High-temperature tensile load-bearing capacity of unidirectional continuous-fibre composites: significance of a specimen-end effect

NOBUO MASAKI*, KENJI WAKASHIMA, SOKICHI UMEKAWA‡
Research Laboratory of Precision Machinery and Electronics, Tokyo Institute of Technology, 4259 Nagatsuta, Midori-ku, Yokohama 227, Japan

A "specimen-end effect" which is of significance for the determination of high-temperature tensile load-bearing capacity of metal–matrix composites reinforced with continuous metal fibres has been indicated. The effect arises from the fact that differential axial straining of the fibre and matrix can occur at high temperatures due to viscous sliding at the fibre–matrix interface. A model-system study has been carried out using a tungsten fibre–copper composite, whose ultimate tensile stress (UTS) at temperatures up to 1000° C is determined indirectly from four-point bending data as well as directly from tensile test results. It is found that at temperatures above 0.6 of the matrix homologous temperature the UTS thus determined has much smaller values than those estimated on the basis of a simple "rule-of-mixtures" equation. Significance of the result is discussed in terms of potential turbine-blade applications of heat-resistant metal–matrix composites, such as tungsten fibre-reinforced superalloys.

1. Introduction

Tungsten fibre-reinforced superalloys (TFRS) are attractive high-temperature metal–matrix composites especially as potential turbine-blade materials in turbojet engines. Reinforcing fibres embedded along the length of a blade are expected to sustain most of the uniaxial tensile load to be generated in engine-operating conditions.

The theoretical strength of a unidirectional continuous-fibre composite in the fibre direction is usually calculated in terms of a simple "rule-of-mixtures" (ROM) equation, in which the fibre and matrix are assumed to undergo equal strains. This equal-strain assumption actually holds at room and moderately elevated temperatures if the fabrication of composites is successful in the sense that the fibre and matrix are well bonded together. At very high temperatures, however, the fibre–matrix interface exhibits a viscous nature as does the grain boundary in metallic polycrystalline aggregates, so that interfacial bonding loosens to some extent. For example, when subjected to hundreds of thermal cycles between, say, 0.5 and 0.8 of the matrix homologous temperature, a tungsten–copper composite fabricated by means of molten copper infiltration, which is an experimental model system for TFRS, deforms such that the copper matrix alone extends plastically leaving the tungsten fibres behind, as shown in Fig. 1. This deformation (occurring under no external loading) is called "thermal ratcheting", which is

**Present address*: Bridgestone Tyre Co, Ltd, 2800-1 Ogawa-Higashi, Kodaira, Tokyo 187, Japan.
‡*Present address*: Department of Mechanical Engineering, Faculty of Science and Engineering, The Science University of Tokyo, 2641 Yamazaki-Higashikameyama, Noda, Chiba 278, Japan.

0022–2461/85 $03.00 + .12

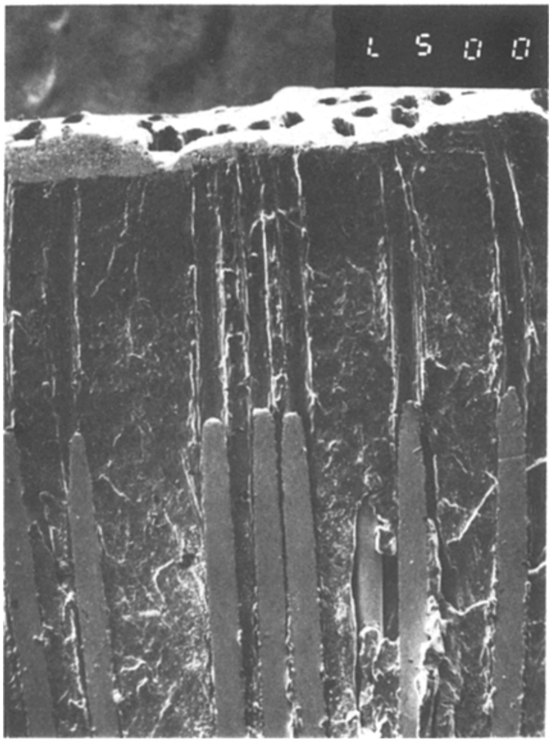

Figure 1 Evidence of interphase boundary sliding in tungsten fibre-reinforced copper. The fibre and matrix underwent unequal axial strains without interface decohesion (Yoda *et al.* [1]).

associated with the relaxation of thermal stress by a viscous interface-sliding mechanism [1].

