JOURNAL OF MATERIALS SCIENCE **29** (1994) 2526–2530

Tensile behaviour of a niobium/alumina composite laminate

J. T. BEALS, V. C. NARDONE
United Technologies Research Center, East Hartford, CT 06108, USA

The tensile properties of a ceramic/metal laminate were examined. Alternating layers of niobium and alumina were fabricated into composite material and tensile tested at room temperature and 982 °C (1800 °F). The tensile behaviour of the laminate material was predicted using established models based on classical laminate theory. The estimated and measured properties showed excellent agreement. Relative to monolithic niobium, the composite material was found to have lower density and substantially higher stiffness.

1. Introduction

The purpose of this research was to determine and interpret the tensile behaviour of a composite laminate consisting of alternating layers of a ductile metal and a ceramic. The system selected for study utilized niobium as the ductile metal and alumina as the ceramic. Two principal advantages of this particular system are that the two materials are in thermodynamic equilibrium and they have very similar thermal expansion coefficients. Further, the high melting point of niobium allows for processing at temperatures suitable for the consolidation of Al_2O_3. The selected system may also be of practical importance, as the potential exists to cover the entire surface of the composite with alumina in order to improve oxidation resistance. Room and elevated temperature tensile tests were performed on both the Nb/Al_2O_3 composite and a baseline monolithic niobium sample processed in the same manner as the composite. The measured properties were compared with results calculated using simple laminate theory as discussed below.

2. Experimental procedure

The precursor materials used to fabricate the laminated composite were 0.051 mm (0.002 in) Nb–1Zr foil and 1 μm α-alumina powder. A uniform thickness of alumina slurry was applied to the niobium foil to achieve a volume fraction of 0.50. The coated foil was then cut and stacked into graphite tooling.

The laminates were consolidated at 1500 °C (2732 °F) and 104 MPa (15×10^3 p.s.i.) for 1 h by vacuum hot pressing. A ten-ply composite resulted in a 1.02 mm (0.040 in) thick panel. A monolithic laminate using Nb–1Zr foils was fabricated to be the same thickness as the composite panel and was consolidated using the same temperature/pressure profile. The consolidated panels were sectioned into metallographic and mechanical test specimens. Laser cutting was used to minimize damage to the laminates.

Metallographic examination of the laminate cross-section revealed a uniform alternating microstructure of alumina and niobium as shown in Fig. 1. While bonding occurred, no interfacial reaction could be observed between the alumina and the niobium. Small amounts of localized porosity were observed in the alumina layers. Etching of the niobium showed a worked microstructure with a few small carbides in the near surface layers of the composite. Microhardness measurements were made to confirm that the niobium plys did not embrittle during processing.

The composite and monolithic laminates tensile properties were measured at room temperature and 982 °C (1800 °F). For room-temperature tensile testing, fibre glass tabs were adhesively bonded to straight-sided specimens. Strain was measured with both strain gauges and extensometry. The elevated temperature specimen geometry is shown in Fig. 2 as straight-sided specimens with teeth to hold quartz extensometry. The elevated temperature tests were done in vacuum to eliminate environmental effects. An extensometer gauge length of 2.54 cm (1.0 in) was used for both room and elevated temperature tests.

3. Results

Two tensile tests were done for each material at both room temperature and 982 °C (1800 °F), with the results presented in Table I. Representative stress–strain curves are shown in Fig. 3 for the composite specimens and in Fig. 4 for monolithic niobium. Fig. 5 represents the initial portion of the stress–strain curves, indicating the proportional limit (PL) of the laminate material at room temperature and 982 °C.

The composite laminate tensile fracture surfaces were examined metallographically. Longitudinal cross-sections were polished and etched as shown in Figs 6 and 7 after both room and elevated temperature tests. Composite density measured using liquid displacement was found to be equal to 6.3 g cm^{-3} (0.23 lb in^{-3}).

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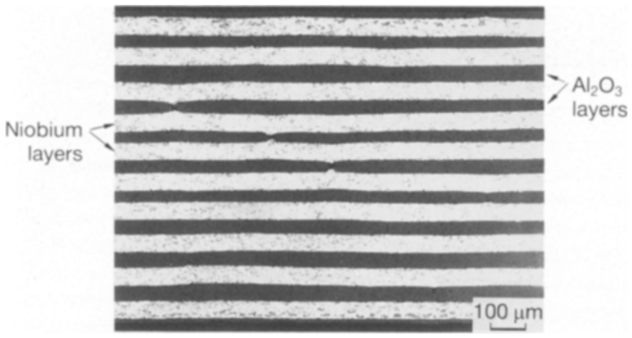

Figure 1 As-fabricated niobium/alumina composite microstructure.

