JOURNAL OF MATERIALS SCIENCE 31 (1996) 164–171

An investigation of the microstructure and mechanical properties of the macro-interface in selectively reinforced aluminium castings

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The ring groove areas of squeeze-cast Al–12% Si alloy pistons can be selectively reinforced with Saffil (Al_2O_3) fibres or SiC whiskers to provide local high temperature strength and wear resistance. Since the reinforced region and the unreinforced alloy typically have different coefficients of thermal expansion, cyclic residual stress may occur at the macro-interface between them when it experiences thermal cycling. This could conceivably result in fatigue induced damage at the macro-interface, making it susceptible to failure. To investigate this, the strength of the macro-interface has been measured before and after thermal cycling using bimaterial tensile samples. Prior to thermal exposure, samples typically failed at the macro-interface with an average strength less than that of the unreinforced alloy alone. The low initial strength has been attributed to several factors, including poor alloy–reinforcement bonding and an accumulation of brittle particles or other material at the macro-interface. After being thermally cycled 1000 times between 50 °C and 275 °C or given an equivalent isothermal exposure, samples typically failed in the unreinforced alloy or at the macro-interface with average strengths less than those measured prior to thermal exposure. However, there was no clear evidence that fatigue induced damage had occurred as a result of thermal cycling and the strength drop associated with thermal exposure has been attributed to alloy overageing.

1. Introduction

There has been a great deal of interest in the development of discontinuously reinforced metal–matrix composites (MMCs) for a variety of applications. The automotive industry is particularly interested in developing Al-alloy based MMCs for use in cast engine components because of their potential for weight savings over conventional iron components. However, cost and manufacturing feasibility have played a large role in determining the extent to which composites have been used for such applications. This has given rise to the practice of selective reinforcement in which only those areas of a component which require increased strength or wear resistance are reinforced. This practice has been applied in the production of squeeze-cast Al–12% Si alloy pistons which are reinforced only in the ring groove areas. The pistons are made by first placing a preform of fibres or whiskers into a die in the appropriate location. Liquid alloy is then cast under pressure to infiltrate the preform and fill out the rest of the die. This method has been shown to produce quality castings with a minimum of porosity and good alloy–reinforcement bonding [1]. However, there is some concern about the mechanical integrity of the macro-interface between the composite region and the unreinforced alloy. For example, it has been observed that the preform surface may act as a filter for solid particles in the infiltrating liquid alloy, causing their accumulation at the macro-interface [2]. It is conceivable that such a situation could influence its strength. Also, the macro-interface may be the site of residual stress because of the thermal expansion difference between the composite and the unreinforced alloy. This is a special concern for selectively reinforced components, such as pistons, that experience thermal cycling because the magnitude of the residual stress will change with temperature changes. It is conceivable that the cyclic residual stress, produced by thermal cycling, could lead to fatigue induced damage at the macro-interface and possibly failure of the component.

The objective of the present work was to investigate the microstructure and strength of the macro-interface in selectively reinforced castings and to determine if thermal cycling causes fatigue induced damage at the macro-interface, thereby degrading its strength.

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TABLE I Nominal element composition of the alloy [3]

Element	Si	Fe	Cu	Mn	Mg	Zn	Ti	Ni	Al
Composition %	11.0–13.0	0.9	1.5–3.0	0.5	0.6–1.5	1.0	0.25	0.5–1.5	balance

2. Experimental procedure

2.1. Material and heat treatment

Selectively reinforced Al–12% Si alloy squeeze-castings were obtained from the General Motors Advanced Material Development Center. All of the castings contained a disc-shaped region approximately 9 cm in diameter and 1.5–2.0 cm thick reinforced with either SiC_w or Saffil (Al_2O_3) fibres. The nominal element composition of the alloy is listed in Table I [3]. Prior to casting, the SiC_w and Saffil preforms were heated to 850 and 500 °C, respectively. The metal superheat was 760 °C. During casting, an infiltration pressure of 172 MPa was used.

Three different alloy–composite groups were used in the present study and were distinguished by alloy type and reinforcement:

1. Refined alloy–19% v/o SiC_w composite;
2. Unrefined alloy–16% and 19% v/o SiC_w composite;
3. Unrefined alloy–18% v/o Saffil composite.

"Refined" or "unrefined" refers to whether or not the Si phase in the alloy was refined such that it formed blocky particles. Refinement is accomplished by adding phosphorus to the molten alloy where it reacts with aluminium to form AlP crystals. These crystals then act as nuclei for the growth of primary Si particles since they have a similar crystal structure and lattice constant [4].

Test samples fabricated from the castings were given an initial T5 heat treatment prior to mechanical testing or thermal exposure. This consisted of holding them at 210 °C for 8 h followed by an air cool. This is a precipitation heat treatment used to ensure dimensional stability and provide some relaxation of residual stresses [5].

2.2. Mechanical testing

The most straightforward method of measuring the strength of the macro-interface, as used by other investigators, was to test bimaterial tensile samples [2, 6]. A simple flat, rectangular geometry was selected for the tensile samples used in the present study (Fig. 1).

