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Composite strengthening in 6061 and Al–4 Mg alloys

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Metal matrix composites using prealloyed 6061 Al (containing 1% Mg) and elemental blend Al–4Mg alloys with 10 vol% SiC particulate reinforcements were fabricated using powder metallurgy techniques. The consolidation of the powders was effected by the section rolling process recently developed at the Defence Metallurgical Research Laboratory. This process involves the successive steps of cold isostatic pressing, vacuum sintering and special canning followed by section rolling. This resulted in a high-integrity composite product. An interfacial layer containing magnesium-rich precipitates observed in both the composites is suggested to be the major reason for the low (compared to the value predicted by the rule of mixtures) modulus and strength values in these composites. This layer also appeared to promote interfacial failure at the alloy/SiC interface. The Al–4 Mg alloy, which is known to be non-heat treatable, was found to respond to precipitation hardening heat treatment in the composite. The enhanced generation of dislocations due to the presence of SiC, promoting a more homogeneous precipitation of the second phase and the possibility of an inhomogeneous distribution of magnesium (as a result of elemental blending) are suggested to be the major factors responsible for rendering the Al–4Mg alloy amenable to the precipitation hardening heat treatment.

1. Introduction

Aluminium and its alloys, discontinuously reinforced with SiC particles have been the focus of worldwide attention in the development of metal matrix composites for some time [1, 2]. The compatibility of SiC, as a reinforcement, with the aluminium matrix and the significant improvement in properties of the composite over aluminium and its alloys are the two major reasons for the extensive use of SiC. Although various techniques are employed in the fabrication of aluminium matrix composites, these can be broadly classified into two groups, namely powder metallurgy and liquid metallurgy. The powder metallurgy route is particularly attractive because of the possibility of obtaining a more homogeneous distribution of the reinforcement in the matrix and limiting the matrix-reinforcement reactions which is a problem when employing the liquid metallurgy technique.

There have been, over the years, numerous powder metallurgy techniques that have been developed for the fabrication of aluminium matrix composites. By far the most widely used technique is that where the matrix and the reinforcement particulates are blended, compacted and then hot pressed. These compacts are consolidated further by extrusion. Conform extrusion has been developed more recently [3] and has been used to produce Al/SiC composites. In this process, the powders are blended and extruded in a continuous manner. The properties of the composites obtained by employing conform extrusion have been quite attractive. Another process which has been tried is the spray-rolling process [4], where the reinforcement is injected into an atomized stream of molten metal which is then sprayed on to a substrate. The major problem in this process is that the interfacial bonding is primarily mechanical and therefore composites with spherical particles were weaker than those with irregular particles. This becomes significant because a three-dimensional configuration is built up by spraying layers of molten metal mixed with the reinforcement. The Ospray process [5] is essentially a modification of this spray-rolling process. Mechanical alloying of the matrix and reinforcement [6] has been used to fabricate composites. This process has the advantage of improving the matrix-reinforcement bonding and distribution in addition to the possibility of developing a finer microstructure in the matrix. Rapid solidification by melt spinning [7] is yet another process that has been used to make aluminium composites. This is classified under powder metallurgy techniques because the melt-spun ribbons are first pulverized and then consolidated by hot pressing and subsequent metal working operations (forging, rolling, extrusion, etc.).

At the Defence Metallurgical Research Laboratory, a technique has been developed where a sintered compact of the aluminium matrix composite is consolidated by direct rolling into sections. This has been achieved by a special canning technique which essentially provides a constraint to the deformation of the outer surface layer of the compact. This process has

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been successfully employed for the fabrication of aluminium matrix composites with different reinforcements, which show excellent properties [8].

The primary consideration in the choice of the matrix material is its compatibility with the reinforcement. Because the properties of the composites are significantly influenced by the interface, much attention has been paid to the choice of alloying elements that may have a beneficial effect on the characteristics of the matrix reinforcement interface. A strongly adherent interface is a prerequisite for a good composite structure. While there needs to be sufficient wetting of the reinforcement by the matrix, there should be no excessive reaction between the two which may result in the degradation of the composite properties. Because different alloying elements contribute to these factors in different ways [9], the choice of a composition which strikes the right compromise between the wettability and excessive reaction is often very difficult.