4. Discussion

The composite material follows three distinct regimes of stress–strain behaviour: (1) the purely elastic regime, (2) the regime where the Al_2O_3 layers in the composite become progressively cracked, and (3) tensile overload of the niobium layers once the Al_2O_3 layers are completely cracked along the entire specimen length. All of the above regimes can be characterized quite well from constituent properties and some simple analysis as reported below.

4.1. Elastic behaviour

The elastic portion of the stress–strain curves very closely follows a rule of mixtures. For example, using the constituent volume fractions of $V_{Nb} = V_{Al_2O_3} = 0.5$, the room-temperature rule of mixture modulus is calculated to be E_{RT} (ROM) = 247 GPa (35.7 × 10^6 p.s.i.), compared to the averaged measured modulus, E_{RT} = 241 GPa (34.9 × 10^6 p.s.i.). Note that the composite room-temperature modulus is over twice that of niobium, which has a modulus of 99 GPa (14.3 × 10^6 p.s.i.). In addition, the composite density is 6.3 g cm^{-3}(0.23 lb in^{-3}) versus a monolithic niobium density of 8.57 g cm^{-3} (0.31 lb in^{-3}). The net result is a specific stiffness value of 38 GPa cm^3 g^{-1} for the composite laminate compared to only 12 GPa cm^3 g^{-1} for niobium.

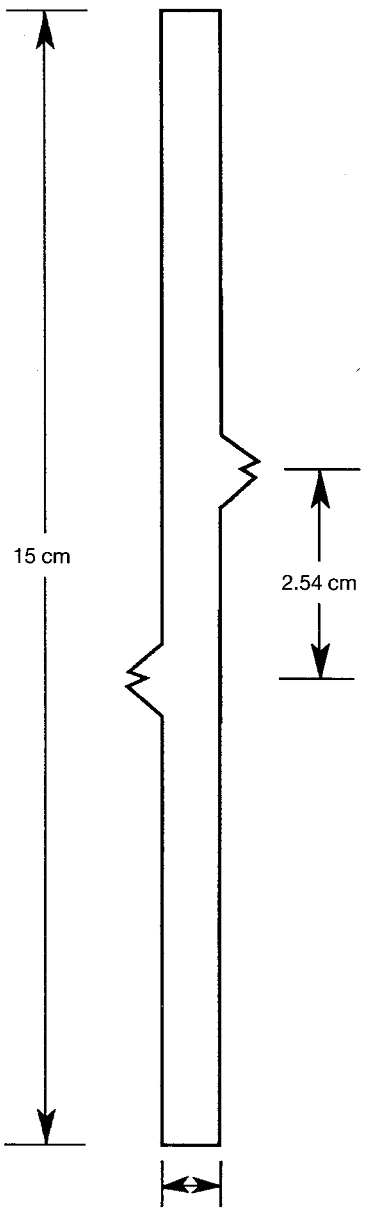

Figure 2 Elevated-temperature tensile specimen geometry.

The elastic modulus of the composite falls from approximately 241 GPa (34.9 × 10^6 p.s.i.) at room temperature to approximately 203 GPa (29.5 × 10^6 p.s.i.) at 982 °C (1800 °F). By comparison, the monolithic niobium modulus falls from 101 GPa (14.6 × 10^6 p.s.i.) to approximately 42 GPa (6.1 × 10^6 p.s.i.). Note that the difference in modulus for the niobium sample between room and elevated temperature is 57 GPa (8.2 × 10^6 p.s.i.). Because the niobium occupies 50 vol % of the composite, it would be anticipated that the decrease in composite modulus due solely to a

TABLE I Tensile test results

Material	Temp.	Elastic modulus	Yield strength	Tensile strength	Failure strain
	(°C) (°F)	(GPa) (10^6 p.s.i.)	(MPa) (10^3 p.s.i.)	(MPa) (10^3 p.s.i.)	(%)
Composite	RT	24333.9	25637.0	28941.9	0.76
	RT	24735.9	25036.3	30844.7	2.18
	9821800	21230.8	19227.8	19430.8	0.59
	9821800	19428.2	18426.6	19028.2	0.67
Niobium	RT	10114.6	15622.5	29642.9	15.0
	RT	10114.6	15322.1	29542.7	14.6
	9821800	436.3	12017.3	21931.7	8.3
	9821800	405.8	13119.0	22332.4	14.1