2.3. Thermal cycling

Thermal cycling was performed using an automated thermal cycling apparatus. Test samples were held in a fixture attached to a movable arm which was raised and lowered into and out of a resistance type furnace when the samples reached pre-set temperature extremes. The temperature range used was between 50 and 275 °C, the latter comparable to the temperature of a piston when an engine is running. A typical thermal cycle lasted roughly 14 min (Fig. 2). Test samples were given a maximum of 1000 thermal cycles in the present study.

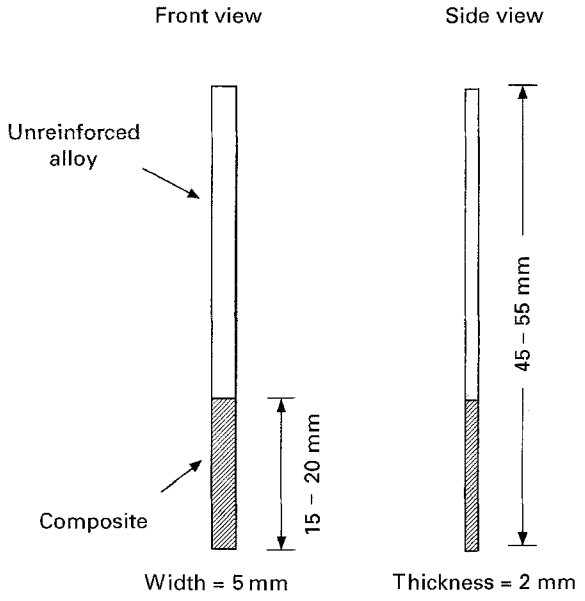

Figure 1 A schematic of a bimaterial tensile sample.

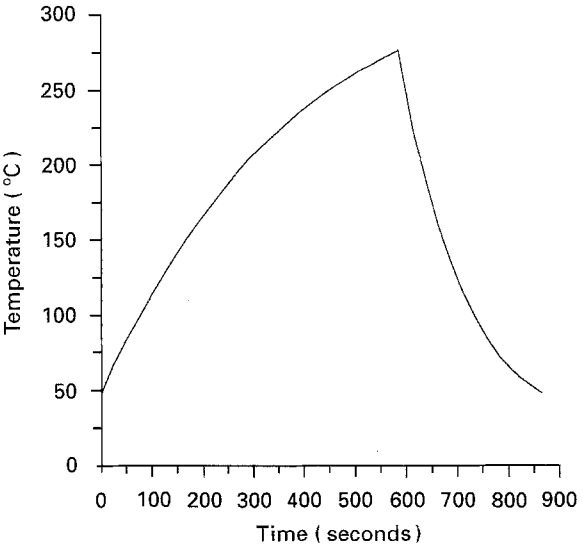

Figure 2 A plot of temperature versus time for the thermal cycle used in the present study.

2.4. Isothermal exposure

Isothermal exposure of test samples was accomplished by holding them at an elevated temperature for a time equivalent to that for 1000 thermal cycles to occur. The purpose of this was to provide a control to distinguish the effects of thermal cycling on the strength of the macro-interface from the possible effects of alloy ageing. The calculation to determine the isothermal exposure temperature was based on the rate of

diffusion of Si in Al. It is the temperature at which Si will diffuse the same distance that it does during thermal cycling in the same amount of time. The isothermal exposure temperature calculated for the thermal cycle shown in Fig. 2 was 237 °C.

3. Results and discussion

3.1. Microstructure evaluation

Particles of primary Si and an undesirable Fe–Mn–Cr intermetallic phase, commonly known as "sludge" [7], were present in both the unreinforced alloy and at the macro-interface in the refined alloy–19% v/o SiC_w composite castings (Fig. 3). The density of these particles was generally greater at the macro-interface than in the alloy, suggesting that they had been filtered by or preferentially nucleated at the preform surface during casting. Small AlP particles were also present at the macro-interface, both within the larger particles and in the aluminium.

In the unrefined alloy–16% and 19% v/o SiC_w composite castings, the unreinforced alloy and macro-interface were relatively free of the blocky Si and intermetallic particles which were found in the refined-alloy castings (Fig. 4). The small dark particles seen at the macro-interface in Fig. 4 are not AlP crystals, although they have a similar size and appearance. They were identified as being small Ca-rich impurities which were inadvertently introduced during the casting process.

The unrefined alloy–18% v/o Saffil composite castings were also free of particles (Fig. 5). However, there were indications of micro-porosity in both the unreinforced alloy and the composite. The general appearance of the pores suggested that they resulted from gas entrapment in the alloy as well as incomplete infiltration of the preform. Also, there appeared to be a lesser amount of eutectic Si present near the macro-interface compared to that in the unreinforced alloy as a whole.

