JOURNAL OF MATERIALS SCIENCE 29 (1994) 5615–5624

In situ scanning electron microscope studies of fracture in particulate-reinforced metal-matrix composites

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In situ observations of specimen surfaces have been used to characterize the fracture behaviour of particulate-reinforced metal-matrix composites. Composites of silicon carbide particle sizes 3, 10 and 30 μm with volume fractions of 5, 10 and 20% in commercial-purity aluminium and aluminium–1% magnesium matrices were studied. The results of this surface study are compared with complementary metallographic studies of sectioned specimens illustrating behaviour from the bulk. Significant differences between the results of these two studies are outlined. Previous work using these techniques is critically examined and recommendations made for the appropriate interpretation of *in situ* straining experiments.

1. Introduction

Particulate-reinforced metal-matrix composites (PRMMCs) are a class of materials with great potential for structural applications [1]. Light, stiff ceramic particles can be added to metal matrices using a number of relatively straightforward manufacturing processes to produce materials with significant increases in their specific stiffness and strength when compared to the unreinforced matrix alloys [2]. There is a trade-off, however, between improvements in these mechanical properties and degradation of the fracture-related properties. It is the relatively poor values of ductility and toughness which are currently limiting widespread usage of such materials.

Despite the modest macroscopic ductility of PRMMCs, their failure proceeds by a ductile void nucleation, growth and coalescence mechanism through the matrix phase [3]. The influence of microstructural parameters, such as matrix heat treatment [4, 5] or size and volume fraction of reinforcement [6, 7], on the micromechanism of fracture has been studied and attempts have been made to correlate these changes with mechanical properties [8]. However, it is very difficult with these *post mortem* techniques to study the progress of fracture as the chronology of the events is lost or masked. Therefore, it is hard to assess the relative importance of each stage in the fracture process, to know the strains at which fracture events occur, and so it is not possible to make better attempts at tailoring the microstructure to optimize material performance. In response to this, experiments have been performed in parallel to conventional fractography where the fracture process is monitored and observed in real time. Initially, *in situ* SEM studies of the mechanisms of crack initiation and growth were performed [9–18]. More recently other techniques have been employed, such as the monitoring of acoustic emissions [8] and the reduction in stiffness [19] or density [20] on straining, to study damage evolution.

In situ SEM techniques have been used to study the micromechanisms of fracture in many materials. Two arrangements have been used to load PRMMCs: tensile beams, with pre-notched, pre-cracked or plane specimens; and bend- or wedge-loaded specimens. The first, and most popular [9–14], has the advantage that the loading is homogeneous with a one-to-one correspondence with tensile failure. If the load and displacement are recorded during a test, the failure mechanisms can be directly related to the applied far-field strains. However, once a crack is formed it is loaded in an unstable configuration and failure follows quickly. The majority of tests performed under tensile loading did not study the damage accumulation prior to macroscopic crack formation, but instead concentrated on crack propagation [9–12, 14]. This was due to the difficulty of locating the initiation site. Da Silva *et al.* [13], however, were successful in this.

Wedge- or bend-loading has been used by a few researchers [15–18]. The constant displacement, double cantilever (CDDC) test, where a wedge of known angle is advanced into a pre-machined notch (Fig. 1), has a number of attractive features and was employed by the authors in their experiments. It allows the accumulation of microstructural damage before crack initiation to be studied easily. In addition, under idealized geometrical conditions and assuming a linear crack-tip field, a simple relationship exists between the crack extension force $G(c)$ and the crack length c for unit beam width:

$$G(c) = 3Eh^2d^3/4c^4 \tag{1}$$

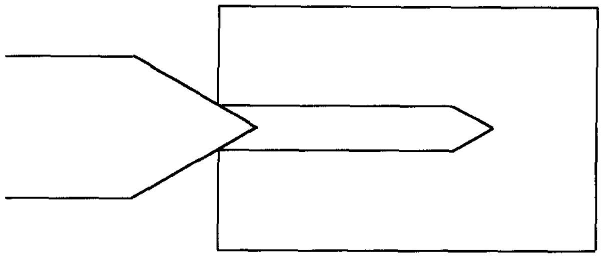

Figure 1 Notch-opening action of CDDC test.

where E = Young's modulus, h = arm opening displacement, d = specimen thickness and c = crack length. Thus, if the crack length is monitored as a function of wedge displacement (and, therefore, arm opening) the crack extension force and material toughness can be evaluated [21]. Another point to note is that here the crack propagates in a stable manner because the crack extension force decreases as the crack length increases. This allows a great deal of control when performing the experiments. In the case of PRMMCs, where there is extensive plastic work at the crack tip and the experimental geometry is not perfect, only a qualitative measure of the toughness can be obtained by this method.

The major emphasis of the *in situ* experiments has been in determining mechanisms. However, these observations are made on a free surface and their validity as representations of processes occurring in the bulk of the material have often not been assessed by performing complementary experiments. One of the principal objectives of this paper is to show that some of the observations are surface-specific or at least influenced by the presence of the free surface. *In situ* experiments have also been used to provide quantitative data on damage initiation and accumulation by counting or sizing fractured particles on the surface. The validity of these results is also examined.

2. Experimental procedure

The material systems examined in this study were silicon carbide particle reinforced aluminium matrices. Composites of three volume fractions (5, 10 and 20 %) and three particle sizes (nominally 3, 10 and 30 μm) of reinforcement in both commercial-purity aluminium (Al-1070) and a simple solution-strengthened aluminium–1 % magnesium alloy (Al-5050) were fabricated by the powder metallurgy route of vacuum hot-pressing and extrusion at AEA Technology, Harwell Laboratories, UK. The details of the fabrication process are given elsewhere [22]. Preliminary investigations of polished longitudinal and transverse sections by optical and scanning acoustic microscopy revealed insignificant damage of the reinforcing phase on extrusion, giving confidence that subsequent observations were representative of the fracture process and not artefacts of material manufacture or specimen preparation [23].