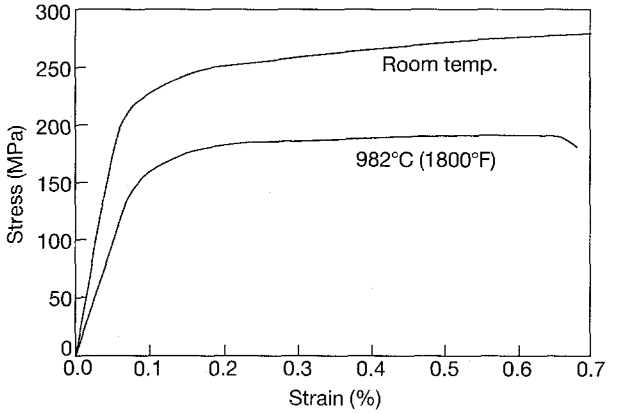

Figure 3 Nb/Al_2O_3 composite tensile stress–strain curves at room temperature and 982 °C (1800 °F).

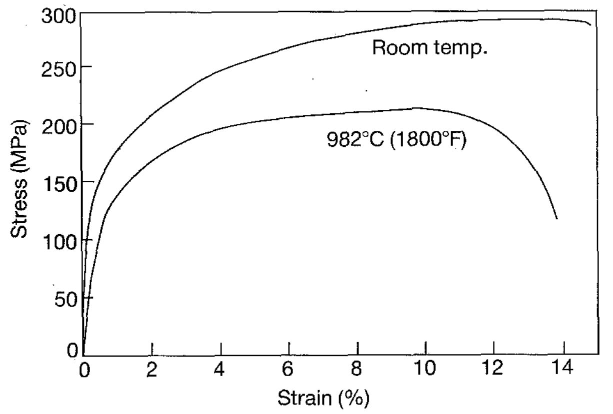

Figure 4 Monolithic niobium tensile stress–strain curves at room temperature and 982 °C (1800 °F).

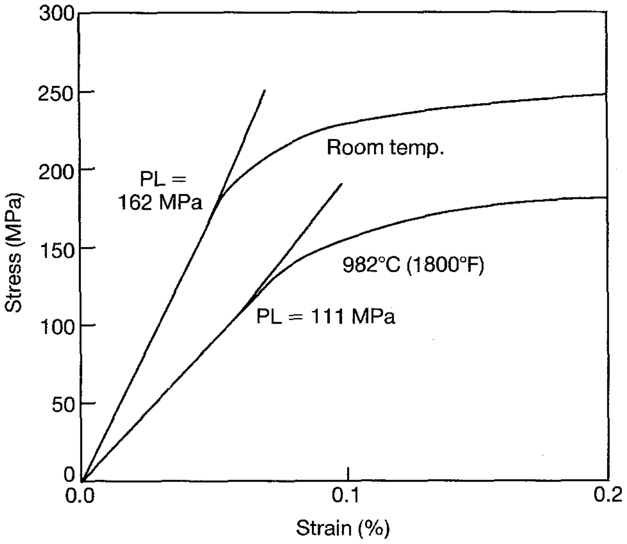

Figure 5 Nb/Al_2O_3 composite tensile stress–strain curves indicating proportional limits at room temperature and 982 °C (1800 °F).

decrease in the niobium modulus, should be 29 GPa (4.1×10^6 p.s.i.). The measured drop in composite modulus between room and elevated temperature was 38 GPa (5.4×10^6 p.s.i.). This suggests that the primary reason for the loss in composite modulus with increasing temperature is a result of a decrease in modulus of the niobium layers.

