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The effect of particulate: fibre ratio on the properties of short-fibre/particulate hybrid MMC produced by preform infiltration

C. M. FRIEND, I. HORSFALL, C. L. BURROWS*
Materials Technology Group, Royal Military College of Science (Cranfield), Shrivenham, Swindon, Wiltshire SN6 8LA, UK

The details of a novel process for the production of short-fibre/particulate hybrid metal matrix composites (MMC) are presented. It is shown that preforms can be produced from various combinations of particulate and fibre reinforcements, and that these preforms can then be liquid-metal infiltrated to produce hybrid MMC. This process is extremely flexible and can produce materials which vary from conventional hybrids, to composites where the fibre fraction is low and the material is essentially a particulate-reinforced composite. Mechanical property data are presented for these hybrids which show that their strength and elastic moduli appear to be controlled primarily by the total reinforcement volume fraction and not by the particulate: fibre ratio. However, it is shown that this ratio does have a strong influence on the fracture toughness of these hybrid materials.

1. Introduction

Currently there is considerable scientific and technological interest in metal matrix composites (MMC) because the artificial reinforcement of alloys with high-strength second phases represents a unique method for improving their mechanical properties. Such composites exploit the high strength and moduli of both high-performance inorganic fibres and particulates [1]. Although the property improvements associated with particulate-reinforced MMC are lower than those observed with other reinforcement types, these materials are of great technological significance because they exhibit pseudoisotopic properties and are potentially extremely cheap [2]. Currently these particulate-reinforced MMC are fabricated by powder metallurgy [3], stir-casting [2, 3, 4, 5] and spray-forming techniques [2]. Following production, these materials are then converted to their final component form by mechanical working such as extrusion or forging. However, an alternative near net-shape forming process for the fabrication of MMC components should also be attractive on the bases of cost and low particulate/matrix reactivity. This process is squeeze-casting, which could produce MMC components via two distinct routes. Firstly it could convert liquid metals containing particulate dispersions to components via conventional squeeze-casting. This would directly produce MMC components containing general reinforcement throughout the product [6]. Secondly, however, squeeze-casting could also produce both the MMC and final product form simultaneously. This route would involve the infiltration of a reinforcement preform by the liquid matrix alloy during squeeze-casting. Such an approach would therefore enable the production of components containing both general levels and local regions of reinforcement and would then be generically similar to materials derived from short-fibre preforms [7]. This is an interesting alternative MMC fabrication technique but has the principal draw-back that it requires preforms containing substantial quantities of particulate. This has led to only limited exploitation of this fabrication route.

The preform infiltration technique requires the production of preforms containing stable dispersions of the reinforcing phase. Conventional preforms are produced from short-fibres which pack together and can be bound to produce a rigid array of well-dispersed fibres [8]. The fibre fractions of such preforms can also be varied over a wide range (typically 0.04 to 0.40). The principal problem with particulates, however, is their low aspect ratio compared to short fibres, which produces a tendency for the particulate to "close-pack" during any attempt at preforming. This means that it is not possible to produce preforms with low particulate contents, and that the particulate volume fractions in the preforms cannot be easily varied.

An alternative route to particulate preforms is to employ some form of particulate/fibre hybrid. In such hybrid preforms the array of fibres could then be used to disperse the particulates. Such preforms would require a minimum volume fraction of supportive fibres, but could be manufactured with varying volume fractions of particulates. Although these preforms could not produce pure particulate-reinforced MMC, this

**Present address*: Department of Materials Technology, Brunel University, Uxbridge, UK.

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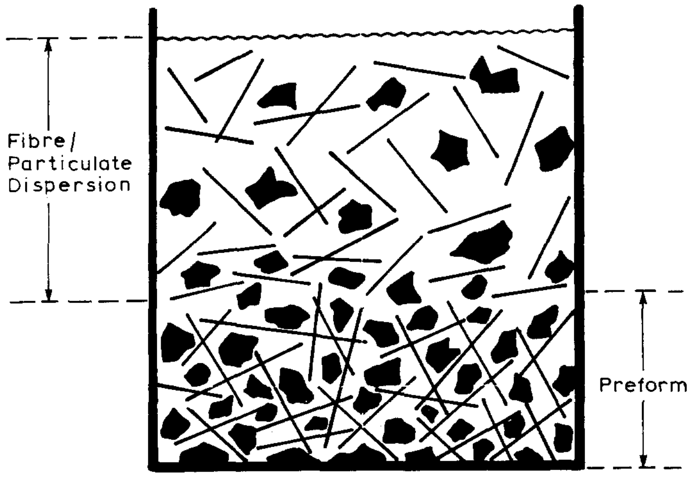