Specimens were made to the geometry of Fig. 2. Slices parallel to the longitudinal direction were taken from the extruded bar by electric discharge machining. The central reduced section and notch were also made by electric discharge machining so that no mechanical damage was introduced at the notch root. This was followed by lapping and polishing of the top surface. After polishing, the specimens were heated to 420 °C at a rate of 200 °C h^{-1} and held there for 2 h before being either water-quenched or furnace-cooled at 10 °C h^{-1} to room temperature, hereafter referred to as quenched and annealed, respectively. The *in situ* studies were undertaken in a Jeol-35X SEM, which has a commercial manually-operated straining stage, using both secondary and back-scattered electron imaging (Fig. 3). The stage was modified to perform the CDDC tests (Fig. 4). The wedge was on the end of a long, fine, hand-driven thread which allows the displacement of the wedge to be measured to a resolution of 2 μm. The specimens were held lightly in place by the wedge and the displacement at zero strain recorded. The wedge was advanced incrementally whilst observing the specimen, the displacement noted and the specimen then examined in greater detail.

3. Results

Several features on the *in situ* tests were common and generic to the fracture process in all the composites studied, and will be described first by considering the Al-1070 composite systems. The effect of the microstructural parameters and thermal treatment on the

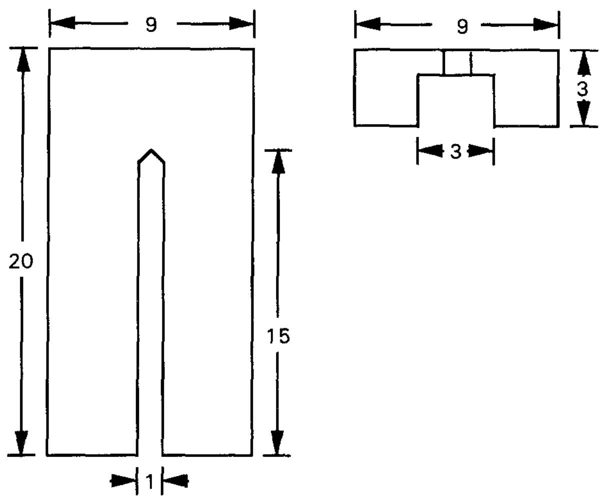

Figure 2 In situ test specimen geometry.

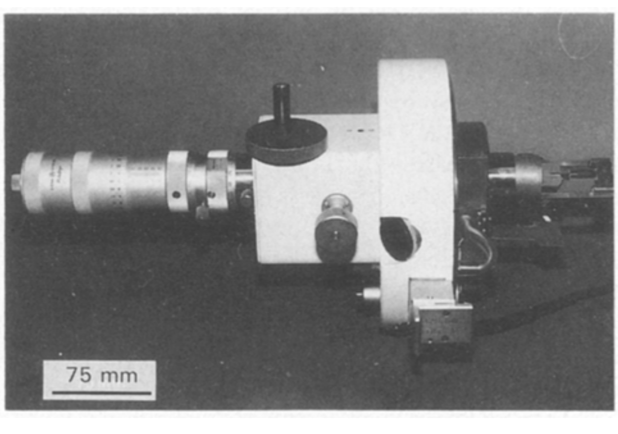

Figure 3 SEM straining stage.

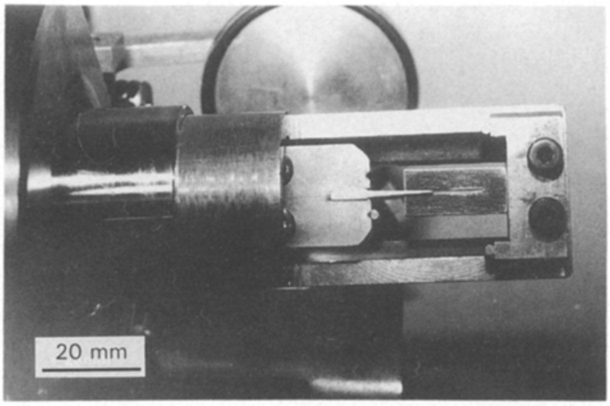

Figure 4 CDDC loading arrangement.

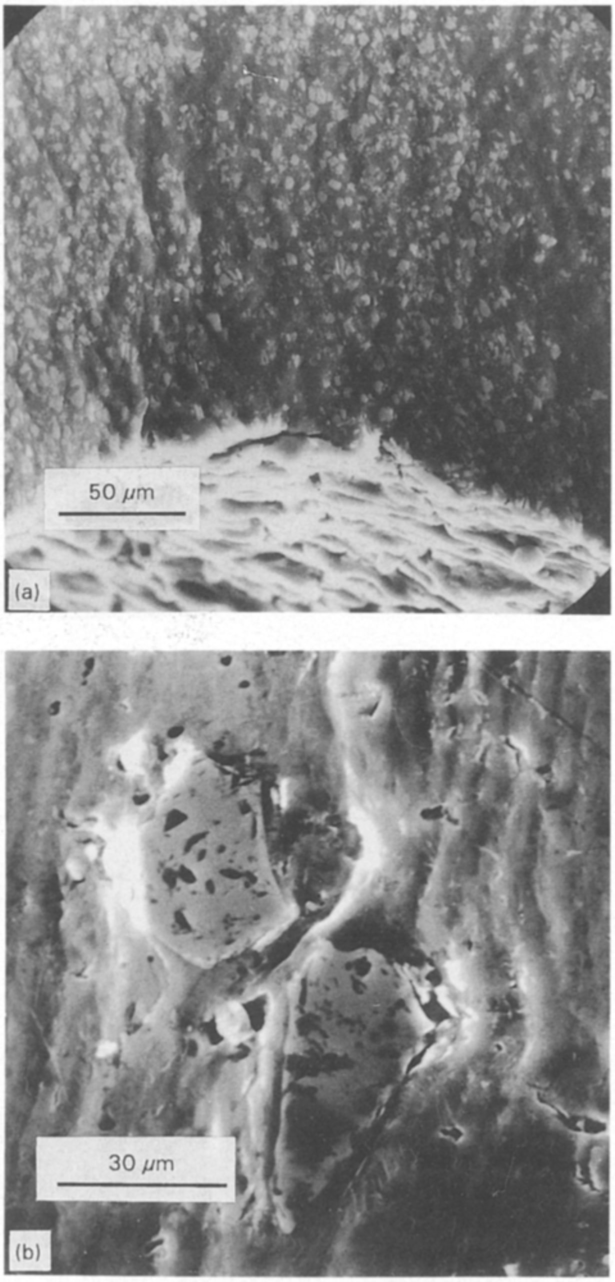

Figure 5(a,b) Extensive surface relief on straining and the effect of the particles on the matrix flow.

mode of failure will be outlined. Then a comparison of Al-1070 and Al-5050 matrix composites will be made.

