

Letters

Thermal fatigue damage in Borsic Al (6061) composites*

Thermal stresses generated in fibre-reinforced composites due to expansion coefficient mismatch ($\Delta\alpha$) between the fibre and the matrix can be a serious problem [1–3]. The magnitude of thermal stresses generated is, of course, proportional to $\Delta\alpha$ and to the amplitude of the thermal cycle, ΔT . However, it has always been a tacit understanding that in metal matrix composites thermal fatigue would never amount to a serious problem as the ductile metal will always be there to accommodate the thermal stresses generated due to temperature changes, deliberate or inadvertent [2–4]. The purpose of this note is to draw attention that in real life things can be a bit different, especially when the number of cycles is quite large (>100).

In a composite material subjected to thermal cycling, thermal stresses would be generated in both the components, fibre and matrix. These thermal stresses can be relieved by one or more of the following means: (i) plastic deformation of the ductile metal matrix; (ii) cracking or failure of the brittle fibre; (iii) failure of the fibre matrix interface.

One should remark here that boron fibre itself, as it is produced currently, is a composite material with a core consisting mainly of borides of tungsten and a shell consisting mainly of elemental boron. The interface between the two can be a source of weakness just as the interface between the boron fibre and the aluminium matrix can be. If the fibre/matrix interface is very strong, and if the boron fibre has a decent, well-developed core, i.e. tungsten boride core/boron interface is not a source of weakness then, of course, plastic deformation of the soft aluminium matrix is the only avenue left to accommodate the thermal stresses generated. In the commercially produced Borsic*/Al composites, these conditions are obtainable up to a few hundred cycles [4]. Above this number, however, these conditions may not be encountered, and sure enough, we observe (as shown below) all the three means enumerated above to be in operation during

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thermal fatigue involving a large enough number of cycles.

Samples of Borsic*/Al (3 mm \times 3 mm \times 32 mm), with fibres aligned at 0° or 90° to the long axis and containing a volume fraction of 10% were cycled from 310 to 625 K at a rate of 6 cycles h^{-1} . The encapsulated samples were lowered in a furnace, held there for some time, withdrawn and cooled in a jet of air. The samples were removed periodically for microstructural examination in scanning electron microscope. Within the first few cycles one observes slip lines in the aluminium matrix, followed by grain-boundary sliding above about 50 cycles. With continued cycling the whole

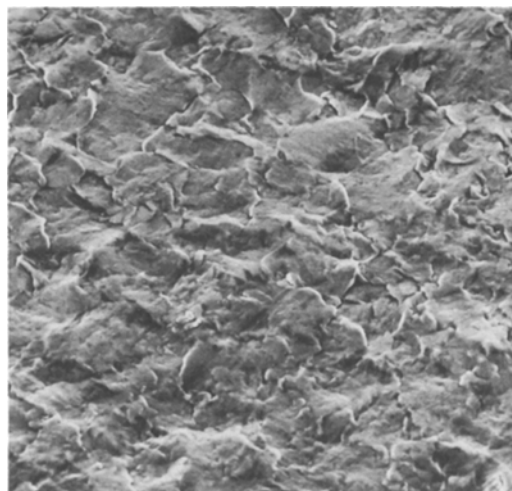


Figure 1 Grain-boundary sliding in Al matrix after 500 cycles, $\times 50$ (subsequently reduced by 30% for reproduction).



Figure 2 Multiple cracking in boron fibre after 1500 cycles, $\times 50$ (subsequently reduced by 30% for reproduction).

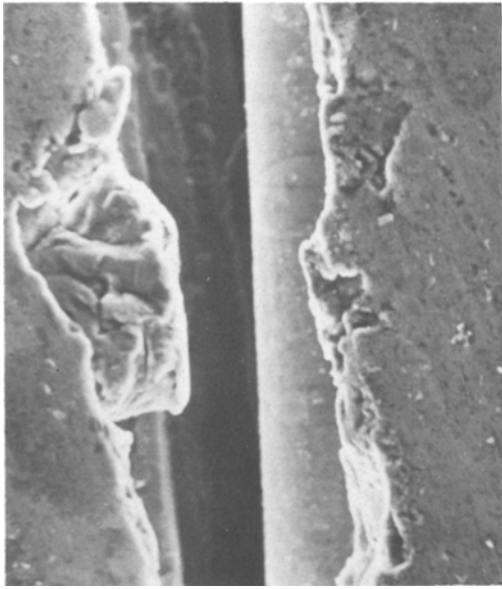


Figure 3 Decohesion at Borsic/Al interface after 1500 cycles, $\times 300$ (subsequently reduced by 20% for reproduction).

of matrix suffers grain-boundary sliding in response to the thermal stresses. Fig. 1 shows the grain-boundary sliding in Al after 500 cycles. This had been found earlier too [4]. With still further cycling, damage to boron fibres and fibre/matrix interface also sets in. Multiple cracking in boron fibre is shown in Fig. 2 while decohesion at fibre/matrix interface is shown in Fig. 3. Both of these figures