Figure 1 Schematic illustration of hybrid preform fabrication.

route would offer increased flexibility because it would allow the production of a variety of preform types. These could vary from conventional fibre/particulate hybrids (where the fibre fraction is substantially higher than that of the particulate), to preforms where the fibre fraction is at a minimum, and the response of the resulting composite is essentially that of a particulate-reinforced MMC. By selection of a suitable binder system it should also be possible to pressure infiltrate these hybrid preforms in an identical manner to that employed for conventional short-fibre preforms.

This paper describes a process route which is capable of fabricating such preforms and shows that these can then be conventionally infiltrated to produce hybrid MMC. It also presents mechanical property data for a series of particulate/fibre hybrid composites produced via this process route.

2. Preform manufacture

Hybrid preforms can be fabricated from a number of different combinations of short fibres and particulates. The fibres investigated in the present programme included both short aluminosilicate and crystalline alumina fibres, and the particulates included a variety of SiC abrasive grits. These materials differed significantly in their physical characteristics; however, they were all preformed by an identical technique. This illustrates the flexibility of this preforming process. In this paper the process route will be discussed in the context of the preforming of Saffil (ICI trademark) fibres and SiC abrasive grits. This will give a systematic illustration of the effect of both particulate:fibre ratio and particulate size on the mechanical properties of the resulting hybrid composites.

The production of hybrid preforms is relatively straightforward and requires the dispersion of the two reinforcements followed by their co-sedimentation to produce a mechanically stable preform. This is illustrated schematically in Fig. 1. The reinforcements must initially be well dispersed to remove agglomerates of fibres or particulates, and this dispersion must then be sedimented to produce a preform consisting of an array of interlocked short fibres which support the particulate fraction.

Preforms containing Saffil fibres (3 μm diameter short δ-alumina fibres) and SiC particulates (commercial abrasive grits) were produced by a process route similar to that employed for conventional short-fibre preforms [8]. This process consisted of two stages: (i) the dispersion of the constituents into an aqueous medium, and (ii) the settling of this dispersion to produce a preform. In the first stage, milled Saffil (with an average fibre length of ~ 500 μm) and SiC particulates (600 and 320 grit) were dispersed into an aqueous medium using agitation. The level of agitation was carefully selected to break down fibre agglomerates without causing significant damage to the fibres themselves, and this agitation was also sufficient to disperse the particulates amongst the suspension of short fibres. The aqueous medium employed contained a mixed binder system consisting of an organic constituent, in this case latex (~ 2 wt % of the solids content) and an inorganic constituent, consisting of reagent grade colloidal silica (also ~ 2 wt % of the solids). Following this stage the preforms were fabricated by settling the dispersion on to a screen by means of vacuum-assisted filtration. The particulate:fibre ratios in the final preforms were controlled by altering the relative amounts of the species dispersed in the aqueous medium, and the total reinforcement fraction was controlled by two factors. These were: (i) the natural settling characteristics of the short-fibres and particulates, and (ii) the application of a load during settling. After settling the preforms were then carefully dried at 120 °C to drive off residual free water, and following this stage the preforms exhibited significant "green-strength" due to the presence of the latex binder. After drying, the preforms were then fired at 1200 °C in air. This firing had the dual role of burning out the organic binder and also generating strength in the inorganic silica binder. Following firing the preforms were then capable of resisting the metallostatic pressures applied during liquid-metal infiltration.

In the context of this paper, three types of hybrid preforms will be considered. These were manufactured with total reinforcement fractions of 0.30, and contained varying ratios of particulate and fibre. The ratios varied from 1:1 ($V_p = 0.15$:$V_f = 0.15$) to 5:1 ($V_p = 0.25$:$V_f = 0.05$). Preforms consisting of only Saffil fibres were also manufactured for comparison with the hybrid materials.