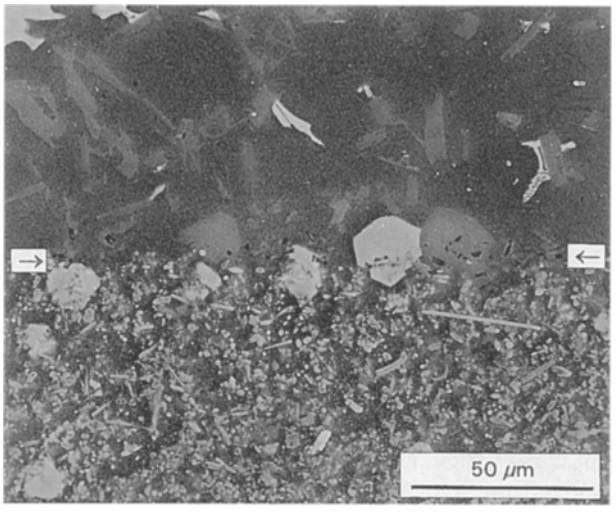

Figure 3 A section of the macro-interface in the refined alloy–19% v/o SiC_w composite castings (indicated by the arrows) showing accumulated Si (dark grey), intermetallic (white) and A1P (small black) particles.

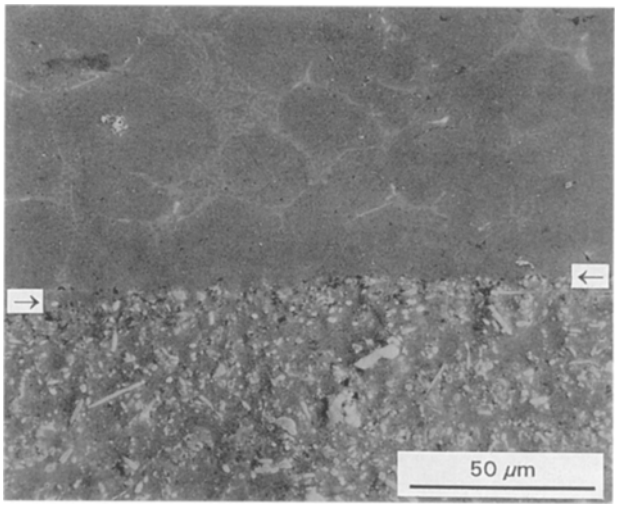

Figure 4 A section of the macro-interface in the unrefined alloy–19% v/o SiC_w composite castings (indicated by the arrows).

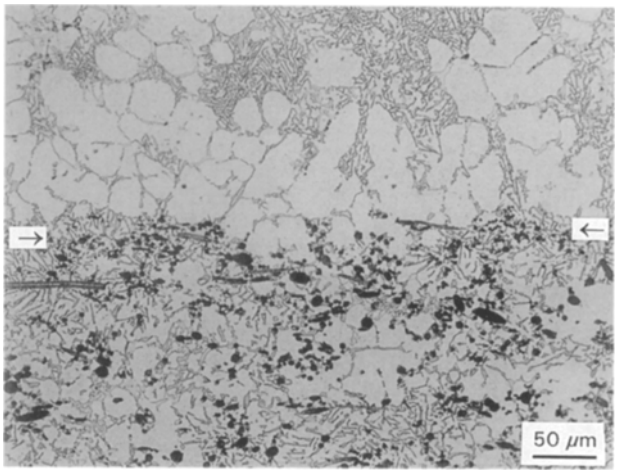

Figure 5 A section of the macro-interface in the unrefined alloy–18% v/o Saffil composite castings (indicated by the arrows).

3.2. The strength of the macro-interface at various temperatures

It was of interest to determine the strength of the macro-interface at the various temperatures that a selectively reinforced piston might experience in use. Bimaterial tensile samples fabricated from the unrefined alloy–19% v/o SiC_w composite castings were used for this experiment. At least three samples were tested at each of the following temperatures: 50, 75, 100, 125, 150, 200 and 250 °C. Fig. 6 shows the strength and break points of each sample tested.

Sample failure typically occurred at the macro-interface at temperatures below approximately 125 °C. Above this temperature, samples typically failed in the unreinforced alloy, away from the macro-interface. The fracture surfaces of the samples which failed at the macro-interface all had the same characteristic appearance (Fig. 7). Whiskers were present on the composite-side fracture surfaces and only their impressions were on the alloy-side fracture surfaces. Subsequent tensile tests of the macro-interface were conducted at room temperature where the strength of the

