MEASUREMENT OF FIBER FRACTURE AND FIBER-MATRIX INTERFACE SHEAR STRENGTHS IN METAL MATRIX COMPOSITES

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INTRODUCTION

Metal matrix composites (MMC's) are receiving considerable attention as candidate materials for aerospace applications. By combining the high elastic stiffness of ceramics such as silicon carbide, alumina, and graphite with an elastically soft, ductile light metal such as aluminum, magnesium, or titanium, materials of very high specific modulus and acceptable crackgrowth resistance are in principle attainable. In practice, high specific modulus has to date been achieved at the expense of acceptable toughness. This has stimulated renewed interest in the fundamental fracture micromechanisms of MMC's.

Studies of MMC fracture micromechanisms usually must contend with very complex stress distributions and slip mechanisms within a heterogeneous deforming body and the absence of non-invasive methodologies for observing the fracture processes at the microscale. The complex internal stress distributions arise from the elastic incompatibility of the matrix and reinforcement constituents, the spatial distribution of reinforcement, and spatially varying residual stresses due to differential thermal contraction of the two constituents during cooling from process temperatures.

For basic studies of MMC fracture micromechanisms, we have attempted to eliminate some of these problems through the design of a model composite that still retains the essential features of import, but in a configuration more amenable to analytic work. The model composite consists of a single ceramic fiber grown along the center of a single crystal aluminum tensile sample. Using acoustic emission, interrupted test metallography and digitally recorded stress-strain behavior, we are exploring methodologies for quantitative determination of key microstructure parameters such as the fiber fracture strength, the fiber-matrix interface cohesion and the matrix slip behavior.

EXPERIMENTAL

Dumbbell geometry single crystal tensile samples were used for this study. They were prepared from 99.99% aluminum and 140µm
FOR ADHESION MEASUREMENT

ACOUSTIC EMISSION INSTRUMENTATION
diameter silicon carbide fibers with unmodified surfaces using a modified Bridgman technique shown schematically in Figure 1. A high density graphite mold was internally machined to accommodate the growth of an aluminum single crystal with a 57mm gauge length and 4mm gauge diameter. The fiber was centrally located down the longitudinal axis of the mold, retained at the top by means of a screw and at the bottom by graphite glue attachment to a plug. The plug acted as a weight on the fiber, maintaining it centrally during subsequent crystal growth.

The mold was attached to a water-cooled pedestal, the system evacuated and backfilled with argon and then heated using a radio frequency induction furnace. The mold was heated to approximately 900°C and solidification achieved by lowering through a water-cooled copper chill at constant velocity. A combination of steep temperature gradient and slow growth speed was used to ensure a single crystal solidification (1). Two growth velocities, with drastically different resulting reinforcement-matrix interfaces, were used. Samples with shallow reaction zones were grown at 0.41mms⁻¹ while samples with extensive reaction zones were grown at a velocity of 0.0083mms⁻¹. Samples with no fibers were also grown so that fiber effects could be separated from matrix behavior.

The oxide normally present on an aluminum single crystal is a potential extraneous acoustic emission source during tensile testing. It was removed by electropolishing the entire sample in a 6% perchloric acid/methanol solution at -30°C.

The specimens were mechanically tested in tension using a screw type machine driven at constant crosshead velocity (0.5mm/min), Figure 1. The effective machine stiffness was measured to be 0.96MNm⁻¹ using the maximum load rate method (2). The load cell and strain gauge voltages were continuously digitized throughout the tests, the data stored on a minicomputer for later analysis.

Acoustic emission was continuously measured during testing using the system shown schematically in Figure 1. Conical PZT-5 piezoelectric elements were used as transducers attached to either sample end. The signals from these were amplified, bandpass filtered and passed to a high speed digital rms-to-dc converter with a 17ms averaging time. The rms voltage was digitized at 14 bits at 1.5ms intervals and recorded on a minicomputer. The largest transient bursts exceeded the amplifier linear range (200 mv rms) and are therefore nonlinear in amplitude.

A dual waveform recording system was also used in parallel with the rms measurement system so that signals could be later evaluated in detail. This system featured 8-bit digitization with a 50ns sampling interval.
Figure 2. Bridgman-type apparatus from growing directionally solidified single crystal aluminum/SiC monofilament composites.

RESULTS

SEM micrographs showing the transverse sections of undeformed single crystal composites grown at two velocities are shown in Figure 3. It can be seen that extensive reaction occurred at the fiber-matrix interface in the slowly grown sample. This resulted in the formation of an approximately 10μm thick Al₄C₃ layer at the interface. Much less evidence of reaction was resolvable with an SEM in the more rapidly solidified material. Any reaction zone present was <1μm in thickness.