3. Composite fabrication and evaluation

Preforms of the type described above have been infiltrated with a variety of aluminium alloys ranging from commercially pure aluminium to high-strength precipitation hardening alloys. However, in the context of this paper the fabricated MMC will be compared on the basis of a 7039 (Al–4.0Zn–2.0Mg) matrix.

The experimental details of short-fibre MMC production using this alloy have been described in two earlier papers [9, 10], and in the present work similar infiltration conditions were employed. The preforms were preheated to 350 °C and infiltrated with 7039 (superheated to 1000 °C) under a metallostatic pressure of 25 MPa. The duration of this pressurization was 1 min. Following infiltration the fabricated MMC was then allowed to cool partially within the infiltration rig, and then finally air-cooled to room temperature. The composite properties were then determined with the matrix alloy in the as-cast condition to avoid possible problems associated with degraded age hardening in the composite matrices [11].

The quasi-static mechanical properties of the composites were determined by tensile testing, and the dynamic fracture responses by instrumented impact testing. Round tensile specimens with 6 mm diameters and 30 mm gauge lengths were machined from the cast composites using polycrystalline diamond tooling, and these specimens were tested in a conventional screw-driven tensile machine. The strains of the specimens were monitored throughout the tests using straingauges. This enabled measurement of both their elastic moduli and failure strains. Two gauges were attached to opposite sides of the specimens which compensated for any bending which developed during the tensile tests. The dynamic fracture responses of the composites were determined by instrumented Charpy testing on un-notched specimens diamond machined from the MMC castings. The test instrumentation consisted of a transducer on the rear surface of the impact tup composed of four straingauges in a Wheatstone bridge arrangement. Following calibration this transducer directly monitored the force on the tup during the fracture events. This enabled the determination of the force/time relationship during fracture. The impact data were captured by a transient recording system which processed the data and provided digital measurements of force, energy and displacement, as well as graphical data such as force/time, energy/time, and force/displacement. This allowed a more detailed analysis of the fracture events in these materials.

Optical, quantitative and scanning electron microscopy were also employed to characterize the composites and the fracture surfaces of the impact specimens.

4. Results

Fig. 2 shows the macroscopic appearance of these preforms prior to melt infiltration. The preforms were similar to conventional short-fibre preforms with nominal dimensions 100 mm diameter and 15 mm thickness. Figs 3 and 4 show the microstructures of the conventional short-fibre composite and the various hybrids. These composites were all well-infiltrated with no signs of residual porosity. The short-fibre material (Fig. 3a) exhibited a two-dimensional random distribution of fibres which was typical of the microstructures obtained from commercially available short-fibre preforms [7, 9, 10]. The hybrid composites (Figs 3b to d) also contained a two-dimensional random arrangement of short fibres, and this network supported a well-dispersed distribution of SiC particulates. Fig. 3 shows the hybrids containing the 600 grit SiC (with an average particulate size of ~ 8 μm) and Fig. 4 shows a composite containing the coarser 320 grit SiC (which exhibited an average size of ~ 18 μm). There were no signs of reaction between the fibres and matrix alloy, nor between the particulates and the matrix.

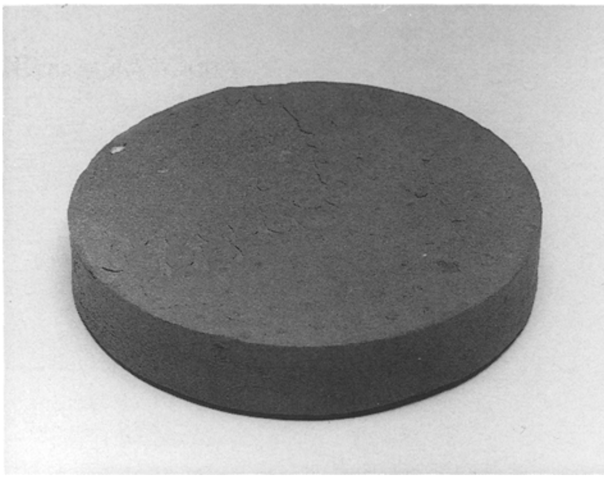

Figure 2 SiC particulate/Saffil fibre hybrid preform.