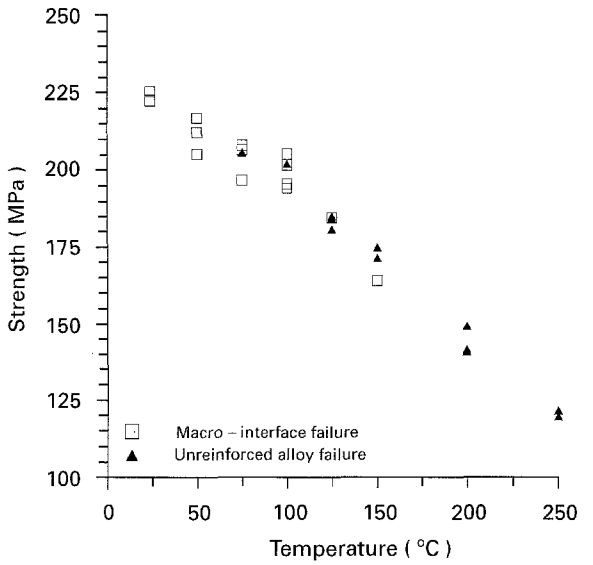

Figure 6 A plot of the measured tensile strengths of unrefined alloy–19% v/o SiC_w composite samples as a function of temperature. The break point of each sample is also indicated. □ Macro-interface failure; ▲ unreinforced alloy failure.

macro-interface was likely to be less than that of the alloy alone.

3.3. The strength of the macro-interface with thermal exposure

To investigate the possibility of fatigue induced damage occurring as a result of thermal cycling, the strength of the macro-interface in each of the three casting groups was measured in the "as fabricated" condition and after 1000 thermal cycles or an equivalent isothermal exposure. It was hypothesized that if fatigue damage occurred, it would be reflected in a greater strength decrease for the thermally cycled samples. The average strength of both the refined [8] and unrefined alloy was also measured in the "as fabricated" condition for comparison and is shown in Table II. A summary of the strength data obtained for each macro-interface is shown in Table III.

3.3.1. The refined alloy–19% v/o SiC_w composite macro-interface

Examination of the thermally cycled samples of this alloy–composite system showed that many of the accumulated primary Si and intermetallic particles at the macro-interface had cracked (Fig. 8). In most cases the cracks appear to have branched from one AlP particle to another, cutting the larger particles into two or more pieces. Particles at the macro-interface in the isothermally exposed samples did not exhibit this type of cracking.

Samples tested in the as fabricated condition all failed at the macro-interface with an average strength of 184 $\pm$ 5 MPa, approximately 15% less than that of the refined alloy alone. Thermally cycled and isothermally exposed samples also failed at the macro-interface with average strengths of 166 $\pm$ 3 MPa and 164 $\pm$ 8 MPa, respectively.

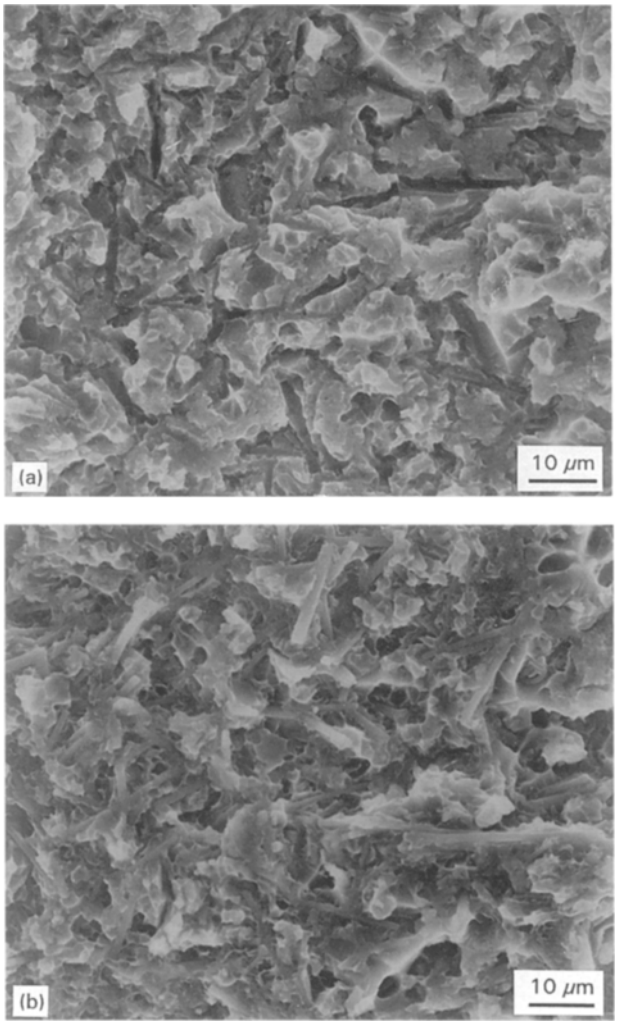

Figure 7 Matching alloy-side (a) and composite side (b) fracture surfaces of an unrefined alloy–19% v/o SiC_w composite sample which failed at the macro-interface.

TABLE II The as fabricated strength of each alloy type

Alloy type	Average tensile strength
Refined	216 MPa [8]
Unrefined	254 $\pm$ 2 MPa

The fracture surfaces of the as fabricated, thermally cycled and isothermally exposed samples were generally indistinguishable from one another. Furthermore, for any given sample, the alloy-side fracture surface was virtually indistinguishable from that of the composite-side fracture surface (Fig. 9). The fracture surfaces were covered with large numbers of cleaved primary Si and intermetallic particles with small areas around them showing ductility where the alloy failed. Whiskers were not found on any of the fracture surfaces.

