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Thermal-mechanical fatigue behaviour of alumina fibre/aluminium–lithium composite

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Metal matrix composites are candidates for elevated temperature applications. For this reason, it is important to understand their behaviour under thermal-mechanical fatigue conditions. Thermal cycling of a composite material creates thermal stresses in the composite because of thermal expansion mismatch between the fibre and the matrix. This can lead to plastic deformation of the matrix, interface damage, and fibre fracture. Mechanical cyclic loading of the composite during thermal cycling can aggravate the situation even more. A computer-controlled servo-hydraulic thermal-mechanical fatigue test system was used to perform tests on α-Al_2O_3(FP)/Al–2%Li metal matrix composite specimens. The volume fraction of unidirectionally aligned fibres was 35%. The tests performed were free-expansion tests, fast and slow thermal fatigue tests, and isothermal fatigue tests. Large reductions in the composite strength were observed under thermal fatigue conditions. This degradation can be attributed to observed microstructural damage of the fibre/matrix interface and fibre fracture.

1. Introduction

In any assessment of the mechanical properties of a metal matrix composite reinforced by ceramic fibres, strength of the fibre/matrix interfacial bond is a very important parameter. Thus any treatment which may alter the interfacial region of the composite will also change its mechanical properties. Thermal treatments are especially important because of the large thermal expansion mismatch between the metal matrix and the ceramic fibre materials. Stresses arising from this thermal expansion mismatch can degrade the fibre/matrix interface, thus decreasing the efficiency of load transfer from the matrix to the fibre [1]. The thermal stresses generated can also plastically deform the matrix and work-harden it. Thermal stresses are greatest during changes in temperature and thus are most evident in thermal cycling experiments.

The effects of thermal treatments on the interface microstructure and mechanical properties of α-Al_2O_3 (FP)/Al–Li composites have been studied by some researchers [2–5]. Kim *et al.* [2] described a trend toward decreasing transverse tensile strength in thermally cycled specimens and attributed it to interfacial failure initiated by void formation; these voids resulted from the thermal expansion mismatch between the fibre and the matrix. Greater reductions in the transverse tensile strength occurred for thermally cycled specimens with equal total time at the maximum temperature, T_{max}, than for isothermally treated samples. Hunt [3] found the same decreasing trend with thermal cycling and attributed this drop to a reduction in interfacial shear strength caused by the thermal expansion mismatch. X-ray diffraction studies showed interfacial reaction products to be $Li_2O \cdot 5Al_2O_3$. Hall and Barrailler [4] described similar effects in their study involving isothermally heat-treated samples. Chawla [5] studied samples treated isothermally and by thermal cycling. A degradation in strength resulted from both treatments, but with a greater effect from thermal cycling. Thermal cycling also caused a preferential loss of lithium for the fibre/matrix interface regions near the surface.

In the studies on FP alumina/Al mentioned above, the samples were subjected to heat treatment in an unloaded state. If the composite sample is in a loaded state during the thermal treatment, we expect the strength degradation to occur at an accelerated rate because of the combined effect of applied mechanical stresses and internal thermal stresses. In most elevated temperature applications, a part made of a composite material is likely to be subject to simultaneous thermal and mechanical loading. The purpose of the present work was to characterize the response of α-Al_2O_3(FP)/Al–Li composites to simultaneous mechanical cyclic loading and thermal cycling.

2. Material

The material used in this study was α-Al_2O_3(FP)/Al–2% Li composite, with a fibre volume fraction of 35%. The FP fibre is a polycrystalline α-Al_2O_3 fibre with an average diameter of 20 μm and a purity of greater than 99%. The ultimate tensile strength and elastic modulus are approximately

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1400 and 380 GPa, respectively [6]. The alumina (FP)/Al–Li composite was fabricated commercially by liquid metal infiltration of a fibrous preform in vacuum. The FP fibres, in the form of a fibre prepreg with a fugitive binder, were loaded into a mould. The mould was then infiltrated with molten metal, after burning the fugitive binder, to form the composite.

