JOURNAL OF MATERIALS SCIENCE 19 (1984) 2239–2245

Fatigue crack growth behaviour of Ti–6 Al–4 V metal matrix/continuous SiC and B_4C/B fibre composites

Y. H. PARK*, D. NARAYEN, M. SCHMERLING, H. L. MARCUS
Department of Mechanical Engineering/Materials Science and Engineering, The University of Texas at Austin, Texas, USA

The fatique crack growth (FCG) behaviour of SiC and B_4C/B reinforced Ti–6 Al–4 V metal matrix composites loaded in the transverse direction as a function of modifications of the interface between the fibre and matrix was studied. The interface chemistry, modified by sulphur diffusion during thermal cycling treatment, changed the FCG in air, dry nitrogen and hydrogen environments when compared with the as-received specimens. The FCG rates tend to be higher in a humid environment. The SEM fractrography indicates that the FCG in humid air was by an increased amount of fibre splitting. The FCG in dry nitrogen environment was more often by interface debonding with some fibre splitting and fiber fracture. The FCG rates in dry hydrogen for both as-received and heat-treated specimens were intermediate between the observed rates for dry nitrogen and humid air. During FCG in laboratory air, the sulphur-enriched interface of the specimens thermal cycled in a sulphur environment reacts with the humidity in air to degrade the interface cohesion, resulting in complete separation of the interface from the matrix and the fibre at low strains. This inability of the interface to sustain any strain further increases the FCG rates in the matrix. The results show that the interface does transfer load during fatigue cycling either in an inert environment or if the interface has a minimal amount of impurities.

1. Introduction

The fatigue behaviour of metal matrix composites (MMCs) differs from that of unreinforced metals in several ways. MMCs exhibit various failure modes such as debonding of the interface between the fibre and the metal matrix, fibre splitting, fibre fracture and matrix fracture, which occur both independently or interdependently.

Because of their high strength-to-weight ratio, high modulus and toughness, titanium MMCs have long been considered for applications in dynamic structures. However, like other MMCs, titanium MMCs are characterized by anisotropy, heterogeneity and interface. Tensile properties of titanium MMCs reinforced with continuous SiC, Borsic and B_4C/B fibres have been studied [1–3] and the results indicated that the mechanical properties are dependent on the interface or the reaction zone between the fibre and the metal matrix.

In this study, various experiments were performed to study the effect of temperature and other environments on the interfaces and the mechanical properties of the titanium metal matrix composites reinforced with SiC and B_4C/B fibres.

The mechanical properties studied were transverse loading residual strength after thermal exposure and fatigue crack growth (FCG) under Mode I loading after thermal treatment. The condition of the matrix–fibre interface as a function of the above treatments was studied using both

*Present address: Materials Research Corporation, Orangeburg, NY 10962, USA.

0022–2461/84 $03.00 + .12

scanning Auger microscopy (SAM) and scanning electron microscopy (SEM). The fibre interface chemistries were modified by thermal treatment in oxygen and sulphur-bearing environments.

2. Experimental details

2.1. Materials

The metal matrix composite (MMC) systems used in this study were SiC or B_4C/B continuous fibre in a Ti–6 Al–4 V alloy matrix. The composite panel was four-ply, approximately 0.84 mm thick. The composite had a fibre volume fraction of about 40% with a diameter of 150 μm. Detailed specimen preparation appears in [4].

2.2. Heat treatments

With the intention of studying the effect of a modified interface on the fatigue behaviour and failure modes of the titanium MMCs, heat treatment in a sulphur environment was used. Specimens were thermally cycled in a sulphur-rich atmosphere between 25° C and 550° C in a fluidized bath furnace filled with alumina particles [4]. An automatic timing device was used to maintain heating and cooling periods of five minutes at each temperature. All specimens were placed in pyrex tubes with dry powdered sulphur, evacuated to a pressure of 10^{-5} torr and sealed. Thermal cycling was carried out for a period of 85 h for a long sulphur diffusion distance [4].

