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High-Strain Fatigue Studies of a Composite Material

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The reinforcement of metallic matrices with stronger, stiffer ceramic fibres is one approach which is currently being pursued in an effort to develop materials which will withstand the demanding conditions imposed by present-day engineering requirements. The gains in tensile strength which might be obtained by suitable combination of fibres and metal have been widely speculated upon [1] and, recently, experimental verification of some of these predictions has been obtained [2]. As yet, however, little attention has been directed towards assessing the limitations of such composite systems, isolating their causes and attempting to overcome them.

1. Introduction

Ideally, the modulus of the fibre should be as high as possible relative to that of the matrix, so that on loading, as the components are equally strained, the fibres carry the major part of the load on the system. The modulus ratio between the resin matrix and the glass fibres of fibre-reinforced plastics is generally of the order 10 to 20:1, ensuring that the proportion of load on the weak matrix is small. In the case of structural metals reinforced with ceramic fibres, modulus ratios of the order of 2:1 to 5:1 are more likely. However, in the case of reinforced metals, it is possible to transfer a greater proportion of the load to the fibres by allowing the matrix to deform plastically. In this way, pure aluminium has been reinforced with silica fibres, even though the moduli of both are 10×10^6 lb/in.^{2} (7×10^3 kg/mm^2) [3].

This mechanism of load transfer could impose severe limitations on the use of fibre-reinforced metals. Continued excursions into the plastic range, such as a component subjected to cyclic stressing would undergo, could have a marked effect on the fatigue properties. Even at the low stresses (± 9 ton/in.^{2} $= \pm 14.7$ kg/mm^2) already investigated [4], the matrix is in fact subjected to high levels of plastic strain, and cracks are formed in the matrix at endurances somewhat similar to those predicted by Coffin's [5] empirical relationship,

$$N_f = \sqrt{\left(\frac{\epsilon_f}{4\Delta\epsilon_p} \right)}$$

where N_f is the number of cycles of fatigue; $\Delta\epsilon_p$ is the plastic strain range in fatigue; ϵ_f is the elongation to fracture in $\frac{1}{4}$ cycle.

Fibre-reinforced metals may be superior to more conventional alloys in terms of stiffness/weight and strength/weight, but, if full advantage is to be taken of the high strength which can be obtained in fibres, these fibres must be loaded to high fractions of their breaking stress, which in general will correspond to $\sim 2\%$ elastic strain. Thus, an investigation of the fatigue properties of fibre-reinforced metals at strains of this order is necessary. It is quite apparent that, at these high strains, cracking of the matrix will occur after relatively few cycles, but for some applications this need not necessarily condemn the material. The matrix cracks formed at low cyclic strains were seen to be deflected by the fibre, and it is only if matrix cracks pass into the fibre at higher stress amplitudes, or if some other mode of failure emerges, that complete fracture of the composite will take place.

Early results on the aluminium-silica system showed that cyclic strain amplitudes of 0 to

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+1% did in fact produce failure of the material. Thus, this investigation is primarily concerned with determining the mechanism of failure of the strong fibres by high amplitude fatigue, and to this end the three possible sources of damage, i.e. matrix, interface, and fibre itself, have been studied.

2. Experimental Procedure

2.1. Composite Fabrication

Silica fibres, precoated with aluminium by a freezing process [6], were hot pressed to form solid composites. For this investigation, all composites were consolidated by a pressure of 6000 lb/in.^{2} (4.2 kg/mm^2) applied for 1 h at 450° C in an atmosphere of 5 torr hydrogen. Test pieces were then file-cut and polished to shape (fig. 1) for fatigue testing.

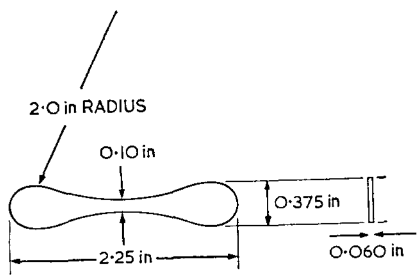


Figure 1 Composite specimen shape.

2.2. Composite Testing

Fatigue tests were carried out in the Instron tensile-testing machine, using the load-cycling device. All tests were carried out in "pull-pull" along the fibre direction, the minimum stress in all tests being 1 ton/in.^{2} (1.57 kg/mm^2).

