# OPTIMUM PARAMETERS FOR MINIMUM RESIDUAL POROSITY IN Al/SiC<sub>P</sub> COMPOSITES

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## ABSTRACT

The effect of the following processing parameters on the residual porosity of Al/SiC<sub>p</sub> composites was investigated: SiC particle size, SiC type and Mg content in the aluminum alloy. The contribution of each of the aforementioned parameters and their interactions was determined using analysis of variance and the effect of the levels of each parameter was investigated using surface response analysis. Results from surface response analysis are in excellent agreement with those obtained with ANOVA. Anova results show that particle size is the parameter that most significantly impacts the residual porosity, followed by the interaction between SiC type and SiC particle size. Surface response analysis indicates that for both types of SiC, residual porosity diminishes with decreasing particle size. Using 20 µm powders, none of the two types of SiC significantly affects residual porosity; nonetheless, a composite with C SiC exhibits the minimum residual porosity. Using 75 µm powders, variation in type of SiC, significantly impacts residual porosity. Regardless of the Mg level in the alloy, the lower the particle size, the lower the porosity. At a given particle size, variation in Mg content does not significantly affect residual porosity; however, an alloy 3 wt.% Mg produces the highest porosity. Accordingly, the optimum parameters for minimum residual porosity are: 20um SiC type C, with an alloy 6 wt.% Mg.

## **1. INTRODUCTION**

Two of the major problems frequently encountered in the processing of  $Al/SiC_p$  composites by the pressureless infiltration method are the presence of considerable levels of residual porosity and the development of unwanted reaction products ( $Al_4C_3$ ) [1-3]. Residual porosity is related to an inadequate wetting of silicon carbide by molten aluminumand and unwanted phases are developed from the dissolution of the SiC reinforcement by the liquid aluminum. At temperatures above 1000 K, SiC dissociates into  $Al_4C_3$  and metallic silicon rejected into the matrix [3]. Both problems are related to the processing parameters such as alloy chemistry, temperature, atmosphere, preform porosity, particle size, etc [4] . In this work, the effect of the following processing parameters on the residual porosity of  $Al/SiC_p$  composites was investigated: SiC particle size, SiC type and Mg content in the aluminum alloy.

## 2. EXPERIMENTAL

## Design of experiment

The effect of the following processing parameters on the residual porosity of  $Al/SiC_p$  composites was investigated: SiC particle size, SiC type, and Mg content in the aluminum alloy. These parameters were investigated at two levels: 20 and 75  $\mu$ m, C and GC, and 3 and 6 wt.% Mg, respectively. The difference between C and GC SiC is the impurity level, being GC the type of SiC with the least amount of impurities. Typical impurites in SiC powders are SiO<sub>2</sub>, C, Fe and free Si. A full factorial experiment design of the kind 2<sup>3</sup> was used. Factorial designs allow determining the effect of a given factor in various levels on one or more response variables [5]. The contribution of each of the aforementioned parameters and their interactions on the variability of the residual porosity was investigated using analysis of variance (ANOVA) and the effect of each of the levels was determined using surface response analysis. In Table 1, a standar 2<sup>3</sup> factorial design showing the established parameters and levels is shown.

Trial	Mg (wt. %)	SiC <sub>p</sub> type	Particle size (µm)
1	3	С	20
2	6	С	20
3	3	GC	20
4	6	GC	20
5	3	С	75
6	6	С	75
7	3	GC	75
8	6	GC	75

Table 1 Standar  $2^3$  factorial design showing the parameters investigated.

Materials and procedures

Silicon carbide preforms 40 and 60 % porosity were prepared with 75 and 20  $\mu$ m particle size powders, respectively [6]. Both kinds of preforms were prepared by mixing thoroughly a predetermined amount of the SiC powders with 5 wt. % dextrin and distilled water. The mix was then compacted in a steel die to produce 3 cm x 4 cm x 0.5 cm slabs. The preforms were then dried at 120 °C in a forced air drier for two hours, then cured at 225 °C for two more hours. A preform was placed on top of a plate of the aluminum alloy (about 40 g) into a ceramic container that was previously coated with boron nitride. The chemical composition of the alloys are shown in Table 2.

Table 2Chemical composition (wt. %) of the alloys used in the experiment.