In this study, two alloys containing magnesium have been chosen for the matrix. One is 6061 Al which contains about 1% Mg as the major alloying element. The low strength and good ductility of 6061 Al make it an ideal candidate for a considerable amount of composite strengthening. The other alloy is Al–4% Mg which is known to have a high capacity for work hardening. This alloy was made using a variation of the elemental blend technique. Powders of an Al–Mg master alloy were diluted with that of pure aluminium to achieve the desired composition, the benefits of which are further described in this paper. Pure aluminium was also used as a matrix for comparison with the alloys. The composites were consolidated by the "sinter + rolling" technique described previously.

2. Experimental procedure

The composites of pure aluminium, 6061 Al and Al–4 Mg with SiC particles were fabricated using powder metallurgical techniques. Pure aluminium and 6061 Al powder was prepared by ultrasonic gas atomization in an inert environment. A master alloy of Al–Mg was prepared and blended with the pure aluminium powder to obtain the desired composition of Al–4 Mg. The chemical analysis and the average particle sizes of the powders are shown in Table I. The aluminium alloy composites containing 10 vol% SiC were made by blending the powders of the matrix with −500 mesh SiC particles in a polar solvent medium, using a horizontal roller mill which gave a tumbling action to the powder slurry. This blended slurry was dried and the powders were cold isostatically pressed at 200 MPa. These compacts were then degassed in vacuum for 1 h. Pure aluminium and 6061 Al powders were also processed in an identical manner for comparison with the composites. The vacuum degassing temperature was 450 °C for pure aluminium and Al/SiC while it was 400 °C for 6061 Al, 6061 Al/SiC and Al–4 Mg/SiC. These compacts which were degassed were subsequently sintered in vacuum for 1 h. The sintering temperature was 600 °C for pure aluminium and Al/SiC and 510 °C for 6061 Al/SiC and Al–4 Mg/SiC.

TABLE I Chemical analysis and size of powders

Powder	Chemical analysis	Average particle size (μm)
Al	0.25 Fe, 0.04 Si, bal Al.	45
6061 Al	0.83 Mg, 0.27 Si, 0.25 Cu, 0.06 Fe, bal Al.	44
Al–4 Mg	4 Mg, 0.018 Si, 0.013 Fe, bal. Al	45