2.3. Fibre Pretreatments

In an attempt to isolate the factors contributing to the failure process in the fibre, batches of fibres were tested:

(a) As coated; at room temperature; at 400° C; and after soaking at 500° C.

(b) After the following treatments, applied subsequent to the coating process, fibres were tested at room temperature only.

Metal removal Fibres were etched in a solution of 25 ml H_2SO_4, 70 ml H_3PO_4, and 5 ml HNO_3, at 85° C, to remove the aluminium coating.

Simulation of compositing conditions Batches of fibre were subjected to the environmental and physical conditions used during composite manufacture. The effects of both the temperature, 450° C, and the pressure, 6000 lb/in.^{2} (4.2 kg/mm^2), were determined independently, along with the effect of the combination of temperature and pressure. To prevent consolidation of the fibres, these treatments were carried out on fibre embedded in tungsten disulphide powder. This powder was chosen because of its low hardness and its stability under the conditions used.

(c) In addition to fibre coated by the conventional process, the following modifications were made to the coating process to influence the interfacial conditions.

(*i*) In an effort to modify the interaction between aluminium and silica [7], fibre was coated with alloys containing various percentages of bismuth.

(*ii*) Fibre was coated with an alloy inoculated with 5% Al_2O_3 powder in an attempt to increase the foreign particle concentration at the interface.

(*iii*) Fibres were precoated with a thin graphite layer prior to coating with the usual aluminium alloy.

2.4. Fibre Testing

Fibres were tested after the various treatments either in tension in the Goodbrand thread-testing machine, or in fatigue in the apparatus shown in fig. 2. In this apparatus, an oscillating load A was applied to the fibre B, in an approximately square-wave form, by the cam rod D activated by the cam C. A count of the cycles was kept by a pulse counter, activated by a microswitch on the cam rod, which was depressed by the load A. A similar device was used for high temperature testing; the only difference being that the fibres were stressed horizontally within a furnace.

In order to keep the grip failures to a minimum, for room temperature testing, the fibre ends were potted in glass tubes with araldite, as shown in fig. 2 (inset), while for tests at 400° C, the ends were held with high temperature resin.

Fatigue and tensile stresses were based on the mean silica cross-section, calculated from measurements of the mean fibre diameters on a cross-section through a composite specimen. In an average cross-section, fibre areas varied within ±10% of the mean.

In addition to tensile and fatigue tests to destruction, some fibre batches were tensile tested after fatigue stressing, and others tested

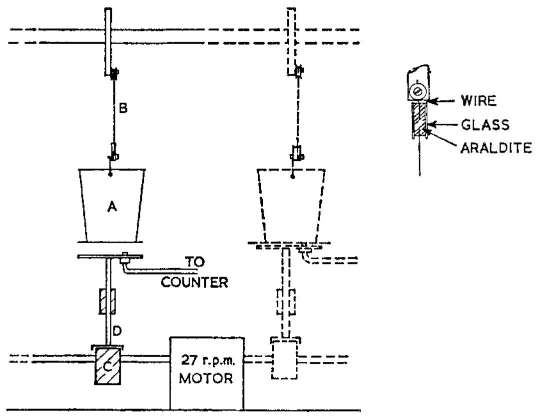


Figure 2 Fibre fatigue apparatus.

in fatigue at room temperature after prior fatigue at high temperature.

2.5. Metallographic Observations on Fibres

Surface and subsurface observations were made on fatigued fibres. Subsurface observations were made on taper sections through fibres mounted in a cold-setting plastic. Owing to the small size and the hardness difference between the components of the fibre, great care was required during polishing, to avoid pulling the fibres out of the mount and producing flaws at the glass-metal interface.

3. Experimental Results

3.1. Composite Strengths

The results of fatigue tests at various stress amplitudes are plotted in fig. 3. Owing to the scatter in the lives obtained in these tests, the *S-N* curves are drawn through the points representing 25%, 50%, 75%, and 100% failures. This plot demonstrates quite clearly that there is a fatigue effect in the composite material.

3.2. Fibre Strengths

3.2.1. Properties of Silica Fibres

Attempts to produce fatigue failure at a stress amplitude of 0 to 60 ton/in.^{2} (0 to 94 kg/mm^2) in the fibre, from which the aluminium had been completely removed, were unsuccessful. Only one instance of gauge length failure in these fibres was observed.

