JOURNAL OF MATERIALS SCIENCE 27 (1992) 1801–1810

Interfacial properties of a SiC fibre-reinforced Ti alloy after long-term high-temperature exposure

W. WEI
Motoren- und Turbinen-Union GmbH, D-8000 Munich 50, Germany

The effect of long-term high-temperature exposure on the interfacial properties of a typical SiC continuous fibre-reinforced titanium alloy was investigated. Specimens were annealed in air and in vacuum under simulated operating conditions using temperatures up to 500 °C for times up to 700 h. The interfacial chemistry and fracture morphology of the specimens were determined using Auger and scanning electron microscopy. It was shown that high-temperature long-term exposure to air resulted in embrittlement of the fibre–matrix interface through combined oxidation and attack of the Ti matrix on the fibres and fibre coating. Exposure to operating temperatures in vacuum did not lead to a significant degradation of the interfaces. The degradation of the interfaces in high temperature service can lead to a reduction of the service life of components made of reactive system metal–matrix composites such as SiC-reinforced Ti alloys.

1. Introduction

Titanium-based alloys have found increased use in high-temperature applications due to their excellent strength-to-weight ratio. In many cases, substantial increases in specific strength and stiffness can be obtained through continuous fibre reinforcement. Of primary interest in many applications are the SiC fibre-reinforced titanium alloy (Ti/SiC) composites.

One of the major problems faced in the development of Ti/SiC composites for high-temperature applications is the degradation of the fibre–matrix interface, and hence of composite properties, by the reaction of the titanium matrix with silicon carbide fibres at high temperatures. Much of the literature dealing with interfacial reactions in Ti/SiC composites has been concerned with optimizing fabricating conditions to reduce the extent of these reactions and the resulting degradation of mechanical properties [1–10]. Typical fabrication conditions result in the formation of a reaction zone consisting of TiC near the fibre surface, and various amounts of brittle titanium silicides, including Ti_5Si_3, TiSi or $TiSi_2$, resulting from Si diffusion towards and reaction with the matrix [2, 5–7, 9]. The thickness of the reaction zone increases with temperature and processing time, resulting in a corresponding decrease in mechanical properties such as tensile strength and elastic modulus [1–3, 8, 11]. A number of coatings have been investigated to inhibit the formation of the reaction zone by providing a diffusion barrier to Si. Coating materials which have been tried include TiC and TiN [5, 8], Al or Ti_3Al [3], and Y_2O_3 [10]. The most successful coatings produced to date appear to be the pyrolytic carbon coatings found on the SCS series of fibres produced by AVCO Specialty Materials (now Textron, Lowell, MA).

It would be expected that the long-term use of Ti/SiC composites at temperatures up to 700–800 °C would also result in changes in interfacial chemistry, resulting in a gradual degradation in mechanical properties. The mechanical performance of components would then be dependent not only on temperature and interfacial chemistry, but also on time. Such a time dependence of interfacial chemistry would be analogous to the time-dependence of microstructure at high temperature seen in metals such as stainless steels and nickel base superalloys under creep conditions.

Most high-temperature investigations and data on metal–matrix composites (MMCs) deal mostly with short-term exposure to temperature. Typical tensile and fatigue tests last, at most, of the order of several days. Little work has been done, however, on the effect of long-term high-temperature exposure on reactive composite systems such as Ti/SiC, or on the long-term chemical stability of fibre coatings [12–14]. A preliminary investigation on the SiC/Ti-6Al-4V system has indicated that long-term exposure to high-temperature operating conditions can reduce the low cycle fatigue life of MMCs [12].

This paper reports the results of an investigation which examines the changes in interfacial chemistry of a typical Ti/SiC composite exposed to high temperatures for extended periods of time. The interfacial chemistry was characterized using Auger electron spectroscopy (AES), while changes in interfacial

0022–2461 © *1992 Chapman & Hall*

strength were inferred from the fracture morphology of Auger specimens fractured in vacuum. The consequences of long-term interfacial chemistry changes for composite development, and for component lifetime prediction, are discussed.

2. Experimental procedure

The material used for this investigation was SiC (SCS-6) continuous fibre-reinforced Ti–6Al–4V plate produced by Textron. The characterization of the interfaces was conducted using (AES) and scanning electron microscopy (SEM). Standard Auger fracture specimens (18 × 2 × 2 mm with a notch at 9 mm) were cut from the plate using a diamond cut-off wheel. Specimens were isothermally annealed in air or in vacuum at several temperatures for times up to approximately 700 h as shown in Table I. The temperatures used were typical of those seen in service by monolithic Ti alloys. The specimens annealed in vacuum were sealed in evacuated quartz tubes. Annealing in air was intended to simulate the interfacial behaviour of uncoated, small components or the edges of composite components. Annealing in vacuum was intended to simulate the changes in chemistry expected in the middle of larger components where oxygen concentrations may not reach the equilibrium levels of the component edges, or in components with oxidation coatings.

TABLE I Annealing conditions for Ti/SiC composite

Temperature (°C)	Time in air (h)	Time in vacuum (h)
RT – as received	–	–
300	670	500
400	500	500
500	580	500
600	–	500

The specimens were examined in a Perkin Elmer PHI 660 scanning Auger microprobe. They were fractured *in situ* at a vacuum of about 10^{-7} Pa. The fracture surfaces were characterized by AES using a primary electron-beam voltage of 10 kV and a primary beam current of about 100 nA. Sputter profiles were performed on selected specimens using argon with an accelerating voltage of 5 kV. Sputter rates were calibrated using Ta_2O_5 as a reference. Secondary electron microscopy was conducted with the Auger electron microscope at 10 kV, as well as with a conventional SEM at 10 kV. Although the specimens were small in cross-section, analyses were made in the centre of the fracture surfaces to avoid end effects. Metallographic sections of selected specimens were also examined to complement the surface analysis results.

3. Results

3.1. Samples annealed in air

3.1.1. Fractography

SEM micrographs and results of the Auger analyses of the as-received materials and specimens annealed in air are shown in Figs 1–5. The morphology of the *in situ* fracture surfaces is strongly dependent on temperature. The surface of the as-received material showed a