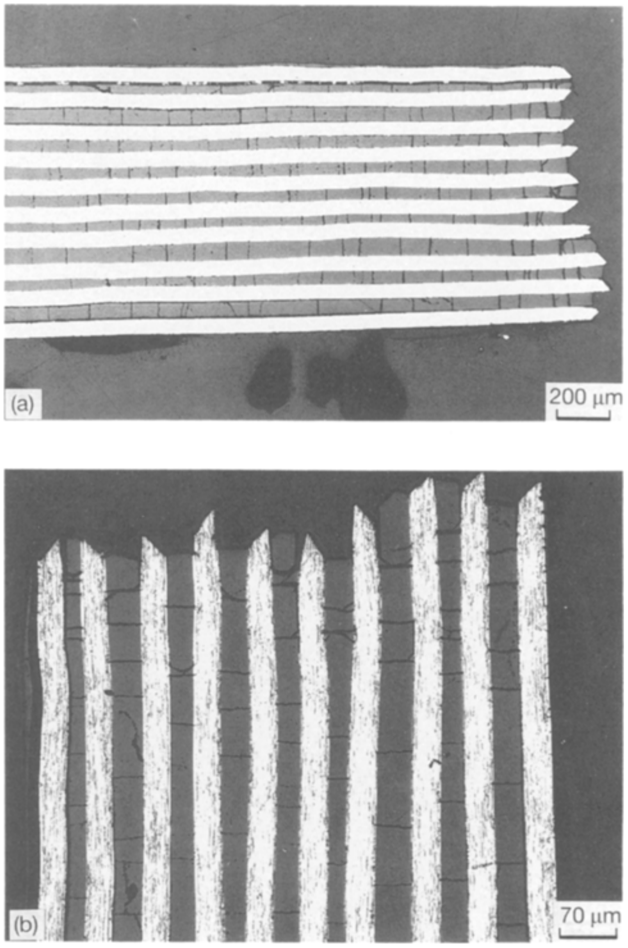

Figure 6 Room-temperature tensile fracture: (a) polished longitudinal section, and (b) higher magnification etched longitudinal section.

It should also be noted that the specific modulus of the Nb/Al_2O_3 laminate at 983 °C (1800 °F) is 32 GPa cm^3 g^{-1} compared to only 5 GPa cm^3 g^{-1} for niobium.

4.2. Brittle failure of the alumina layers

The composite stress–strain curve departs from linearity when the alumina layers first start to crack. This stress at which matrix microcracking occurs can be estimated rather easily. The alumina with its low strain to failure will crack well before the failure of the composite. Assuming linear behaviour to failure, the strain to crack the alumina, $\varepsilon_{\text{crack}}$, can be expressed as

$$\varepsilon_{\text{crack}} = \sigma_{Al_2O_3}/E_{Al_2O_3} \qquad (1)$$

where $\sigma_{Al_2O_3}$ is the ultimate tensile strength and $E_{Al_2O_3}$ is the elastic modulus of alumina. Based on data reported elsewhere [1], the appropriate value of $\sigma_{Al_2O_3}$ is 267 MPa (40×10^3 p.s.i.), and $E_{Al_2O_3}$ is 394 GPa (57×10^6 p.s.i.) respectively. Assuming that there is no debonding in the composite elastic regime such that the strain in the niobium equals the strain in the Al_2O_3, the stress at which the composite deviates from linearity (the proportional limit) will be equal to the tensile failure strain of the Al_2O_3 layers. Note that it is also being assumed that Al_2O_3 layers crack prior to plastic yielding of the niobium layers. Based on the

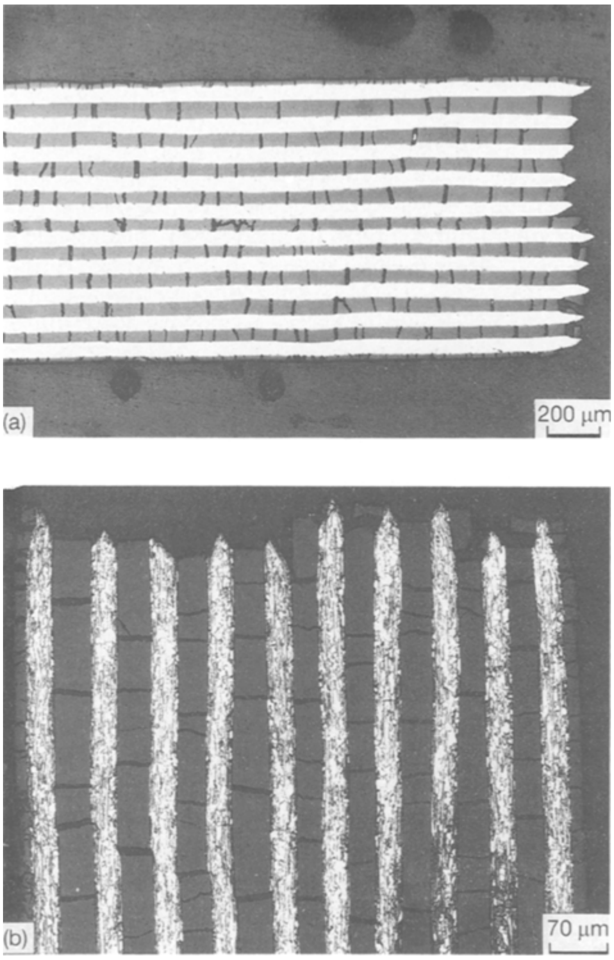

Figure 7 982 °C (1800 °F) tensile fracture: (a) polished longitudinal section, and (b) higher magnification etched longitudinal section.