The fatigue and tensile strengths obtained for these fibres are included in table I, along with similar results for coated fibres. The effect of etching the aluminium from the surface does not produce an inherent strengthening of the silica due to smoothing the surface, as in the case of etching in hydrofluoric acid [8].

3.2.2. Coated Fibres

The effect of stress range on the life of the

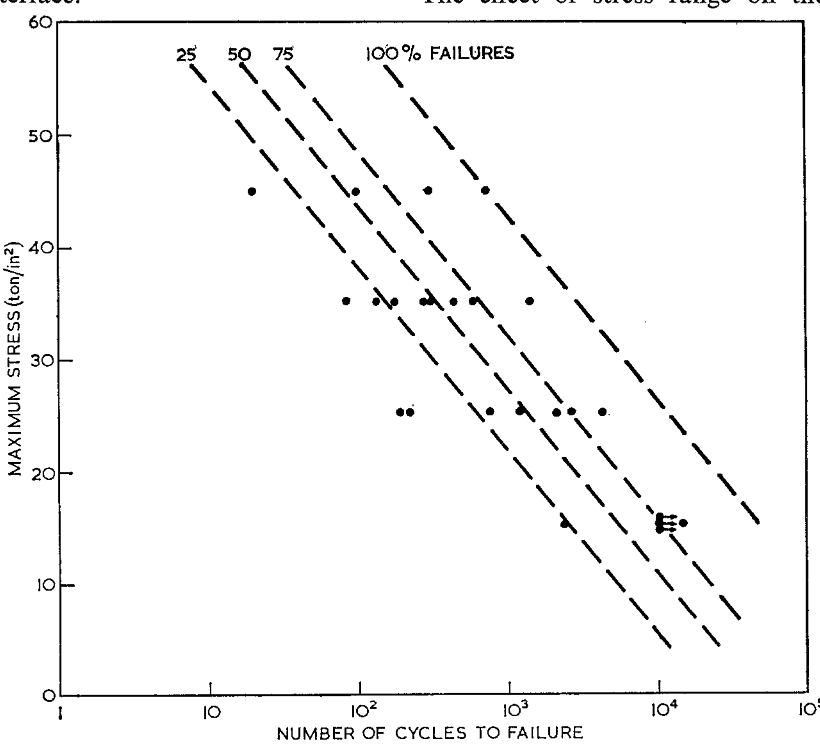


Figure 3 The relationship between stress and number of cycles to failure of composite specimens (fibre batch 149).

TABLE I Etched fibre. Fatigued at 60 ton/in.^{2}

Fibre batch number	Mean life cycles			Mean breaking stress (ton/in.^{2})	
	Unetched	Etched		Unetched	Etched
		Gauge length failures	Junction failures		
127	913	No failures	1142	230	78
133	659	No failures	2509	212	Not tested
137	3519	3375 (1 result)	6767	388	Not tested

coated fibres in repeated tension (i.e. 0 to $+\sigma$) is plotted in fig. 4. The usual dependence between stress and number of cycles to failure is shown by the full line in this figure.

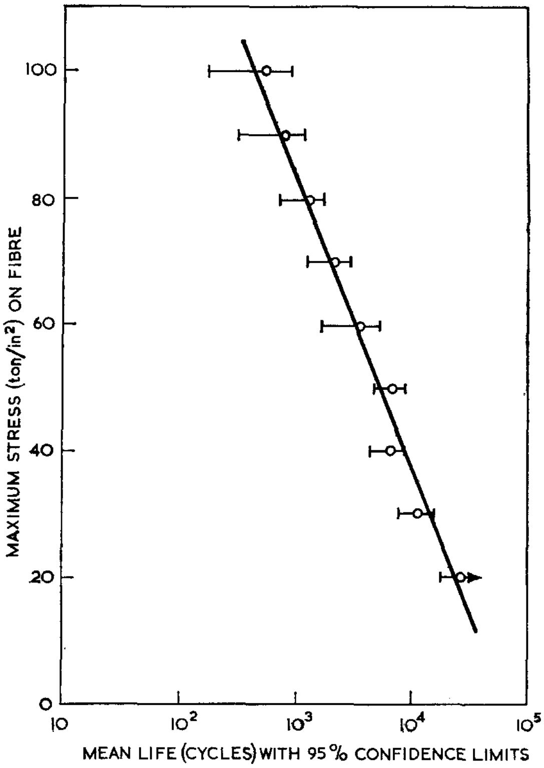


Figure 4 The relationship between stress and the mean life of individual fibres (fibre batch 137).