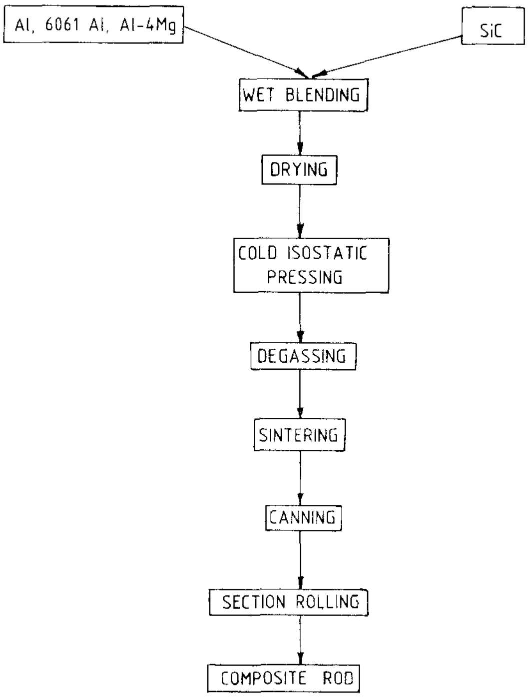


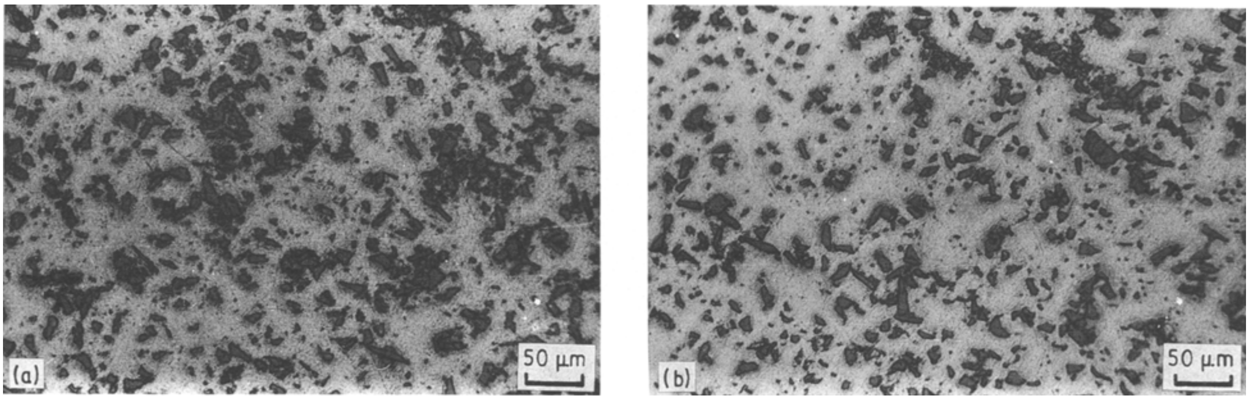
Figure 1 Processing sequence of composites.

The sintered compacts were further consolidated by directly rolling them into round sections having a diameter of 6 to 8 mm. The rolling temperature was 600 °C for aluminium and Al/SiC and 550 °C for the other sintered compacts. The rolling was preceded by a special canning process. Canning would, most likely, provide the constraint to deformation on the surfaces of the sintered compacts. The entire processing sequence is schematically shown in Fig. 1. The composite rods appeared sound and were further used for metallography and tensile testing.

One sample each from the composites was mounted and polished metallographically for examination of the microstructure under both an optical and a scanning electron microscope.

The elastic modulus of each of the samples, produced by rolling, was measured on rods, 5 mm diameter, using a resonance technique (by analysing the vibrational behaviour of the sample following an impulse excitation).

Samples for tensile testing were machined from the different rods. These samples had a gauge diameter of 4 mm and a gauge length of 25 mm. Tensile testing was done at a strain rate of 6.6×10^{-4} sec^{-1}. The fracture surfaces of the sample were examined in an



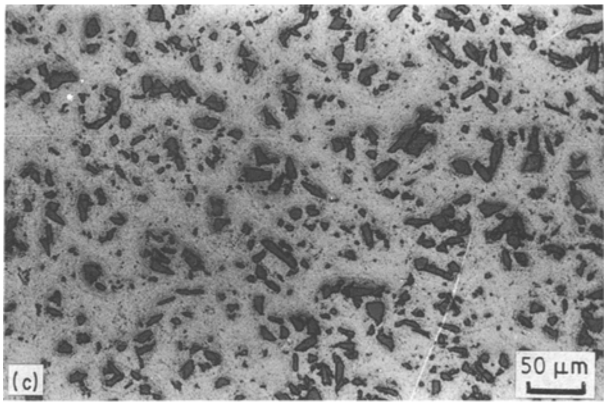


Figure 2 Optical micrographs of the composites: (a) Al/10 vol% SiCp; (b) 6061 Al/10 vol% SiCp, (c) Al–4Mg/10 vol% SiCp.

SEM. Because the alloy 6061 is heat treatable, both 6061 Al and 6061 Al/SiC tensile samples were heat treated to the T6 condition prescribed for the alloy 6061. The heat treatment involved solution treatment at 530 °C for 3 h followed by ageing at 160 °C for 18 h. The Al–4 Mg/SiC was given an identical heat treatment to that of 6061 Al. Although Al–4 Mg alloy is not known to be heat treatable, in this case the composite responded to heat treatment. The possible reasons for this behaviour are discussed later.

Transmission electron microscopy was performed on ion-thinned composite samples to examine the interface between the matrix and the reinforcement to correlate it with the observed properties of elastic modulus and tensile strength.

