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Microstructure and Mechanical Properties of Al/Sic$_p$ Composites with Multimodal Size Distribution of Reinforcements

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Abstract. The effect of particle size distribution and particle size ratio of SiC$_p$ in SiC$_p$/SiO$_2$ preforms on the microstructure, microhardness of SiC$_p$ reinforcements, modulus of rupture, and superficial hardness of Al/SiC$_p$ composites produced by pressureless infiltration has been investigated. SiC$_p$/SiO$_2$ preforms in the form of plates (4cm x 3cm x 0.5cm) have been pressureless infiltrated by the alloy Al-15.52 Mg-13.62 Si (wt. %) at 1100 ^oC for 60 min under inert atmosphere. SiC powders with average particle size of 10, 68 and 140 μm are mixed with SiO$_2$ powders and preforms of 40 % porosity with unimodal, bimodal and trimodal size distributions are prepared by uniaxial compaction. The bimodal (small: large) and trimodal (small: medium: large) preforms are prepared with different particle size ratios in the following levels: 1:1, 3:1, 1:3, 2:2:2, 3:2:1, 3:1:2. Results from characterization by XRD, SEM and energy dispersive X-ray spectrometry show that the typical microstructure of the composites contains the MgAl$_2$O$_4$ (spinel), AlN and MgO phases formed during processing as well as partially reacted silica, SiC, Si and Al. It is found that the density, reinforcement microhardness, modulus of rupture and superficial hardness of the composites increase all with wider particle size distribution. However, whilst the modulus of rupture is mainly affected on going from unimodal and bimodal to trimodal distribution, superficial hardness and microhardness are mostly influenced on going from unimodal to bimodal and trimodal distribution.

Introduction

Aluminum matrix composites with a high volume fraction of a ceramic reinforcement have been the subject of intense investigations in the last years due to improved strength, stiffness, thermal conductivity, abrasion resistance and dimensional stability. The Al/SiC system has attracted the attention of many researchers particularly for those applications demanding a low coefficient of thermal expansion (CTE) and a high thermal conductivity [1-3]. It is well known that in order to achieve large volume fractions of the ceramic in a metal/ceramic composite, it is necessary to use reinforcements of substantially different sizes [3-6]. From the available processing techniques for the production of metal matrix composites, the infiltration of ceramic preforms by liquid metals is the most convenient route for the manufacture of composites with a high volume fraction of the reinforcements. By the infiltration route, it is possible to produce near-net shape composites with high dimensional stability and a uniform distribution of the reinforcements.

Most of the work done on the fabrication of composites with a high volume fraction of reinforcements has been connected to the pressure- or vacuum-assisted infiltration techniques [3-6]. However, in order to abate processing costs, the development of alternative non-assisted or pressureless infiltration routes is of paramount importance. The feasibility of

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producing Al/SiC composites with thermal conductivities similar to those exhibited by aluminum alloys has been reported by Arpón et al. [3] from investigations with pressure-assisted infiltration, and Cui [7] reported the fabrication of Al/SiC composites with a SiC volume fraction of 0.68 by the pressureless infiltration technique. However, an inconvenience of the method is that long processing times are used for the preparation of the preforms (5 hr) and the composites (1-3 hr) [7].

In order to take advantage of the thermal properties (CTE and thermal conductivity) of Al/SiC composites with high volume fractions of the reinforcements, it is also of vital importance to fully characterize the mechanical properties of the composites. The aim of this work was to investigate the effect of particle size distribution and particle size ratio of SiC_p in SiC_p/SiO_2 preforms on the microstructure, microhardness of SiC_p reinforcements, modulus of rupture, and superficial hardness of Al/SiC_p composites produced by pressureless infiltration.

Experimental Procedure

Preforms with a SiC_p volume fraction of 0.60 were prepared with silicon carbide powders of three different particle sizes [10 (small), 68 (medium) and 140 (large) μm], three different particle size distributions (unimodal, bimodal and trimodal) and different particle size ratios for bimodal (small: large) and trimodal (small: medium: large) distributions: 1:1, 3:1, 1:3, 2:2:2, 3:2:1, 3:1:2. The designation for particle size ratio involves both the type of preform (bimodal or trimodal) and the proportion of each particle size. For instance, 3:1:2 stands for a trimodal preform made of three parts of small, one part of medium and two parts of large SiC particles. Unimodal preforms are designated with only the corresponding particle size. The SiC powders were mixed with 10 wt.% of SiO_2 powders (particle size, 346 μm) and 8 wt.% of dextrin as a binder. The mixtures were placed into a steel mold and uniaxially compacted using a pressure of about 3.5 MPa to produce preforms with the geometry of plates of 3 x 4 x 0.5 cm. With the aim of partially eliminating the dextrin, the preforms were heated in an air furnace for two hours at 125 ºC and then for two more hours at 225 ºC. An Al-Mg-Si alloy was fabricated in an induction furnace with commercial Al, Mg and Si materials. Table 1 shows the chemical composition of the fabricated alloy.