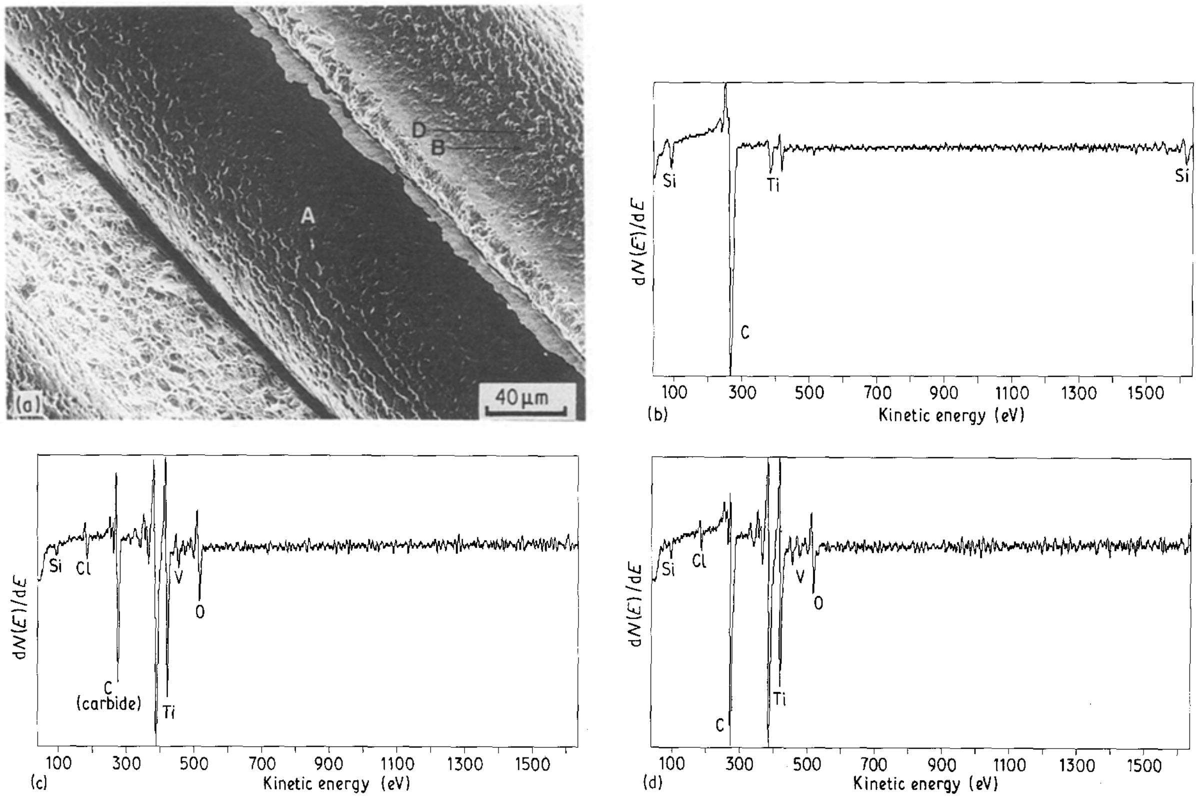

Figure 1 Fracture surface of as-received Ti–6Al–4V/SCS-6 material. (a) SEM micrograph (beam voltage 10 kV); AES spectra of (b) fibre, area A; (c) smooth area in trough, point B; (d) stringers in trough, point D.

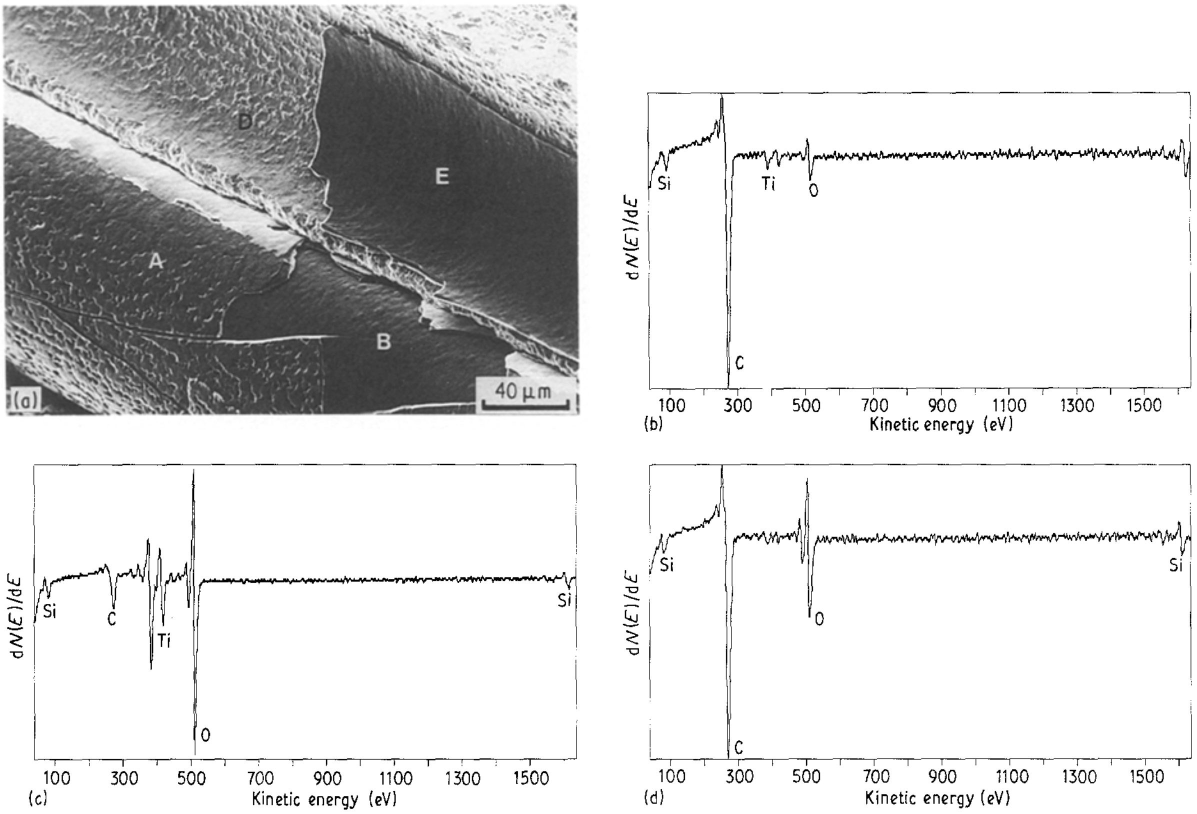

Figure 2 Fracture surface of Ti–6Al–AV/SCS-6 material annealed in air for 670 h at 300 °C. (a) SEM micrograph (beam voltage 10 kV); AES spectra of (b) fibre coating, area A; (c) trough, point D; (d) inside of fibre coating lying in trough,-point E.

fairly clean fracture between the fibre and the matrix (troughs), Fig. 1a. The fibre surfaces are rough, having an array of depressions: these depressions correspond to the array of stringers which cover the trough surfaces. The specimen annealed in air at 300 °C also shows areas of fairly clean fracture. There are, however, areas in which the carbon coating of the SCS fibre has split or broken away from the fibres (see Fig. 2a). At the higher annealing temperatures in air, the fracture surfaces take on a distinctly mottled appearance. At 400 °C, parts of the fibre coating have spalled off and remained in the troughs (Fig. 3a, b). The outer surface of the coating remaining on the fibres shows a rough, flaky surface (Fig. 3a). The stringers in the troughs stand out against the dark, smooth layer, which was identified with Auger analysis as the inside of the fibre coating (Fig. 3b). At 500 °C much of the fibre coating has broken away from the fibres and lies in troughs (area D in Fig. 4a, b). The troughs still contain the stringers seen at all other temperatures, but they tend to be brighter in the SEM, indicating a slight charging effect. The remaining fibre core is relatively smooth but contains white flecks on the surface. The white flecks were observed at higher magnification to lie slightly below the darker fibre core surface.

