properties in general.

The role of constraint effects in composite creep should be examined in more detail utilizing a composite of well-characterized fibres and matrix. The additional strengthening may prove most advantageous in certain composite applications.

The precise nature of localized thermal instability in eutectic composites under stress (or strained) requires amplification, particularly from a rupture standpoint. An in-situ microscopic approach utilizing high voltage electron microscopy would appear to be the most useful for quantitative assessment.

6.2 Pb/Phosphor Bronze Discontinuous Fibre Composite

6.2.1 Summary and Conclusions

The creep behaviour of the Pb/phosphor bronze fibre composite was found to depend on whether the fibres were continuous or discontinuous. When continuous fibres were present, the logarithmic creep of the fibres was reflected in the overall composite behaviour. However, for discontinuous fibres, the relative independence of matrix and fibres allowed the existence of a steady-state. In the extreme, such a regime has been found for rigid discontinuous fibres.

Due to variations in fibre volume fraction over the composite length, composite deformation was inherently unstable. The weak cross-sections were further magnified by the high stress sensitivity of the matrix. Considerable scatter in both creep and rupture characteristics was found to originate from this factor.

It follows that composite rupture was controlled by localized behaviour. The major portion of the rupture process was associated with interlaminar shear failure of the matrix which occurred over long periods of time. The presence of broken fibres at the rupture surface only was attributed to localized high strain rates which tended to strengthen the matrix and hence reduce the critical aspect ratio. The lack of correlation between composite behaviour and single fibre pullout results was in qualitative agreement with the existence of weak cross-sections.

A theory for the steady-state was developed for both rigid and creeping fibres. Based on an assumption of the form of the laminar

shear creep zone parallel to the fibres, the fibre stress distribution was calculated and subsequently the composite carrying capacity was obtained. Various design parameters were extracted from the theory. Under appropriate conditions, the discontinuous theory degenerated to that set down earlier for continuous fibre composites. Distinct similarities were found between the theory and available expressions for the viscosity of fluids containing inclusions. Also, for some conditions, the theory of Mileiko (1970) for rigid fibres only is similar to the present development.

The measured creep strengths of Pb/phosphor bronze were less than the predicted behaviour of the rigid fibre model. Qualitatively, this was explained by the fact that the fibres themselves experienced creep. Utilizing the creeping fibre model, reasonably good agreement was found between theory and experiment. In addition, no major disagreement was apparent between the theoretical predictions and available literature data.

A direct extension of the creep theory produced expressions for the tensile behaviour of a discontinuous fibre composite. At one limit, the predictions simplified to those of Kelly and Tyson (1965). The latter was shown elsewhere to agree with experiment.

6.2.2 Suggestions for Further Work

Utilizing the theory as a basis for design, additional studies should be performed on another composite mixture to provide a more complete comparison between theory and experiment. A range of aspect ratios, volume fractions and temperatures would test the limiting conditions of the theory.

A more simple indirect test of the theory could be obtained from tensile experiments on a composite with a low stress sensitivity matrix. The predicted secondary modulus could likewise be checked.

As the theory inferred that, in general, matrices with a stress sensitivity of unity were most efficiently reinforced, consideration should be given to fibre reinforced superplastic materials.

The basic problem of creep rupture of discontinuous fibre composites remains to be solved. Single fibre constant load pullout tests may be a useful model for composite behaviour.

Appendix I

Discussion of Literature Dataon Creep Characteristics of Al-Al₃Ni at 250°C

Salkind et al (1966) reported some creep results at constant stress for Al-Al₃Ni at 250°C which displayed an abnormally large scatter. No origin for the scatter was proposed. A second feature of the basic data was the proposed existence of a fibre spacing dependence of creep rate.

Reanalysis of their data revealed a relation of the form as shown in Fig. 1,

$$\dot{\epsilon}_{\min} = \dot{\epsilon}_{co} \left(\frac{t_r}{t_{ro}} \right)^{-r} \quad \dots (1)$$

The exponent $r = 1.5$ or 0.96 for $\sigma_c = 88.3$ or 103 MN/m^2 respectively. However, the specimens solidified at two different rates were characterized by the same relation. One conclusion that can be drawn is that the factor controlling the apparent spacing dependence of $\dot{\epsilon}_{\min}$ is simply the variable rupture strength.