3. Results and discussion

Fig. 2 shows the microstructure of the composite as observed under an optical microscope. These micrographs clearly show that the distribution of SiC is uniform across the matrix. They also show that the polar solvent medium used in blending is effective in preventing any form of agglomeration of the SiC particles. The microstructure does not show any porosity and therefore it is concluded that the "sinter + rolling" technique for the consolidation of these composites is extremely effective in producing an integral composite with uniform distribution of the reinforcement.

The elastic modulus of each of the samples, measured by the resonance technique, is shown in Table II. The predicted value of the elastic modulus for each of these composites, based on the rule of mixtures is shown alongside the measured value under the heading ROM-*E*. A value of 480 GPa has been used in the calculation for SiC [10]. The values for pure aluminium and 6061 Al were measured in this study. In pure aluminium, the addition of 10 vol% SiC particles increases the modulus by almost 50%. The observed value of 102 GPa is very close to the theoretically predicted value of 111 GPa. This is indicative of the integrity of the interface between aluminium and SiC. A strongly adherent interface, void of any interfacial reaction product, would ideally be the most effective in transferring the load from the matrix to the reinforcement. In the Al/SiC composite, the transmission electron micrograph shows (Fig. 3) that the interface is clean and well delineated, thus appearing sound.

In the 6061 Al alloy, the addition of 10 vol% SiC particles leads to a substantial increase in the elastic modulus (Table II), though it is not as much as in the case of pure aluminium. It is also interesting to note that the measured value falls far short of the predicted value. This is quite consistent with the observation of a very thin layer at the interface between the matrix and the SiC (Fig. 4). Although this layer was not analysed for its composition, it is most likely a magnesium-rich phase resulting from the segregation of

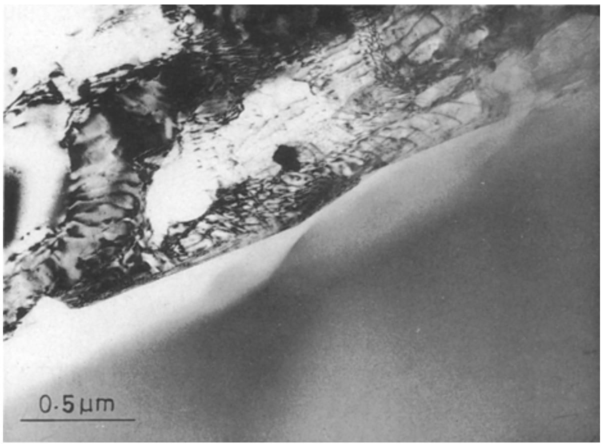


Figure 3 Transmission electron micrograph of the Al/SiC interface.

TABLE II Mechanical properties of aluminium matrix composites

Material	Processing condition	*E* (GPa)	ROM-*E* (GPa)	YS (MPa)	UTS (MPa)	Elongation (%)
Al	Section rolled	70	–	64	90	21
Al/10 vol% SiCp[a]	Section rolled	102	111	91	140	20
Al/10 vol% SiCp	Extruded	–	–	75	115	22
6061 Al	Section rolled	–	–	110	170	24
6061 Al	Section rolled + T6	68.5	–	243	290	15
6061/10 vol% SiCp	Section rolled	–	–	148	202	11
6061/10 vol% SiCp	Section rolled + T6	94	109	304	327	8
Al–4 Mg	Rolled + annealed	67 [13]	–	125 [13]	225 [13]	20 [13]
Al–4 Mg/10 vol% SiCp	Section rolled	–	–	136	258	21
Al–4 Mg/10 vol% SiCp	Section rolled (heat treated)	91	108	289	350	7.2

[a] SiCp = SiC particles.

magnesium to the interface of 6061 Al and SiC. Because the matrix contains 0.8% Mg, it is quite likely that there is some segregation of magnesium which is consistent with the suggestion of Nutt and Carpenter [11]. It is, therefore, inferred that this interfacial layer is responsible for the elastic modulus value falling short of the predicted value. This fact also underlines the importance of the interface in influencing composite properties. It is also worthwhile to note that the elastic modulus of 94 GPa for this composite (with 10 vol% SiC particles) is still superior to the 88 GPa obtained for 6061 Al with 15 vol% SiC particles in previously reported work [12].