Now a viscous nature of the fibre–matrix interface is recognized, we expect that the tensile stress carried by each individual fibre considerably lowers at high temperatures because an applied tensile load is transmitted to the fibre through an interfacial shear force. In the present paper, we first consider the tensile load-bearing capacity of a unidirectional continuous-fibre composite and point out a significant "specimen-end effect" to be encountered in experimental determination of the composite tensile strength at high temperatures. We next show experimentally determined tensile strength values of the tungsten–copper composite in the temperature range up to 1000° C, which have been obtained indirectly from four-point bending data using a method proposed by Laws [2] as well as from direct measurement by conventional tensile tests. Finally, the experimentally determined tensile strength values are compared with the ROM predictions.

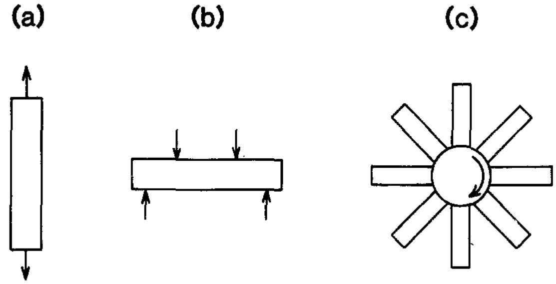

Figure 2 Various test methods adoptable for determination of the tensile strength of a material. (a) Conventional tensile test, (b) four-point bending test, and (c) centrifugal tensile test.

2. Composite load-bearing capacity in the presence of a viscous interphase boundary layer

The composite to be considered is supposed to consist of a "continuous" metal fibre, a metal matrix, and in between a very thin boundary layer which behaves in a viscous manner. Determining experimentally the tensile load-bearing capacity of the composite at high temperatures, one should take notice of such a "specimen-end effect" as shown below.

To begin with, let us consider a material whose tensile strength is to be determined experimentally. Using a rectangular strip of the material, we may adopt various test methods, which include a conventional tensile test, a four-point bending test and a centrifugal tensile test, as shown in Fig. 2. If the material is monolithic, i.e. the material contains no reinforcing fibre, the above three tests would yield consistent tensile strength values. Needless to say, the conventional tensile test is simplest among all and thus has been widely used.

Now, in the case of the composite, a problem arises when the determination of high-temperature tensile strengths is concerned. To see this problem, let us consider conventional tensile tests under two different thermal conditions: (1) uniform heating along the full length of a specimen to a desired test temperature, and (2) concentric heating of the centre gauge portion to the test temperature with forced cooling of the specimen shoulders. If the test temperature is so low that viscous shear flow within the boundary layer is virtually negligible, equal strength values, corresponding to the ROM prediction, are expected in the above two cases.

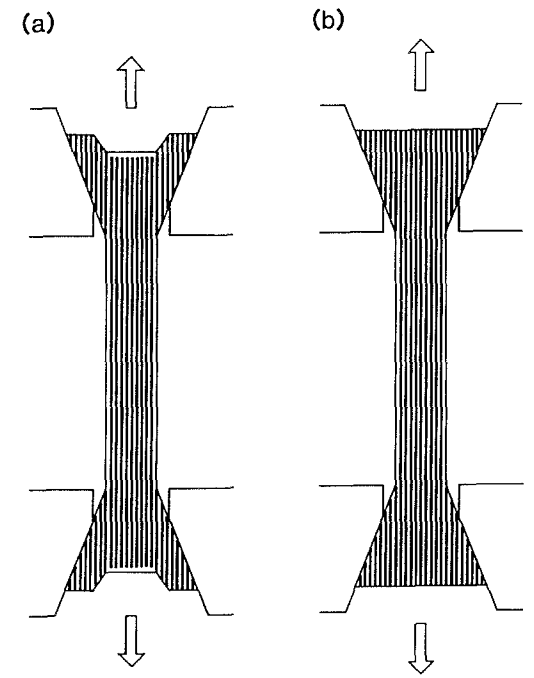

Figure 3 Possible modes of deformation of unidirectional continuous-fibre composites in elevated-temperature tensile testing. (a) Uniform heating throughout the specimen, and (b) concentric heating of the centre gauge portion to a given high temperature.