Table I compares the nominal reinforcement fractions of the hybrid composites (600 grit SiC) with the same fractions measured by quantitative optical microscopy. It is clear from this table that the nominal and true reinforcement fractions agree well. This agreement is significant because it implies that the final reinforcement fractions of the composites can be predicted from the amounts of the constituents dispersed during preform fabrication.

Identical hybrid microstructures were obtained in composites containing both fine and coarse particulates. The particulates were generally well dispersed, however, the fibre fraction did affect the homogeneity of their distribution. In low fibre fraction materials the

TABLE I Comparison of the nominal and true reinforcement fractions for composites containing a nominal total reinforcement fraction of 0.30

Fibre fraction, V_f		Particulate fraction, V_p		True total reinforcement fraction
Nominal[a]	True	Nominal[a]	True	
0.05	0.05	0.25	0.26	0.31
0.10	0.09	0.20	0.20	0.29
0.15	0.14	0.15	0.16	0.30

[a]Nominal fractions were determined from the mass ratio of the constituents dispersed during preforming.

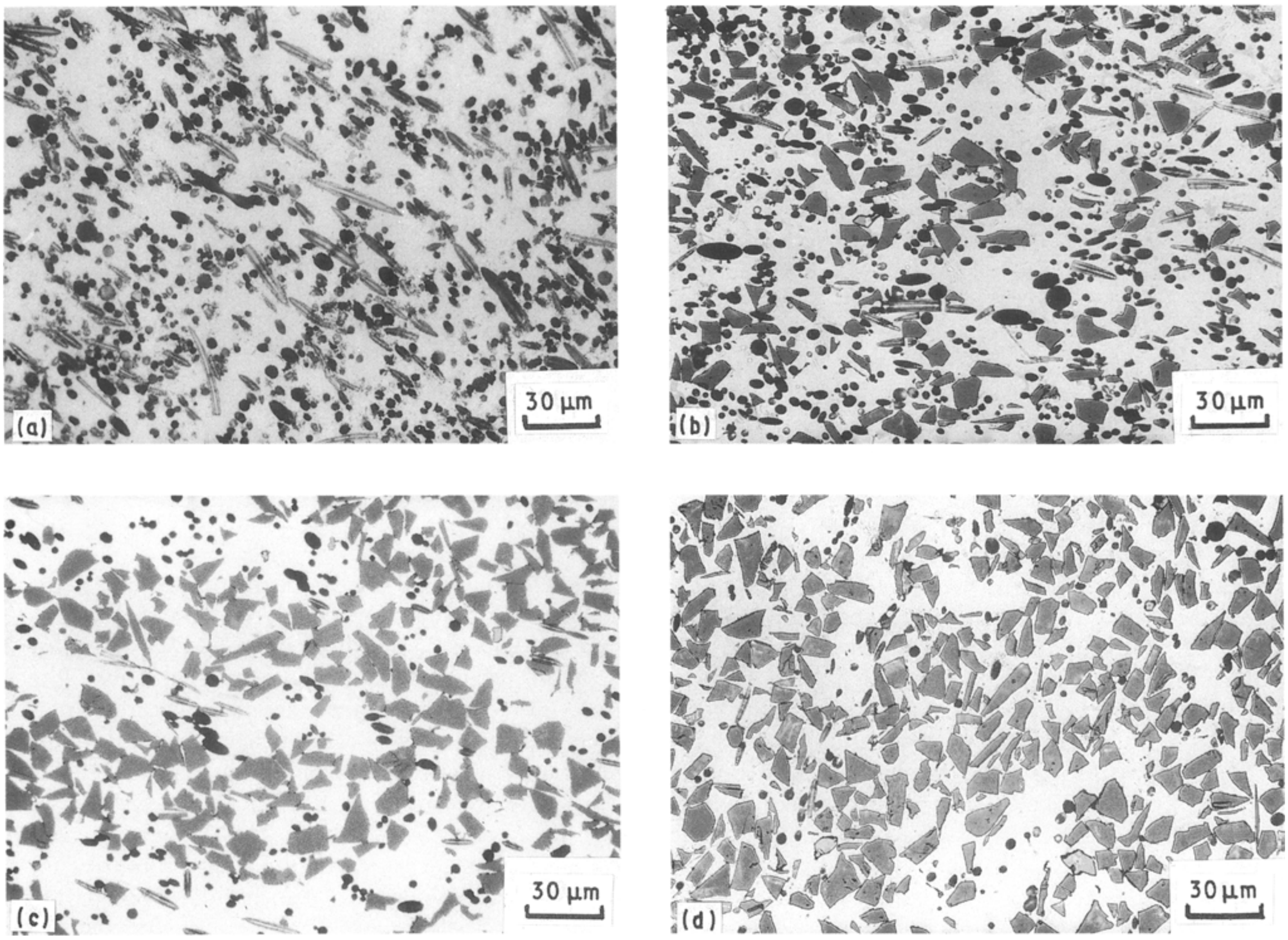

Figure 3 Optical micrographs of a conventional Saffil and SiC particulate/Saffil fibre hybrid composites, (a) $V_f = 0.30$, (b) $V_p = 0.15$: $V_f = 0.15$, (c) $V_p = 0.20$: $V_f = 0.10$, (d) $V_p = 0.25$: $V_f = 0.05$ (particulate size ~ 8 μm).