The matrix material was an aluminium alloy containing 2% lithium. The lithium is added to allow the molten aluminium to wet the FP fibres, but also has the effect of hardening the matrix. There is an optimum value for lithium concentration; too little lithium will not allow proper wetting of the fibre, giving void formation at the interface, while too much lithium will lead to fibre degradation and a broad interfacial zone [6]. Fig. 1, an optical micrograph, shows a transverse section of the as-received FP/Al composite. Note the fairly uniform distribution of fibres and a very thin, peripheral zone on fibres where lithium has reacted with alumina forming a minimal interfacial zone.

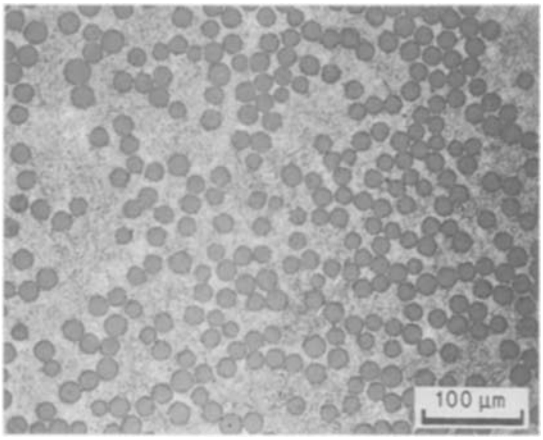

Figure 1 Cross-section of as-received α-Al_2O_3(FP)/Al–Li composite.

3. Experimental procedure

Fatigue specimens, Fig. 2, were machined from the as-cast composite material. A computer-controlled thermal fatigue test system [8], installed at Sandia National Laboratory was used. The system consisted of a closed loop servo-hydraulic test frame controlled by a DEC PDP 11/34 computer through an MTS 433 interface. The heat was applied by an r.f. induction heating coil designed specifically to eliminate thermal gradients in the gauge of the specimen. A blower was used to cool the specimen during the downramp in the temperature cycle.

Several tests may be performed with this test system. The tests used in this study were free-expansion, thermal fatigue, and isothermal fatigue tests. Free-expansion tests involved free expansion of the sample with no external loading during a temperature cycle. The strain induced by the thermal expansion of the material was measured. Also during the free-expansion test, the modulus of the specimen was measured. This was done by first rezeroing the extensometer and then loading the sample to 50 MPa and back to zero. The slope of the resulting stress–strain curve was measured and the elastic modulus calculated. This procedure was repeated 25 times for each temperature and the average value was taken. Thermal fatigue tests involved cycling the temperature of the sample while the sample gauge length was kept constant. This constraint resulted in a stress on the composite sample that varied with temperature. The peak stresses for each cycle were recorded and plotted versus number of cycles. Two types of thermal fatigue tests were used: slow and fast thermal fatigue tests, which differed in the length of the thermal cycle. These tests gave data on stress required to keep the specimen gauge length constant during a temperature cycle. The isothermal fatigue tests were traditional mechanical fatigue tests performed at an elevated temperature. These gave data on cycles to failure of the material at a given temperature. The fatigue tests performed by this system were strain-controlled, i.e. the sample was cycled between fixed values of strain.

After testing, the samples were sectioned with a low-speed diamond saw and polished with diamond paste down to 0.1 μm. The samples were then examined using optical and scanning electron microscopy to observe any microstructural changes in the matrix or at the fibre/matrix interface, or to observe for evidence of fibre fracture.