2.3. Fatigue crack growth (FCG) testing

All FCG testing was performed on transverse specimens. Edge-notched specimens with an initial notch length of 6 mm, specimen width 25 mm and specimen length 62.5 mm were used. Aluminium doublers were glued at the ends using an epoxy glue to prevent load point failure. Fatigue cycling was performed on an MTS closed loop electrohydraulic system in the tension–tension mode, between 9.1 and 92 kg at a frequency of 2 Hz. Fatigue crack growth was monitored visually with the aid of several equidistant vertical lines normal to the notch inscribed on the specimen surface. Stress intensity calculations [5] showed the thickness requirements for the plane strain FCG testing, as specified in the ASTM Standards [6], could only be satisfied up to a K value of 11 MPa $m^{1/2}$. Actual K values ranged from about 0.9 to 27 MPa $m^{1/2}$ with a load ratio of 0.1.

The first set of experiments was performed on both as-received and heat-treated transverse specimens in laboratory air of approximately 50% r.h. In addition, to study the effects of gaseous environments on the FCG, experiments were also performed in atmospheres of dry nitrogen and dry hydrogen. For this purpose, a high vacuum system (sorption and ion pumps) connected to an environment chamber was employed. The chamber was evacuated to 5×10^{-4} Pa (4×10^{-6} torr) and backfilled with the gas of interest.

Finally, all the fatigue fractured surfaces were viewed in a Jeol 35 SEM to determine the failure paths and mechanisms and the effects of interface modification and gaseous environments on the failure mode. Multiple specimens were run in each set of experiments to permit duplication of results.

3. Results and discussion

3.1. FCG for SiC/Ti–6 Al–4 V composites

The first set of experiments was carried out on both as-received and thermally cycled SiC/Ti–6 Al–4 V specimens in laboratory air of 50% r.h. It was observed that the rate of crack growth in the heat-treated specimens slightly exceeded that of the as-received specimens, indicating that the thermal cycling in sulphur environment may be instrumental in degrading the fatigue resistance. The as-received specimen showed the presence of both fibre splitting and interfacial debonding, while the heat-treatment specimens exhibited only interfacial debonding. These observed fracture modes are in agreement with Mahulikar's [5] results for Borsic/Ti–6 Al–4 V composites, where he noticed a transition from fibre splitting for the as-received condition to interfacial splitting for the heat-treated condition.

To investigate the effect of different gaseous environments on the FCG and failure mode, room temperature experiments were performed in dry nitrogen gas. Both the as-received and thermally cycled specimens showed considerably lower FCG rates in nitrogen than in humid air (Figs. 1 and 2). This observation indicates that it is the reaction of sulphur with humid air, more than likely the dissociated hydrogen, that results in further degradation of the fibre–matrix interface and consequent reduction in fatigue crack growth resistance. The presence of an inert atmosphere such as nitrogen renders the sulphur from the thermal cycling treatment relatively ineffective in increasing the FCG rates (Fig. 2). Fractography on both sets of fracture surfaces revealed the

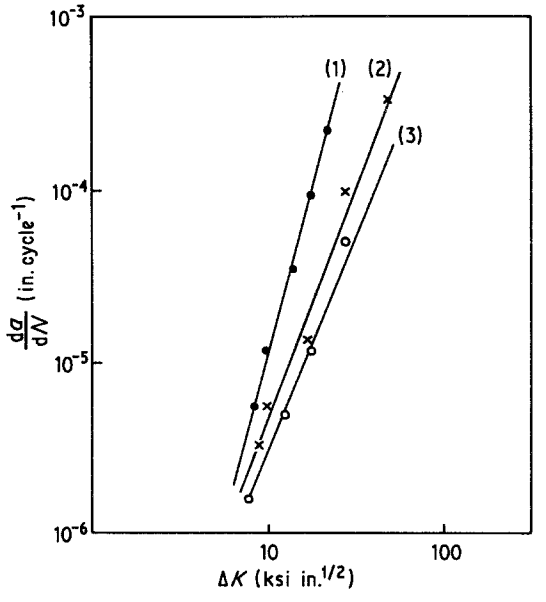

Figure 1 FCG of as-received transverse SiC/Ti–6 Al–4 V specimens in various gaseous environments. (1) Humid laboratory air; (2) dry hydrogen; (3) dry nitrogen.

presence of fibre splitting. This is in contract to Mahulikar's [5] results for Borsic/Ti–6 Al–4 V, where fibre splitting was seen only in humid air. The difference can be attributed to better fibre–matrix bonding in the SiC composites, or that the SiC are more brittle than the Borsic fibres, causing fibre splitting in preference to debonding from the matrix.