On straining, the surfaces of all the specimens (which were planar initially) became rippled, lifting the reinforcing particles proud of the surface. The particles localize and intensify the applied strain but, under the approximately plane stress conditions at the surface, can relieve this strain by deformation out of the plane of the surface. This intense deformation occurred along well-defined directions which were maintained throughout a test. These appeared to be along the loci of expected maximum shear stress. This was most clearly seen in the studies on the composites with 5 vol % of particles (Fig. 5). Evidence for the onset of failure by shearing at the interface and particle cracking may also be seen in this figure.

As the strain was increased, void nucleation events at the particles were seen in all composite systems before the formation of a macroscopic crack (Fig. 6). This implies that the model of You *et al.* [24], which has fracture initiating in the matrix distant from the particles, is inappropriate for these low-strength matrix alloys. The wedge was advanced further before nucleation at the 10 μm particles than in the 30 μm particles, implying an increase in the strain to void nucleation on reducing the particle size. This is consistent with an increase in particle fracture stress on decreasing the size of these brittle ceramic reinforcements, as expected from models of defect-controlled particle strength. The extent of wedge advance before nucleation was also a function of the volume fraction of reinforcement, decreasing with increasing volume fraction. For composite systems where conventional fractography has shown the void nucleation mechanism to be by decohesion at the particle–matrix interface (Table I), there was some evidence of failure by particle cracking during the *in situ* straining. Matrix failure was sometimes seen in the composites containing 3 μm particles at these low strains, although this was not as common as the particle cracking seen in other systems (Fig. 7).

The damage zone around the notch root increased on further straining until a macroscopic crack was formed (Fig. 8). Note the very large spatial extent of the damaged region around the crack. The cracks were discontinuous on the surface and, in general, followed one of the directions of intense matrix deformation detailed earlier. Microcracked areas were formed ahead of and to the side of the crack tip associated with enhanced strain levels there. These regions extended some tens of interparticle spacings from the crack tip and were of the same order of size in both the quenched and the annealed material. As other *in situ* studies have reported [9–13, 15–17], crack propagation was by failure of matrix ligaments between microcracked regions ahead of the crack tip, containing either fractured particles or failed matrix, and the main crack. As the adjoining matrix failed, additional microcracked regions were formed ahead of the "new" crack tip. This can be seen in Fig. 9. There were two features associated with this propagation mechanism. First, crack bridges were formed by the discontinuities in the crack due to the intact matrix

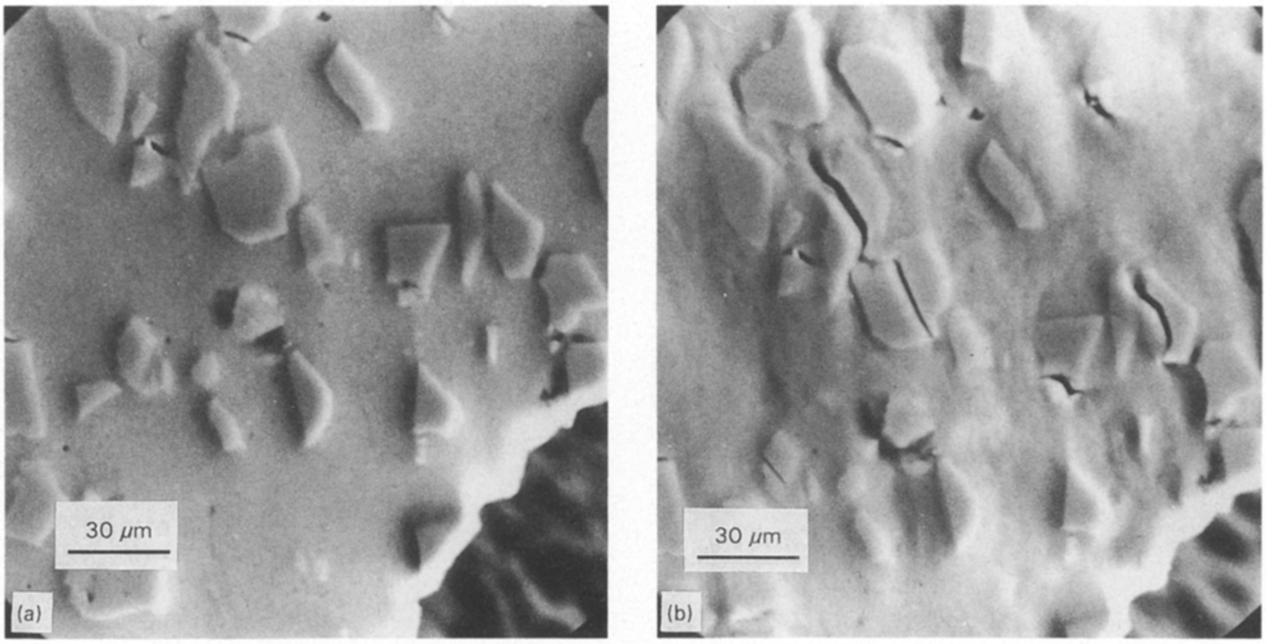

Figure 6 Void nucleation events at the reinforcing phase on straining from (a) zero strain to (b) small strains.

TABLE I Summary of fractographic results giving the nucleation mechanism at the reinforcing particles

Material	Particle size (μm)	Volume fraction (%)		
		5	10	20
Al-1070	3	Decohesion	Decohesion	Decohesion
	10	Fracture	Fracture	Fracture
	30	Fracture	Fracture	Fracture
Al-5050	3	Decohesion	Decohesion	Decohesion
	10	Decohesion	Fracture	Fracture
	30	Decohesion	Fracture	Fracture

ligaments (Fig. 10). Secondly, the crack often branched by separating into two or more paths (Fig. 11). There was increased crack tip advance for a given wedge increment for the composites of higher volume fraction of reinforcement. As stated earlier, loading in CDDC allows quantitative values of toughness to be obtained if the crack tip extension is known as a function of wedge advance [21]. Due to the discontinuous nature of crack growth in these PRMMCs and the extensive plastic deformation at the crack tip, only qualitative information could be obtained. The results, therefore, imply increasing toughness with reduced volume fraction of reinforcement. Indeed, it was very difficult to propagate a crack in the composites containing 5 vol % of reinforcement. There was no measurable difference between the two heat-treated conditions (Table II).