3.1.2. Chemistry

The chemistry of the fracture surfaces also depends strongly on temperature. This dependence can be qualitatively described by correlating the Auger spectra with the corresponding fracture features in Figs 1–4. The resulting picture is shown schematically in Fig. 5.

3.1.2.1. As-received. The chemistry of the as-received material agrees with literature studies on this and similar materials in the as-received condition [6, 7]. The fibre surface (point A in Fig. 1a) consists primarily of C with small amounts of Si and Ti (Fig. 1b). The smooth areas of the trough (point B) contain mostly C, the matrix elements Ti and V, and traces of Cl, Si and O (Fig. 1c). The C KLL (272 eV) signal shows the shape for carbide [15], while the Ti LMM peaks are characteristic of the non-elemental form (peak-size ratio Ti 387/Ti 416 > 1). These areas probably comprise the interfacial layer of TiC formed during the fabrication of the composite. The traces of Cl and O are assumed to come from processing. The stringers (point D) show the same elements as the smooth areas (Fig. 1d), but the C signal has the mixed shape of the carbide layer and the carbon layer on the fibre surface. It is assumed that silicides are present in the interfacial zone, but these could not be resolved under the analysis conditions used in this work. The observed chemistry of the interface of the as-received material is summarized schematically in Fig. 5a.

3.1.2.2. 300 °C Anneal. For the specimen annealed at 300 °C, slight oxidation of the fibre–matrix interface was observed. The Auger spectra of the fibre surface (coating; point A in Fig. 2a) show signals for Si and C, as well as traces of Ti and O (Fig. 2b). The underlying

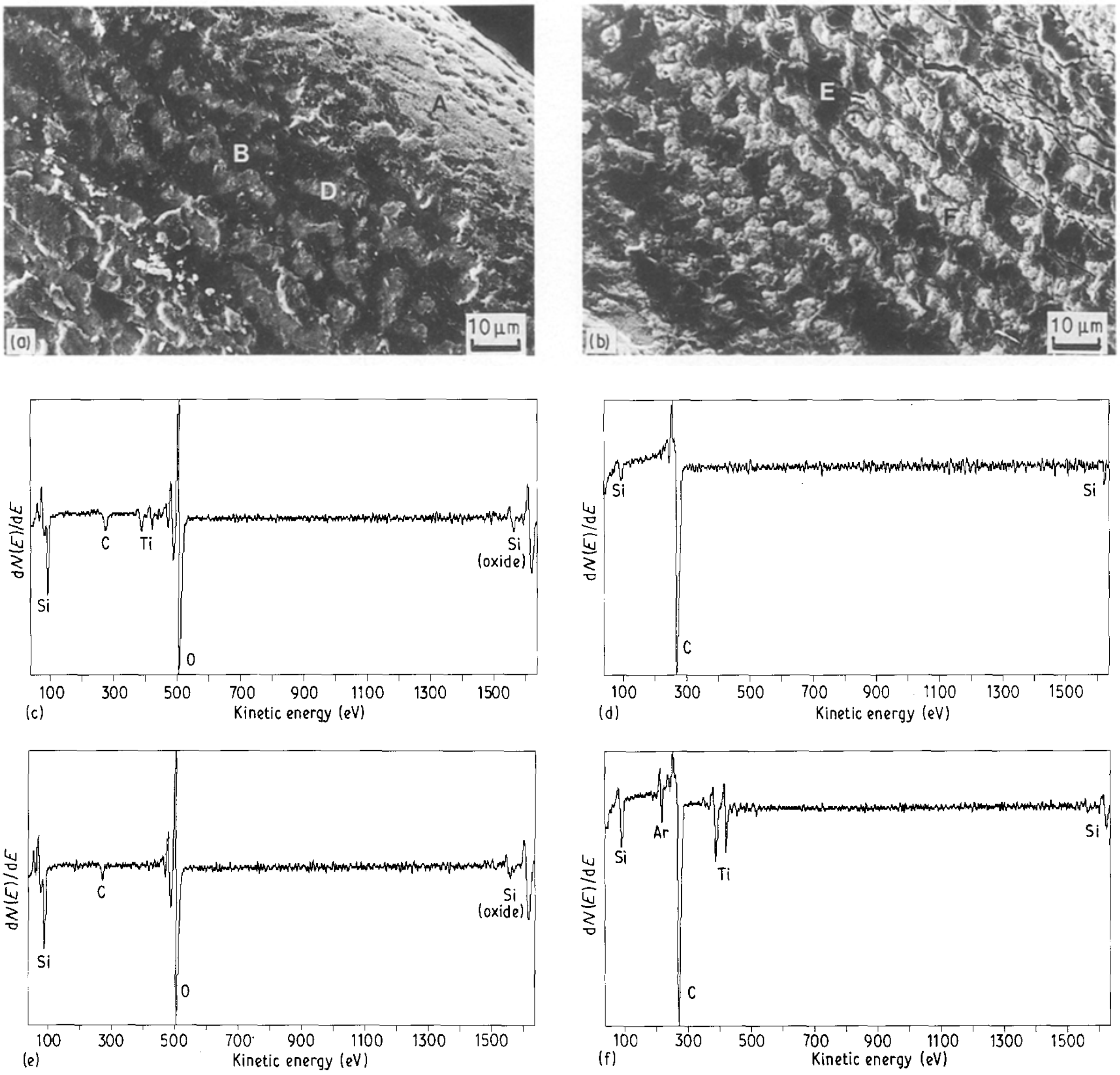

Figure 3 Fracture surface of Ti–6Al–4V/SCS-6 material annealed in air for 500 h at 400 °C. SEM micrographs of (a) fibre (beam voltage 10 kV); (b) trough (beam voltage 10 kV); AES spectra of (c) light, raised area on fibre, area A; (d) dark area on fibre, area B; (e) grey area on fibre, area D; (f) area A after depth profiling.

fibre core (point B) also shows a trace of O. The trough surface (point D) corresponding to the fibre surface (point A) shows the presence of oxidized titanium with some Si and C (Fig. 2c). The dark layer (point E) is the inside of the fibre coating which lies next to the fibre core (point B). This layer contains Si and C, with a large O signal as compared with the trace amount found on the fibre core (Fig. 2d). A sputter profile of the fibre coating surface indicated that the oxygen layer was fairly thin, ~ 25 nm. The titanium oxide layer in the trough was, on the other hand, too thick to be sputtered through in a reasonable time. Oxygen gettered by the matrix during the anneal could also have prevented a precise determination of the oxide thickness in the trough. The presence of the oxidized Ti signal and O, along with the typical Si and C signals for the fibre, indicate that oxidation of the interface of the material annealed at 300 °C has occurred preferentially in the TiC layer or on the matrix side of the interface. This is shown in the schematic diagram of the interface in Fig. 5b.