The progressive nature of the damage resulting from fatigue stressing is demonstrated in fig. 5. This shows that the tensile strengths of fibres after cycling lie within an envelope between the fatigue stress and a maximum value which decreases with increased number of cycles, between 0 and 52 ton/in.^{2} (0 to 82 kg/mm^2).

The fall-off in tensile strength with increasing numbers of cycles (N) of prestress is most noticeable with low values of N, and virtually ceases as N increases. This form of relationship is not surprising, since the higher N values of prestressing in fatigue overlap with the scatter in lives of fibres at the fatigue stress. Thus, in any pretreatment, only the stronger fibres survive the full N cycles of fatigue to be tested in tension. Furthermore, as the severity of the pretreatment continues, the process naturally selects stronger and stronger fibres. If it is assumed, as is quite likely, that fibres strong in tension will be those that are also strong under fatigue stressing, it is hardly surprising that the true content of the damaging process is hidden at higher N values.

The fatigue properties at 400° C were superior to those of fibre tested under similar conditions at room temperature (fig. 6). However, fatigue at these high temperatures resulted in catastrophic failure when attempts were made subsequently to fatigue these fibres at room temperature. Soaking at 400° C for a similar period of time had little effect on room temperature fatigue properties; although soaking at 500° C for 1 h gave a reduction in fatigue and tensile properties.

3.3. Effect of Manufacturing Variables on Fibre Strength

The effect of composite manufacturing conditions, i.e. pressure and temperature, when applied separately, and concurrently, on fibre tensile and fatigue strengths are shown in table II.

Three points emerge from this table.

(a) The application of temperature alone causes a slight reduction in the mean fibre fatigue life and an appreciable reduction in mean tensile strength.

(b) Although pressure alone causes fibre breakage, the tensile and fatigue strengths of the fibre remaining unbroken show an increase over the untreated fibre. This apparent anomaly suggests that pressure has a slight tendency to break weaker fibres.

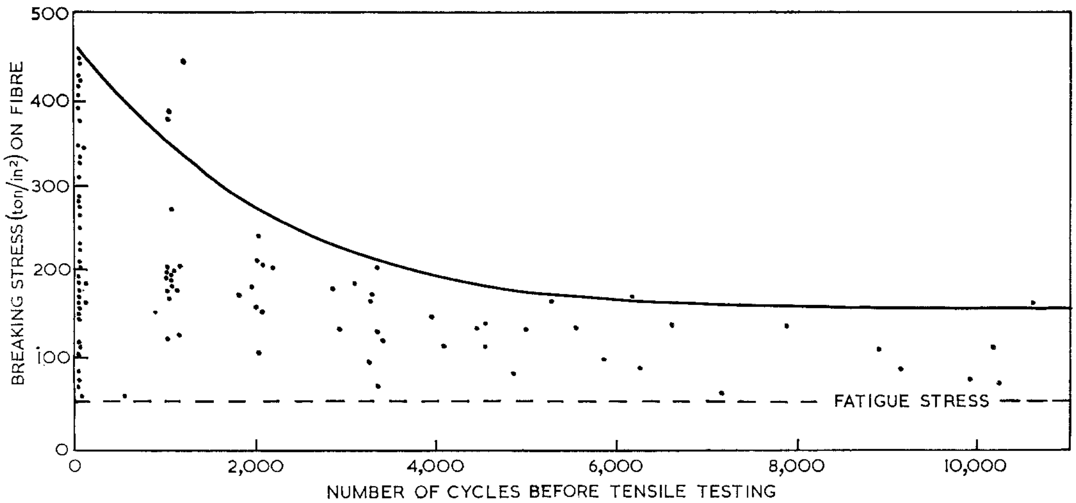


Figure 5 The relationship between fibre breaking stress and number of cycles of prestressing (fibre batch 146).