fibre was not uniformly dispersed and the particulates collected in the higher fibre fraction regions. This resulted in decreased homogeneity at higher particulate:fibre ratios. This effect is illustrated in Fig. 3c and d. The minimum fibre fraction which was able to support the particulates was ~ 0.05 and this therefore limited the maximum particulate:fibre ratio in this study to 5:1 ($V_p = 0.25$: $V_f = 0.05$).

Fig. 5 summarizes the mechanical properties of these composites. The UTS of the hybrids were marginally lower than those of the Saffil fibre composites, and exhibited little variation with particulate:fibre ratio. Similar effects were also observed for the yield and 0.2% proof stresses. The yield and proof stresses of the hybrids did not depend on the particulate size, exhibiting values of 26 ± 6 and 177 ± 9 MPa, respectively. These were marginally below the values observed for the $0.3V_f$ composite (35 ± 3 and 192 ± 10 MPa). However, the UTS did depend on the particulate size, decreasing with coarser particulates

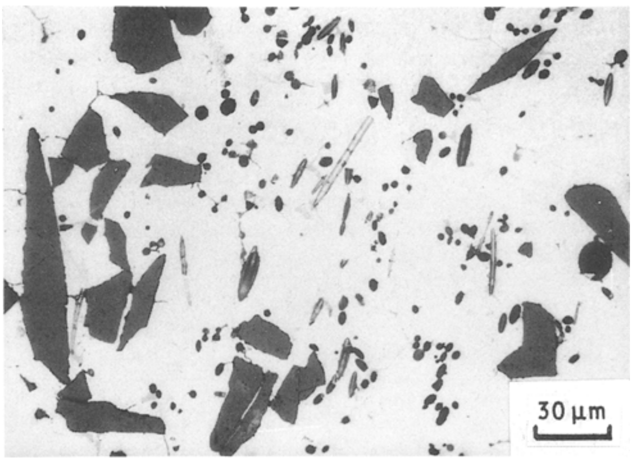

Figure 4 Optical micrograph of a SiC particulate/Saffil fibre hybrid composite (particulate size ~ 18 μm).

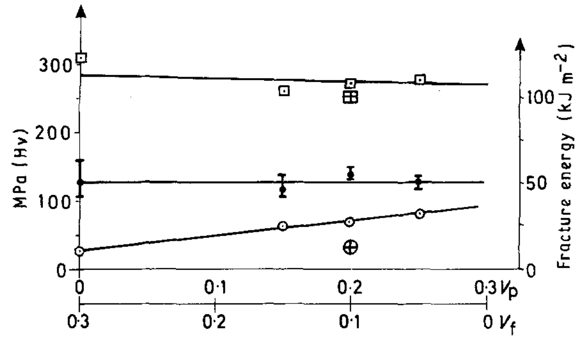

Figure 5 The effect of particulate:fibre ratio on the (⊡) UTS, (⧗) hardness and (⊙) toughness of SiC particulate/Saffil fibre hybrid MMC. (⊞ ⊕ Data for 18 μm particulate hybrids, total reinforced fraction = 0.3.)