3.1.2.3. 400 °C Anneal. The gradual break-up of the interfaces at higher temperatures complicates the analysis of the surface chemistry of the fracture surfaces. For the specimen annealed at 400 °C, the chemistry of the fibre coating varies with depth. This can be seen by comparing the various levels seen in Fig. 3a remaining after partial or complete spalling. The chemistry of the light, raised areas (area A in Fig. 3a) shows SiO_2 with small amounts of C and Ti (Fig. 3c). The darkest areas (B) show the original fibre coating chemistry, primarily C with a trace of Si (Fig. 3d), but do not appear to be the lowest level. The grey areas (D) appear to penetrate below the coating surface and show SiO_2 with a trace of C (Fig. 3e). Depth profiling of the raised area (A) revealed that the oxide layer was approximately 70-nm thick. The chemistry at that depth consisted of a mix of SiC and Ti (Fig. 3f). The profile on area D revealed a thinner oxide layer of about 30 nm, covering the SiC core of the fibre. The smooth, dark areas in the trough (area E) contain C with Si and a trace of O, corresponding to the inside of the fibre

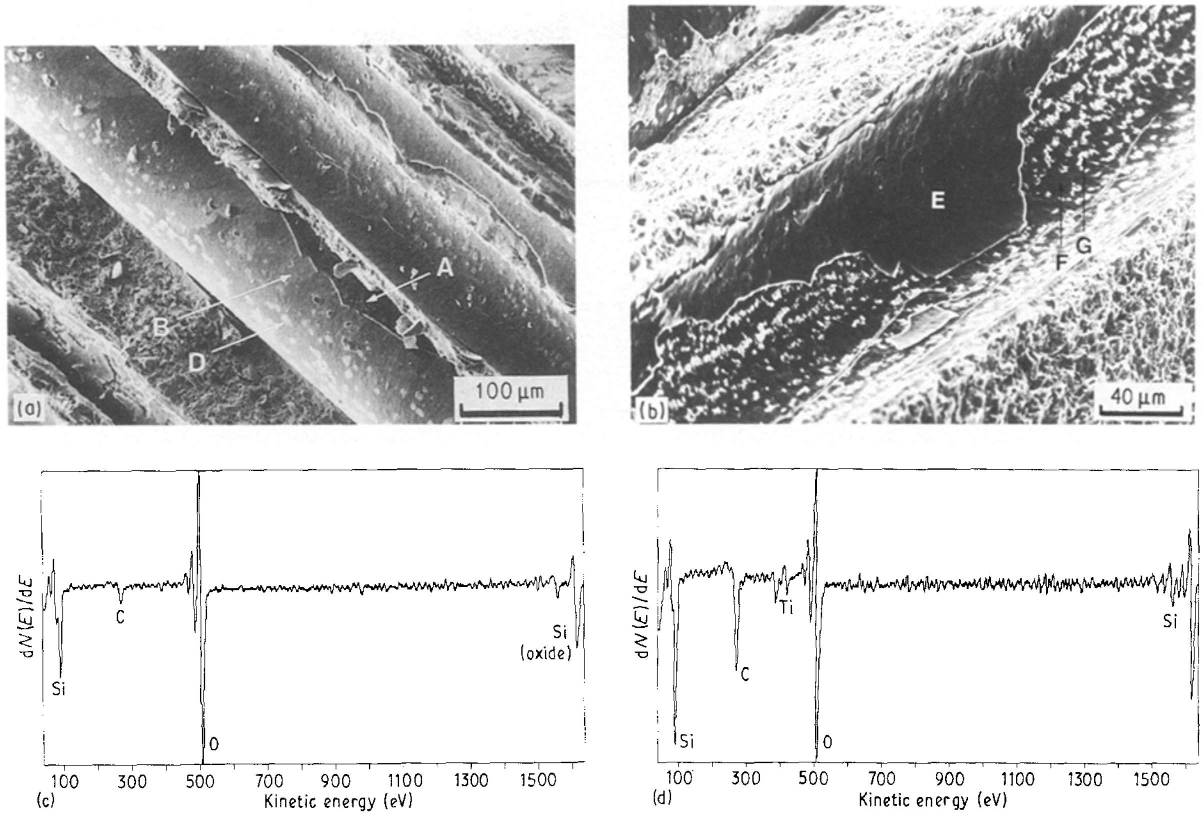

Figure 4 Fracture surface of Ti–6Al–4V/SCS-6 material annealed in air for 580 h at 500 °C. SEM micrographs of (a) fracture surface (beam voltage 10 kV); (b) trough (beam voltage 10 kV); AES spectra of (c) fibre core, area B (oxidized Si valence peak at 76 eV is partially reduced by the electron beam); (d) white flecks on the fibre core, point D.

coating (see Fig. 3d). The stringers (F) appear to be a Ti oxide with traces of Si and C, similar to those found at 300 °C (Fig. 2c). These rather complicated observations are shown schematically in Fig. 5c. It appears that at 400 °C, the oxidation proceeds most rapidly at the interface between the depressions in the fibre and the stringers in the trough. The coating is thinnest there, and oxidation as well as diffusion of Ti can occur most readily at those locations. The increased presence of oxidized Si in the spectra indicates that the oxidation of the fibre surface proceeds at a rate comparable to that of the matrix or TiC layer at this temperature. The oxidation also appears to have undercut the coating, where it was observed that non-oxidized coating was found lying above oxidized fibre. Oxygen could reach the interface between the coating and the fibre core through cracks in the coating or through the reaction zone at the depressions. The tendency for separation of the coating from the fibre at the various annealing temperatures indicates that this interface is relatively weak. Rapid diffusion of oxygen along that interface from the outside atmosphere (specimen edge) could lead to the undercutting oxidation observed.

The smooth, dark areas in the trough correspond to the inside of the fibre coating. It is conceivable that the chemistry through this layer would be the same as the variable chemistry observed for the different layers of coating found on the fibres. The chemistry of the surface of the trough coating would then lie in the middle of the reaction zone, the level C + Si + O shown in Fig. 5c.