The elastic modulus of Al–4 Mg, quoted in Table II has been obtained from published literature [13]. Comparing this value (67 GPa) with that of the composite (91 GPa), reveals the beneficial effects of SiC. Fig. 5a shows a layer containing precipitates at the interface between the matrix and SiC. This layer was analysed and found to be rich in magnesium (Fig. 5b). The presence of silicon, however, could not be detected. The presence of this magnesium-rich layer is, most likely, responsible for the improvement in modulus being considerably lower than the predicted value.

In tensile testing it is found that SiC has a significant effect on composite-strengthening pure aluminium. An addition of 10 vol% SiC leads to a 55% increase in the tensile strength while the ductility remains almost the same. While the nature of the interface (Fig. 3) is certainly beneficial in this system, the significant observation is that the strengthening comes with no penalty to the ductility (% elongation). This is also indicative of the nature of consolidation and the efficiency of the "sinter + rolling" technique in producing sound composites. The strength and ductility of extruded Al/SiC (processed at DMRL and given an extrusion ratio of 10:1) is shown in Table II. The values obtained by section rolling are comparable to that obtained by extrusion. The strength value of 140 MPa, obtained by section rolling, is also comparable to values reported elsewhere [14] for the extruded product. The fracture surface of the composite

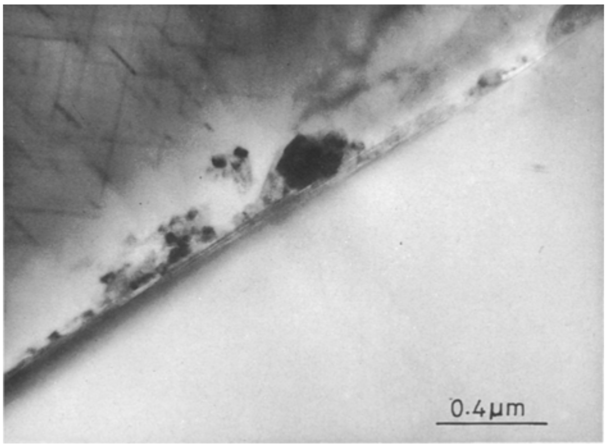


Figure 4 Transmission electron micrograph of 6061 Al/SiC interface.

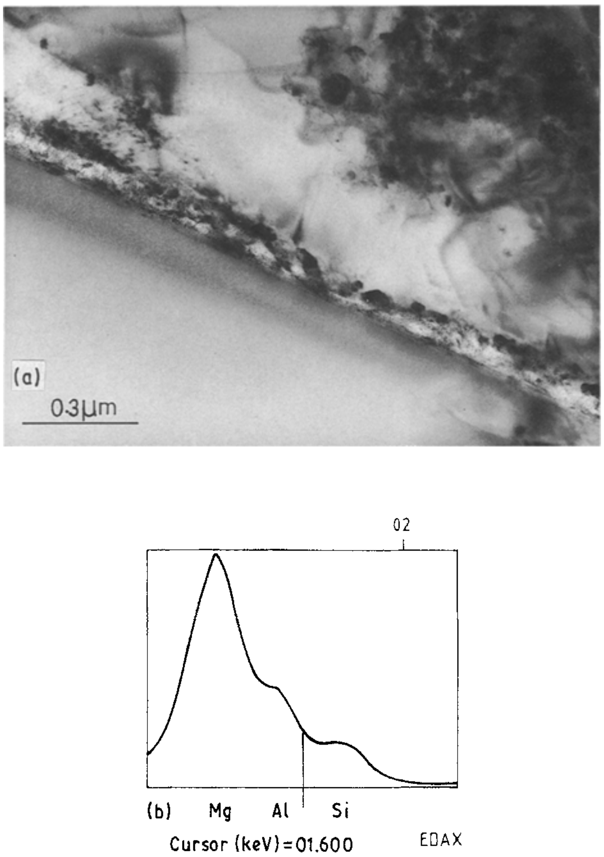


Figure 5 (a) Transmission electron micrograph of Al–4Mg/SiC interface. (b) EDAX analysis of interfacial precipitates.