In high-temperature testing, on the other hand, heating of type 1 would make it possible for the viscous interfacial shear flow to occur along the full length of reinforcing fibres, whereas heating of type 2 would not because of complete blocking of the shear flow at the low-temperature specimen ends, as depicted in Fig. 3. In other words, differential axial straining of the fibre and matrix occurs in the former, so that the assumption on which the ROM equation is based no longer holds. Moreover, local shearing at the specimen shoulders gives rise to complexities and thus the conventional tensile test is not always best suited to the case concerned.

The tensile strength of the composite can be deduced indirectly from bending data [2]. When a rectangular specimen uniformly heated to a given high temperature is subjected to pure bending, a situation such as that shown in Fig. 4 is expected to arise. It is then evident that the embedded fibres have little effect on axial reinforcement of the material especially at very high temperatures.

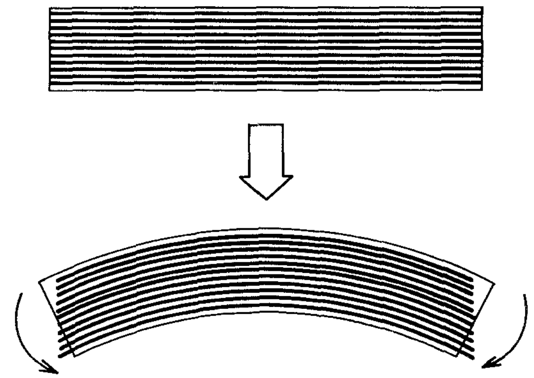

Figure 4 Possible deformation mode in pure bending of a uniformly heated, unidirectional continuous-fibre composite beam.

3. Experimental details

The tungsten–copper system was chosen as an experimental model material for TFRS for the reasons that (i) an ideal interfacial bond can be obtained because molten copper wets solid tungsten without detrimental interfacial reactions, and (ii) the maximum test temperature can be considerably lowered in terms of the matrix melting temperature. The composite was fabricated by means of vacuum infiltration, the details of which were described earlier [1, 3, 4]. The reinforcing fibre used was a commercially pure (undoped) cold-drawn tungsten wire of 0.1 mm diameter supplied by Japan Tungsten Co, Ltd. OFHC copper of 99.99% purity was infiltrated at 1200° C into wire bundles wound on a graphite spool and then directionally solidified to eliminate macropores due to freezing shrinkage.

In practising tensile tests at elevated temperatures, we adopted the uniform heating method described in the preceding section. The test piece used was of the form shown in Fig. 5, in which well-aligned continuous tungsten fibres were embedded along the loading direction with random and quite uniform distributions in transverse sections. The test piece was attached to "hanger-type" machine grips, so that upon loading, gauge sections are subjected to a tensile load which balances with a shear force generated in the specimen shoulder. Our preliminary experiments, however, showed that in elevated temperature testing some test pieces were drawn out from one of the machine grips; the determination of the composite tensile strength was then unsuccessful. Accordingly, following the

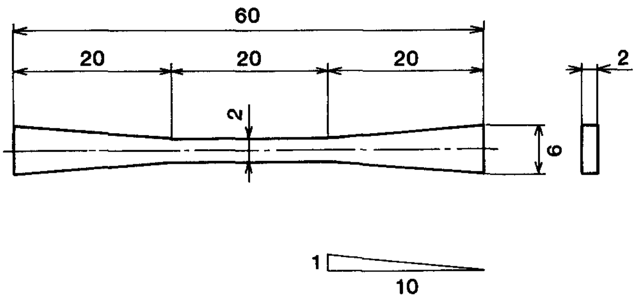

Figure 5 The tensile specimen geometry.