above, we can write the following equation to calculate the composite laminate proportional limit, σ_{pl}

$$\sigma_{pl} = E_c(\sigma_{Al_2O_3}/E_{Al_2O_3}) \qquad (2)$$

where E_c is the composite elastic modulus. Using the measured composite laminate modulus, Equation 2 calculates a composite proportional limit of 173 MPa (25.0×10^3 p.s.i.) at room temperature and 144 MPa (20.9×10^3 p.s.i) at elevated temperature. These calculated values correlate very well to the measured proportional limits for the composite laminates 162 MPa (23.4×10^3 p.s.i.) at room temperature and 111 MPa (16.1×10^3 p.s.i.) at 982 °C (1800 °F), as shown in Fig. 5.

The anticipated length of ligaments in the cracked alumina layers can also be estimated. An analysis by Piggott [2] based on slip length results in the following equation

$$m_c = (V_{Al_2O_3}\,\sigma_{Al_2O_3})/(2V_{Nb}\,\tau_i\,S_{Al_2O_3}) \qquad (3)$$

where m_c is the fraction of ligament subject to slip, τ_i is the interfacial shear strength, and $S_{Al_2O_3}$ is the aspect ratio of the cracked matrix ligaments. Cracking is complete when $m_c = 1$. Therefore, the aspect ratio of cracked alumina (ligament length/ply thickness) can be estimated by substituting $m_c = 1$ into Equation 3 and rearranging to result in the following

$$S_{Al_2O_3} = (V_{Al_2O_3}\sigma_{Al_2O_3})/(2V_{Nb}\,\tau_i) \qquad (4)$$

When the interfacial shear strength, τ_i, is assumed to be half the niobium yield strength, the critical aspect ratio for room-temperature testing is calculated to be 1.6. For an average alumina ply thickness, $t = 51$ µm (0.002 in), the predicted cracked matrix length is 81 µm (0.0032 in). Image analysis on a longitudinal section of the room-temperature tensile fracture measured a mean matrix length of 123 µm (0.0045 in). Similar cracked matrix lengths were measured for the elevated temperature specimen, which would be predicted to be similar to the room-temperature results as there is only a small drop in the niobium yield strength (and therefore interfacial shear strength) at the elevated test temperature.

4.3. Ductile failure of the niobium layers

After cracking of the Al_2O_3 layer occurs, the niobium must carry the total load for the composite to prevent failure. For the case of the laminate tested at room temperature, the tensile strength measured for the composite was 299 MPa (43.3×10^3 p.s.i.). This stress would correspond to the niobium (0.50 V_{Nb}) carrying twice that amount or 598 MPa (86.6×10^3 p.s.i.). However, the ultimate tensile strength measured for monolithic niobium is only 296 MPa (42.8×10^3 p.s.i.). This increase in strength can be attributed to the failure mode of the composite niobium plys, as discussed below.

Two effects have been documented that can explain the increased *in situ* niobium strength level in the laminate. Recent work has analysed tensile failures when testing lead sandwiched between glass slides [3], and compared the response resulting for strong/weak interfaces and for ductile shear. Moderate increases in laminate strength were observed for a strong interface or constrained system. The strongly bonded interface does not allow extensive deformation of the ductile phase and therefore decreases the composites work of fracture. If the interface is allowed to debond, a significant increase in work of fracture is observed along with some decrease in composite strength. However, another fracture mode was found to contribute to both increased strength and work of fracture. As shown schematically in Fig. 8, when the brittle layers crack they do not all crack within the same plane. This forces a shear failure in the ductile regions where the angle of shear is dependent on the location of the cracks in the brittle regions. In the Nb/Al_2O_3 laminates reported here, the predominant composite tensile failure mode was this shear failure of the ductile niobium regions between cracked alumina regions as shown in Fig. 6.