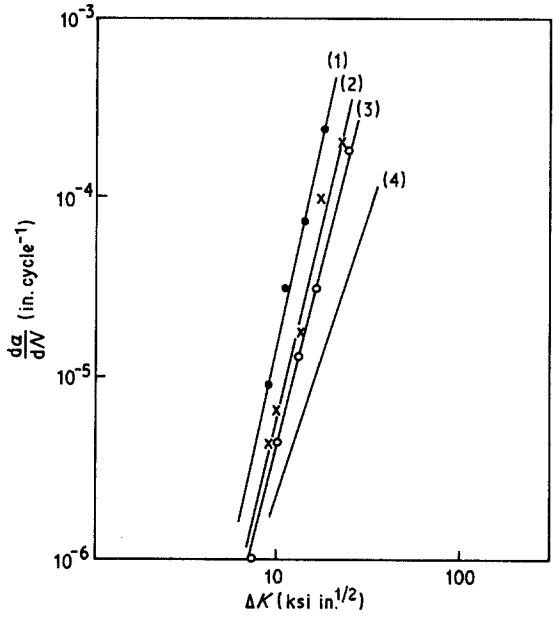

Figure 2 FCG of thermal cycled transverse SiC/Ti–6 Al–4 V specimens in various enviornments. (1) Humid laboratory air; (2) dry hydrogen; (3) dry nitrogen; (4) from (3) in Fig. 1.

The third set of experiments was performed in dry gaseous hydrogen to evaluate the hydrogen effect. The FCG rates of both the as-received and thermally-cycled specimens were intermediate between the observed rates for dry nitrogen and humid air (Figs. 1 and 2). The FCG rate of heat-treated specimens slightly exceeded that of the as-received specimens. Fractography revealed the presence of some fibre splitting in each case, which can be explained by reasoning similar to that for the dry nitrogen case.

In all cases the fibre direction along the interface or through the split fibres was found to be the least resistant path for the FCG in the transverse specimens. In the case of mixed mode loading [5], the FCG was in the direction of the fibre, indicating that the direction of the fibre–matrix interface was the least resistant path for crack propagation.

3.2. FCG for B_4C/B/Ti–6 Al–4 V composites

Fig. 3 shows the results for the FCG tests in a transverse direction for as-received B_4C/B/Ti–6 Al–4 V specimens and subsequently loaded cyclically at a load ratio of 0.1 in both laboratory air and gaseous nitrogen. FCG data for the annealed Ti–6 Al–4 V alloy in air is also included for comparison. The FCG of Ti–6 Al–4 V alloy in air is also included for comparison. The FCG of Ti–6 Al–4 V alloy in air and inert dry argon at room temperature yield almost identical cyclic crack growth rates [7].

When the data are presented in a plot of da/dN against ΔK, as shown in Fig. 3, it is clear that the Stage II FCG of the composites along with a Ti–6 Al–4 V alloy can be expressed by Paris' law

$$da/dN \propto \Delta K^m$$

The slope of the logarithmic plot (m) is identical for the Ti–6 Al–4 V alloy and the composites tested in the transverse direction. The results for composites show little difference in FCG between this study and Mahulikar's [5]. The fractography, shown in Fig. 4, indicates that the FCG in humid air was by an increased amount of fibre splitting, as reported here and from Mahulikar's results [5]. FCG in a dry nitrogen environment, on the other