3.1.2.4. 500 °C Anneal. The oxidation attack of the interfaces at 500 °C is a natural progression of the mechanisms occurring at 400 °C. The oxide is primarily SiO_2 at this temperature. The fibre coating remaining on the fibre (area A in Fig. 4a) has a similar chemistry to that of the coating at 400 °C (Fig. 3c), namely SiO_2 with the presence of C and Ti. The chemistry varied somewhat with position on the coating, with SiO_2 detected at some points, and a mixed SiO_2, SiC and Ti signal at other points. The fibre core itself (point B) showed SiO_2 with a trace of Ti (Fig. 4c). The white flecks (point D) on the fibre core surface consisted of SiO_2 with some Ti present, with the Si peak showing a mixed elemental and oxide character (Fig. 4d). Recall that the flecks were observed at higher magnification to lie below the surface of the fibre core. The inside of the fibre coating lying in the troughs (areas E and F in Fig. 4b) showed only SiO_2. The stringers in the troughs (point G in Fig. 4b) charged up under the electron beam, but based on the analysis conducted on the specimen annealed at 400 °C, are thought to consist of a Ti oxide or mixed Ti–Si oxide with traces of C (Fig. 3d). The thickness of the various oxide layers could not be determined due to charging of the surfaces during sputtering. Sputtering was discontinued after removal of about 0.5 µm with no change observable in the surface chemistry.

The interfacial chemistry of the specimen annealed at 500 °C is shown in Fig. 5d. The oxidation process at 500 °C has apparently completely undercut the coating, so that the adhesion of the coating to the fibre core is weaker than the adhesion of the coating to the

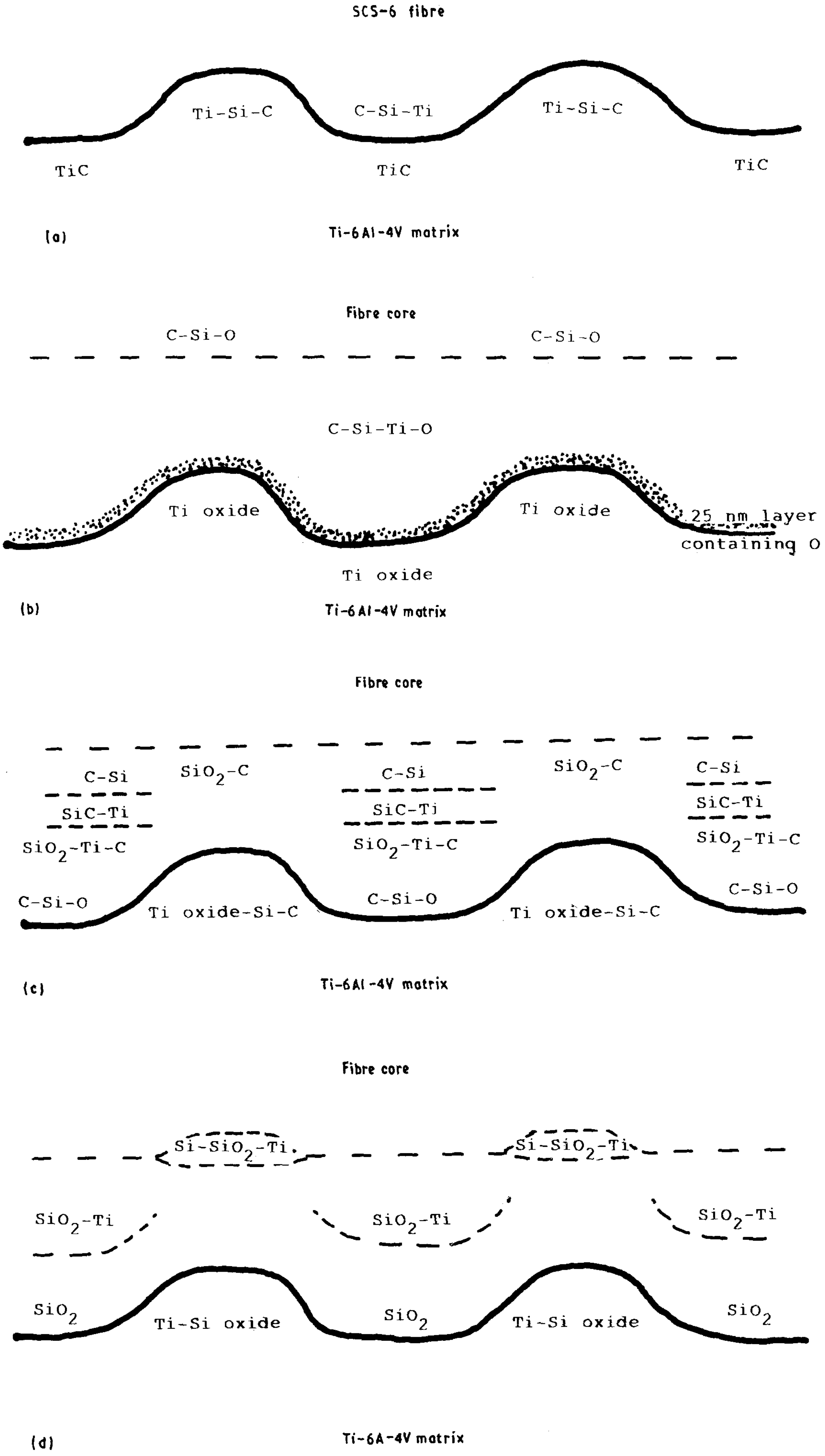

Figure 5 Schematic diagram of oxidation of the interface of Ti–6Al–4V/SCS-6 material as a function of temperature (dashed lines indicate observed fracture boundaries). (a) As-received material; annealed at (b) 300 °C for 670 h; (c) 400 °C for 500 h; (d) 500 °C for 580 h.

matrix. Upon fracture, there is an increased amount of coating separation from the fibre, and more coating layers are observed in the troughs. As at 400 °C, the chemistry of this layer corresponds to that of the inside of the coating, that is the chemistry at the fibre/coating interface.

3.2. Samples annealed in vacuum

Results of the analyses of specimens annealed for 500 h at various temperatures in vacuum are shown in Figs 6–9. The changes in fracture morphology at the different temperatures are not as dramatic as those in air. They are fairly similar to those of the as-received and 300 °C air specimens, as can be seen by comparing Fig. 6a, b with Figs 1a and 2a. There is some breakaway of the fibre coating (areas A and F), and partial thin layers of coating were observed adhering to the troughs (point E in area D).

The chemistry of the interfaces does not change as much with temperature for the specimens annealed in vacuum. The changes appear to be the low-temperature analogue to the chemistry changes observed at much higher fabrication temperatures. The fibre and trough chemistry are similar to the as-received material, with the fibre coating surface (point A in Fig. 6a) showing SiC with the presence of Ti (Fig. 7a), and the trough (area E) containing a TiC layer with the presence of Si from the fibre (Fig. 8a). However, Ti was also observed within the fibre coating (point B and Fig. 7b), and the dark flecks of coating in the trough (point F) showed a strong Ti signal mixed with the C and Si peaks for the fibre (Fig. 8b). The fibre core (area D) and the inside of the coating (area G) showed C with a trace of Si, as would be expected from unreacted fibre material (Fig. 7c). The trace of O detected at the interfaces was of the order of several nm thick, and may have come from trace oxygen in the annealing tubes. Only at 600 °C did the interfacial chemistry depart slightly from that of the lower temperatures. The trough showed only TiC and only a trace of Si (Fig. 9a). The dark flecks in the troughs showed much less Ti and more C and Si from the fibre (Fig. 9b) than at the lower temperature.