work by Laws [2], we supplementarily adopted a means of deriving, indirectly from bending data, a tensile strength value which should have been obtained directly if the tensile test had been valid. A rectangular beam, of length 40 mm, thickness (d) 2 mm, and width (b) 6 mm, containing parallel tungsten fibres was used in a four-point bending test, the outer and inner spans (L_o and L_i) being 30 and 10 mm, respectively. The data required in deducing the composite flow curve at the bending moment M and the strain on the tensile face ε, the former being related to the applied load P such that $M = P(L_o - L_i)/4$ and the latter to the crosshead displacement δ such that $\varepsilon = \delta d/\{[(L_o^2 - L_i^2)/4]^2 + \delta^2(L_o^2 + L_i^2)/2 + \delta^4\}^{1/2}$ provided the condition of pure bending applies. For details of the calculation procedure, see Laws [2].

Static tensile and bending tests were performed on an Instron-type testing machine equipped with a quadrupole elliptical reflector-type infrared image furnace. The load and crosshead displacement were recorded using a standard method. The majority of strength data were obtained at crosshead moving rates of 0.5 and 3 mm min^{-1} for the tensile and bending tests, respectively, which gave an approximately equal rate of tensile strain increase (5×10^{-4} sec^{-1}). Elevated temperature tests up to 1000° C were carried out in an argon atmosphere to inhibit oxidation.

4. Results

4.1. Tensile data

Tensile flow curves for the fibre volume fraction V_f = 0.06, 0.18, 0.35 and 0.45, are shown in Fig. 6. The vertical and horizontal axes are the load divided by the initial cross-sectional area (nominal stress) and the crosshead displacement, respectively.

At room temperature, the reinforcing fibres break in a brittle manner, so that the composite fractures at rather small strains except the case of $V_f = 0.06$. When V_f is very low, multiple fibre breakage occurs and, because of local work-hardening of the matrix in the vicinity of broken fibre ends, the composite can sustain increasing applied loads, displaying a considerable elongation as shown in Fig. 6 ($V_f = 0.06$). Values of the ultimate tensile stress (UTS) are plotted against V_f in Fig. 7 (open circles). The data points lie on a straight line, indicating the ROM relation to hold. The fibre strength deduced by extrapolation is 1750 MPa.

At elevated temperatures, the fibres behave plastically as the ductile-to-brittle transition temperature of the tungsten wire used is slightly below 200° C. The composite still has small elongations at moderate temperatures (200 and 400° C) except, again, in the case of $V_f = 0.06$. With further increase in temperature, a drastic change occurs in the composite flow behaviour. As can be seen from Fig. 6 (V_f = 0.18, 0.35 and 0.45), the crosshead displacement reaches a considerable extent, the load level being held nearly constant. Such a flow curve is anticipated if a specimen deforms by local shearing in its shoulder rather than in the tensile mode in the centre gauge portion. In fact, all specimens that showed this type of flow curve were drawn out from one of the machine grips without breaking into two pieces. The condition for the tensile fracture to occur may be given by

$$\tau/\sigma \geqq w/2L_s \qquad (1)$$

where σ and τ are the composite tensile and shear strengths, respectively, w is the width of the centre gauge portion and L_s is the length of the shoulder part. This indicates that the deformation in the tensile mode could have occurred if the specimen geometry had been able to be changed so as to reduce the shape factor $w/2L_s$. The shape factor for the specimens used was