4. Results

4.1. Free-expansion tests

The variation of thermal strain as a function of temperature during a single thermal cycle is shown in

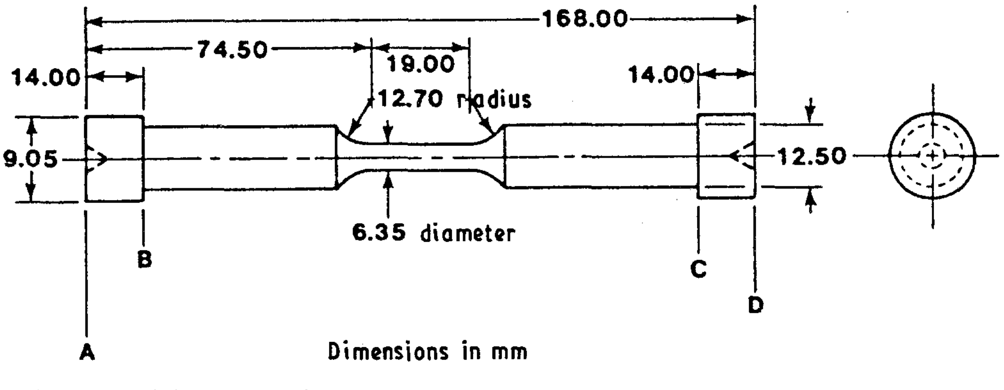

Figure 2 Mechanical-thermal fatigue test sample.

Fig. 3. For a temperature change from 25 to 500 °C, the strain induced by thermal expansion was 0.004%. The Young's modulus, E, of the composite was measured in this free-expansion test as a function of temperature. Fig. 4 shows the variation of E with temperature. As expected, the modulus of the composite decreased slightly with increasing temperature from 167.5 GPa at 50 °C to 145.8 GPa at 500 °C.

4.2. Slow thermal fatigue tests

The slow thermal fatigue tests were run from 50–500 °C with a 10 min cycle time. Because of time constraint, the number of cycles for the test was limited to 100. Fig. 5 shows the maximum tensile and compressive stress versus the number of cycles. The maximum stresses in the sample levelled out during the first few cycles and remained about constant for the duration of the test. It can be assumed that the sample would have withstood several more cycles before failure, as the microstructure did not show any large-scale damage development at this stage. Fig. 6 shows an optical micrograph after 100 cycles showing little or no damage.

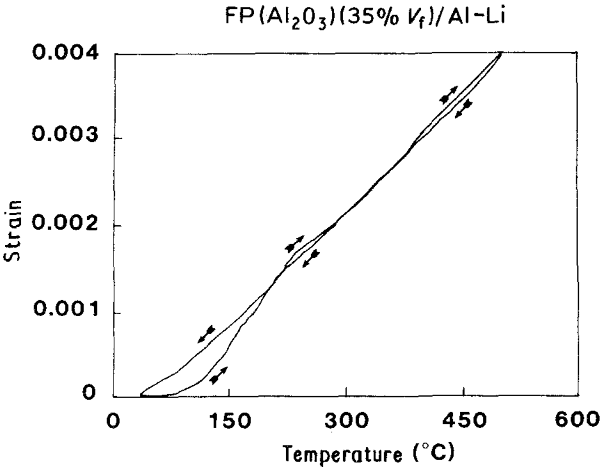

Figure 3 Strain versus temperature of FP/Al during a single free-expansion thermal cycle.

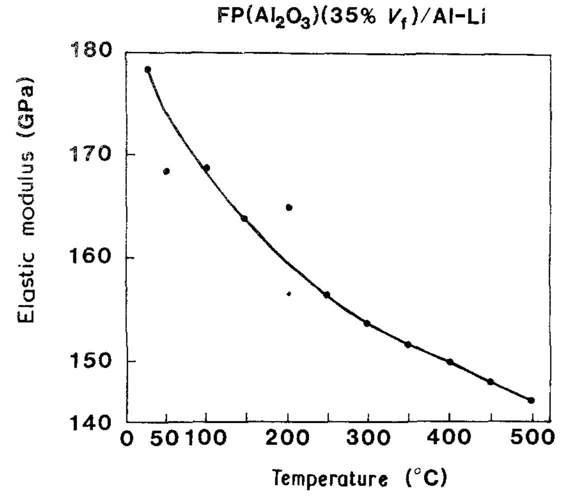

Figure 4 Young's modulus versus temperature measured during free-expansion tests.