Table 1 Chemical composition of the aluminium alloy [wt.%].

Al	Mg	Si	Fe	Mn	Zn	Others
Balance	15.5	13.6	0.99	0.15	0.13	2.61

Infiltration trials were performed in a tube furnace with control of the process atmosphere. The preform and alloy were placed in a ceramic container and the whole assembly was positioned in the furnace. The system was heated at a rate of 15 ºC/min up to 1100 º C, held at this temperature for 60 min and then cooled down at the same rate to room temperature. In order to enhance the wetting of the preform by the liquid alloy during heat up, a change in the processing atmosphere (Ar $\rightarrow N_2$) was made on reaching 1000 ºC. Once the system was cooled down to room temperature, the composites were prepared for physical, mechanical and microstructural characterization. The density was evaluated using Archimedes´ principle. Specimens were characterized by X-ray diffraction (XRD), optical microscopy (OM), scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDX). Mechanical characterization was conducted using four-point bending tests (ASTM C1161-94 standard) and Rockwell superficial hardness. Vickers microhardness tests were performed on SiC particles of 68 (medium) and 140 (large) μm incorporated into the composites.

Results and Discussion

Phase Analysis and Microstructure. Results from XRD reveal the presence of the $MgAl_2O_4$, AlN, MgO and Mg_2Si phases formed during processing, in addition to Al, Si and SiC. These phases were formed regardless of the particle size distribution and particle size ratio. A typical XRD pattern from a composite specimen with bimodal size distribution is shown in Fig. 1. It is noteworthy that the unwanted Al_4C_3 phase is not detected in the composites microstructure.

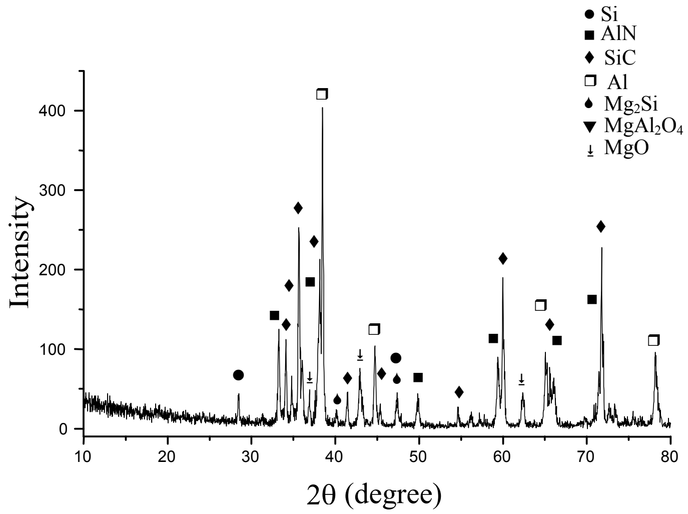


Figure 1. XRD pattern of a composite with bimodal size distribution (1:1) (SiC, 10 and 140 μm).

The magnesium aluminate (spinel) is formed by the reaction of the silica added to the preforms with magnesium and aluminum in the alloy, according to:

$$2\ Al_{(l)} + 2\ SiO_{2(s)} + Mg_{(l)} = MgAl_2O_{4(s)} + 2\ Si_{(s)} \quad (1)$$

Analysis by SEM shows that, in a typical microstructure of the composites, the spinel is present at the periphery of partially reacted SiO_2 particles. Figure 2 shows the typical microstructure of the composites and Figure 3a is a photomicrograph showing a partially reacted particle of SiO_2. Figure 3b is an EDX spectrum on the region corresponding to $MgAl_2O_4$.

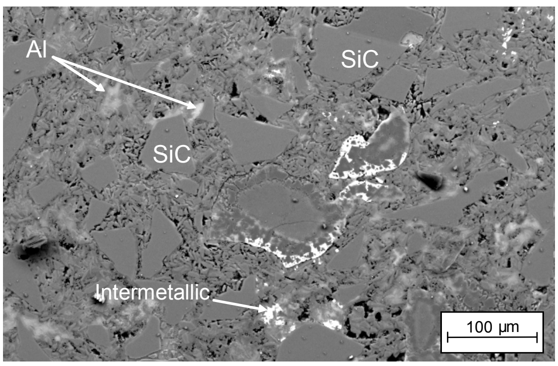
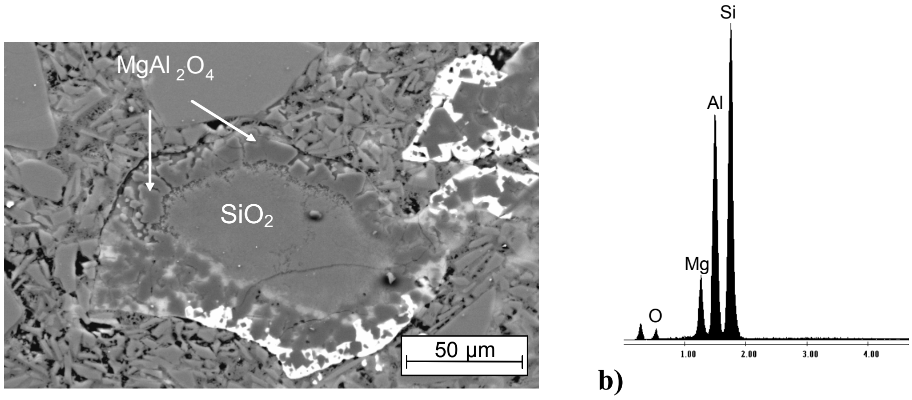