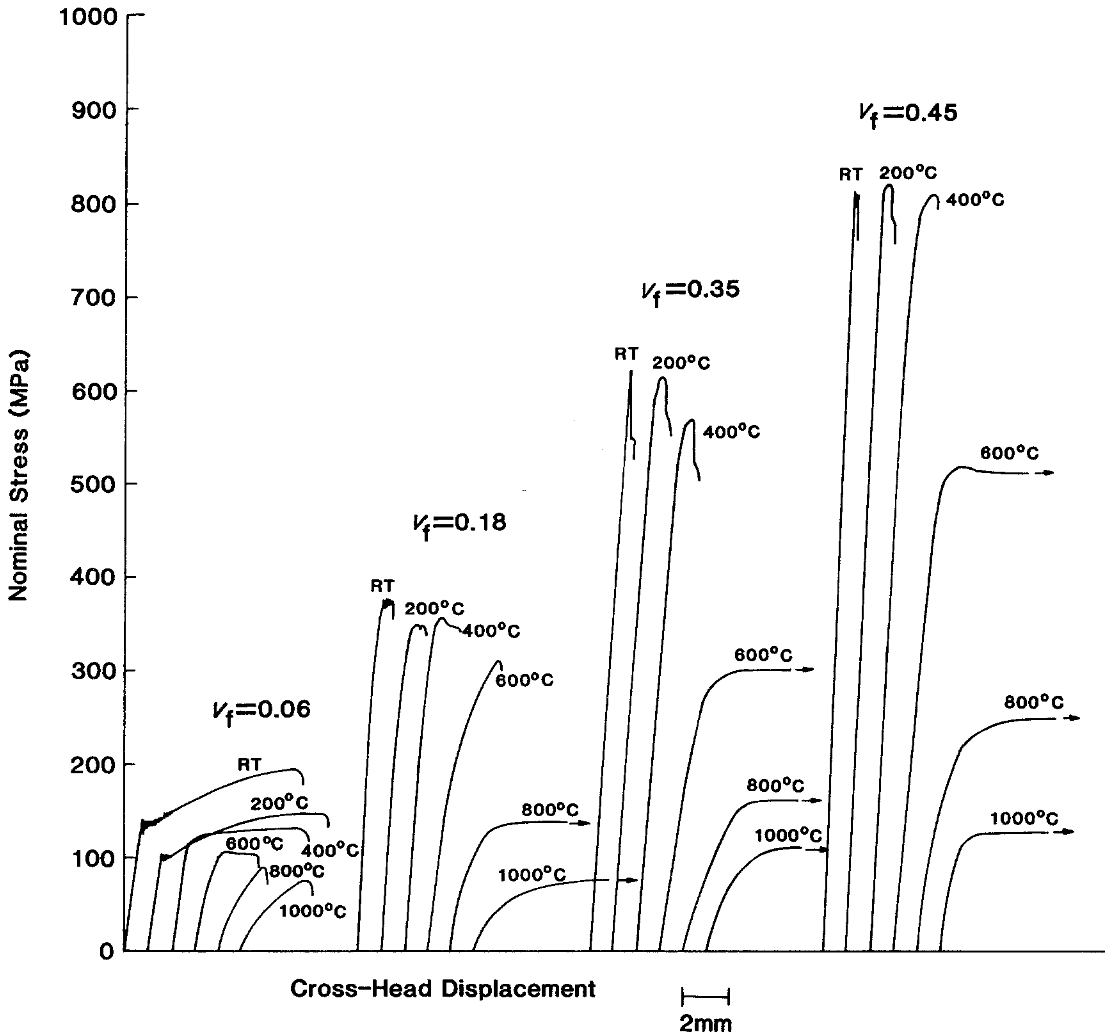

Figure 6 Load–displacement curves in tension at various test temperatures.

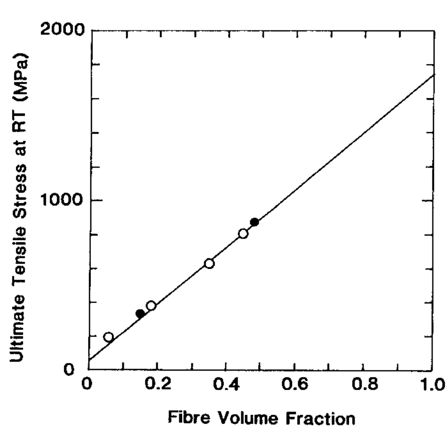

Figure 7 Plot of the composite ultimate tensile stress at room temperature against the fibre volume fraction. Open circles, determined directly by tensile tests. Solid circles, determined indirectly from four-point bending data.

0.055 (Fig. 5) and this value was the smallest to be attainable within geometric limitations imposed by the machine grip used. Accordingly, the composite tensile strength was practically undeterminable for all pull-out specimens. In Fig. 8, the observed UTS values are plotted against the test temperature, in which half-filled circles represent, from the above reasoning, ones not exceeding the real UTS values.