Stress-strain and acoustic emission-strain behaviors for the two composites and an aluminum single crystal with no fiber are shown in Figures 4, 5, and 6. These results show a number of interesting features:

* The aluminum single crystal containing no fiber had a smooth stress-strain and acoustic emission-strain behavior. The data was in agreement with that reported for similar material in the past (3).
Figure 3. SEM micrographs of transverse sections of SiC filament in Al showing development of Al4C3 interface with increasing solidification times.

(a) rapidly solidified materials (140s)
(b) more slowly solidified (1.9 h).
Figure 4. Stress-strain and AE-strain curves for single crystal Al specimen with no SiC Fiber.

Figure 5. Stress-strain and AE-strain curves for monofilament SiC/Al single crystal composite grown at slower rate (1.9 h solidification time).
The more slowly grown composite exhibited a smooth stress-strain behavior but large "burst" acoustic emissions were observed superimposed on a smooth background behavior.

The more rapidly grown composite exhibited a discontinuous stress-strain curve with 23 large load drops. Strong acoustic emission signals accompanied each load drop.

Figure 6. Stress-strain and AE-strain curves for monofilament SiC/Al rapidly solidified single crystal composite (140 s solidification time).
Figure 7. Amplitude distribution of AE events in Fig. 4-6. The horizontal axis gives the index number of the event and is cumulative.

These samples were tested to a plastic strain of 5%. Following testing, samples were longitudinally sectioned and polished to reveal the state of the SiC fibers. Figure 7 shows a typical result indicating multiple transverse fractures of the fiber. To determine the number of fractures, samples were immersed in a 10% NaOH solution at 50°C to dissolve the aluminum matrix, leaving behind the fragmented fiber. It was found that in the rapidly grown sample 30 fractures occurred, whereas 55 fractures were evident in the more slowly grown composite. Thus, the average fragment length after 5% plastic strain was 2.4 and 1.4 mm for the rapidly and slowly solidified samples respectively.
The distributions of amplitudes of the AE events is shown in Figure 7. For the more rapidly grown material, there is a close correlation between the number of events and number of fiber fractures as indicated by the arrow. This number (30) is also close to the number of load drops (23), some of which might have resulted from multiple fractures. Fiber fractures can only account for about half of the AE events observed during testing of the more slowly grown material. Since plasticity sources are probably unaffected by solidification, we suggest that these extra signals are associated with interface cracking.

DISCUSSION

The magnitude of a load drop on a "hard" tensile testing machine can be used to estimate fiber strength. The analysis given in the Appendix gives the fiber strength:

\[ \sigma_f = \Delta P (A_s/A_f) (d\sigma/d\varepsilon)/k l_c \]

where

- \( \Delta P \) = load drop (N)
- \( A_s/A_f \) = ratio of cross-sectional areas of sample and fiber
- \( d\sigma/d\varepsilon \) = workhardening rate (Nm\(^{-2}\))
- \( k \) = machine stiffness (Nm\(^{-1}\))
- \( l_c \) = critical length of fiber (m)

The last three load drops of the rapidly solidified composite averaged 4.5N (0.31MPa), \( A_s/A_f = 943 \), \( d\sigma/d\varepsilon = 0.18\)GPa, \( k = 0.91\)MNm\(^{-1}\) and \( l_c = 2.4\)mm. Thus, the fiber strength \( \sigma_f \) is ~0.3GPa, only 10% of that of a virgin fiber prior to composite processing.

Fiber loading theory indicates that the critical length for fiber fracture, \( l_c \), is given by the relation (4)

\[ l_c = \sigma_f d/2\tau \]

where \( \tau \) is the interfacial shear strength and \( d \) the fiber diameter. Substituting \( l_c = 2.4 \)mm, \( \sigma_f = 300 \) MPa and \( d = 0.14 \)mm gives a value for \( 2\tau \sim 17.5 \) MPa. Equating \( 2\tau \) with the tensile stress indicates that the interfacial shear strength in the rapidly solidified sample is approximately equal to the matrix shear strength at 5% strain. This suggests that fiber-matrix failure
occurs here by matrix shear, and that it may be lessened if the matrix shear strength is increased (by solution or precipitation hardening, for example).