The presence of brittle particles at the macro-interface is most likely to be responsible for its low initial strength. It is also apparent that the particles were susceptible to cracking during thermal cycling. However, the strength of the macro-interface decreased to comparable levels with both thermal cycling and

TABLE III Summary of the macro-interface strength data

Macro-interface	0 TC	1000 TC	Isothermal exposure equal to 1000 TC
Refined alloy–19% v/o SiCw	184 ± 5 MPa (MI)	166 ± 3 MPa (MI)	164 ± 8 MPa (MI)
Unrefined alloy–16% v/o SiCw	198 ± 10 MPa (MI)	165 ± 4 MPa (MI)	158 ± 1 MPa (MI)
		162 ± 5 MPa (alloy)	160 ± 1 MPa (alloy)
Unrefined alloy–18% v/o Saffil	214 ± 7 MPa (MI)	197 ± 15 MPa (MI)	193 MPa (MI)
		193 ± 10 MPa (MI/C)	203 ± 3 MPa (MI/C)

MI Failure occurred at the macro-interface.
Alloy Failure occurred in the unreinforced alloy away from the macro-interface.
MI/C Failure occurred near the macro-interface in the composite.

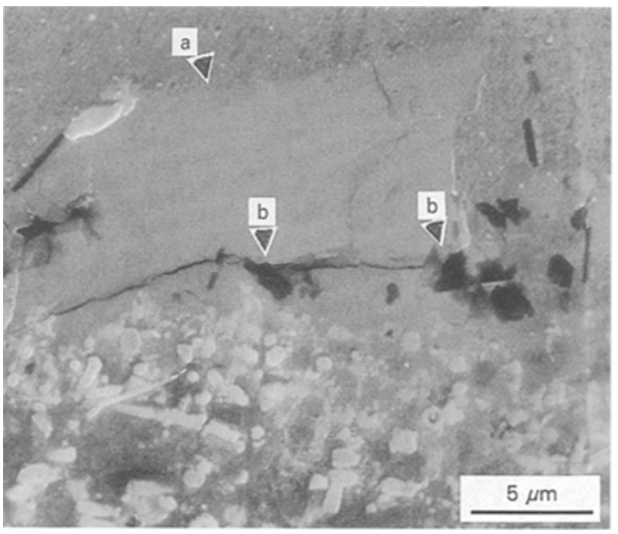

Figure 8 A cracked primary Si particle (a) and smaller A1P particles (b) at the macro-interface in a refined alloy–19% v/o SiC_w composite sample which had been thermally cycled 1000 times.

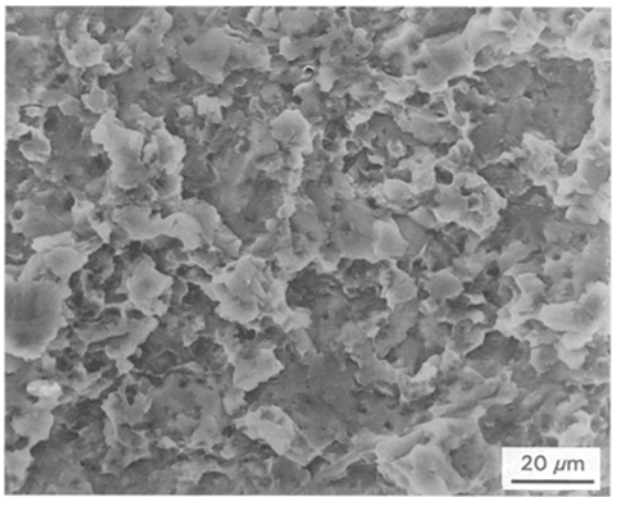

Figure 9 A section of the alloy-side fracture surface of a refined alloy–19% v/o SiC_w composite sample showing cleaved Si and intermetallic particles.

isothermal exposure, even though the isothermally exposed samples did not display particle cracking. This suggests that a metallurgical change such as alloy overageing was responsible for the strength decrease, not particle damage.

3.3.2. The unrefined alloy–16% v/o SiC_w composite macro-interface

Examination of both the isothermally exposed and thermally cycled samples of this alloy–composite system did not reveal any damage caused by thermal exposure.

Samples tested in the as fabricated condition all failed at the macro-interface with an average strength of 198 ± 10 MPa, approximately 22% less than that of the unrefined alloy alone. This is also approximately 12% less than the room temperature strength of the unrefined–19% v/o SiC_w composite macro-interface. Thermally cycled test samples failed at both the macro-interface and in the alloy away from the macro-interface with average strengths of 165 ± 4 MPa and 162 ± 5 MPa respectively. Isothermally exposed samples also failed in both locations with average strengths of 158 ± 1 MPa and 160 ± 1 MPa, respectively.