As the crack grew it did not in general pass through failed particles in the microcracked region. The fractured particles remained in the wake of the main crack. Thus, while the strain field associated with the crack caused nucleation, the crack tip itself was not attracted to these paths of apparently easy growth. This occurred in all systems including those where

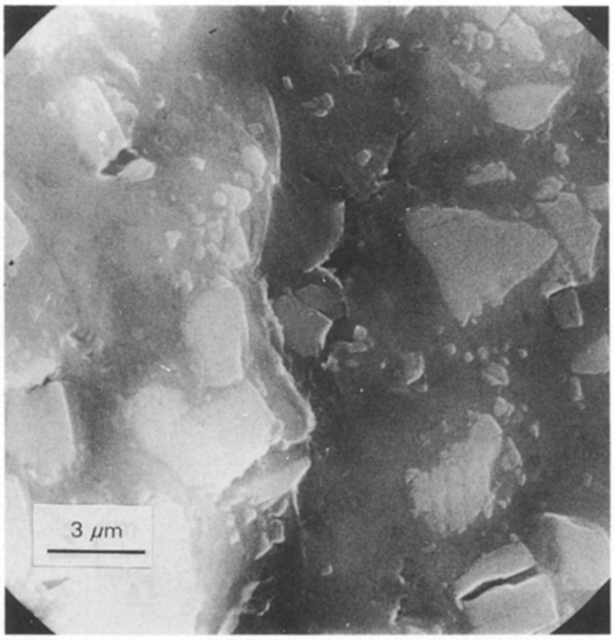

Figure 7 Local matrix failure in the 3 μm particulate composites.

fractography has clearly shown the presence of fractured particles on the fracture surface. Indeed, in the systems where void nucleation is by decohesion at the particle–matrix interface, especially those containing 3 μm particles, the crack path was almost entirely in the matrix with no clean interfacial failure (Fig. 12).

The same basic microstructural features of crack initiation and propagation were seen during the testing of the Al-5050 matrix composites. However, there were qualitative differences. First, the crack tip advance was much greater for a given wedge increment in the Al-5050 matrix composites than in the corresponding Al-1070 matrix composites (Table II). Therefore, it can be inferred that they have a lower toughness than the Al-1070 matrix composites. Secondly, the Al-5050 matrix composites often had a

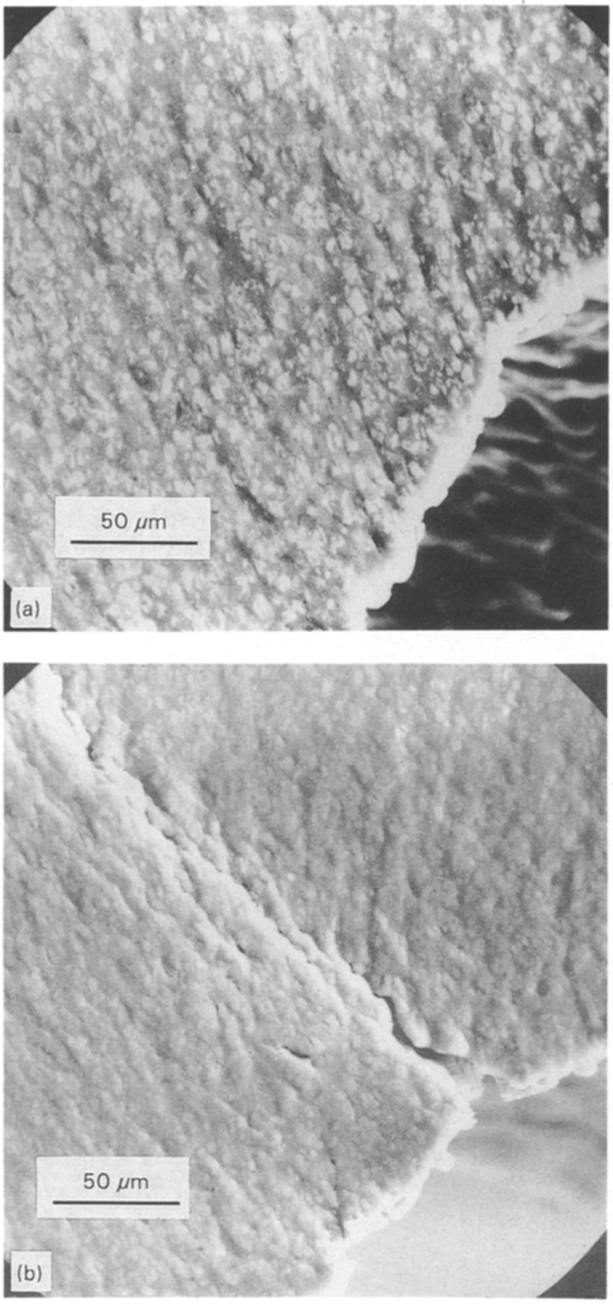

Figure 8(a,b) The macroscopic crack follows the extensive deformation in approximately the direction of the greatest resolved shear stress.

"stepped" or slightly serrated crack path. These observations are consistent with both the results of mechanical testing, which shows a marked reduction in the ductility of the Al-5050 matrix composites, and fractography [8] and will be discussed more fully later.

4. Discussion

The results of the *in situ* studies on PRMMCs are in many respects wholly consistent with those on any material which fails by a ductile rupture mechanism and contains a significant population of second-phase particles. On straining, inhomogeneous deformation occurs because of the presence of the second-phase particles. These either fail themselves or pull away from the surrounding matrix, forming voids [25].