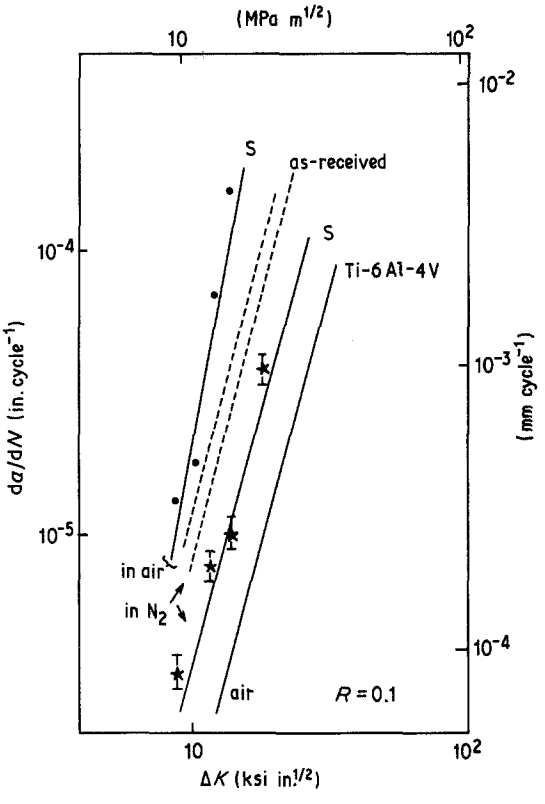

Figure 3 Transverse FCG rates for B_4C/B/Ti–6 Al–4 V thermally cycled in sulphur compared with the initial condition and Ti–6 Al–4 V plate.

hand, was more often by interface debonding with some fibre splitting and fibre fracture (Fig. 5).

Specimens thermally cycled in sulphur show higher FCG rates in laboratory air than the as-received sulphur-free specimens. The slope (m) is greater for those specimens. When sulphur is at the interface following thermal cycling in the

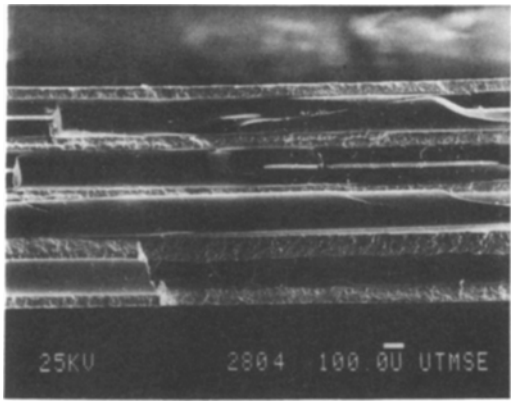

Figure 5 SEM fractograph of FCG in dry nitrogen for as-received B_4C/B/Ti–6 Al–4 V, transverse.

sulphur environment, the FCG rate in humid air increases and is accompanied by interface debonding. SEM fractography in Fig. 6 shows well-defined interface debonding.

The FCG results for the samples thermal cycled in a sulphur environment and tested in dry nitrogen gas indicated a slight decrease in growth rate with the same slope as the dry nitrogen FCG rates of the as-received specimens. The fractography of nitrogen tested sulphur-enriched B_4C/B/Ti–6 Al–4 V composites, shown in Fig. 7, shows fracture modes such as fibre splitting, fibre fracture and matrix fracture similar to those for FCG in nitrogen of as-received specimens. This similar fracture mode correlates with the similar value of m with the as-received specimen.

When ΔK is normalized using the rule of mixtures with the elastic moduli of the respective materials [$E_{\text{composite}} = 140\,\text{GPa}$ (20×10^6 psi), $E_{\text{Ti}} = 116\,\text{GPa}$ (16.8×10^6 psi)], to have compar-

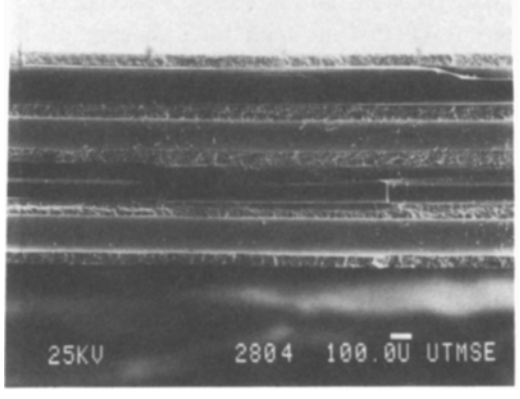

Figure 4 SEM fractograph of FCG in air for as-received B_4C/B/Ti–6 Al–4 V, transverse.