The fracture surfaces of the samples which failed at the macro-interface had the same general appearance as those of the unrefined alloy–19% v/o SiC_w composite system (Fig. 7). Closer inspection of individual whiskers on the fracture surfaces of both the 16% and 19% v/o SiC_w systems showed portions of an unidentified coating adhering to many of them (Fig. 10). To investigate this further, an untested sample was immersed in a 3% solution of NaOH to dissolve away the alloy so that the bare macro-interface could be observed. It was found that a layer of extraneous material was present at the macro-interface that extended several microns into where the composite previously existed (Fig. 11). It is hypothesized that the layer is actually excess binder material, silica, which is used to hold the whiskers together during the manufacture of the preform [9].

The presence of extraneous material at this macro-interface appears to be one explanation for its low initial strength (compared to the alloy alone). It is not known at this time why the initial strength of the 16% v/o SiC_w system is lower than that of the 19% v/o SiC_w system. The comparable strength decrease for both the thermally cycled and isothermally exposed samples again suggests that alloy overageing may be responsible. This is also supported by the fact that samples failed in the alloy away from the macro-interface only after thermal exposure.

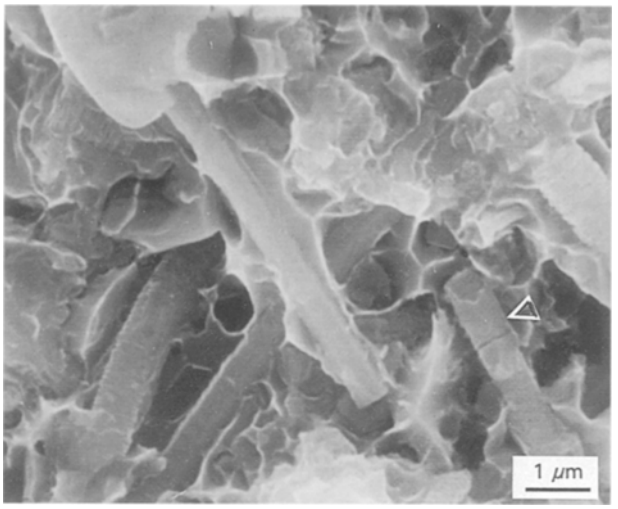

Figure 10 A section of the composite-side fracture surface of an unrefined alloy–16% v/o SiC_w composite sample which failed at the macro-interface. The arrow indicates the fragmented coating observed on many of the whiskers.

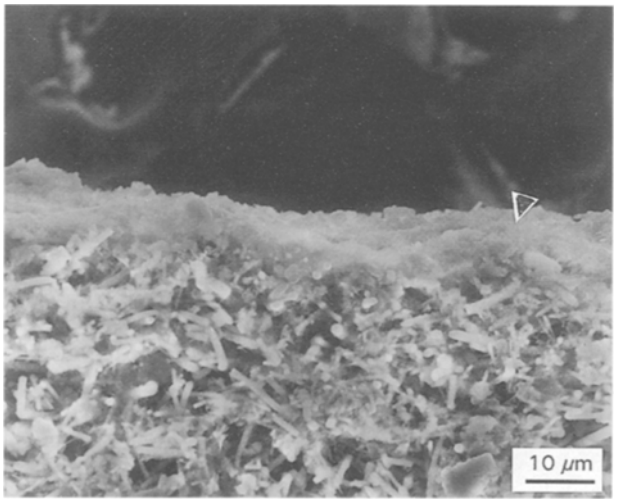

Figure 11 A section of the macro-interface in a heavily etched unrefined alloy–16% v/o SiC_w composite sample. The layer of extraneous material, believed to be silica binder, is indicated by the arrow.

3.3.3. The unrefined alloy–18% v/o Saffil composite macro-interface

As with the previous system, examination of both the isothermally exposed and thermally cycled samples of this alloy–composite system did not reveal any damage caused by thermal exposure.

Samples tested in the as fabricated condition all failed at the macro-interface with an average strength of 214 ± 7 MPa, approximately 16% less than that of the unrefined alloy alone. Thermally cycled test samples failed at both the macro-interface and in the composite near the macro-interface, with average strengths of 197 ± 15 MPa and 193 ± 10 MPa, respectively. Isothermally exposed samples also failed in both locations with average strengths of 193 MPa and 203 ± 3 MPa respectively.

The samples which failed at the macro-interface had fracture surfaces similar in appearance to those observed in the previous alloy–composite system (Fig. 12).