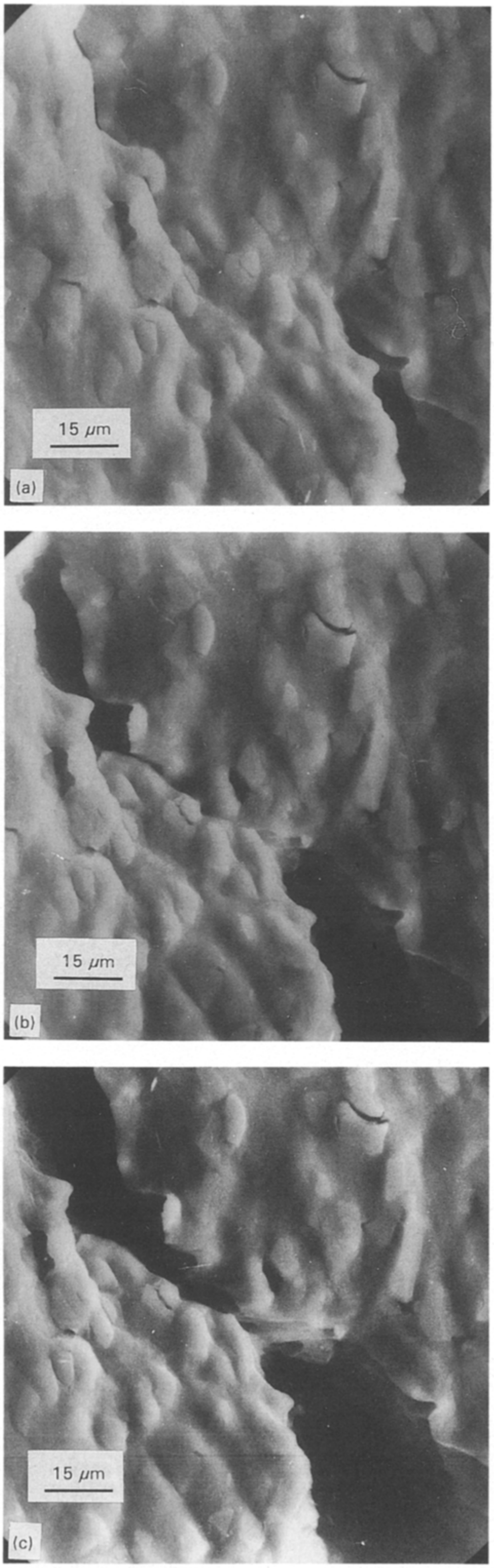

Figure 9(a–c) Crack propagation sequence at three strains.

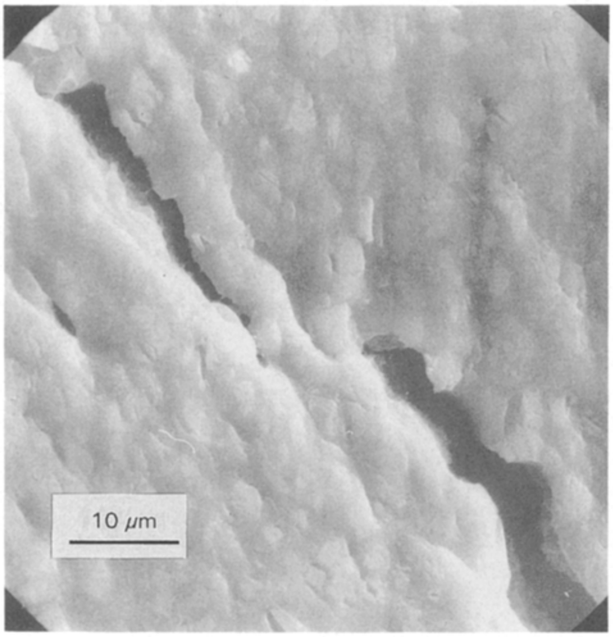

Figure 10 Discontinuous nature of cracks.

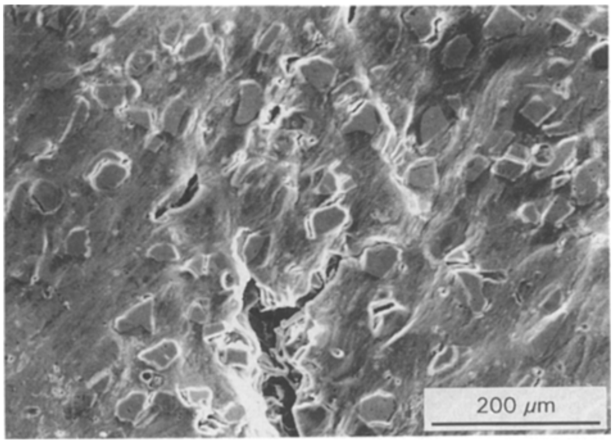

Figure 11 Crack branching.

TABLE II The effect of microstructural parameters on damage initiation and crack tip advance

Material	Wedge increment before nucleation (mm)	Crack tip advance per wedge increment ($\mu m\, mm^{-1}$)
20%, 30 μm, Al-1070, Qu	0.630	42
20%, 30 μm, Al-1070, An	0.650	38
20%, 30 μm, Al-5050, Qu	0.440	76
20%, 10 μm, Al-1070, Qu	1.140	15
20%, 10 μm, Al-5050, Qu	0.870	28
20%, 3 μm, Al-1070, Qu	2.338	4
5%, 30 μm, Al-1070, Qu	6.820	2
5%, 30 μm, Al-5050, Qu	4.370	6

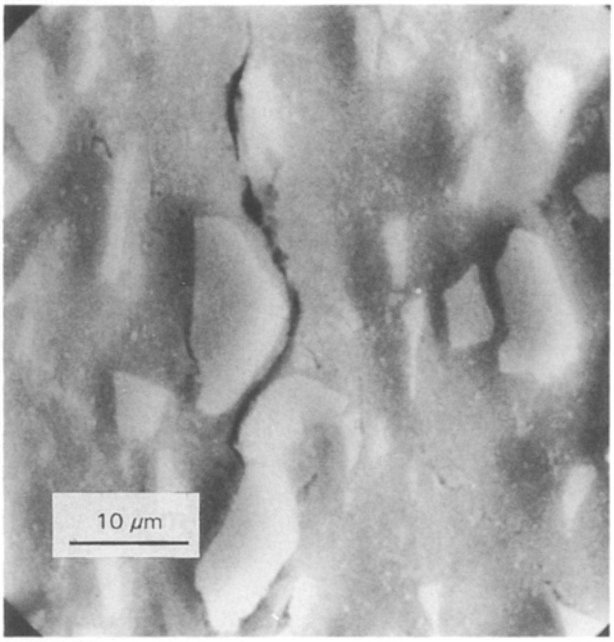

Figure 12 The crack passes through the matrix away from the interface.

Additional strain is required for the matrix to fail in the absence of the second phase. On further straining these voids grow whilst new voids are nucleated elsewhere. Eventually a macroscopic crack is formed by the linkage of these voids. Once the crack has formed, the strain fields ahead of the tip are sufficient to cause locally the initiation of failure at nearby second-phase particles. These become the new crack tip when the adjoining matrix ligaments can no longer sustain the locally high strain levels and the flow localizes, thereby leading to matrix failure and coalescence.