4.3. Fast thermal fatigue tests

The fast thermal fatigue tests involved a 30 s cycle time and a temperature range from 300–500 °C. Fig. 7 shows the stress versus temperature during a single fast thermal cycle. The hysteresis loop corresponds to a single cycle. Fig. 8 shows a plot of the maximum stress in tension and compression versus number of cycles for two samples in this test. It can be seen that

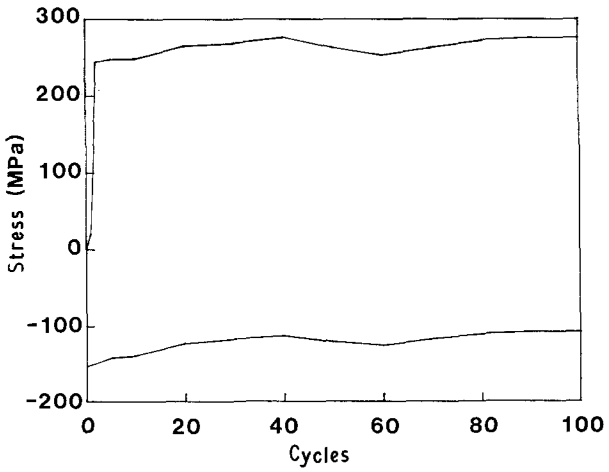

Figure 5 Maximum stress versus number of cycles during slow thermal fatigue testing.

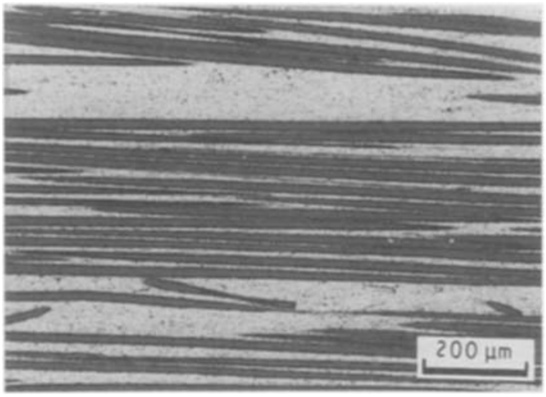

Figure 6 Optical micrograph of a slow thermal fatigue specimen showing little or no damage development after 100 cycles between 50 and 500 °C.

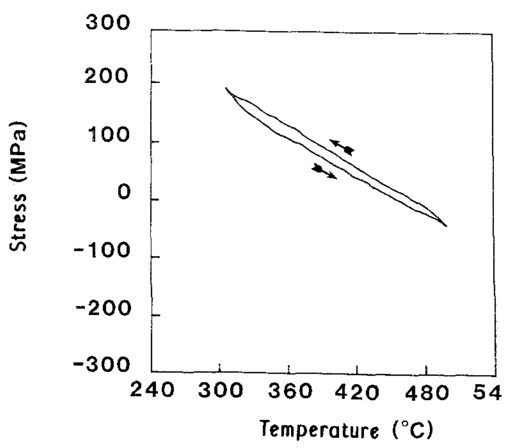

Figure 7 Stress versus temperature during a fast thermal fatigue temperature cycle.

the stress first increased and then dropped off. The initial increase in the curve is evidence of work hardening of the matrix caused by the thermal stresses arising from the thermal expansion mismatch between the components of the composite. The decrease in the stress levels is attributed to widespread fibre fracture in the specimen. Fig. 9, a scanning electron micrograph, shows transverse cracks in alumina fibres in a longitudinal section of a sample after 489 cycles.