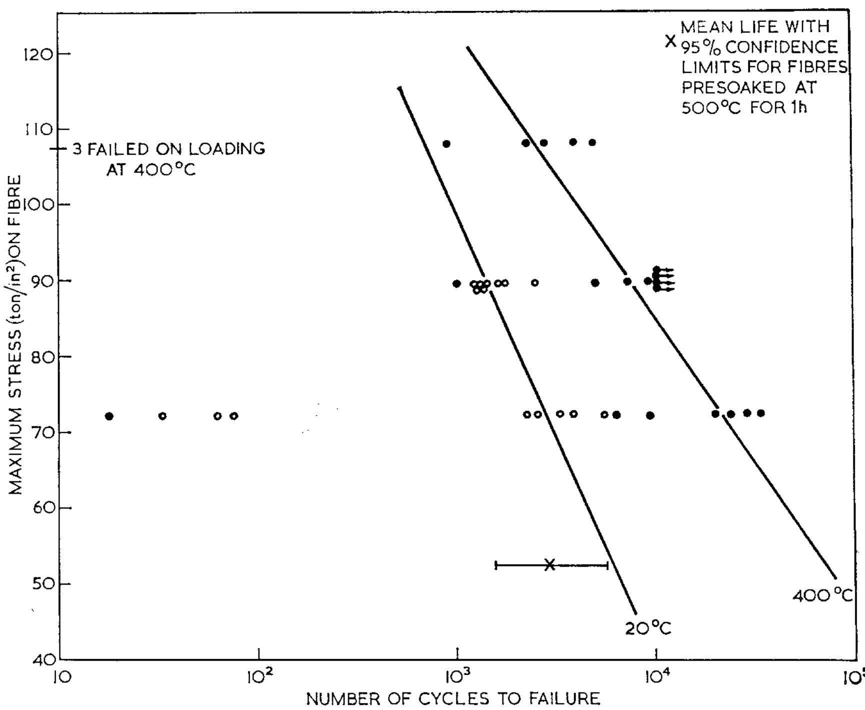


Figure 6 The relationship between stress and number of cycles to failure for individual fibres fatigued at room temperature and at 400° C (fibre batch 146).

(c) The combined application of temperature and pressure results in a marked reduction in both tensile and fatigue properties of fibre remaining unbroken after the treatment. This reduction is more pronounced if this comparison is made against the fibres subjected to pressure alone, i.e. the batch similarly up-graded by the fracture of the weaker fibres.

3.4. Modifications to the Coating Process

Fibre strengths produced by the three modified coating processes are shown in table III. The addition of inhibitor to the alloy produced no significant changes in mechanical properties of the fibre, nor were any differences in the interfacial structure resolved by optical microscopy. It is not possible to draw any definite conclusion

TABLE II Effect of manufacturing conditions on fibre batch 146. Fatigued at 60 ton/in.^{2}

Treatment	% broken fibres after treatment	Mean fatigue life of unbroken fibres (cycles)	Mean breaking stress of fibres unbroken (ton/in.^{2})
Untreated	—	3750	282
450° C for 1 h in powder	0	3300	198
6000 lb/in.^{2} for 1 h in powder	10	4600	334
450° C for 1 h at 6000 lb/in.^{2} in powder	30	2650	186

TABLE III Effect of modified coating processes on fatigue life and tensile strength. The fibres were fatigue tested at 60 ton/in.^{2}

Batch number	Fibre	Mean life (cycles)	Mean breaking stress (ton/in.^{2})
1085	0% Bi	1803	282
133	0.005% Bi	659	212
137		3519	388
127	0.01% Bi	1106	256
316	0.03% Bi	2024	226
844		647	336
845		576	444
250	0.1% Bi	2647	340
251		2441	366
252		1893	308
A	Standard	3011	270
B	5% Al_2O_3	617	206
651	Carbon barrier	> 20000	292

from the attempted inoculation with alumina particles.

However, the application of the carbon film between the silica and the matrix produced marked improvement in the fatigue characteristics of the fibres.

3.5. Metallographic Examination

3.5.1. Interfacial Conditions

Attempts were made to examine the interfacial regions between fibre and matrix for the presence of particles, by both electron and optical microscopy. However, owing to difficulties in specimen preparation, no unambiguous evidence of particles could be observed.

3.5.2. Fatigue Behaviour

The surfaces of fibres after fatigue were seen to be heavily deformed, and extrusion-intrusion pairs, such as that in fig. 7a, were observed. In fibres tested at high temperature, large voids and cracks were widespread in the matrix (fig. 7b). These micrographs indicate that matrix cracks can exist in contact with the fibre without causing fibre damage.