The details of the crack propagation mechanism in PRMMCs are influenced by the microstructural parameters. As mentioned earlier, the initiation of failure at the reinforcing particles at lower wedge increments for larger particles is consistent with a defect-controlled nucleation criterion. Similarly, the influence of the higher volume fraction of reinforcements is to decrease the interparticle spacing and hence to increase the constraint on matrix flow. The local stress is, therefore, also increased for a given wedge increment, producing earlier initiation of failure and larger crack tip advance. Additionally, there are locally high volume fraction regions in the material where increased microstructural damage introduced during fabrication is expected before deformation. In addition to cracked particles, higher dislocation densities may be found there due to the mismatch in thermal expansion coefficients between the matrix and reinforcement. However, we found no conclusive evidence for crack path selection through such regions of high local reinforcement density.

The effect of the matrix alloy on the results requires more careful consideration. There are two observations to consider: the extended crack tip advance, and the "stepped" crack profile. Optical microscopy has shown that the Al-5050 matrix grains were often elongated during extrusion, the Al-5050 starting powder size being somewhat larger than that of the Al-1070. TEM studies have shown the presence of an additional phase, principally MgO, at the particle–matrix interface and at certain grain boundaries after processing [26]. Comparison of matched fracture halves of failed tensile specimens has similarly indicated failure initiation at this additional phase. Thus, it appears that the MgO provides a favourable nucleation site. The presence of voids in the matrix lowers the local constraint on matrix flow and promotes void coalescence between those voids formed at the reinforcing particles. Therefore, lower local strains are required before the adjoining matrix ligaments fail and so the crack tip advances further for a given wedge increment. The shape of the crack path may, therefore, also be associated with failure at these weakened grain boundaries, the elongated shape of the grains producing the "stepped" appearance.

The effect of the microstructural parameters on failure initiation and crack propagation is mirrored in the mechanical properties of the composites. Earlier initiation and extended crack tip advance are seen in the composites of lower ductility [8]. This gives some confidence in the qualitative results of the *in situ* experiments.

However, the technique necessarily studies a free surface in plane stress which may be unrepresentative of the bulk of the specimen which is in a stress condition closer to plane strain. Indeed as described earlier, a number of the experimental observations cast doubt on the validity of using the results of *in situ* investigations either in isolation to describe failure completely or to provide quantitative data on the fracture process. First, before crack initiation the surface of the specimens became highly distorted, relieving the strain at the particles by deformation out of the plane of the surface. This deformation mechanism would be unavailable in the bulk of the material. Secondly, the propagating crack rarely passed through a silicon carbide particle even if the particle was already fractured, preferring instead to pass through the matrix away from the interface. This behaviour is inconsistent with that seen on examining fracture surfaces of these PRMMCs. Thirdly, the extent of damage ahead of the crack tip was larger than predicted by well-established models developed for ductile rupture processes in other materials.

To assess the validity of the *in situ* investigations, stable cracks were introduced into bulk specimens (10 mm square) by loading in the same CDDC manner. The specimens were electrolessly nickel-plated to support the crack faces and sections perpendicular to the crack plane at least 3 mm from the free surface were made by electric discharge machining. These sections were prepared for optical microscopy. Again features common to all material systems were observed, so initially the results from a representative composite will be reported here for illustration.

Figs 13 and 14 are micrographs of an Al-5050 matrix composite containing 20 vol % of 30 μm particles. At low magnifications (Fig. 13) one can see several regions of fairly extensive void growth linked by failure through joining matrix ligaments. These features would be formed by a discontinuous crack growth mechanism with the opening of microcracked regions ahead of the crack tip. The crack path is not tortuous but follows the damage region ahead of the crack tip. There is little evidence of microcracking to the side of the main crack, only in front of it. These observations qualitatively support the crack propagation mechanism proposed by considering the *in situ* studies.

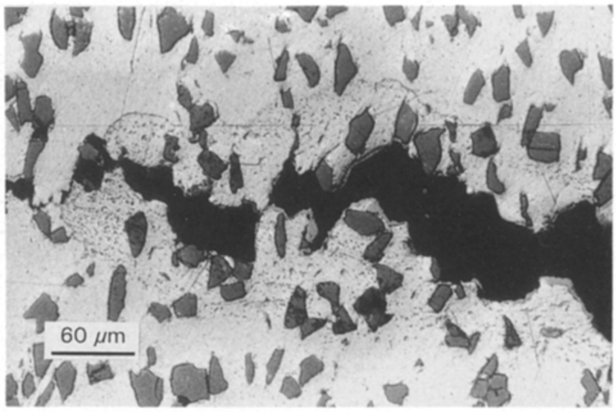

Figure 13 Section through a stable crack loaded in CDDC, showing discontinuous nature of crack growth with areas of large void growth joined to areas of small growth.

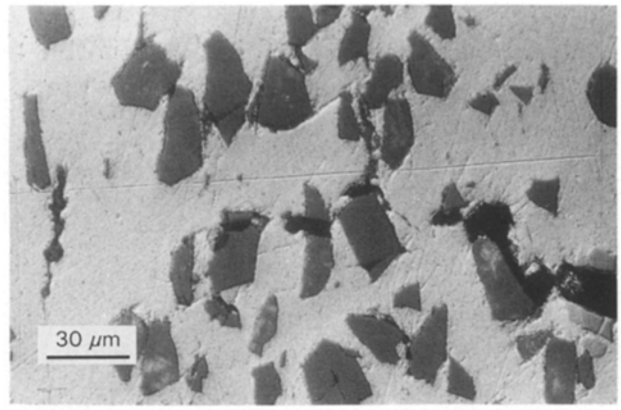

Figure 14 Damage confined to 1–2 interparticle spacings ahead of crack tip.