Figure 2. SEM photomicrograph showing the typical microstructure of the composites.

Effect of particle size distribution on density. Figure 4 shows the effect of particle size distribution on the composites density. In this and the subsequent figures, the numbers 1, 2 and 3 in the *X*-axis refer to unimodal, bimodal and trimodal distributions, respectively. The measured densities (in the range from 2.77 to 2.91 g/cm^3) are similar to those reported by Chen et al. for composites prepared by the pressure-assisted infiltration technique [6]. The



a) **b)**

Figure 3. (a) SEM photomicrograph showing a partially reacted SiO_2 particle, and (b) EDX spectrum in a zone corresponding to $MgAl_2O_4$.

change in the density of the composites with different particle size distributions is explained in terms of the degree of packing attained when using particles with different sizes in the preforms. In regard to the influence of the particle size ratio in a given preform on the density of the composites, no significant effect is observed.

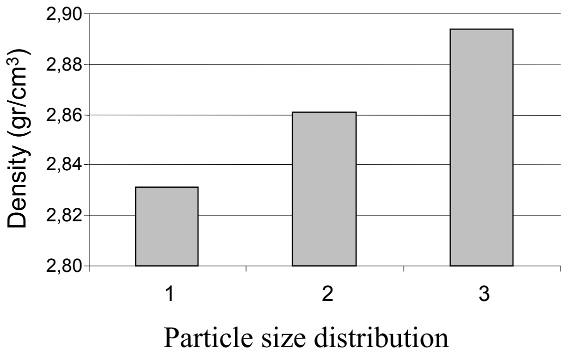


Figure 4. Average densities of the composites as a function of particle size distribution.

Modulus of rupture (MOR). Results from four-point bending tests reveal that the MOR does not significantly increase when the size distribution varies from unimodal to bimodal. However, an increase of about 16 % is observed when a change is made to trimodal size distribution, as shown in Fig. 5. The average values of the moduli of rupture of the composites are 117 ± 8, 119 ± 17 and 145 ± 13 for unimodal, bimodal and trimodal size distributions, respectively. Typical values of MOR for some specimens are shown in Table 2.

Table 2. Modulus of rupture of composites with different size distribution of reinforcements.

Particle size ratio and distribution	MOR [MPa]
Unimodal (68 µm)	117 ± 8
1:1	136 ±17
1:3	102 ±18
3:1:2	129 ±12
3:2:1	113 ± 14
2:2:2	194 ± 10

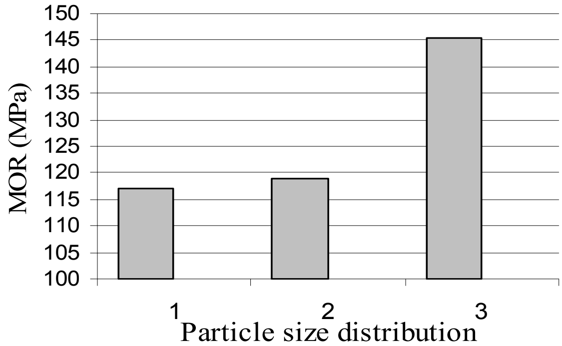


Figure 5. Modulus of rupture as a function of particle size distribution.

Fracture surface analysis revealed that, irrespective of the size distribution, there existed good bonding between the aluminum matrix and the SiC reinforcements. Figure 6 exemplifies the typical fracture surface in the composites, showing the aluminum matrix and a SiC particle.

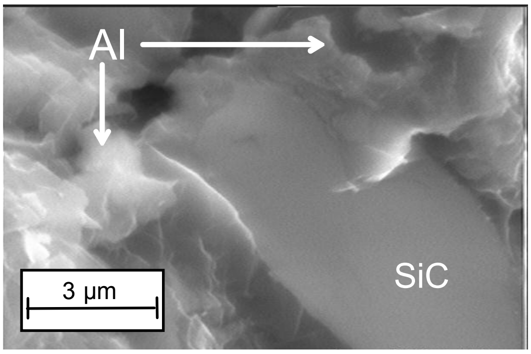


Figure 6. SEM photomicrograph showing the aluminum matrix and a SiC particle on the fracture surface of a 2:2:2 specimen.