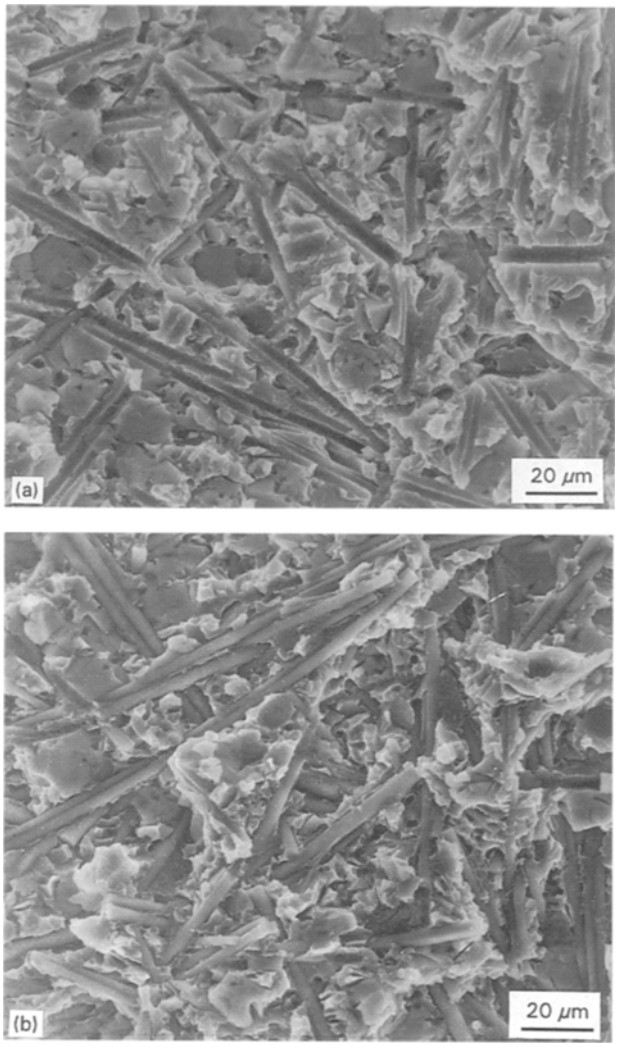

Figure 12 Matching alloy-side (a) and composite-side (b) fracture surfaces of an unrefined alloy–18% v/o Saffil composite sample which failed at the macro-interface.

Fibres were present on the composite-side fracture surfaces and their impressions were on the alloy-side fracture surfaces. Examination of individual fibres did not indicate the presence of a coating. In fact, most of the fibres appeared to be completely bare. This indicates that they were poorly wet by the liquid aluminium during casting. There was also evidence of microporosity on the fracture surfaces of samples which broke at both the macro-interface and in the composite.

It has been already stated that there was a region near the macro-interface which appeared to have a lesser amount of eutectic Si. Since the fibres appeared to be poorly bonded to the aluminium, this may explain why failure occurred at the macro-interface. However, it was not understood why most of the fibres remained on the composite-side fracture surfaces in this alloy–composite system. To investigate this, an untested sample was etched in a similar manner as that previously described to examine the bare macro-interface. Inspection of the etched sample did not reveal a material layer similar to that observed in the unrefined alloy–SiC_w reinforced composite systems. However, examination of the etched composite showed that the fibres were interconnected with a network of Si and intermetallic material (Fig. 13).

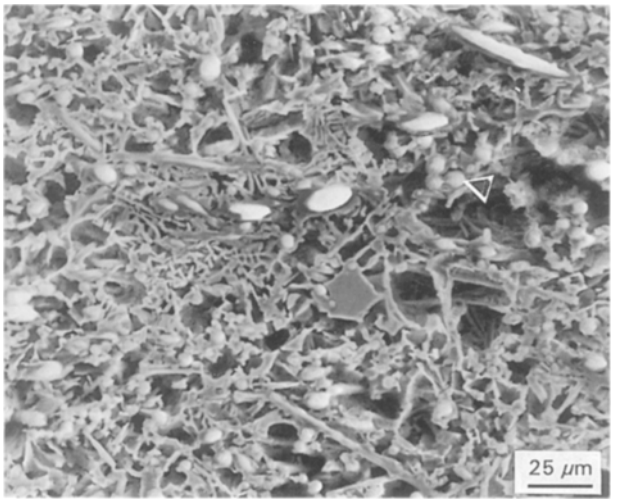

Figure 13 A section of the 18% v/o Saffil composite that was heavily etched to show the intermetallic network which exists around the fibres. The fibres are indicated by the arrow.

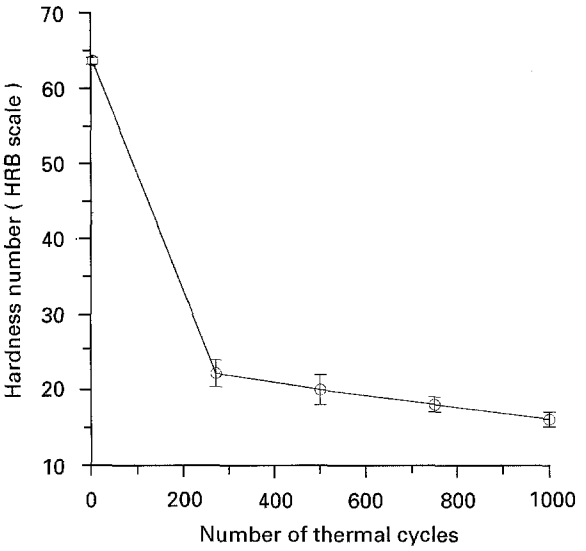

Figure 14 A plot of the hardness of the unrefined alloy as a function of the number of thermal cycles.