Nucleation events can be seen ahead of the crack tip, but the damage region is now confined to one or two interparticle spacings (Fig. 14). There is also no evidence of crack branching or bridging. Ritchie and co-workers [27–29] have made similar observations of the crack profile on the surface during their studies of fatigue crack propagation. As ΔK was decreased, they noted that the number of fractured particles ahead of the crack tip, giving a measure of the damaged zone, was reduced. They also reported a transition from crack bridging and branching at large ΔK to the absence of these features at low ΔK. Indeed, they postulated that a critical ΔK exists below which there is no crack bridging by intact matrix ligaments. They attributed these results to a reduction in the plastic zone ahead of the crack tip as ΔK is lowered and, therefore, the volume of material containing brittle particles sampled by the crack. If a small volume is sampled by the crack, the opportunities for substantial microcracking leading to crack branching and bridging are reduced until these features are no longer observed.

Kamat *et al.* [30], Crowe *et al.* [31] and Manoharan and Lewandowski [11, 12] have devised models to estimate the extent of damage ahead of a stable crack propagating in a PRMMC. These are based on variants of the plane-strain asymptotic crack tip solutions for a power-hardening solid by Hutchinson [32] and Rice and Rosengren [33] which were modified by the blunting solutions of Rice and Johnson [34] and McMeeking [35]. With a knowledge of the fracture toughness and yield stress of the material, the size of the zone of enhanced matrix deformation ahead of the crack tip was predicted to be of the order of the interparticle spacing in all models. This is supported by the sections through the crack but not by the *in situ* observations [9–18].

Two possible explanations for this behaviour have been advanced. Humphreys [17] reported similar results on comparing *in situ* studies from a pre-cracked bend configuration with those from within the bulk of a material. His results were interpreted by considering a surface relaxation of the compressive thermal residual stresses exerted on the particles after cooling from fabrication or heat-treatment temperatures. In the bulk of the material this additional stress must be overcome before the particles are fractured. Thus, more fractured particles are seen in the vicinity of the crack tip on the surface of the specimen. A study of the relaxation at free surfaces of these thermal residual stresses in PRMMCs, however, indicates that they are only reduced within a very narrow band, some 7 μm, from the surface [36]. Thus, particles larger than this may well experience some if not all of the residual stresses. In the present study the material was studied in two heat-treated conditions, quenched and annealed, in an attempt to remove or reduce the influence of the thermal residual stresses. The heat treatment had a marked effect on the tensile properties of the composites [37]. In the *in situ* studies, however, the damage zone was of similar size, suggesting that other influences may need to be considered.

Mummery and Derby [16] have suggested that the smaller damage zone is due to the reduced plastic zone at the crack tip on moving from the plane stress state on the free surface to plane strain in the bulk. The failure of intact silicon carbide particles must be due to plastic deformation in the matrix as it is not possible to generate sufficiently large elastic stresses in aluminium to cause their fracture. Humphreys [17] noted that the damage was much more localized on increasing the matrix yield strength dramatically. As the plastic zone size ahead of a propagating crack is inversely proportional to the square of the yield stress [38], this observation also supports the above proposal. Further experimental evidence is shown in Fig. 15 of a composite containing 20 vol % of 10 μm particles. The measured yield strength of this composite is higher than that of the composite considered so far which contains 30 μm particles. In addition, the interparticle spacing is much reduced leading to a lower matrix mean free path. This introduces additional constraint on the deformation at the crack tip,

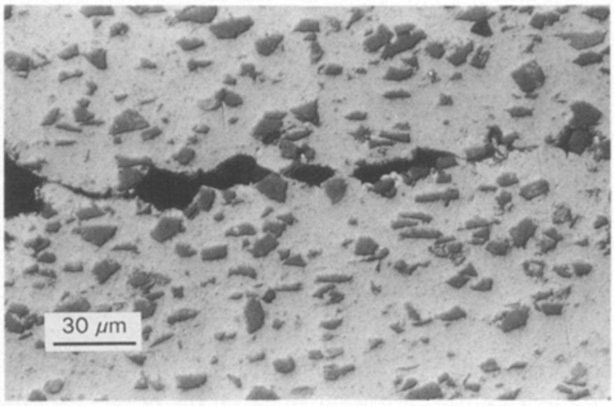

Figure 15 Straighter crack path in composite containing 10 μm particles.

effectively reducing the plastic zone there. The smaller highly strained sampling volume can be seen as a straighter crack path. Indeed, the progressive reduction in stress intensity employed by Ritchie and co-workers [27–29] may be considered to be analogous to taking sections through a crack at different depths below the free surface, initially in plane stress, passing through mixed-mode states before achieving effectively plane strain conditions in the interior of the specimen. However, on adoption of this approach, the predicted increase in plastic zone is only by a factor of 2 to 3 [38]. The measured damage zone on the surface is still somewhat larger than this. Thus it seems most likely that a combination of the relaxation of thermal stresses and the increase in plastic zone is needed to rationalize the experimental observations.

These observations do imply, however, that quantitative measurements of the extent or progression of damage on straining by counting or sizing fractured particles on the surface of specimens [39, 40] are likely to be subject to error. Additionally, it has been shown that damage extends further below the fracture plane on the surface of failed tensile specimens than in the bulk [41]. However, these studies also showed a larger number of failed particles in the centre of the sections in the necked region, where there was increased stress triaxiality, than towards the edge.

Fig. 16 shows the crack passing through the failed silicon carbide particles in accord with study of matched fracture halves in this system. Similarly, Fig. 17 shows the crack leaving cleanly decohered interfaces in a composite containing 30 μm particles in Al-5050 at two magnifications. Other *in situ* studies have also indicated that the crack path and failure mode at the reinforcing phase found on the surface may not be representative of those in the bulk. Wu and Arsenault [9, 10] stated that the propagating crack did not cause the failure of any particles on the surface but rather the crack was attracted to the particles that had already failed, either in processing or preparation for examination. They further implied that since conventional fractography was a *post mortem* technique, it could not be determined whether particles on the fracture surface were caused to fail by the straining process. They therefore stated that failure of PRMMCs was due to the growth and coalescence

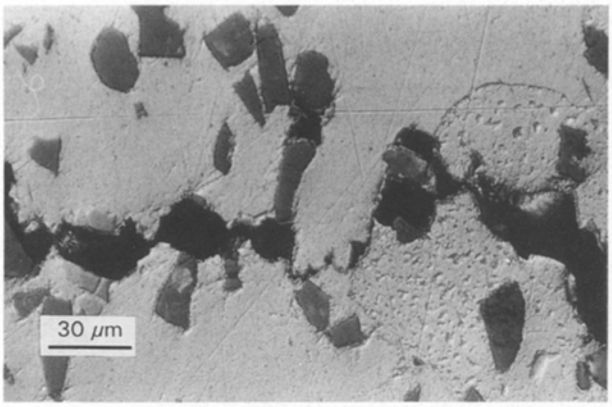

Figure 16 Crack passing through 30 μm particles.