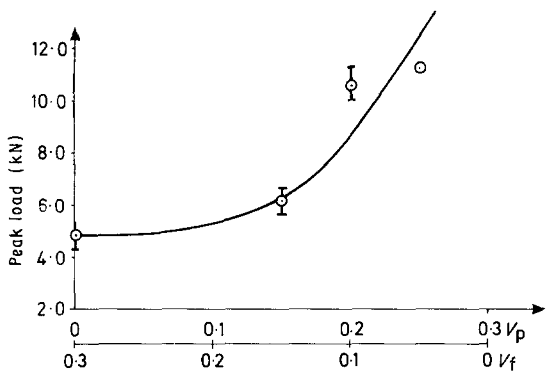

Figure 6 The effect of particulate:fibre ratio on the peak-load developed during instrumented impact testing. (Particulate size = 8 μm, total reinforced fraction = 0.3.)

(Fig. 5). Measurement of the elastic moduli of these materials was difficult. However, values of Young's modulus generally lay between 110 and 120 GPa. These were similar to values obtained for a $0.3V_f$ Saffil-reinforced composite. Fig. 5 also shows that the hardness of these hybrids was independent of the particulate:fibre ratio.

The final data in Fig. 5 shows the effect of particulate:fibre ratio on the dynamic fracture energy. It is clear from Fig. 5 that as the particulate:fibre ratio increased, fracture was associated with increased dynamic fracture energy. This indicated an improvement in composite toughness with increasing particulate:fibre ratio. The form of the impact (force/time) curves for the hybrids were similar to those of the short-fibre composites. The nature of short-fibre MMC impact curves have been discussed previously [10] and this analysis implies that the hybrids exhibited an essentially brittle response with a low energy associated with the propagation of fracture. The impact curves for all the composites exhibited an elastic response of similar "compliance" but a peak-load which increased with particulate:fibre ratio. The variation of this peak-load is shown in Fig. 6. The size of the particulate also had a significant effect on the toughness (Fig. 5), with coarser particulates resulting in lower toughness levels.

Figs 7 and 8 show fractographs from the impact specimens. There was a continuous transition in the fracture surfaces with increasing particulate:fibre ratio (Fig. 7). At low ratios the fracture was dominated by the short fibres and there was little evidence of matrix ductility. This contrasted with higher ratios where the fracture surface became less dominated by fibre failure, and instead by the presence of the particulates. Increasing the fraction of the particulates also resulted in more extensive matrix regions which exhibited clear signs of plastic deformation. Fig. 8 shows the fracture surface of a material containing the coarser particulate. It is clear that in these hybrids the