Recently Breinan et al (1970) obtained a near identical set of results at 250°C as shown also in Fig. 1. He noted that relation (1) existed and that its form was the same as that found by Monkman and Grant (1965). However, this would appear to be purely fortuitous as the latter observation necessitated variable applied stresses. The independence of fibre spacing in (1) was not reported but is confirmed by their results, Fig. 1.

The possibility that the Al-Al₃Ni data represented a transient behaviour of the form

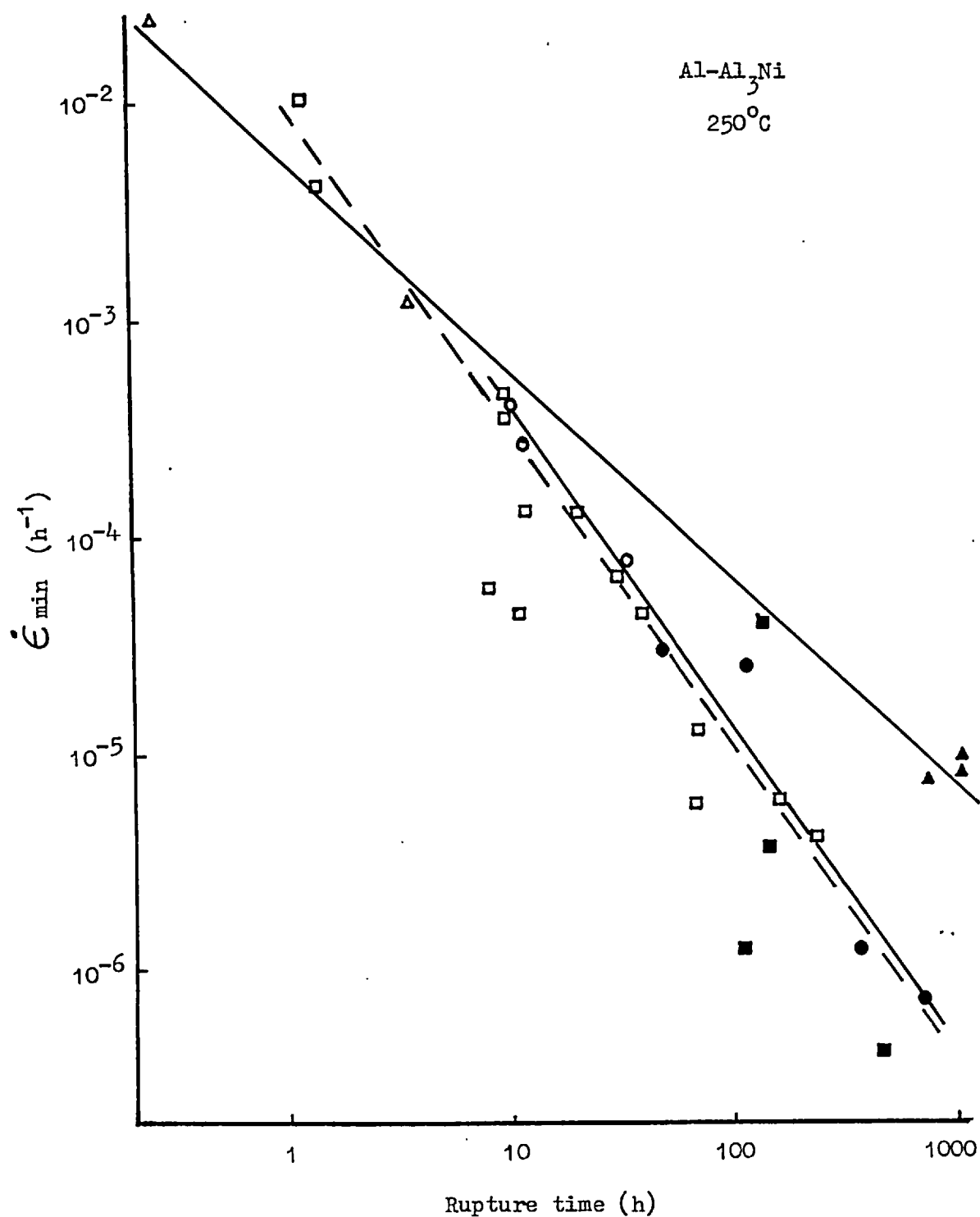
$$\epsilon = A'' t^q \quad \dots (2)$$

or

$$\dot{\epsilon} = q A'' t^{q-1} \quad \dots (3)$$

was examined. Although (3) would be linear in a double logarithmic plot of $\dot{\epsilon}$ against t , the slope must be $-1 < (q - 1) < 0$. This is not in accordance with Fig. 1. A second plot of some of Breinan's data where $\dot{\epsilon}_{min}$ was measured at prerule times is given in Fig. 2. The scatter is considerable but the slope is approximately -1. It is possible then that the period of tertiary creep distorted the data of Fig. 1.

The origin of the general scatter may be due to the role of matrix grain boundaries as described in Chapter II.



Line	Stress (MN/m ²)	R (cm/h)	R (cm/h)	Slope	Reference
—	88.3	○ 2	● 11	-1.5	Salkind (1966)
—	103	△ 2	▲ 11	-0.96	Salkind (1966)
---	93	□ 3.5	■ 11	-1.5	Breinan (1970)