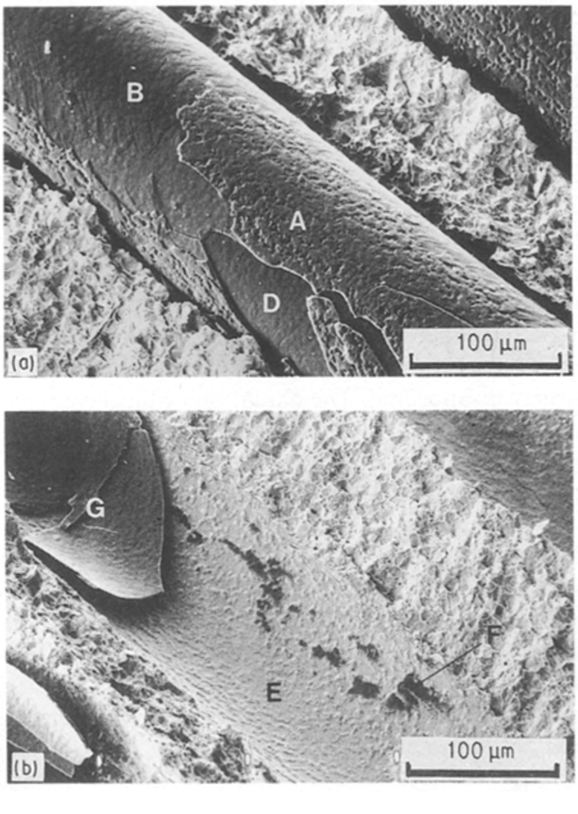

Figure 6 SEM micrographs of specimens annealed in vacuum. (a) Fibre surface of specimen annealed at 400 °C for 500 h; (b) trough surface of specimen annealed at 600 °C for 500 h.

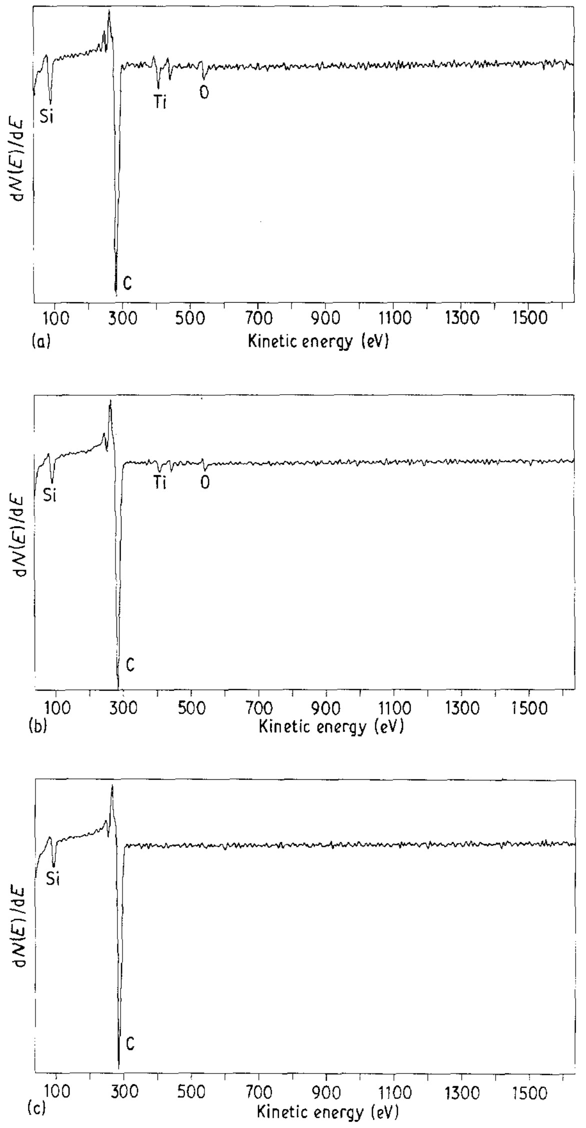

Figure 7 Typical Auger spectra of fibre surface features for specimens annealed in vacuum. (a) Fibre coating surface (500 °C, point A in Fig. 6a); (b) Middle layer in fibre coating (400 °C, point B in Fig. 6a); (c) fibre core (500 °C, point D in Fig. 6a).

4. Discussion

The results of this investigation show that the long-term exposure of a reactive composite system to operating conditions can significantly degrade the interfacial properties of the material. The kinetics of the oxidation reactions which occur at the various temperatures are determined by the oxidation of the matrix, the fibre coating and fibre core, and the interfacial chemistry originally resulting from fabrication.

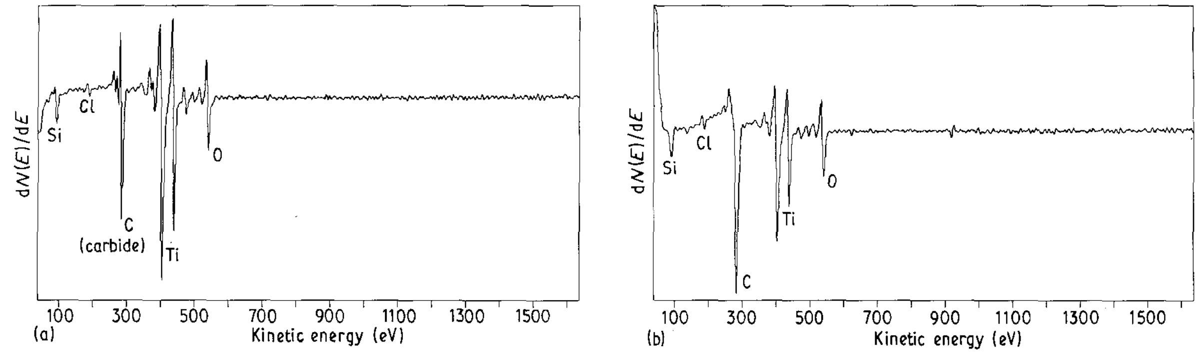

Figure 8 Typical Auger spectra of trough surface features for specimens annealed in vacuum. (a) Trough (500 °C, point E in Fig. 6b); (b) dark flecks of fibre coating adhering to trough (400 °C, point F in Fig. 6b).