Although fibres with the metal completely removed were not subject to fatigue failure, those in which the metal had not been completely dissolved did fail in fatigue. Metallographic examination of these fibres showed that failure always occurred at the point where the aluminium remained adhering to the fibre (fig. 8).

4. Discussion

4.1. Source of Damage

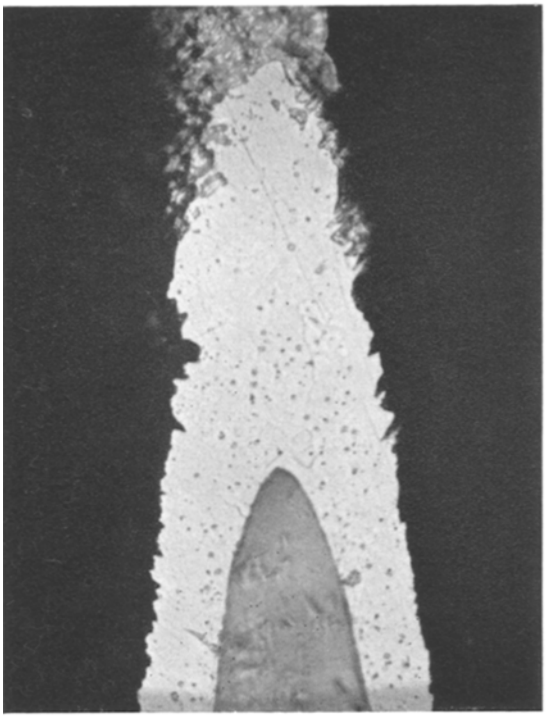
It is clear, from the tests on the fibres with coatings completely removed, that silica fibres themselves definitely do not suffer inherently from a fatigue mechanism. This observation confirms previous work in this laboratory [9], and the conclusions reached by Gurney and Pearson [10].

Once the fibres are coated with metal, there is a definite cyclic damaging effect which at any stress level increases as the number of cycles applied increases, and eventually leads to failure of the fibre. Further evidence that the damaging process is associated with the presence of the metal on the fibre arises from the fact that, in the case of incomplete removal of metal, failure always occurred at the point of adherence of the remaining metal.

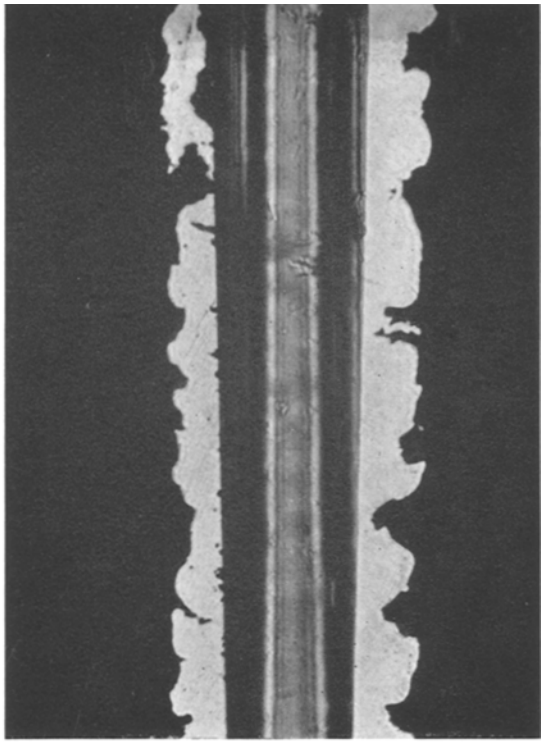
The damage produced within the metal matrix by fatigue stressing the composite has already been demonstrated, and the model used to explain the behaviour should apply to the behaviour of individual fibres [11].

The possibility of a matrix crack extending until it meets a fibre and, despite elastic moduli and surface tension differences, producing in the fibre a stress concentration of sufficient magnitude to fracture it has been postulated for the case of whisker-reinforced metals [12]. From the metallographic evidence to date, there is no reason to believe that mechanism is operative either in the aluminium-coated silica fibres or in the composite made from these fibres.