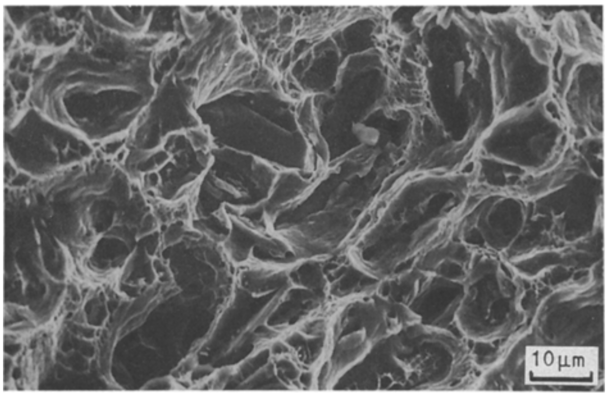


Figure 6 Fractograph of Al/SiC.

sample (Fig. 6) shows dimpled failure and also some indication of failure at the Al–SiC interface although it is certainly not as in the cases of 6061 Al and Al–4 Mg composites, as will be seen later in this discussion.

In the as-rolled condition, the 6061 Al alloy displays a tensile strength of 179 MPa, whereas in the peak-aged condition it is 290 MPa. In spite of the fact that the silicon content of this alloy is lower than the prescribed limits, this value compares well with the 300 MPa reported by McDanels [12]. 6061 Al/SiC, in the as-rolled condition appears superior to the 6061 Al in the same condition, except for ductility which is about 50% lower in the composite. In the peak-aged condition, SiC has a greater influence on the yield strength in 6061 Al than on the tensile strength. Compared to 243 MPa for the matrix, the composite shows a yield strength of 304 MPa. The ultimate tensile strength of the matrix is 290 MPa and that of the composite is 327 MPa. However, the overall effect of adding SiC to 6061 Al does not appear to be as dramatic as in the case of pure aluminium. The ductility (% elongation) of 8% for the 6061 Al/SiC composite in the T6 condition is comparable to that of 6% reported previously [12], where the reinforcement was 15% SiC particles compared to 10% in this study. This is again indicative of the efficiency of the section rolling process as a means of consolidation of the sintered compacts of these composites. The fracture surface (Fig. 7) of the 6061 Al/SiC composite shows ductile failure but also indicates failure at the alloy–SiC interface. This is consistent with the observation in Fig. 4 of the presence of a thin interfacial layer in the composite. It can also be seen that there is a precipitate-free zone adjacent to this layer. Some coarse precipitates are also visible at the interface. It is likely that the interfacial precipitates are Mg_2Si and the matrix precipitates are magnesium and aluminium [15].

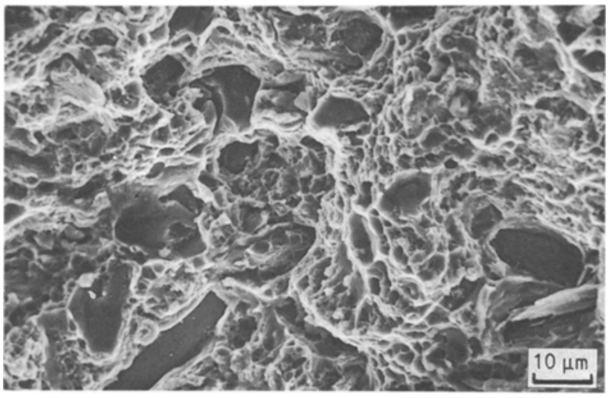
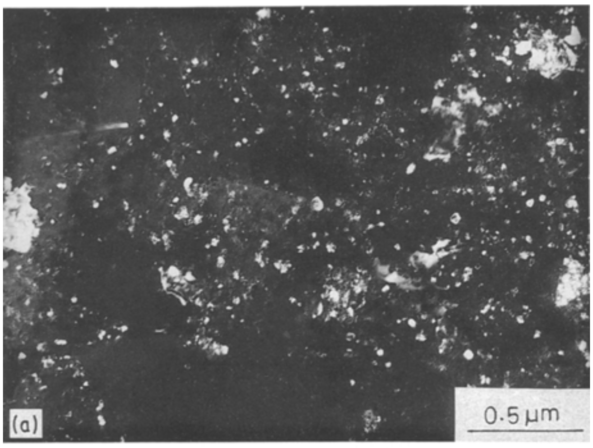


Figure 7 Fractograph of 6061 Al/SiC.