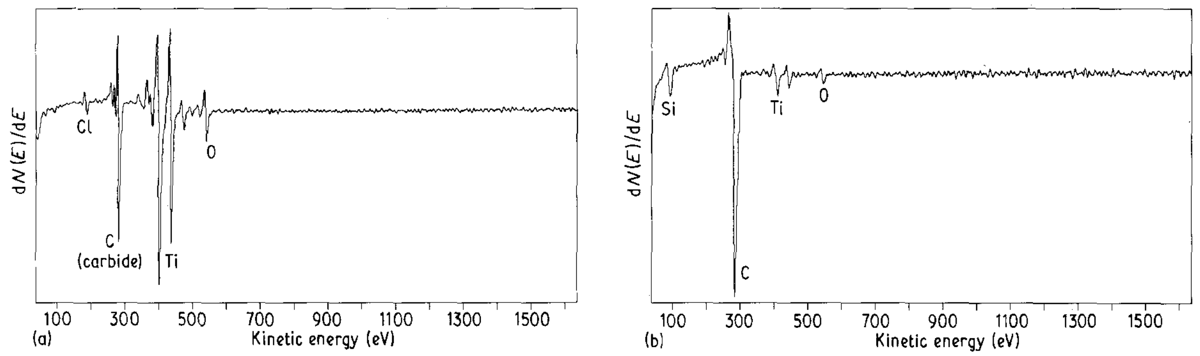

Figure 9 Auger spectra of specimen annealed in vacuum at 600 °C for 500 h. (a) Trough; (b) dark flecks of fibre coating adhering to trough.

Under the experimental conditions used, the details of the individual reactions and their contribution to the overall process cannot be determined. The reactions can, however, be qualitatively described as shown in Fig. 5.

At 300 °C, the interfacial zone is only slightly oxidized. It appears that the Ti Matrix and/or TiC reaction product is preferentially oxidized with respect to the fibre. At this relatively low temperature, the strong affinity of titanium for oxygen predominates. At the higher temperatures, the changes in interfacial chemistry are a result of the interaction of a number of simultaneous processes including:

(a) diffusion of oxygen along the interface between fibre and matrix;
(b) diffusion of oxygen along the interface between fibre core and fibre coating;
(c) oxidation of the various components in the interfacial zone (matrix, TiC fabrication reaction product, fibre coating);
(d) degradation of the fibre coating at the thinner depressions by oxidation and reaction with Ti;
(e) attack of the fibre core through the coating by oxidation and reaction with Ti (at 500 °C).

These processes result in the gradual embrittlement of the interfacial region as can be inferred from the fractographs in Figs 1–4.

The results of this investigation further show that protective fibre coatings are not only important in MMC fabrication, but must be further developed to provide protection over long periods of time at high temperatures. Even the proven coating of the SCS-6 fibre degrades at operating temperatures, as seen in Figs 1–4. This observation is further supported by the metallographic sections shown in Figs 10 and 11. An SEM micrograph of a section of the as-received material (Fig. 10a) shows by mass contrast that the fibre coating consists of three layers. As indicated in the literature, the outermost layer is carbon-rich [4, 7] with several stoichiometric variations lying underneath. A C (272 eV)-line scan across the interface shows this clearly (Fig. 10b) where there appear to be three separate C-rich layers on the fibre core. After long-term air exposure at high temperature, the outermost coating has been degraded and is no longer visible, as seen for example in Fig. 11a for a specimen annealed in air at 600 °C for 500 h. The corresponding C-line scan (Fig. 11b) shows that the outermost layer has changed in chemistry where the C signal has largely disappeared. The oxidation of this layer could not, however, be clearly shown by line scan due to unavoidable smearing of the interface during metallographic preparation.

The interfacial chemistry did not change to a great degree during the vacuum anneals. The interfacial reactions would be expected to be similar to those occurring during processing which is also conducted under vacuum conditions. At the lower temperatures, the reaction kinetics would be expected to be much lower (Arrhenius relationship) and therefore the stability of the interfaces is not surprising.

These changes in interfacial properties with time and oxygen content of the operating environment have important consequences for the design of high-temperature components with reactive metal–matrix

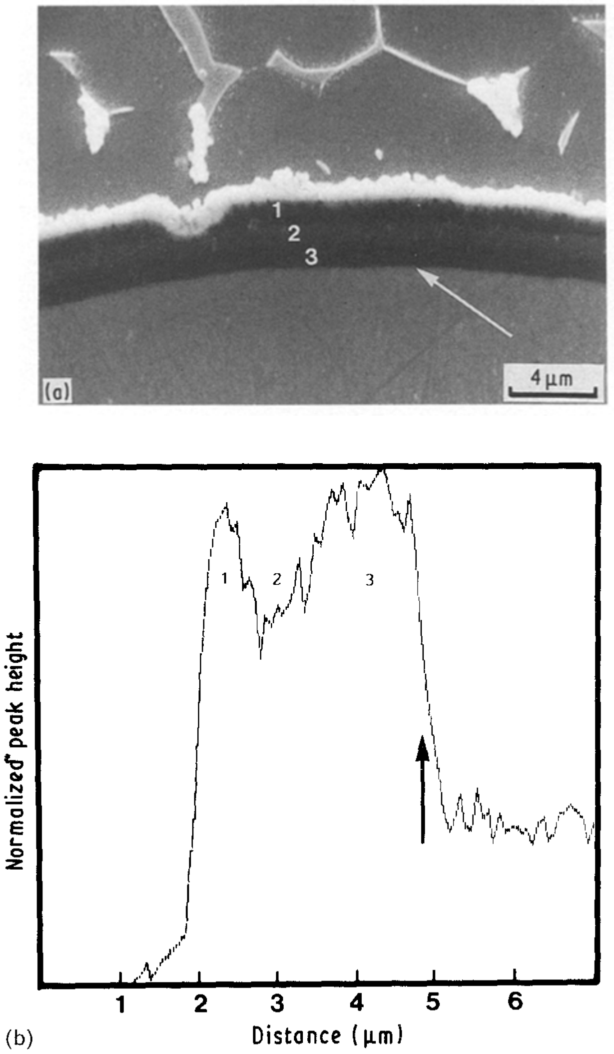

Figure 10 Auger line scan of fibre/matrix interface of as-received material. (a) SEM micrograph (arrow indicates fibre coating/fibre core interface); (b) C (272 eV) line scan: layers 1, 2 and 3 and arrow correspond to layers and interface in (a).