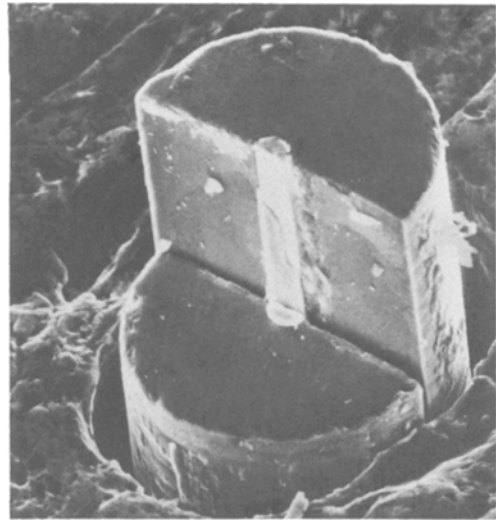


Figure 5 Diametrically split boron fibre has one part slid past another, $\times 400$ (subsequently reduced by 30% for reproduction).

are from 0° samples after 1500 cycles. Cracking in boron fibre starts at the boron-tungsten boride core interface. This is demonstrated in Fig. 4a and b of the boron fibre after 1500 cycles. One can notice the crack starting at the boron/core interface in Fig. 4a. Fig. 4b, a higher magnification of the core region of Fig. 4a, shows vividly the microcracks present at the boron/core interface. Boron fibre can also split diametrically due to thermal stresses, and the two separated parts can slide past

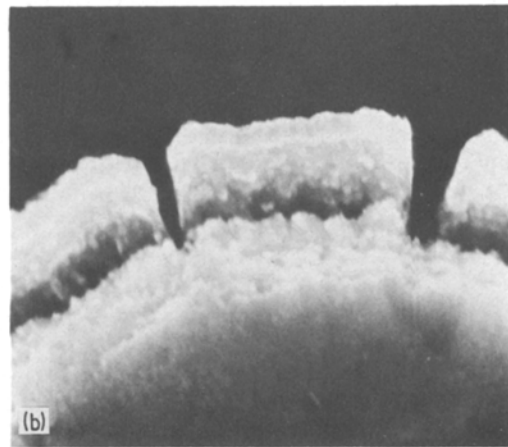
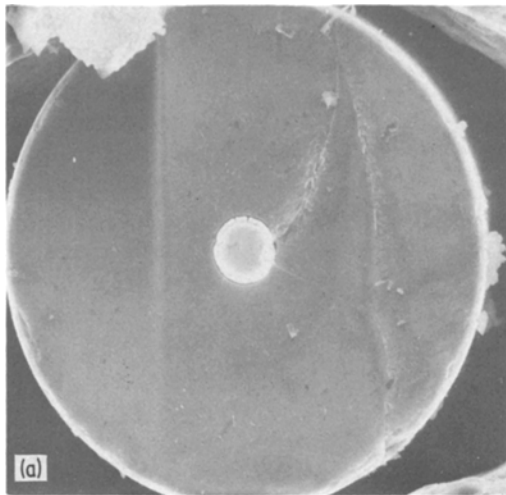


Figure 4 Borsic fibre after 1500 cycles. Notice the crack nucleation at boron/core interface. (a) $\times 500$, (b) $\times 10\,000$ (both subsequently reduced by 30% for reproduction).

each other due to the shear stresses generated. A dramatic evidence of this phenomenon is seen Fig. 5. Again, one notices that crack started at the boron/core interface. Also to be noted is the separation of the boron fibre/aluminium matrix interface. In conclusion then, all three means enumerated above operate as result of thermal fatigue involving greater than 100 cycles and lead to a much severe microstructural damage in these commercially produced Borsic*/Al composites.

Acknowledgements

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References

1. E. SCALA, in "Fiber Composite Materials" (ASM, Metals Park, Ohio, 1965) p. 131.
2. K. K. CHAWLA, *Metallogr.* 6 (1973) 155.
3. *Idem*, *Phil. Mag.* 28 (1973) 401.
4. *Idem*, in "Grain Boundaries in Engineering Materials", Proceedings of 4th Bolton Landing Conference, edited by J.L. Walter, J.H. Westbrook and D.A. Woodford (Claitor's Baton Rouge, La., 1975) p. 435.

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The etching of (1 1 1) As surfaces of GaAs with nitric acid

Chemical etching is a common process used for the study of the properties of semiconductor materials. In the case of GaAs dislocations are usually detected on the (1 1 1) Ga plane using a nitric acid solution (Schell etch). However little attention has been paid to the reaction of nitric acid on (1 1 1) As surface apart from the observation that As_2O_3 crystals are deposited in it [1].

Results are presented here which show that under certain conditions of solution strength and volume a different etching behaviour is found.

The material used for these experiments was Cr doped (1 1 1) orientated bulk GaAs with the two (1 1 1) surfaces being etch polished using sodium hypochlorite [2] to produce a mirror-like surface finish. The material was then cleaved into samples about 2 mm × 1 mm and cleaned in acetone, trichloroethylene and methanol in an ultrasonic cleaner.

The etched surfaces were studied in an SEM (Cambridge stereoscan Mk IIA) and in some case it was found advantageous to coat the samples with a thin (~300 Å) gold film, due to the insulating nature of the surface films.

The dilution ratio of the etchant being the ratio of the volume of water to that of the concentrated acid.

When the samples were etched in 25 ml nitric

acid solutions of dilution ratios 1.5 and 2 for times between 4 and 60 min without any agitation at room temperature cubic crystals were produced on their surfaces in a random manner (see Fig. 1). A microprobe analysis of these crystals showed them to contain As and subsequently X-ray diffraction showed these crystals to be Arsenolite (As_2O_3). According to Kyser and Millea [1] these adhere to the sample surface by means of a raised pyramidal base of GaAs, although here this was only found when a short etching time (25 sec) and a nitric acid solution dilution ratio of 1 were



Figure 1 Cubic As_2O_3 crystals, × 1327.