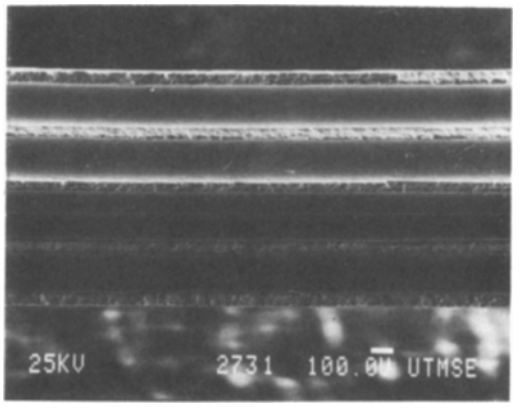

Figure 6 SEM fractograph of FCG in air for B_4C/B/Ti–6 Al–4 V thermally cycled in sulphur, transverse.

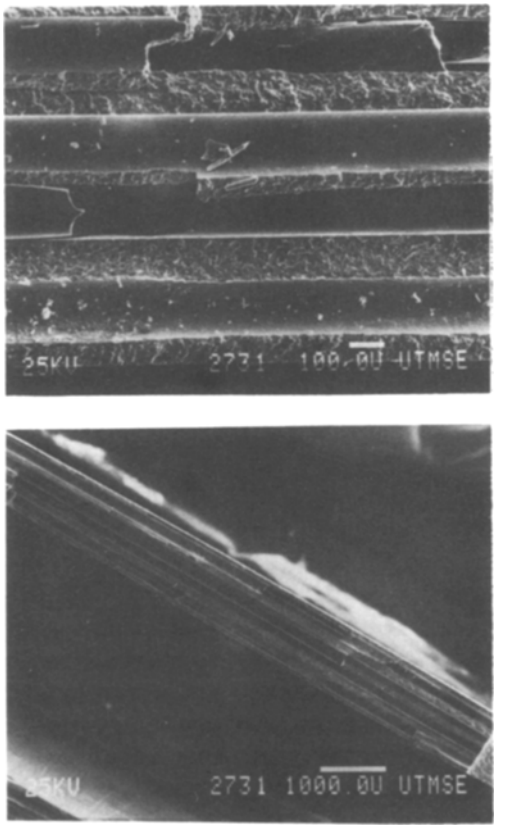

Figure 7 SEM fractographs of FCG in dry nitrogen for B_4C/B/Ti–6 Al–4 V thermally cycled in sulphur, transverse.

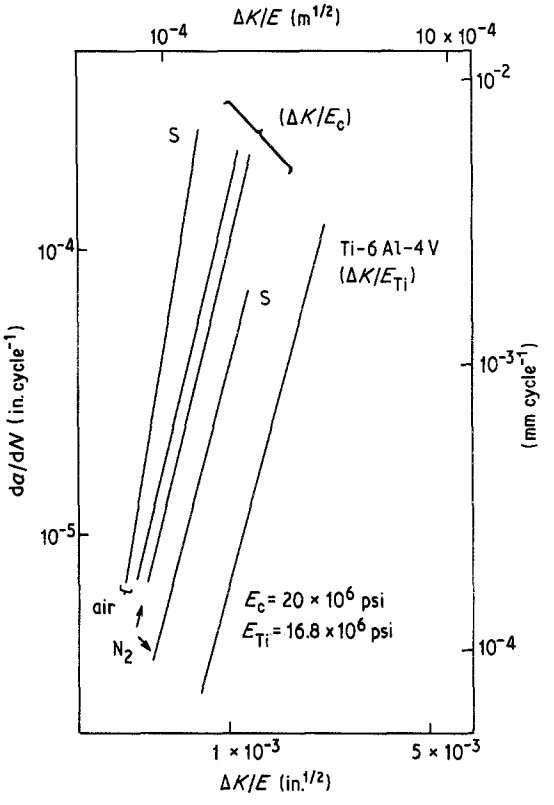

Figure 8 FCG rates after the normalization of ΔK by the elastic modulus, for B_4C/B/Ti–6 Al–4 V composite.

able crack opening displacement (COD) [8], the composite FCG is even greater than that of the Ti–6 Al–4 V matrix with the slopes (m) virtually unchanged, as shown in Fig. 8. The fibre directions along the interface or through split fibres are the least resistant paths for the transverse FCG. This can be explained by the fact that the brittle fibre and interface are not carrying much of the stress. If it is assumed that all the cyclic stress and COD are accommodated by the matrix material, the equivalent $\Delta K/E$ would translate the results to match closely the Ti–6 Al–4 V results, with the exception of the sulphur/humid air measurements. The amount of translation of ΔK_{tr} will be $1.7\,\Delta K_{composite}$, since the load is carried by the matrix with a volume fraction of 60% for the transverse specimen (Fig. 9).