The one possible remaining source of damage is the interface between the fibre and matrix. Movement, during cyclic loading, of hard



(a)



(b)

Figure 7(a) Taper section of a fibre showing superficial surface damage in the aluminium. (b) Taper section of a fibre showing large voids and cracks in the matrix. (× 450)

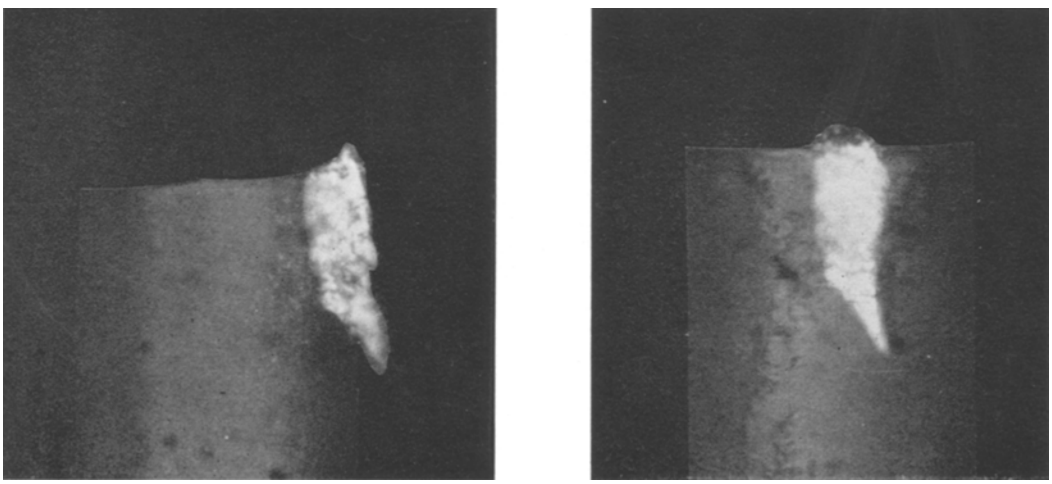


Figure 8 Micrograph showing residual aluminium at the fracture point of an etched fibre. (× 750)

particles at the interface, relative to the fibre, could cause abrasion of the fibre surface resulting in severe degradation in strength. Interaction between the fibre and the metal during the coating process is one possible means whereby hard particles could be located at the interface. The fact that the damaging process is accelerated in fibres which have been soaked at temperatures where chemical interaction can occur [13] lends support to the hard-particle hypothesis. The temperature at which damage occurs is lowered by the application of cyclic stressing. A second possible source of hard particles may be inclusions present in the coating alloy, such as alumina particles carried in from the surface of the coating meniscus.

Unfortunately, although hard particles appear to be the most likely source of damage, the evidence is largely circumstantial, as attempts to observe particles at the interface by optical and electron microscopy were unsuccessful. However, no significant changes in fatigue strengths were observed after attempts to inhibit interaction. Innoculation of particles of Al_2O_3 may have produced changes in fatigue strength but, if so, these could arise as a consequence of the lower tensile strengths after this treatment.

The one direct piece of evidence suggesting that damage on the surface of the fibre is responsible for the fatigue process is that, by applying a mechanical and a chemical barrier between the fibre and metal, the fatigue properties of the fibres are markedly improved. In this case, a graphite layer of a few Ångströms thick was sufficient to provide the necessary barrier.

4.2. Nature of Damaging Process

Indirect evidence suggests that fatigue damage is due to relative to-and-fro motion of hard particles at the fibre-matrix interfaces. These particles may be foreign inclusions picked up during the coating process, but it is most likely that they originate from reaction between the fibre and matrix and are therefore silicon and alumina. The movement of these particles results in progressive surface damage to the fibre. When this damage is such that the critical crack length is exceeded for the fatigue stress, failure occurs.

Movement of the particles is associated with cyclic plastic deformation of the matrix. A simple model for the production of particle movement may be visualised in terms of dislocation theory. During a portion of the strain cycle, slip occurs, and dislocation groups pile up against the obstacle; the build-up in stress at the tip of the pile-up causes the particle to move away. On reversing the direction of strain, the particle moves back to its equilibrium position, and is then pushed in the other direction as reversed slip occurs owing to the compression produced on the matrix by the fibre [11]. The resulting dislocation pile-up in the opposite direction causes the particle to continue its motion in the reversed direction.

On this basis, the application of high temperature under these conditions may be expected to have two effects. The growth of particles at the interface would be facilitated by increased atomic mobility within the metal matrix. The pile-up of dislocations at any significant stress may not occur, as relief mechanisms such as climb of edge dislocations or cross-slip of screw dislocations seem more feasible. In fact, the evidence suggests that both these mechanisms occur: the high temperature fatigue properties are somewhat superior to those at room temperature, although on subsequent testing at low temperature the fibres were observed to be severely damaged.