Fig. 1 Minimum creep rate vs rupture time

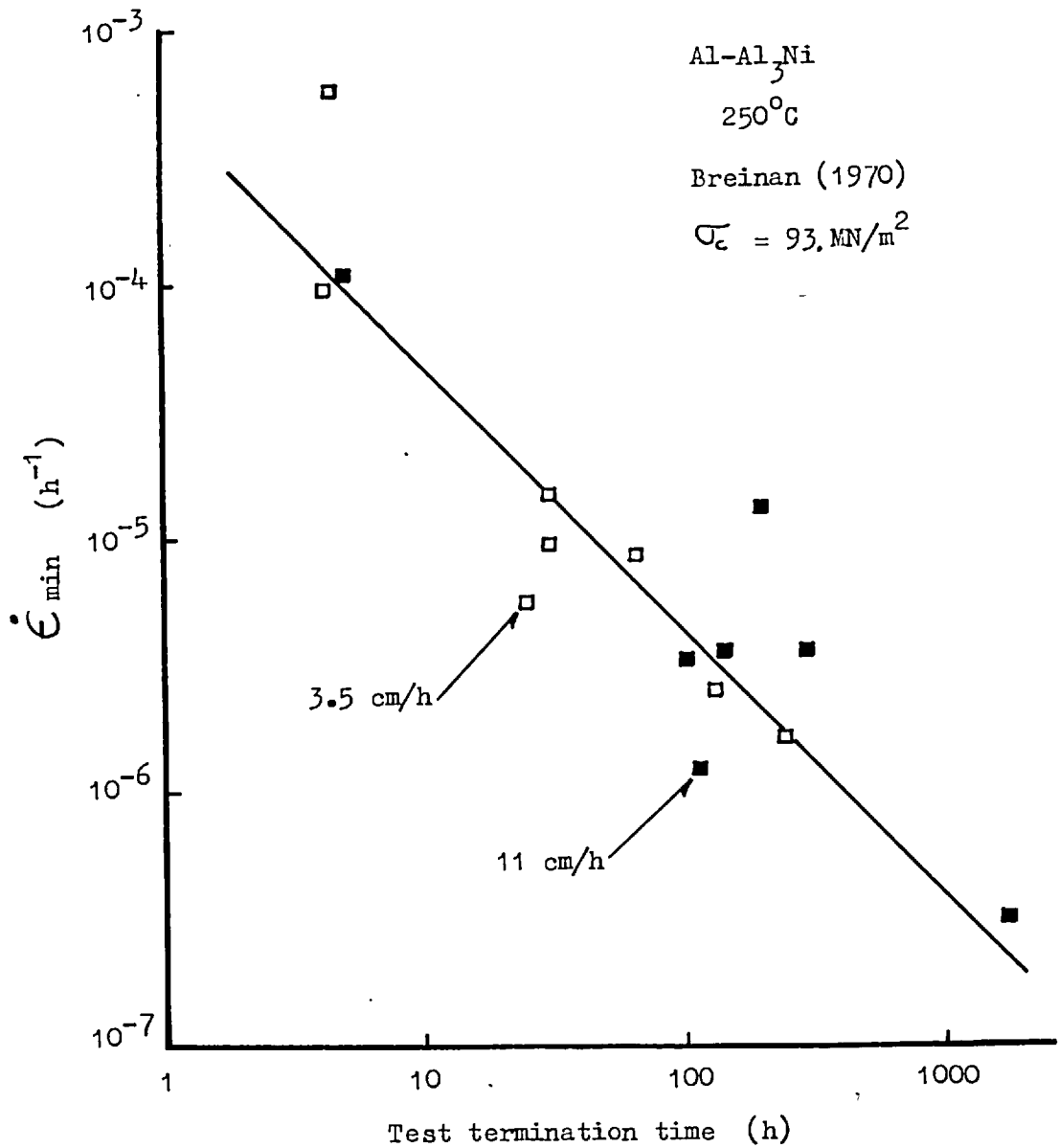


Fig. 2 Minimum creep rate vs time of termination of the creep test (specimens unbroken).

Appendix II

Temperature Dependence of Al-Al₃Ni and Al₃NiElastic Moduli

Because of the complexity of extracting whiskers and measuring their static modulus individually, the composite modulus was determined with the intention of utilizing the rule-of-mixtures formulation to calculate fibre properties. Incremental unloading at elevated temperatures generally allows determination of moderately true moduli (Lubahn and Felgar 1961). Such a procedure was carried out on a bicrystalline creep specimen over the temperature range 20°C - 550°C. The linear primary composite modulus E_{CI} is given in Fig. 1 along with the pure Al matrix modulus which was determined from the data of Sutton (1953). The modulus of Al₃Ni was calculated from the relation

$$E_{Al_3Ni} = \frac{E_{CI} - E_{Al}(1-V_f)}{V_f} \quad \dots (1)$$

and the values are plotted also in Fig. 1. At ambient temperature, the value obtained is close to that determined by Hertzberg et al (1965) on extracted whiskers.

Whereas the matrix data were linear over the entire temperature range, the composite moduli were found to be either constant or increasing slightly to about 100°C. This characteristic was observed on the cooling down cycle also. A similar result has been reported for the primary modulus of B-Al continuous fibre composite by Schaefer (1967). Alexander (1969) suggested this unique characteristic may be due to the development of a superior bond or relaxation of residual stresses in

the matrix. Al-Al₂Cu did not display this type of behaviour (Crossman 1969). The elastic behaviour of B fibres alone as a function of temperature does not appear to have been measured. Dickson et al (1969) observed a more normal monotonic variation of elastic constants of Ni₃Al up to 850°C. Consequently, although the present composite modulus may behave as measured, it would appear unlikely that the modulus of Al₃Ni increases with temperature as calculated. The origin of the findings is unknown.