The sulphur-enriched interface of the specimen thermally cycled in sulphur reacts with the humidity in the air during FCG in laboratory air to degrade the interface cohesion, resulting in complete separation of the interface between the matrix and the fibre at low strains. This inability of the interface to sustain any significant strain further increases the fatigue crack growth rate in the matrix. The increase in the value of the slope may be due to the degradation of the interface by titanium sulphides creating a volume of brittle materials.

4. A model of the failure mechanism for the transverse FCG

The results presented here demonstrate that the interface between the matrix and the fibre does transfer load during fatigue cycling in either an inert environment or if the interface has a minimal amount of impurities. On the basis of the results of the experiments described above, a model for the failure mechanism for the FCG of the transverse titanium MMCs can be proposed. The model is shown schematically in Fig. 10. Only the matrix deforms plastically. Due to the high degree of anisotropy for the MMCs, the plastic zones are elongated in the direction of the fibres. Thus the fibres in front of a sharp crack can be assumed to be surrounded by a plastic sheath of the matrix. During

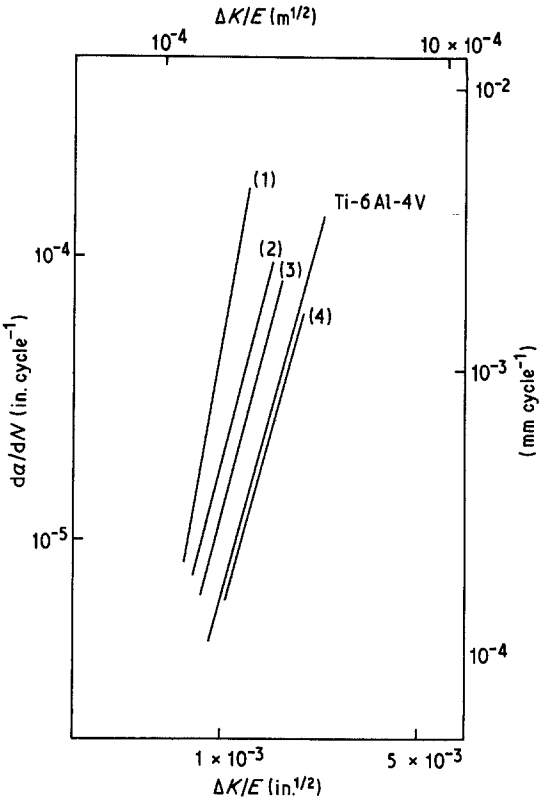

Figure 9 FCG rates after the translation by a factor of 1/0.6, for B_4C/B/Ti–6 Al–4 V. (1) TF, S/FCG in air; (2) as-received, in air; (3) as received, in nitrogen; (4) TF, S, in nitrogen.

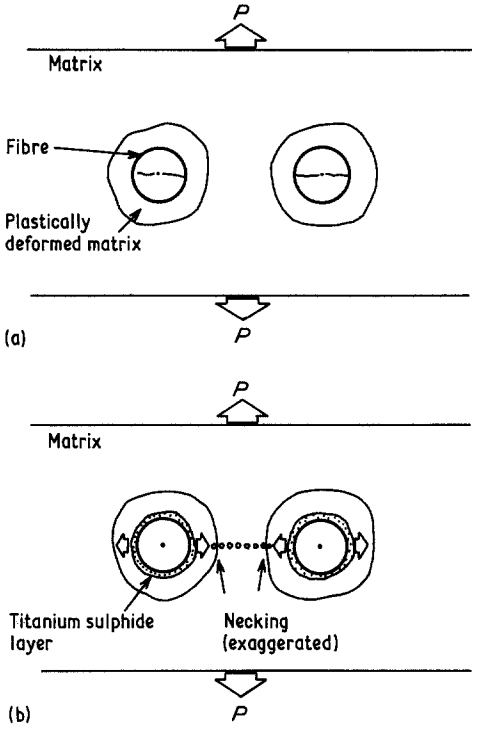

Figure 10 A model for FCG fracture modes in laboratory in (a) as-received and (b) thermally-cycled conditions.

cyclic loading of as-received materials in humid air, the matrix deformed plastically under tension closes over the fibres which do not deform plastically ($\epsilon_{fibre} = 0$). When the interface transfers the load the fibres split as described earlier (Fig. 10).