The Al–4 Mg/SiC composite shows heat-treatable characteristics. As compared to the “as-rolled” condition, there is considerable improvement in the strength on ageing from 136 to 189 MPa in the yield strength and from 258 to 350 MPa in the ultimate tensile strength. The average strength and ductility values reported in the literature for the Al–4 Mg, rolled and annealed product [13], is also shown in Table II for comparison. There is more than a 50% improvement in the ultimate tensile strength due to the addition of SiC and the nature of this elemental blend process. Fig. 8a shows the particles that contribute to strengthening in this composite. These particles were analysed and found to be rich in aluminium and magnesium in almost equal proportions (Fig. 8b). In general, only alloys with magnesium contents above 5% to 6% are expected to respond to heat treatment [13]. In aluminium alloys with higher concentrations of magnesium, coarse precipitates of Mg_5Al_8 form heterogeneously at preferred sites like slip bands, dislocations and grain boundaries [16]. Therefore age hardening does not occur. In the case of this composite, the large number of dislocations generated by the presence of SiC particles is most likely to result in a more “homogeneous” precipitation in the matrix, thus giving improvements in strength on heat treatment. Another possible reason for this unexpected response to heat treatment is that the magnesium concentration may not be uniform throughout the matrix due to the nature of the elemental blend process. This is probably the reason for the presence of localized regions where clusters of magnesium-rich precipitates have been observed (Fig. 8c). Localized heterogeneities of the magnesium concentration could also promote precipitation of phases that strengthen the composite on heat treatment. No attempt was made, however, to optimize the heat-treatment schedule. Comparing the strength values obtained in this composite with that of 5083 Al composites [12], it is found that the UTS of 350 MPa with 10 vol% SiC particles compares well with the 360 MPa obtained in 5083 Al reinforced with 20 vol% SiC particles. It is of interest to note that 5083 Al has 0.7% Mn and 0.15% Cr besides 4.4% Mg which would be expected to promote strengthening. The 7% ductility in the Al–4% Mg composites is also respectable. Fig. 9 shows the fracture surface of this composite. Failure still appears predominantly dimpled, although interfacial failure can be observed. This is consistent with the presence of the magnesium-rich interfacial layer observed in Fig. 5a.

A comparison of the composites shows that, in the heat-treated condition, the Al–4 Mg/SiC composite is superior to both the 6061 Al and pure aluminium composites, based on tensile strength. A comparison of the specific properties can be seen in Fig. 10. When



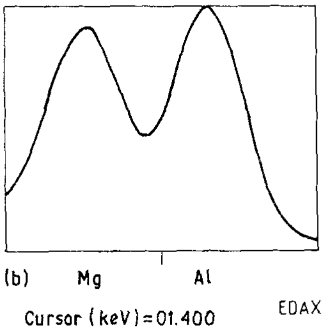
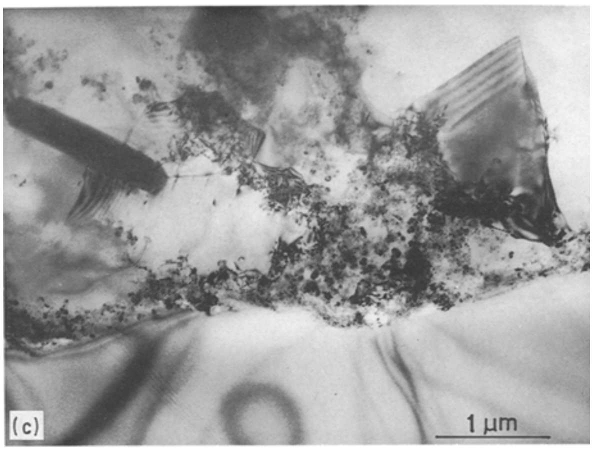