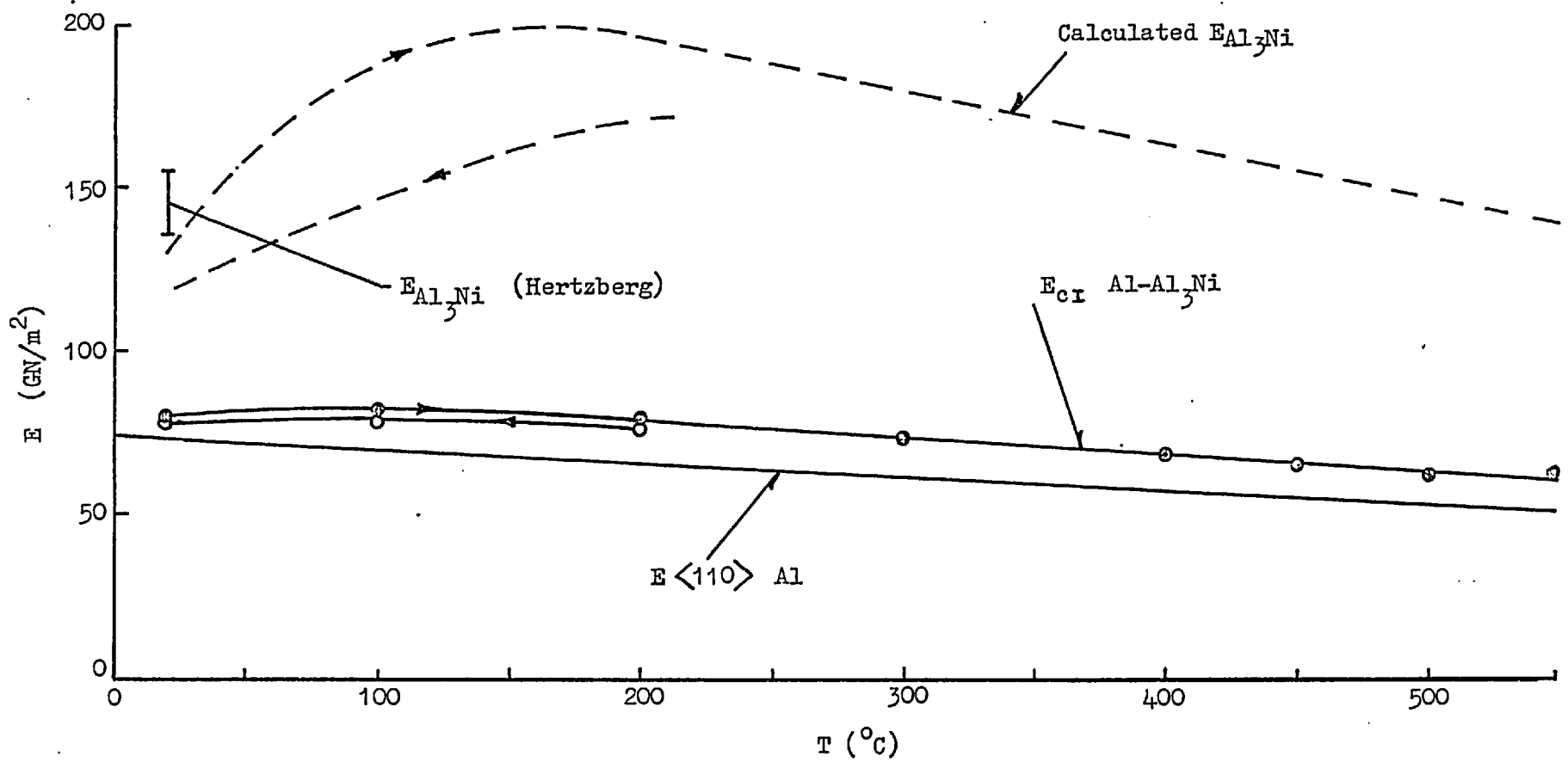


Fig. 1 Temperature dependence of elastic moduli for Al-Al $_3$ Ni, Al and Al $_3$ Ni (calculated).

Appendix III

Thermal Expansion Behaviour of Al-Al₃Ni

The expansion of two Al-Al₃Ni composite specimens, one with aligned whiskers (unidirectionally solidified) and one with quasi-random whiskers (normal solidification) was measured between 0°C - 575°C. The specimens consisted of cylindrical rods, 100 mm in length by 10 mm in diameter.

The results are given in Table I. From 0°C - 200°C, the average expansion coefficient $\bar{\alpha}$ for both aligned and quasi-random structures was $21 - 22 \times 10^{-6}$ per °C. This is comparable to the coefficient of polycrystalline pure Al, $23 - 25 \times 10^{-6}$ per °C (Hodgman, 1961).

For the aligned fibre specimen, a transition region was observed at 200 - 300°C, presumably due to matrix yielding. The random fibre specimen did not display a similar behaviour. At higher temperatures, the average coefficients were significantly different,

$$\begin{aligned}\bar{\alpha} \text{ (aligned)} &\sim 14 \times 10^{-6}/^{\circ}\text{C} \text{ for } 400\text{-}575^{\circ}\text{C} \\ \bar{\alpha} \text{ (random)} &\sim 28 \times 10^{-6}/^{\circ}\text{C} \text{ for } 450\text{-}550^{\circ}\text{C}.\end{aligned}$$

Following heating to the maximum temperature, both specimens exhibited a contraction in length at 0°C of $\sim 0.06 - 0.08\%$, evidence of plastic deformation during the test. Temperature cycling tests such as utilized in creep activation energy analyses may have to take into account the expansion characteristics of the composite.