In the case of the sulphur-enriched specimen, fatigue cycling in humid air reduced the cohesion of the interface due to hydrogen–sulphur interactions. The stress is then carried by the matrix alone and necking forms in the matrix along the interface between two adjacent fibres. This could be initiated from the rough crack surface due to the interaction of the titanium sulphide and the humidity in air. Such rough surfaces come from the radial and branch cracks, as shown in Fig. 11. The fracture mode in the vicinity of the fibres is exclusively interface debonding and the fibres are undamaged, as shown in Fig. 6.

5. Summary and conclusion

The effect on the fatigue crack growth (FCG) behaviour of the SiC and B_4C/B reinforced Ti–6 Al–4 V metal matrix composites (MMCs) by the modification of the interface between the fibre and the matrix by isothermal and thermal cycled treatments was studied. The thermal cycling and isothermal treatments were carried out in environments of air, sulphur and vacuum.

The FCG results demonstrated that the interface transferred the load during fatigue cycling in either an inert environment or if the interface had a minimal amount of impurities. In the case of

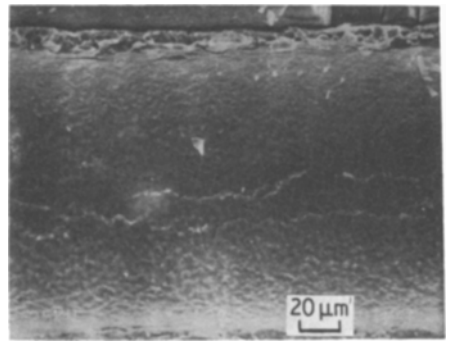

Figure 11 SEM fractograph of SiC/Ti–6 Al–4 V composite thermally cycled in sulphur for one day at temperatures up to 550° C. Branch cracks can be seen along the interface between matrix and fibre.

the sulphur-enriched interface, the humid air FCG environment reduced the cohesion of the interface, resulting in the applied load being carried solely by the matrix. This led to an increased FCG rate with a higher value of the Paris exponent (m). Without the enhanced sulphur at the interface the Paris exponent remained constant, although the FCG rate nominally increased in a humid air environment.

The non-environmental FCG rate change can be largely explained in terms of the volume fraction of the matrix relative to FCG of the monolithic Ti–6 Al–4 V matrix material.

Acknowledgement

This research was sponsored by AFOSR, under contract 80-0052. The titanium metal matrix composites were supplied by the AFML.

References

1. A. G. METCALFE, in "Composite Materials", Vol. 4 (Academic Press, New York and London) p. 269.
2. B. R. COLLINS *et al.*, in "Failure Modes in Composites I", edited by I. J. Toth *et al.* (American Institute of Mechanical Engineers, Town, 1973) p. 103.
3. D. S. MAHULIKAR, Y. H. PARK and H. L. MARCUS, in Proceedings of the Greece/USA Mixed Mode Fracture Conference, Athens, Greece, 1980.
4. Y. H. PARK, PhD Dissertation, University of Texas at Austin (1983).
5. D. S. MAHULIKAR and H. L. MARCUS, *Met. Trans. A.* to be published.
6. W. BROWN and J. SRAWLEY, ASTM STP 410 (American Society for Testing and Materials, Philadelphia, 1969).
7. R. J. BUCCI *et al.*, ASTM STP 513 (American Society for Testing and Materials, Philadelphia, 1972) p. 125.
8. R. W. HERTZBERG, in "Deformation and Fracture Mechanisms of Engineering Materials" (John Wiley, New York, 1976) p. 514.

Received 15 September
and accepted 29 September 1983