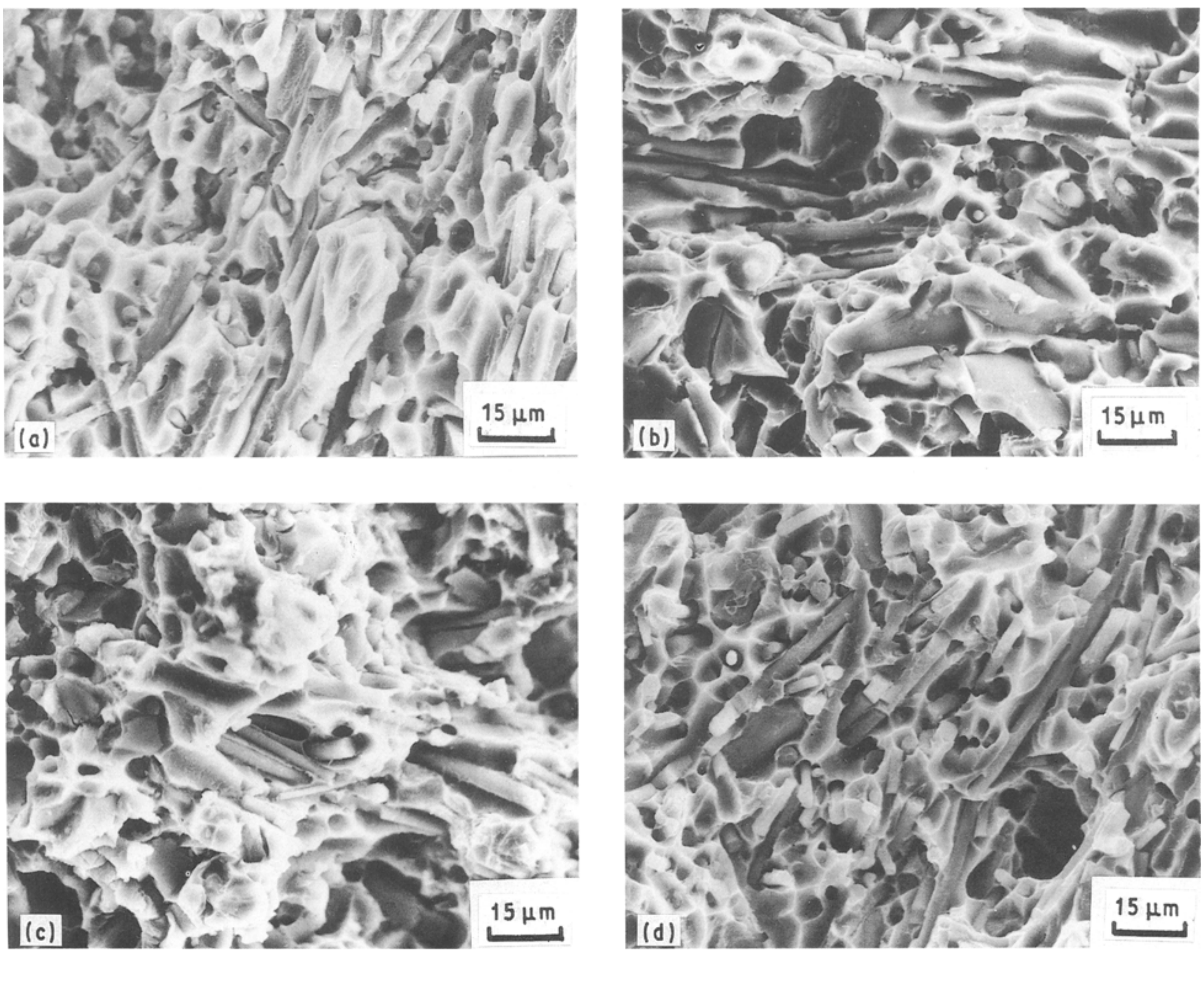

Figure 7 Fractographs of a conventional Saffil and SiC particulate/Saffil fibre hybrid composites. (a) $V_f = 0.30$, (b) $V_p = 0.15$: $V_f = 0.15$, (c) $V_p = 0.20$: $V_f = 0.10$, (d) $V_p = 0.25$: $V_f = 0.05$ (particulate size ~8 μm).

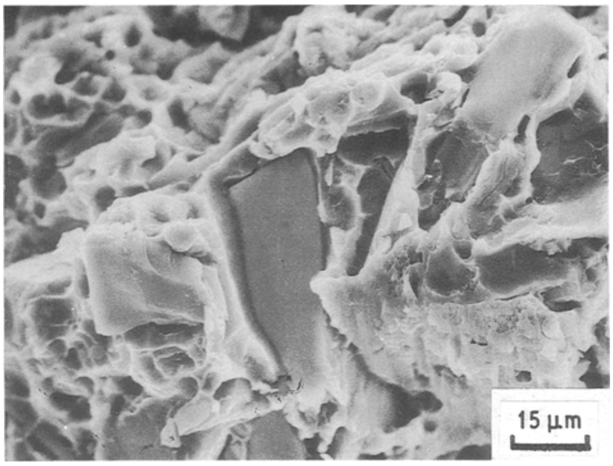

Figure 8 Fractograph of a SiC particulate/Saffil fibre hybrid composite containing $\sim$ 18 μm particulates ($V_p = 0.20 : V_f = 0.10$).

fracture response was dominated by the coarse particulates.