Table I.

The Determination of the Linear Thermal Expansion of Two
Rods of Aluminium Composite Materials Al-Al₅Ni

Aligned		Quasirandom	
Temperature °C	Expansion mm	Temperature °C	Expansion mm
0	0.000	0	0.000
50.0	0.103	50.8	0.114
0	0.000	98.5	0.218
0	0.000	152.9	0.337
50.0	0.100	201.4	0.449
98.8	0.201	0	-0.004
153.2	0.325	0	0.000
199.8	0.427	224.8	0.489
0	-	250.4	0.534
0	0.000	301.8	0.636
50.2	0.100	348.6	0.732
99.2	0.202	397.6	0.841
144.3	0.304	451.2	0.972
200.4	0.428	499.9	1.104
0	0.003	549.1	1.256
0	0.000	574.5	1.348
200.9	0.429	0	-0.085
252.4	0.571		
298.9	0.640		
348.1	0.671		
399.6	0.739		
449.1	0.801		
498.8	0.874		
549.3	0.947		
574.2	0.986		
0	-0.058		
0	0.000		
257.5	0.563		
294.1	0.645		
402.4	0.798		
492.6	0.918		
0	-0.011		

Appendix IV

Kelly and Lilholt Tensile Constraint Data

Kelly and Lilholt (1969) working with single crystalline Cu/W continuous fibre composites calculated the plastic modulus of the in-situ matrix and found it to be considerably larger than that of the pure matrix. A unique feature of their specimens was the relatively small interfibre separations. No origin for the behaviour was evident. Their data are replotted in Fig. 1 in the form of calculated slope $(d\sigma'_m/de)_{\max}$ (from their Table 4) against elastic modulus or effectively orientation (from their Table 1 after matching V_f values). The regular behaviour shows that the slope is orientation dependent in roughly the same manner for both $d = 10 \mu\text{m}$ and $20 \mu\text{m}$ fibres. Also the data are in accordance with the relative orientation dependence of maximum strain hardening rate (stage II) in fcc crystals (McLean, 1962).

To ascertain whether the interfibre separation ($D - d$) itself is an important parameter, the composites are compared in Table 1 at equal values of ($D - d$) or equivalent fibre volume fractions V_f (calculated from equation (5.6)). It is apparent that a strong similarity in behaviour exists at equivalent V_f 's but not at equal V_f 's.

The evidence suggests that the basic strain hardening characteristics of the copper matrix within the composite have been enhanced by the presence of a small interfibre separation. The single crystallinity of the matrix may be an important feature. As Al-Al₃Ni is characterized by fibre separations which are an order of magnitude smaller than those of concern here, the matrix mechanical behaviour including creep may be similarly modified.