The increased strength contribution from shear can be calculated within the shear plane as large plastic shear will work harden the material. The work-hardening behaviour for niobium, $\sigma = A\varepsilon^n$, can be estimated [4] as $A = 545$ MPa and $n = 0.19$. For a shear strength of k, the corresponding axial stress would be $\sigma = k/\sin\phi\cos\phi$. Estimating the peak shear strength as half of the maximum strength the niobium can achieve with work hardening, and using the measured average ϕ from longitudinal cross-sections of 45°,

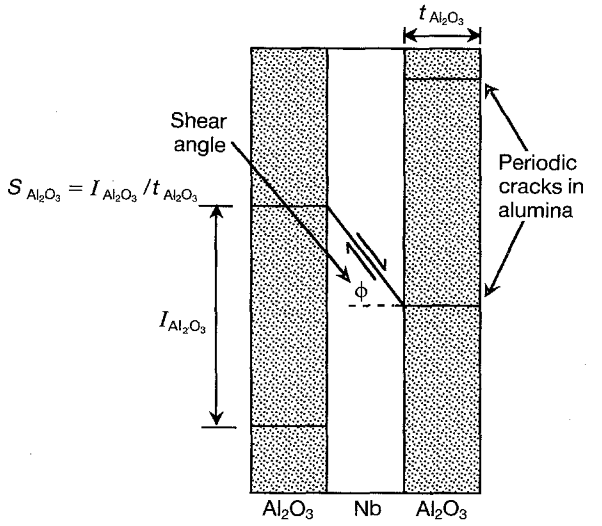

Figure 8 Schematic illustration of non-planar crack formation in brittle Al_2O_3 layers leading to shear failure of ductile niobium layer.

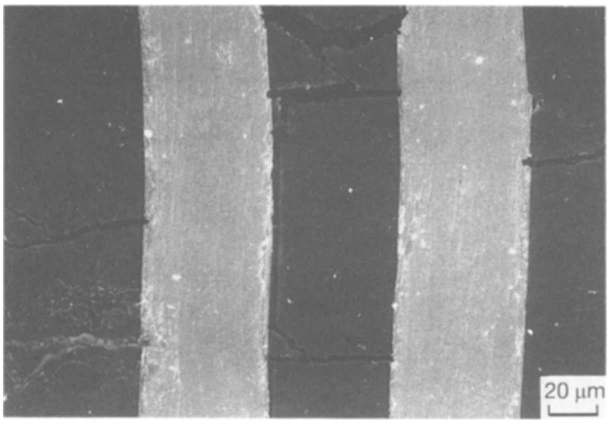

Figure 9 Scanning electron micrograph showing typical debonding in room-temperature tensile fractured side surface.

a calculated peak stress of 545 MPa (78.9×10^3 p.s.i.) results. This axial stress is very close to the stress carried by the niobium in the composite laminate of 598 MPa (86.6×10^3 p.s.i.).

Differences in the composite strain to failure were measured for room and elevated temperature tests. A decrease in strain to failure (and subsequent work to fracture) for the elevated temperature tensile specimens are reflected in their failure mode. As previously mentioned, both shear failure and debonding contribute to an increased work to fracture. Many previous studies [5–7] have discussed the role of debonding in the toughening of ductile fibre-reinforced intermetallics. As the interface debonds, a larger volume of metal is free to deform. While some constraint and therefore work hardening is lost, the increase in strain to failure contributes to the composite work of fracture. Also increasing the work of fracture is the axial contribution related to the niobium shear displacement. For the alumina niobium laminates tested at room temperature, both debonding and the shear failure are present in the tensile failure cross-section. The room-temperature cross-section shown in Fig. 6 exhibits niobium fractures almost exclusively by shear, while a scanning electron micrograph in Fig. 9 also shows debonding in most plys. The elevated temperature niobium plys do show a more ductile fracture, Fig. 7, resulting in greater crack extension. However, fewer niobium plys have failed by shear and no debonding occurs. This is reflected in the reduced strain to failure of 0.63% measured for the 982 °C (1800 °F) tensile specimen, versus 1.45% for the room-temperature specimens.

5. Conclusions

The niobium/alumina composite laminate material has shown benefits in both room and elevated temperature tensile properties compared to monolithic niobium. Significant increases in elastic modulus and decreases in density were measured. Three separate regimes of tensile stress–strain behaviour were identified in this system. The composite behaviour in each regime, at both room and elevated temperature, could be explained using established models based on classical laminate theory.

Acknowledgements

This work has been sponsored by the SDIO Innovative Science and Technology Office and was monitored through the Office of Naval Research (ONR contract N00014-87-C-0406) by Dr Steve Fishman.

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Received 16 September 1992
and accepted 27 September 1993