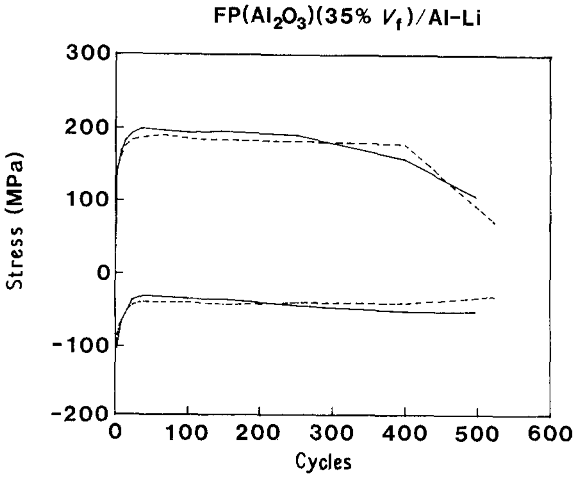

Figure 8 Maximum stress versus number of cycles during fast thermal fatigue testing for two specimens.

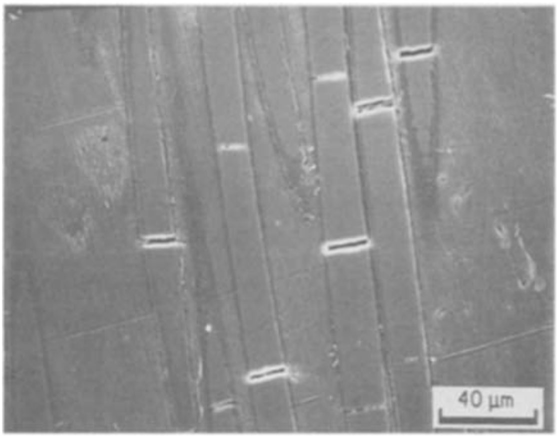

Figure 9 Scanning electron micrograph showing fibre fracture caused by fast thermal fatigue testing.

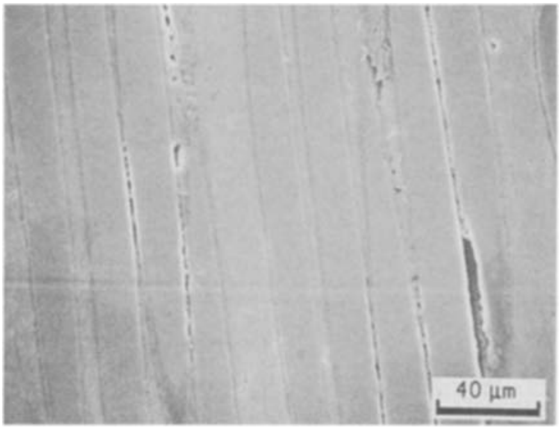

Figure 10 Scanning electron micrograph showing interfacial cracking resulting from fast thermal fatigue testing.

Fig. 10 shows interfacial separation caused by thermal stresses; while Fig. 11 shows the circumferential zone at the fibre surface, from which lithium diffused out preferentially, leaving behind a cracked and voided interface [5].

4.4. Isothermal fatigue tests

Several typical stress versus strain loops for strain-controlled cycling under mechanical fatigue at a constant temperature are shown in Fig. 12. The temperature of the test was set at 300 °C, and the strain range was set as half of the maximum strain observed during the free-expansion test, which was 0.25%. The test ran