Figure 8 (a) Fine Mg–Al precipitates in Al–4Mg/SiC. (b) EDAX analysis of precipitates observed in (a). (c) Clusters of magnesium-rich precipitates in Al–4Mg/SiC.



comparing specific strength, the highest value is observed in the Al–4 Mg/SiC composite although the effect of SiC in composite strengthening is highest in pure aluminium. In pure aluminium, SiC contributes to a 42% increase in yield strength as compared to only 25% in the 6061 Al system. A comparison of the specific moduli shows that the maximum value is observed in the Al/SiC system and the effect of SiC is most pronounced also in that system.

4. Conclusions

The influence of an addition of 10% SiC particles in pure Al, prealloyed 6061 Al and elemental blend Al–4 Mg has been examined. Consolidation by "sintering + rolling" was found to be a viable process for producing these composites.

The addition of SiC resulted in significant improvements in the strength and modulus in pure aluminium as compared to 6061 Al or Al–4 Mg. The lower value of the elastic modulus observed in 6061 Al/SiC and Al–4 Mg/SiC, compared to the predicted value, was attributed to the presence of the interfacial layer between the alloy and SiC.

The elemental blend approach resulted in an Al–4 Mg/SiC composite that was heat treatable, which also yielded strength values superior even to that of the 6061 Al/SiC composite in the peak-aged condition. The generation of a large number of dislocations due to the presence of SiC probably presents a sufficient number of heterogeneous locations to enhance the precipitation of phases that contribute to strengthening. Another possible reason is the nature of

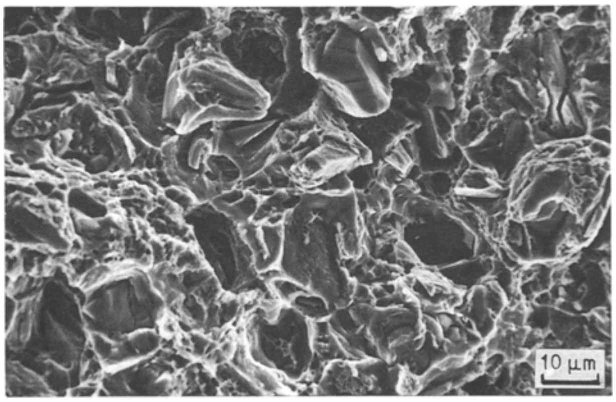


Figure 9 Fractograph of Al–4Mg/SiC.

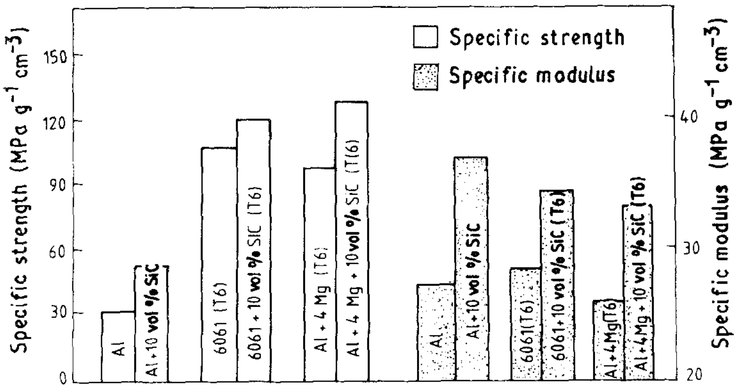


Figure 10 Comparison of specific properties of the composites.

elemental blending to produce Al–4 Mg and the tendency of magnesium to segregate to the interface, resulting in an inhomogeneous distribution of magnesium throughout the matrix, thus enhancing the precipitation of intermetallic phases. Further research is needed to establish clearly the reason. However, it is significant that there is a possibility that non-heat-treatable alloys can be made heat treatable by incorporating reinforcements or elemental blending in such alloys.

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