A similar phenomenon was observed by Bär, Klußmann and Gudladt [10] in their etching experiments with Saffil reinforced Al–Si composites. It is hypothesized that this intermetallic network was responsible for holding the fibres onto the composite-side fracture surfaces during failure at the macro-interface.

Poor alloy–fibre bonding and the presence of micro-porosity, caused by incomplete infiltration of the preform, appears to be the cause of the low initial strength of this macro-interface and why failure sometimes occurred in the composite. Both of these features indicate that insufficient pressure was used during casting to compensate for these effects. Since the thermally cycled and isothermally exposed samples have similar break points and average strengths, alloy overageing was again suspected to be responsible for the strength decrease with thermal exposure.

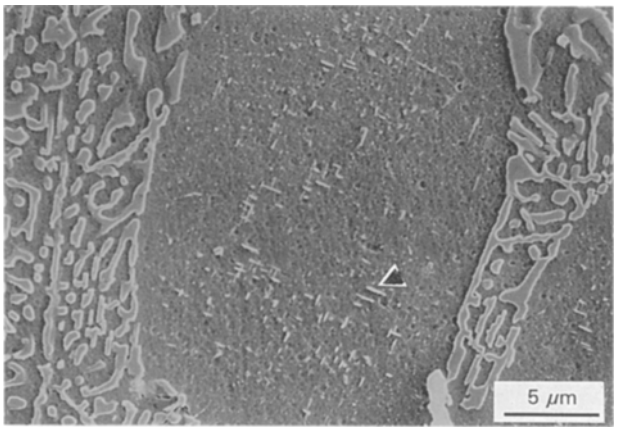

Figure 15 A lightly etched section of an aluminium dendrite in the unrefined alloy showing the precipitate which coarsened during thermal cycling (indicated by the arrow).

3.4. Alloy overageing

The strength decrease with thermal exposure observed for all of the alloy–composite systems was hypothesized to be the result of alloy overageing. To substantiate this, the hardness of the unrefined alloy was measured as a function of the number of thermal cycles (Fig. 14). It was found that there was a decrease in the hardness of the alloy from 63.7 ± 0.5 HRB (hardness Rockwell B) in the 'as fabricated' condition to 22 ± 2 HRB after only 270 thermal cycles. To confirm that the hardness decrease was a result of precipitate coarsening, samples in the 'as fabricated' condition and ones which had been cycled 1000 times were solutionized, heat treated and tested for comparison. The measured hardness of each set was 70.5 ± 0.9 HRB and 64.5 ± 0.7 HRB, respectively. Although there is a slight difference in hardness, the values are comparable.

To determine which precipitate was coarsening, a microstructure comparison was made between the as fabricated and thermally cycled alloy. A sample of each was lightly etched to expose the Si and intermetallic phases for examination (Fig. 15). It was found that small plate-like precipitates had coarsened within the Al dendrites of the thermally cycled sample. Their orientation within the dendrites indicates that they lie on $\{1\,0\,0\}$ type planes since they are either parallel to or perpendicular to the dendrite arms which are known to grow along $\langle 1\,0\,0 \rangle$ type directions [11]. X-ray microanalysis of the precipitate indicated the presence of both Mg and Si. Both of these observations suggest that the precipitate is the β-phase (Mg_2Si) [12].

4. Conclusions

The strength of the unrefined alloy–19% v/o SiC_w macro-interface was found to be less than that of the unreinforced alloy alone at temperatures below approximately 125 °C. It was assumed that this temperature dependence was shared by all of the macro-interfaces examined.

The as fabricated strength of the macro-interface in each of the alloy–composite systems examined was less than that of the unreinforced alloy alone. This appears to be a consequence of a number of factors including poor alloy–reinforcement bonding, an accumulation of brittle particles or other material, and micro-porosity at the macro-interface.

The only evidence of thermal cycling damage was the particle cracking observed at the macro-interface in the samples of the refined alloy system. The effect of this damage was apparently not critical since both the thermally cycled and isothermally exposed samples containing this macro-interface experienced a comparable strength decrease. This was also the case for the other alloy–composite systems. Therefore, there was no clear indication that fatigue damage, resulting from thermal cycling, was responsible for the observed strength decrease. Instead, it was hypothesized that alloy overageing was responsible for the strength decrease.

A layer of extraneous material was found to exist at the macro-interface in the unrefined alloy–16% and 19% v/o SiC_w systems. It was hypothesized that this layer was excess binder material deposited during the manufacture of the preform.

A network of intermetallic material was found to exist in the Saffil reinforced composite. This network is presumably responsible for holding the fibres onto the composite-side fracture surfaces during failure of the macro-interface in the unrefined alloy–18% v/o Saffil composite samples.

Acknowledgements

The authors would like to thank the General Motors Corporation for providing the funding and materials for this research.

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Received 26 July 1994
and accepted 24 May 1995