5. Discussion

The results presented above show that it is possible to produce particulate/fibre hybrid MMC components by the preform infiltration route, and at the highest particulate:fibre ratios these composite materials are almost identical to conventional particulate-reinforced MMC. In these hybrids the strengths and hardness appear to be independent of particulate:fibre ratio, and by implication must have depended only on the total volume fraction of reinforcement. This is interesting and appears to contradict the work of Towata and Yamada [12] who have shown that the presence of SiC particulates can improve the strength of SiC continuous fibre-reinforced composites. However, the present hybrids do differ substantially from those of Towata and Yamada. They investigated continuous fibre/particulate hybrids with particulate/fibre ratios of 0.065:1, whereas the present work concentrated on particulate/short-fibre hybrids with ratios between 1:1 and 5:1. These systems lie at opposite extremes of the range of possible hybrids, and also differ in the nature of the fibre reinforcements. In continuous fibre/particulate hybrids it has been shown [13] that a number of mechanisms interact to provide enhanced mechanical properties. These include (i) improvements in the fibre distribution due to the presence of particulates in the interfibre regions, (ii) the reduction in the formation of meniscus penetration defects found at fibre contact points, and (iii) reductions in the scale of intermetallic compound formation at fibre/matrix interfaces due to the increase in the interfacial area. However, short-fibre based hybrids differ substantially from these materials. In particular, the microstructures of short-fibre MMC are characterized by features such as fibre bridging which is usually deleterious to the properties of continuous fibre materials [13]. Fibre bridging is also a characteristic of the present hybrids where it is used to disperse the particulate fraction. The presence of such microstructural features, therefore, appears to preclude the principal strengthening mechanisms identified above. Therefore, in the case of the present hybrids it can be assumed that hybridizing with particulates produces no enhancement of the quasi-static mechanical properties. The response of MMC to hybridizing, therefore, appears to differ depending on both the particulate:fibre ratio employed and the nature of the fibre reinforcement.

However, in the present work, particulate:fibre ratio did play a strong role in controlling the toughness of the hybrid MMC. The brittle nature of the force/time curves for these hybrids showed that to a first approximation they could be treated by simple linear elastic fracture mechanics. Such an analysis implies that their dynamic fracture energies (Fig. 5) were a measure of the strain–energy release-rates of the composites [14]. Because this energy is controlled primarily by the load required to initiate fracture (Fig. 6), and because the moduli of the hybrids were similar, this suggests that the toughening of these materials with increasing particulate:fibre ratio was associated with an increase in their fracture toughness. Further evidence of such a mechanism comes from the effect of particulate size on the fracture energies. Fig. 5 suggests that the fracture toughness of these hybrids decreases with increasing particulate size. This is consistent with previous observations of the effect of particulate size on the fracture toughness of particulate-reinforced MMC [15].

The improved toughness of these hybrid MMC is interesting because the fracture toughness of particulate-reinforced materials are often reported to be higher than those of short-fibre-reinforced composites [14, 16]. This suggests that by altering the particulate:fibre ratio, the toughness of these hybrid MMC can be continuously changed from the low value associated with short-fibre composites (at low particulate:fibre ratios) to the higher values characteristic of particulate-reinforced materials (at high particulate:fibre ratios). Such an observation is consistent with the fracture surfaces of the impact specimens (Fig. 7) which showed a change in the fracture event from a fibre-dominated process at low particulate:fibre ratios to a particulate-dominated response at high particulate:fibre ratios.

6. Conclusions

1. Particulate/fibre hybrid MMC can be produced by the preform infiltration route.
2. At high particulate:fibre ratios these hybrid MMC are almost indistinguishable from conventional particulate-reinforced materials.
3. The strength, hardness and elastic moduli of these hybrid composites appear to be controlled by the total reinforcement fraction and not by the particulate:fibre ratio.
4. The fracture toughness and dynamic fracture energies of these hybrids depend on the particulate:fibre ratio. These increase at higher ratios due to a change in the fracture process from a fibre, to a particulate-dominated response.
5. The UTS and toughness of these hybrids both

depend on the particulate size, decreasing as the particulate size increases.

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