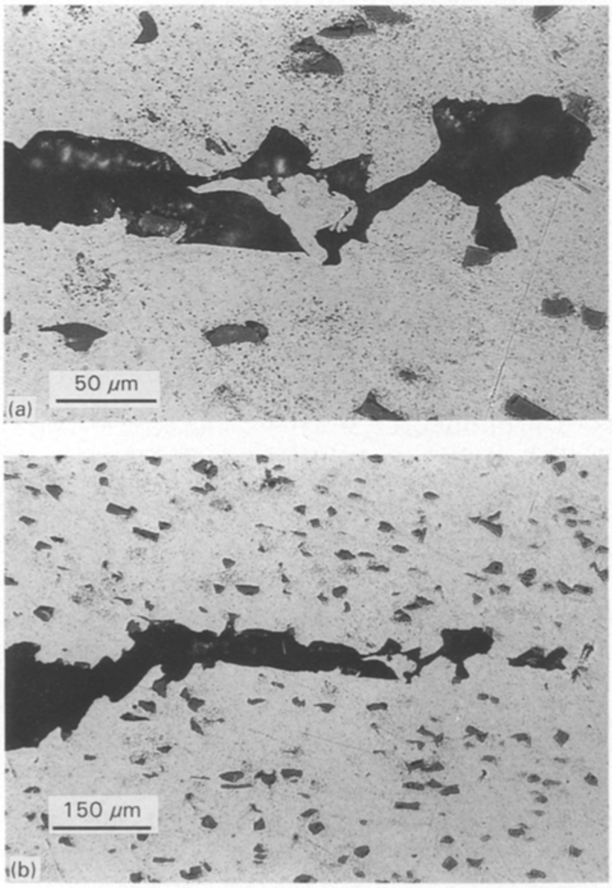

Figure 17(a,b) Crack path through interfaces leaving cleanly decohered particles.

of voids formed at precracked particles. Recent acoustic emission studies have, amongst other experiments, shown this proposal to be incorrect by clearly identifying particle fracture and decohesion during plastic straining of PRMMCs [8]. Humphreys [17], Da Silva *et al.* [13] and Ribes *et al.* [14] noted that existing flaws in the particles were opened readily by the crack at low applied strains. These can be opened by the elastic deformation of the matrix. This may be an additional factor to consider when rationalizing the increased extent of damage on the free surface.

Thus it appears that the observed features of the crack path may only be in qualitative agreement with the processes occurring in the bulk. Studies of the fine details of crack propagation performed on a free surface must be viewed in this light [4]. Complementary studies must also be performed on the bulk of the material by sectioning either stable cracks or failed tensile specimens.

These results also imply that supporting fractography must be performed to confirm the mode of failure at the reinforcing particles. Da Silva *et al.* [13] noted that whilst they found that their fracture surfaces showed a mixture of void nucleation by both interfacial decohesion and particle cracking, the observed failure mechanism on the free surface was predominantly interfacial decohesion. Mochida *et al.* [40] have used *in situ* studies in isolation to identify two modes of particle cracking: shattering and simple cleavage. However, the particles they show as shattering appear to be heavily flawed before straining, implying that the observations may also be affected by faulty specimen preparation. This distinction plays an important part in the development of their paper and so should have been investigated more rigorously. The fractographic results should be considered to be more representative of the fracture of these materials as they sample a far larger volume of the material than these *in situ* crack propagation studies can. Similarly, Kim *et al.* [18] have performed fractography and report that the vast majority of the fracture surface is formed by the ductile failure of the matrix around voids formed at the reinforcing phase. However, they use their *in situ* observations to conclude that whisker failure only occurs as a result of the fracture locally of a large intermetallic particle. Features on the fracture surface associated with the failure of these intermetallics were only few and isolated, again indicating that the *in situ* results should be treated with caution and suggesting that their use by Kim *et al.* is suspect.

The one feature of the *in situ* experiments which cannot be addressed by sectioning techniques is the observations which occur before a crack is initiated. For this, other real-time techniques must be employed. These have included the monitoring of acoustic emissions [8], the reduction in stiffness [19], and the changes in density on straining [20]. All have suggested that failure processes occur before the formation of a macroscopic crack and thus support the *in situ* studies.

5. Conclusions

On first inspection it appears that *in situ* fracture studies can provide a great deal of detailed information on the failure of PRMMCs. However, a closer study has suggested that the free surface significantly affects the observations of the fracture process. The inconsistencies between bulk and surface results may be due to imperfections in material fabrication or sample preparation, but in most circumstances are a result of the plane stress condition. It is important to ascertain which observations and features of these studies are valid and which are not, and also which complementary experiments should be performed.

The preceding discussions have suggested the following observations, measurements and deductions from *in situ* straining experiments are valid:

(i) The onset of damage at the reinforcing phase before the initiation of a macroscopic crack.

(ii) The basic micromechanism of crack propagation. This is by the simultaneous microcracking of regions ahead of the crack tip and failure of the adjoining matrix ligaments.

However, the following are invalid:

(i) Quantitative measurements of particle cracking on the surface used as a measure of PRMMC damage.
(ii) Extent of damage ahead of the crack tip. This may also affect the presence of crack branching and bridging.
(iii) The fine details of the crack path.

These last measurements are better made on sections through stable cracks or failed tensile specimens.

Great care must be taken when preparing specimens for study of their surface. Observations may be erroneously attributed to the fracture process which are in truth artefacts introduced by specimen preparation or material fabrication. It is very important to assess the level of damage prior to testing, ideally by a technique which can show cracks in particles which are held together by the residual compressive thermal stresses. Scanning acoustic microscopy has been shown to be such a technique [23], although careful imaging using back-scattered electrons can be successful.

Acknowledgements

The authors thank the Science and Engineering Research Council for funding this work through the rolling programme GR/F87660 on metal-matrix composites at Oxford and for awarding one of us (PMM) a Post-doctoral Fellowship. Many thanks to Dr J. H. Tweed and other members of his group at AEA Technology, Harwell Laboratories for fabricating the experimental material and for helpful discussions.

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Received 8 November 1993
and accepted 19 January 1994