Turning now to the slowly grown material, we see that though many fiber fractures occurred, no detectable load drops were observed on the stress-strain curve. The noise-limited smallest detectable load drop is ~0.4N (0.03MPa). Thus $\sigma_f<30$MPa, an order of magnitude less than that of the rapidly grown sample. This degradation of strength could have occurred due to the presence of the 10$\mu$m thick (brittle) $\text{Al}_4\text{C}_3$ layer at the interface. Cracks in the layer could cause significant stress intensification in the fiber and thus failure at lower applied stresses (5).

The critical fracture length $l_c$ was also reduced in the slowly solidified material. Typically $l_c=1.4$mm so that $2\tau<3$MPa. This is much less than the local matrix shear strength and is consistent with possible brittle shear failure in the $\text{Al}_4\text{C}_3$ layer. Tensile and shear fractures in the layer may be the source of the additional acoustic emission signals in this material (Figure 7). Such a layer is absent in the more rapidly grown material which does not emit these extra signals. The reduction in fiber strength in slowly solidified composites may be due to cracks in the $\text{Al}_4\text{C}_3$ layer, resulting in a notching effect (5).

CONCLUSION

The combination of metallography observations, tensile tests and the results can be used to estimate fiber strength in situ. The above results suggest that the presence of an interfacial layer can exert a profound reduction of the load-bearing capability of fibers in composites, and that this effect can be strongly influenced by processing conditions. Obviously the failure mode of the interface will affect the macroscopic fracture toughness of the material, since cracks running through the matrix may be blunted by shear failure of the interface, but assisted by tensile failure of the fiber (notch effect).

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APPENDIX

LOAD DROPS DUE TO FIBER FRACTURE AS AFFECTED BY MACHINE STIFFNESS

Consider a tensile specimen with a single fiber oriented along the tensile axis. If the fiber breaks, there will be a localized loss in composite strength. Whether or not there will be a load drop depends on the fiber strength, the machine stiffness, and the load. Notably, for example, if a dead weight ("soft") loading mechanism is used, one gets extensions instead of load drops.

Our analysis assumes use of a "hard" or constant crosshead velocity machine and follows that of Clough (2). The total extension of the crosshead moving at velocity $V$ over time duration $Dt$ is equal to the sum of the extensions $u_p$ of the machine length (pull rods, grips, load cell) and $u_s$ of the specimen:

$$V\Delta t = u_p + u_s = \frac{P}{k} + u_s,$$

where $P$ is the load and $k$ is the machine stiffness ($N/m$). If a fiber fractures, during time increment $dt$ there will be a sudden drop in load with the corresponding extensions:

$$d(V\Delta t) = Vdt = du_s + dP/k.$$
The crosshead displacement \( \Delta t \) will typically be negligibly small during the load drop, for the time over which the load drop occurs corresponds roughly to the difference in arrival times of the longitudinal and surface waves that travel from the specimen up the pull rod to the load cell. If \( L \) is the pull rod length and \( c \) is the longitudinal wave velocity, then \( \Delta t \sim L/2c \), assuming a factor of \( \sim 2 \) difference in wave velocities. Then the crosshead displacement \( \Delta \lambda/t - V\Delta t/2c = (8.33 \times 10^{-6}) \cdot (0.4)/2(5920) = 0.28 \text{nm} \), compared to typical values of \( \Delta \lambda/k \sim 7.2/10^6 = 7200 \text{nm} \) for machine relaxation, where \( \Delta \lambda \) is the load drop. Thus we can consider the load drops to occur at essentially fixed crosshead positions so that \( d(\Delta \lambda/t) \rightarrow 0 \) and

\[
\Delta \lambda = k \Delta \lambda_s.
\]

The fracture of a fiber, with critical length \( l_c \) will cause a localized increase in cross-sectional stress \( \Delta \sigma = A_f \sigma_f/A_s \), the plastic strain extending approximately \( l_c/2 \) along each fiber. Here is the length and \( A_f \) and \( A_s \) are the cross-sectional areas of the fiber and specimen, respectively. This will cause an increase in length of the specimen

\[
\Delta \lambda_s = l_c \Delta \varepsilon = l_c \Delta \sigma/(d\sigma/d\varepsilon) = l_c \sigma_f (A_f/A_s)/(d\sigma/d\varepsilon),
\]

where \( \Delta \varepsilon \) is the localized plastic strain and \( (d\sigma/d\varepsilon) \) is the rate of work-hardening. The fiber strength is then

\[
\sigma_f = \Delta \lambda (A_s/A_f)(d\sigma/d\varepsilon)/k l_c.
\]