Although this mechanism will not necessarily occur in all fibre-reinforced metals, owing to the varying nature of the interfacial regions in different systems, it is possible wherever interaction between components may occur. As the process is strain controlled, increase in modulus of the fibre should enhance fatigue properties.

4.3. High Amplitude Fatigue Failure of the Composite compared to the Fibres

From the experiments on single fibres and the composite, it would appear that failure of the composite material occurs by the linkage of matrix cracks via fractured fibres. It has been demonstrated that fibres are fractured by the cyclic stressing, and these failures will undoubtedly play an important role in the fatigue process of the composite material. However, it is equally true that considerable fibre fracture occurs during composite fabrication, particularly at points where fibres cross over each other owing to slight misalignment [14]. Because of the cushioning effect of the soft embedding powder, this type of damage will not occur in the attempts to *simulate* manufacturing conditions. In order, therefore, to improve the properties of the composite, not only must the high amplitude fatigue damage to the fibres be avoided, but more sophisticated manufacturing techniques will have to be developed to minimise fibre fracture during manufacture.

A comparison of the lives endured by the fibres after being subjected to manufacturing conditions (450° C, 1 h, 6000 lb/in.^{2}), assuming a 50/50 Al:SiO_2 ratio, against those of the composite material is shown in fig. 9a, with 95% confidence limits. The line indicating the lives

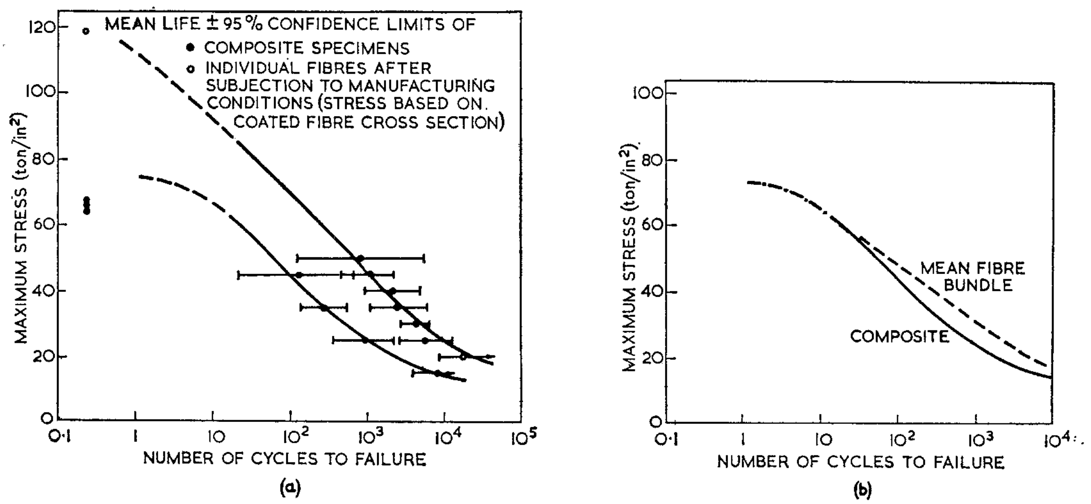


Figure 9 A comparison between the fatigue properties of composite specimens and: (a) individual fibres; (b) a mean fibre bundle (fibre batch 149).

of the fibres is based on the results of the fibres which remained unbroken after the pretreatment. The dotted line in fig. 9b is an estimation of the life of a bundle of fibres assuming: (i) that within the bundle there will be 30% broken fibres (as found after the treatment) which will take no load; and (ii) that all fibres break at the mean life indicated by the full line. This of course means that the stress on the remaining fibres is increased and the mean life reduced. If bundle effects, similar to these discussed by Coleman [15], were taken into account, the dotted line would undoubtedly fall below the composite line in fig. 9b. Thus, it is quite clear that broken fibres are still playing an active role in resisting fatigue stressing, by carrying loads transmitted via the matrix. This can only be true of course in well-bonded specimens; therefore, for any variation in manufacturing techniques to be successful in improving high amplitude fatigue properties, the bond strength cannot be sacrificed at the expense of a reduction in fibre damage.

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