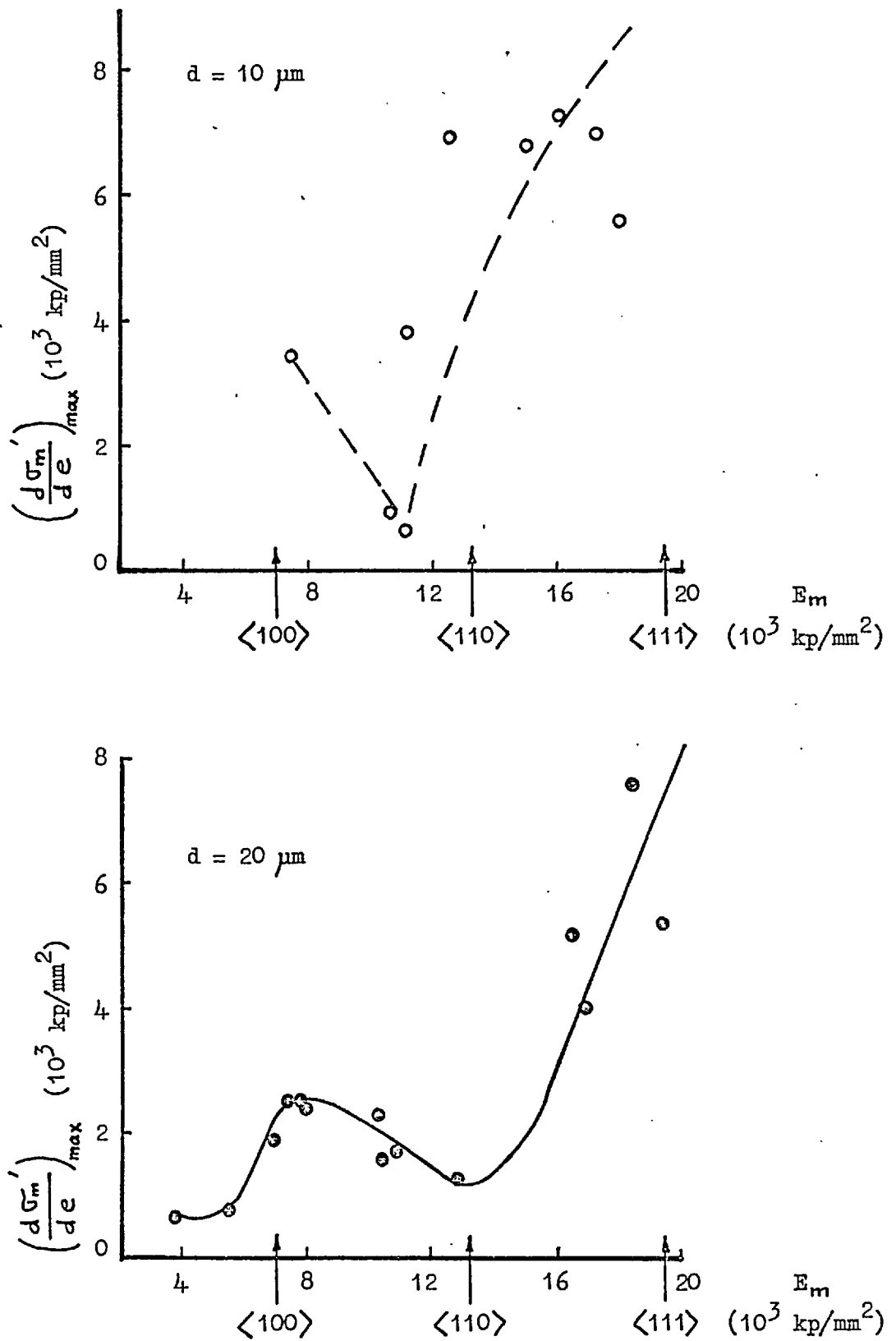


Fig. 1 Maximum calculated slope of stress-stress curve of matrix in composite vs matrix modulus for W/Cu. (after Kelly and Lilholt, 1969). ($1 \text{ kp/mm}^2 = 9.81 \text{ MN/m}^2$).

Table 1

Cu/W Data at Equivalent V_f 's

Matrix Orientation	V_f	E_m (10^3 kp/mm ²)	$\left(\frac{d\sigma_m'}{de}\right)_{\max}$	Equiv. V_f	Actual V_f	E_m (10^3 kp/mm ²)	$\left(\frac{d\sigma_m'}{de}\right)_{\max}$
~ $\langle 110 \rangle$	0.101	10.6	0.9 ± 0.6	0.22	0.208	12.8	1.3 ± 0.7
	0.103	11.1	0.6 ± 0.6				
~ $\langle 100 \rangle$	0.100	7.5	3.4 ± 0.7	0.22	0.183	6.9	2.4 ± 0.7
					0.191	7.9	2.5 ± 0.7
					0.285	7.7	2.5 ± 1.2
~ $\langle 111 \rangle$	0.211	17.9	5.6 ± 1.4	0.37	0.380	18.4	7.6 ± 2.0
					0.358	19.3	5.4 ± 1.7

10 μ m fibres20 μ m fibres

$$1 \text{ kp/mm}^2 = 9.81 \text{ MN/m}^2$$

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