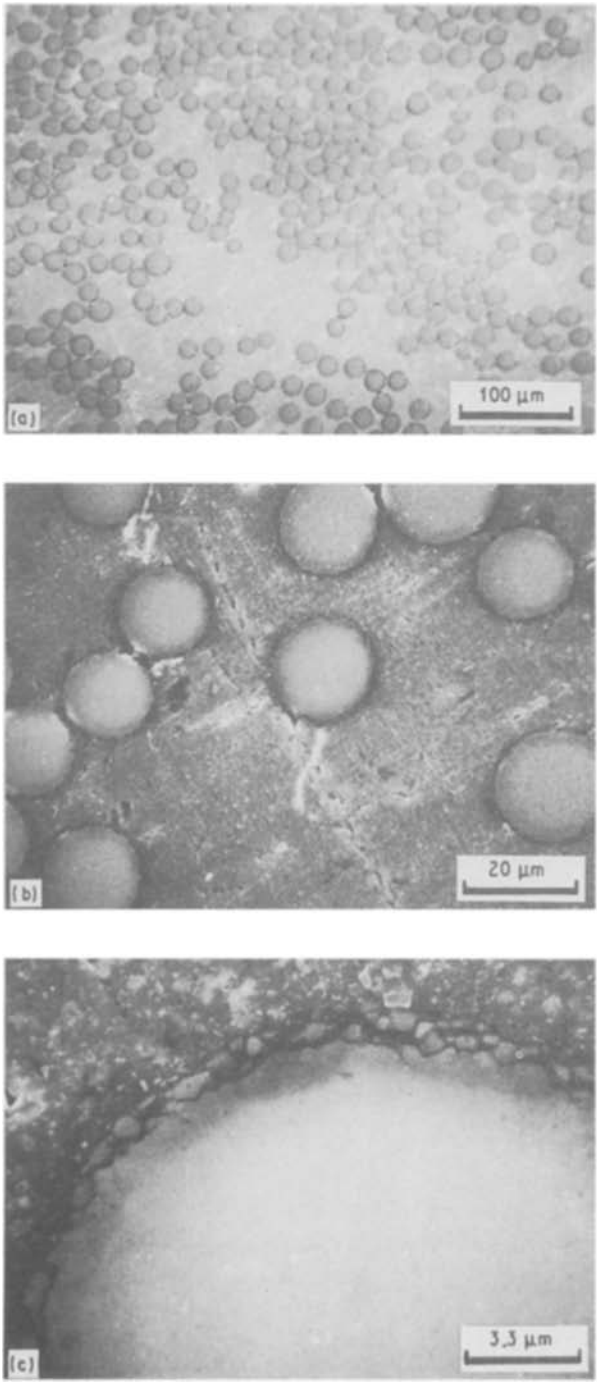

Figure 11 Scanning electron micrographs showing interfacial degradation because of preferential diffusion of lithium from the surface during fast thermal fatigue test.

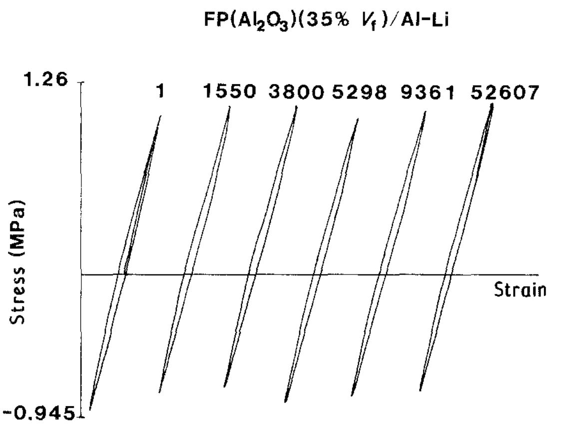

Figure 12 Stress versus strain hysteresis loops for strain-controlled isothermal fatigue test at 0.25% total strain and 300 °C.

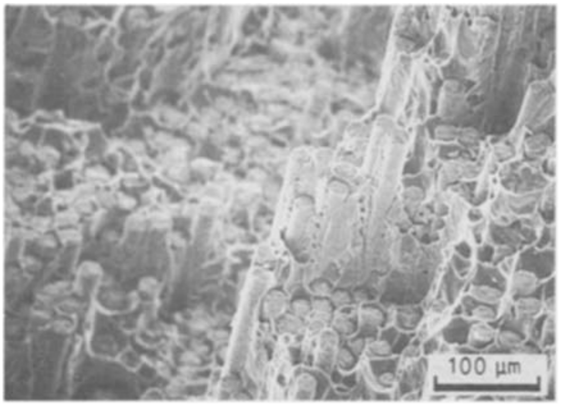

Figure 13 SEM fractograph of an isothermal fatigue specimen tested at 0.5% total strain. Note the fibre pull-out and some porosity at the interface.