4.2. UTS values deduced from bending data

In deducing the composite tensile strength from bending data using the method proposed by Laws [2], the effect of a thermally induced residual stress due to the difference in thermal expansion coefficients between the fibre and

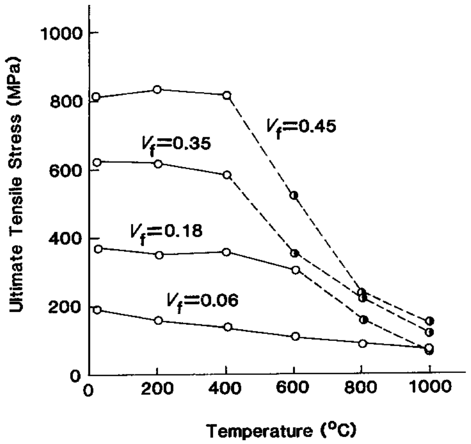

Figure 8 Plot of the composite ultimate tensile stress, determined directly by tensile tests, against the test temperature. Half-filled circles represent invalid data from pull-out specimens.

matrix [3] was neglected, i.e. the stress–strain curve in tension was assumed to be identical with that in compression. Then, the neutral surface of a bent beam coincides with the geometric bisectional surface and the strain on the tensile face is equal in magnitude to that on the compressive face. The face strain was not directly measured but deduced from the crosshead displacement by assuming that the beam was subjected to pure bending. This assumption was not unreasonable in so far as the derivation of UTS value was concerned, for the maximal load was always attained at a small deflection. Results were obtained for V_f = 0.15 and 0.48 and are shown in Fig. 9.

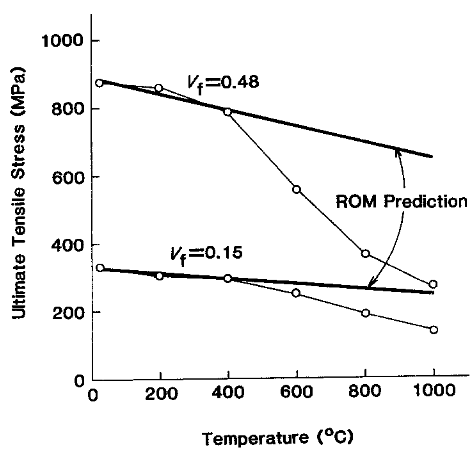

Figure 9 Plot of the composite ultimate tensile stress, determined indirectly from four-point bending data, against the test temperature. Bold lines represent the strength level estimated on the basis of the rule-of-mixtures equation.

The room temperature UTS values for V_f = 0.15 and 0.48 shown in Fig. 9 are plotted in Fig. 7 (solid circles), which also lie on the ROM line. Therefore, we believe that the indirect method of determining the composite tensile strength provides reliable information. As can be seen from Fig. 9, the UTS decreases markedly at around 600° C especially in the case of V_f = 0.48.

5. Discussion

The potential of fibrous metal–matrix composites as high-temperature structural materials has been recognized since McDanels *et al.* [5] first demonstrated the usefulness of fibre-strengthening in metallic materials. Based on the results of room-temperature tensile tests using the tungsten–copper system in particular, earlier studies [6, 7] have established the validity of ROM for predicing the UTS of unidirectional continuous-fibre composites.

We now compare our experimental results of the composite UTS with predictions in terms of the ROM equation:

$$\sigma_c = V_f\sigma_f + (1 - V_f)\sigma'_m \qquad (2)$$

where all symbols have the usual meaning [8]. Since the strength σ_f of the tungsten wire used was not measured in the temperature range covered, we first estimate it from literature data. According to Harris and Ellison [9] who presented their own experimental result along with similar data by other investigators, the UTS of commercial-grade (undoped) as-drawn tungsten wire of 0.0762 mm diameter decreases from 3000 MPa at room temperature to 1350 MPa at 1000° C. This decrease, which occurs almost linearly with increasing temperature, is interpreted as being due to the recovery of cold work. (Note that the recrystallization temperature of the tungsten wire concerned lies in the range 1200 to 1400° C.) Our result for the room-temperature σ_f value, deduced by extrapolation in Fig. 7, was 1750 MPa, which is far apart from the above data. However, the value deduced is quite reasonable in respect that the wire in our composite has experienced a considerable extent of the recovery during infiltration processing at 1200° C. Accordingly, it is likely in our case that σ_f decreases linearly with increasing temperature with values of 1750 and 1350 MPa at room temperature and 1000° C, respectively. On the other hand, σ'_m at room temperature, deduced by extrapolation in Fig. 7, is 70 MPa. Here again, we tentatively assume a linear temperature dependence of σ'_m reaching $\sigma'_m = 0$ at the melting point of copper. Results thus predicted for $V_f = 0.15$ and 0.48 are shown in Fig. 9. As is evident from the figure, increasing underdeviation of the experimentally determined UTS values from the ROM line occurs at temperatures above 600° C. It is likely that the observed decrease in the composite UTS at high temperatures results from reduction in the capability of transmitting loads to fibres due to intrinsic viscosity of the fibre–matrix interface.

The above feature is of significance from an engineering view point if similar results are to be obtained for potential TFRS turbine-blade materials. In blade designing using TFRS, weight-cutting consideration is essential because of a large density of tungsten. One of the possible weight-cutting methods proposed to date is to place heavy tungsten fibres much more densely in the centre "critical zone" than in the top and root of a blade [10]. In such circumstances, it is conceivable that the blade failure occurs at some load level well below the theoretical maximum expected from the ROM equation, though depending on the temperature distribution along the blade. However, one may take an optmistic view of that matter considering a particularly simple nature of the tungsten–copper interface which is based on purely mechanical bonding. In the case of TFRS, the interface is much different because of various metallurgical interactions occurring there during the composite fabrication, which may lead to a strong interfacial bond even at very high temperatures. In any case, engine designers must take that point into account, while engineers in the materials side should provide reliable strength data on TFRS.

6. Conclusion

The present study has indicated an intrinsic problem associated with high-temperature mechanical behaviour of metal–matrix composites reinforced with continuous metal fibres and demonstrated, using the tungsten–copper system, that the theoretical UTS values based on the ROM equation are not always expected even though the fibre–matrix interface is free of extrinsic defects such as imperfect adhesion, interfacial flaw and so on. The tungsten–copper composite is a rare system in respect of its "ideal" interface and hence, it is uncertain at present whether the problem is also fateful to potential TFRS or not.

In order to confirm the presence of a viscous interphase boundary layer, further investigation is needed. For instance, as was the case with the anelasticity of polycrystalline metals due to their intrinsic grain boundary viscosity [11], measurements of the internal friction would provide decisive information. Efforts along this line are being conducted at present in our laboratory using a method of observing the amplitude of flexural vibration under conditions of forced oscillation. Our preliminary measurement of the variation of internal friction with temperature has provided quite encouraging results. Conclusive data will be given elsewhere in the near future.

References

1. S. YODA, N. KURIHARA, K. WAKASHIMA and S. UKEKAWA, *Met. Trans.* **9A** (1978) 1229.
2. V. LAWS, *J. Mater. Sci.* **16** (1981) 1299.
3. K. WAKASHIMA, T. KAWAKUBO and S. UMEKAWA, *Met. Trans.* **6A** (1975) 1755.
4. K. WAKASHIMA and S. UMEKAWA, *ibid.* **7A** (1976) 1952.
5. D. L. McDANELS, R. W. JECH and J. W. WEETON, *Metal Prog.* **78** (1960) 118.
6. *Idem, Trans. Met. Soc. AIME* **233** (1965) 636.
7. A. KELLY and W. R. TYSON, in "High Strength Materials", edited by V. F. Zackey (Wiley, New York, 1965) p. 578.
8. A. KELLY, in "Strong Solids" (Clarendon Press, Oxford, 1966) p. 139.
9. B. HARRIS and E. G. ELLISON, *Trans. ASM* **37** (1966) 744.
10. J. W. WEETON, in "Composites: State of the Art", edited by J. W. Weeton and E. Scala (Transactions of the Metallurgical Society AIME, New York, 1974) p. 255.
11. T. S. KE, *Phys. Rev.* **71** (1947) 533.

Received 8 October 1984
and accepted 10 January 1985