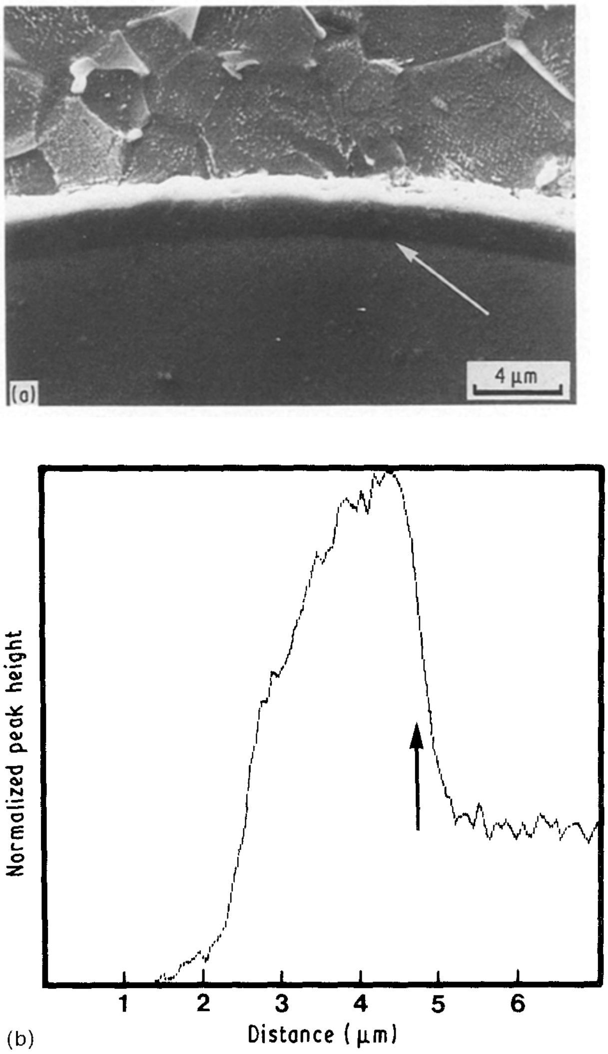

Figure 11 Auger line scan of fibre/matrix interface of specimen annealed in air at 600 °C for 500 h. (a) SEM micrograph (arrow indicates fibre coating/fibre core interface); (b) C (272 eV) line scan: arrow indicates fibre coating/fibre core interface in (a).

composites. The high-temperature data commonly provided by MMC manufacturers are obtained from tests conducted for short times at a given temperature. Tensile tests last at most a few hours between heating up and cooling down, and most fatigue tests last of the order of a few days. However, it was shown in a previously cited study [12] that the embrittlement of the interfaces by long-term exposure to high temperatures in air led to a reduction in the low cycle fatigue life of SiC/Ti–6Al–4V. Premature separation of the fibre from the matrix along the fibre coating/fibre core interface was thought to reduce the beneficial crack-blunting effect of the continuous reinforcement. Thus the use of currently available data for the design of high-temperature MMC components can be misleading and may result in non-conservative life expectations.

Another factor to be considered in the design of such components is the oxidation resistance of the material. One of the purposes of continuous fibre reinforcement of metals is to increase their operating temperature. The matrix alloys currently under consideration were not designed for these temperatures, and do not have the oxidation resistance required for service. In addition, the presence of the fibre/matrix interface allows for rapid diffusion of oxygen and internal oxidation of the MMC. Thus a major effort must be made to develop protective coatings for the matrix materials. As shown in this study, the exclusion of oxygen reduces the degradation of the interfaces at operating temperature.

5. Conclusions

An investigation was conducted to determine the effect of long-term high-temperature exposure on the interfacial properties of a typical SiC continuous fibre-reinforced titanium alloy. It was shown that exposure to air at temperatures up to 500 °C for times up to 700 h resulted in embrittlement of the fibre/matrix interface through combined oxidation and attack of the Ti matrix on the fibres and fibre coating. Exposure to operating temperatures in vacuum did not lead to a significant degradation of the interfaces. The degradation of the interfaces in high-temperature service can lead to a reduction of the service life of components made of reactive-system MMCs such as SiC-reinforced Ti alloys.

Acknowledgements

The author would like to thank Dr M. Weiss of Perkin Elmer, Oberschleißheim (Munich) for his assistance in the Auger work, and Dr H. Klingele of Institut Dr Klingele, Munich for his assistance in the SEM work.

References

1. A. G. METCALFE, in "Metallic Matrix Composites, Composite Materials", Vol. 4, edited by K. G. Kreider (Academic, New York, 1974) p. 269.
2. V. B. RAO, C. R. HOUSKA, J. UNNAM, W. D. BREWER and D. R. TENNEY, in Proceedings of the AIME Symposium – Physical Metallurgy and Composites, October, 1978 (Metallurgical Society of AIME, Warrendale, Pennsylvania, 1978) p. 347.
3. W. D. BREWER and J. UNNAM, in "Mechanical Behavior of Metal–Matrix Composites", edited by J. E. Hack and M. F. Amateau (Metallurgical Society of AIME, Warrendale, Pennsylvania, 1983) p. 39.
4. P. R. SMITH and F. H. FROES, *J. Metals* **36** (1984) 19.
5. H. J. DUDEK, L. A. LARSON and R. BROWNING, *Surf. Interface Anal.* **6** (1984) 274.
6. H. J. DUDEK, R. LEUCHT and G. ZIEGLER, in Proceedings of the 5th International Conference on Titanium, Munich, September 1984, edited by G. Lütjering, U. Zwicker and W. Bunk (Deutsche Gesellschaft für Metallkunde e.V., Oberursel, 1985) Vol. 3, p. 1773.
7. C. G. RHODES and R. A. SPURLING, in "Recent Advances in Composites in the United States and Japan", ASTM STP 864, edited by J. R. Vinson and M. Taya (American Society for Testing and Materials, Philadelphia, 1985) p. 585.
8. R. LEUCHT, H. J. DUDEK and G. ZIEGLER, *Zeitschrift für Werkstofftechnik* **18** (1987) 27.
9. C. G. RHODES, A. K. GHOSH and R. A. SPURLING, *Metall. Trans. A* **18A** (1987) 2151.
10. R. KIESCHKE, R. SOMEKH and T. W. CLYNE, in "Developments in the Science and Technology of Composite Materials", Proceedings of Third European Conference on Composite Materials, Bordeaux, France, March 1989 (Elsevier, London, 1989) p. 265.
11. P. R. SMITH, F. H. FROES and J. T. CAMMETT, in "Mechanical Behavior of Metal–Matrix Composites", edited by J. E. Hack and M. F. Amateau (Metallurgical Society of AIME, Warrendale, Pennsylvania, 1983) p. 143.
12. W. WEI, in "Fundamental Relationships Between Microstructure and Mechanical Properties of Metal–Matrix Composites", edited by P. K. Liaw and M. N. Gungor (Metallurgical Society of AIME, Warrendale, Pennsylvania, 1990) p. 353.
13. M. V. HARTLEY, in Proceedings of the 9th Risø International Symposium – Mechanical and Physical Behaviour of Metallic and Ceramic Composites, Roskilde, Denmark, September 1988, edited by S. J. Andersen, H. Lilholt and O. B. Pedersen (Risø National Laboratory, Roskilde, 1988) p. 383.
14. Y. H. PARK and H. L. MARCUS, in "Mechanical Behavior of Metal–Matrix Composites", edited by J. E. Hack and M. F. Amateau (Metallurgical Society of AIME, Warrendale, Pennsylvania, 1983) p. 65.
15. D. BRIGGS and J. C. RIVIÈRE, in "Practical Surface Analysis by Auger and X-ray Photoelectron Spectroscopy", edited by D. Briggs and M. P. Seah (Wiley, Chichester, 1983) p. 87.

Received 27 November 1990
and accepted 10 April 1991