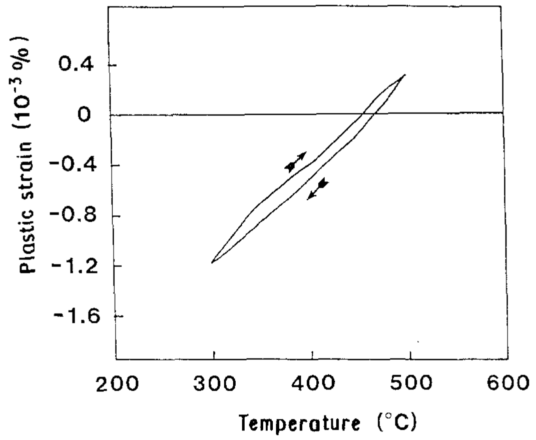

Figure 14 Plastic strain in matrix versus temperature during a fast thermal fatigue cycle.

for 52 607 cycles with no change in the hysteresis loop. The test was then repeated with a strain range of 0.5%. The sample failed after 331 cycles, but again there was almost no change in the hysteresis behaviour. The fracture surface, as seen in the SEM, is shown in Fig. 13. Fibre pull-out, as well as porosity at the interface, indicating interface damage, can be seen.

5. Discussion

Thermal stresses due to temperature changes arise from the thermal expansion mismatch between the fibre reinforcement and the matrix material [1, 8, 9]. The matrix undergoes plastic deformation to relieve the thermal stresses. This deformation starts at the fibre/matrix interface and spreads out radially with increasing number of cycles [1, 8]. With continued cycling, void formation at the interface and/or fibre damage sets in. The composite as a whole cycles between tensile and compressive stresses when subjected to a thermal cycle under a constant gauge length. It goes into overall compression at high temperatures where the matrix (aluminium) would like to expand much more than the fibre, but is not allowed to because of the applied constraint.

We can examine the level of plastic strain in the matrix during a fast thermal cycle in the following manner. The plastic strain in the matrix, ε_p, can be calculated from the following relationship

$$\Delta\varepsilon_{\mathrm{mech}} = \varepsilon_p + \Delta\sigma_c/E \qquad (1)$$

where $\Delta\varepsilon_{\mathrm{mech}}$ is the total mechanical strain, which was held at zero throughout the thermal fatigue cycle, and $\Delta\sigma_c/E$ is the elastic strain in the composite, which was calculated from the stress versus temperature plot in Fig. 7 and modulus versus temperature plot generated during the free-expansion test, Fig. 3. Fig. 14 shows the plastic strain in matrix as a function of temperature in a single fast thermal cycle. It can be seen that most of the strain during a temperature cycle is compressive.

The slow thermal fatigue data showed trends similar to those seen from the fast thermal fatigue tests. Because of the larger cycling temperature difference, it would be expected that the matrix would undergo a greater amount of plastic deformation in a single temperature cycle. Fig. 14 shows that the amount of plastic strain per temperature cycle is much greater than that seen in the fast thermal fatigue tests. But because the cycling rate, and consequently the strain rate, is much lower, the matrix might be able to accommodate the increased strain to a higher level.

The isothermal fatigue tests demonstrated the high fatigue resistance of FP/Al composites, even at elevated temperatures. The sample tested with a 0.25% strain range did not fail after more than 50 000 cycles. A sample subjected to the 0.5% strain range test, however, failed after 331 cycles. As can be seen by the fractograph shown in Fig. 13, fibres were pulled out of the matrix in regions where the fibre/matrix interface was debonded.

6. Conclusions

1. The free-expansion test showed that the elastic modulus of the FP alumina/Al–Li composite decreased with increasing temperature up to 500 °C, mainly because of the decrease in the matrix elastic modulus.

2. Thermal fatigue tests (cycling the sample temperature while the sample gauge length is kept constant)

showed that the composite stabilized in a matter of a few cycles.

3. Although the composite as a whole experienced mostly tensile stresses during thermal cycling, the matrix experienced mostly compressive (plastic) loading. The fibres are loaded elastically in tension during thermal cycling.

4. The microstructural damage involved the embrittlement of some of the fibre/matrix interface regions, void formation at the interface, fibre fracture, and finally matrix fracture leading to the failure of the composite.

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