Superficial hardness. Rockwell hardness tests revealed that the superficial hardness of the composites increases in the order unimodal→ bimodal → trimodal size distribution, the average values being 39 ± 3, 63 ± 3 and 66 ± 1 RH30N, respectively. As shown in Fig. 7, although the change in hardness between the bimodal and the trimodal distributions is not significant, a considerable increase is observed on going from unimodal to bimodal or trimodal distributions. This behavior can be explained in terms of the plastic deformation of the matrix. It is considered that during indentation the matrix will undergo plastic deformation while the SiC particles start interfering with one another up to the point where impingement limits the matrix plastic flow. It is also assumed that after the SiC particle impingement, the situation will be governed by the hardness of SiC. In this context, once the matrix has totally deformed, the hardness will remain nearly constant.

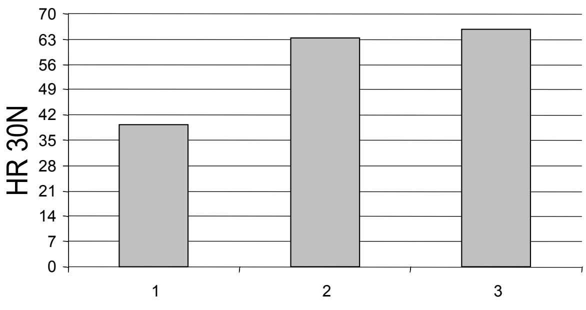


Figure 7. Superficial hardness as a function of particle size distribution.

Vickers microhardness in SiC_p. Results from the Vickers microhardness tests in medium and large SiC particles reveal that the microhardness increases with increasing particle size, being the effect more evident on going from unimodal to bimodal and trimodal distributions (see Fig. 8). However, an explanation for this behavior can not be given from our current results. The average hardness of SiC in composites with unimodal, bimodal and trimodal size distributions of reinforcements is 2339 ± 223, 3360 ± 320 and 3464 ± 368 kg/mm^2, respectively. Figure 9 is a photomicrograph showing typical indentations on the SiC particles.

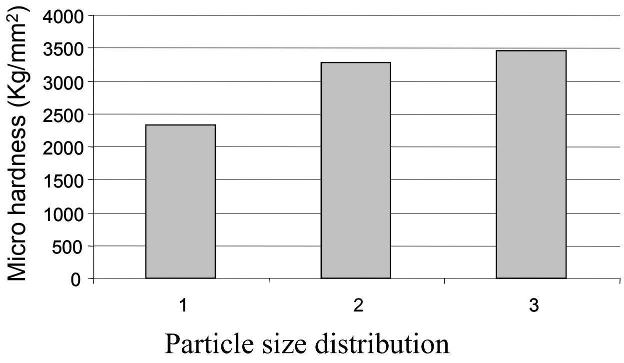


Figure 8. Microhardness of SiC_p as a function of particle size distribution.

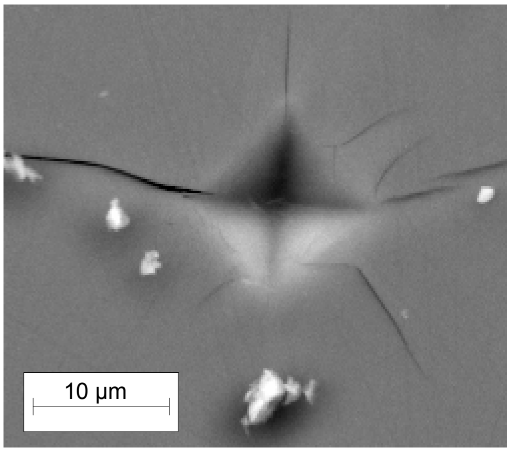


Figure 9. Indentation in a SiC particle of 68 μm.

Summary and Conclusions

Al/SiC_p composites with multimodal size distributions of reinforcements have been produced by the pressureless infiltration route. The microstructure of the composites is characterized by the presence of the phases $MgAl_2O_4$, MgO, AlN and Mg_2Si, which were formed during processing, in addition to Al, SiC, SiO_2 and Si. It was found that the density, SiC_p microhardness, superficial hardness and modulus of rupture of the composites increase all with wider particle size distributions. The effect of particle size distribution on the superficial hardness is explained in terms of the plastic deformation experienced by the metallic matrix and the blockage between particles during indentation. However, more detailed studies would be required to elucidate the effect of size distribution on the microhardness of SiC_p.

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