Alloy	Si	Mg	Total other elements	Al
1	10.23	2.98	0.10	86.68
2	9.82	6.02	0.11	84.04

Infiltration trials were performed in a horizontal tube furnace with a 6.5 cm diameter alumina tube provided with end-cap fittings to controll the process atmosphere. The preforms were heated in ultra high purity argon at a rate of 15 °C/min up to 1150 °C. At this temperature, in order to enhance the wetting of the SiC particles by the liquid aluminum, the atmosphere was switched to ultra high purity nitrogen and the systen was held isothermally for 60 min [4].

After cooling to room tempertaure in nitrogen atmosphere, the infiltrated slabs were removed from the furnace for measuring the density. Specimens were sectioned and polished using standard metallurgycal procedures and microstructural characterization was performed using optical microscopy, scanning electron microscopy (SEM), energy disspersive X-rays (EDX) and X-ray diffraction (XRD). The density of the composites was measured using the Archimedes' principle and the percentage residual porosity was calculated using the following formula:

$$\%P = \left(1 - \frac{\rho_{Composite}}{\rho_{Theoretical}}\right) \times 100$$

Where  $\rho_{Composite}$  is the measured density of the composite and  $\rho_{Theoretical}$  is the theoretical density of the composite calculated using the law of mixtures.

## 2 RESULTS AND DISCUSSION

In Figure 1, the XRD pattern of the replica specimen of trial 2 reveals that in addition to SiC, Al and Si, Al<sub>4</sub>C<sub>3</sub> and AlN phases are also present. This specimen was selected because it exhibited the lowest residual porosity (2.2 %). It should be noted that the area of the composite analyzed by XRD, corresponds to the face that was in direct contact with the aluminum alloy. Since this side of the preform is the region in contact with the liquid aluminum the longest time period, it is the area more susceptible to the development of Al<sub>4</sub>C<sub>3</sub> by the partial dissolution of the SiC particles [7]. On the other hand, the presence of AlN is attributed to a series of chemical reactions between the Mg in the alloy and the nitrogen in the atmosphere, where formation of Mg<sub>3</sub>N<sub>2</sub> is a prerequisite for the formation of AlN [8]. In Figure 2 a photmicrograph showing the typical microstructure in the replica specimen of trial 2 is shown.

## ANOVA and surface response analysis

Results from analysis of variance are shown in Table 3. According to Table 3, the parameter that most significantly affects residual porosity in the composites is particle size, followed by the interaction between SiC type and particle size. In Figure 3, a surface response plot for the interaction between SiC particle size and type is shown. SiC types C and GC are represented by (-1) and (+1) on the type axis, respectively.



Figure 1 XRD pattern corresponding to composite obtained in replica of trial 2



Figure 2 Scanning electron photomicrograph of the composite replica of trial 2.

According to Figure 3, minimum residual porosity can be obtained by using 20  $\mu$ m particle size and SiC type C. In Figure 4 a surface response plot of the interaction between SiC particle size and Mg content in the aluminum alloy is shown. In accordance with this figure, the lowest residual porosity can be attained by using 20  $\mu$ m particle size and an alloy with 6 wt. % Mg. Nevertheless, using an alloy with 3 wt. % Mg does not significantly changes the magnitude of the residual porosity.

	0		0	
Source	Sum squares	D.F	Mean square	Р
(A) Mg content in Al alloy	27.7223	1	27.7223	0.011551 *
(B) SiC type	66.4339	1	66.4339	0.000996 *
(C) Particle size	511.2664	1	511.2664	0.000001 *
AB	18.1203	1	18.1203	0.029966 *
AC	1.0256	1	1.0256	0.548280
BC	137.1428	1	137.1428	0.000088 *
ABC	31.3119	1	31.3119	0.008528 *
Error	20.8857	8	2.6107	
Total	813 9089	15		

 Table 3
 Anova table for percentage residual porosity (\* 1 % significance)



Figure 3 Surface response plot for the interaction between SiC particle size and type.



Figure 4 Surface response plot for the interaction between SiC particle size and Mg content.

## CONCLUSIONS

Results from surface response analysis are in excellent agreement with those obtained with anova. Anova results show that particle size is the parameter that most significantly impacts the residual porosity, followed by the interaction between SiC type and SiC particle size. Surface response analysis indicates that for both types of SiC, residual porosity diminishes with decreasing particle size. Using 20  $\mu$ m powders, none of the two types of SiC significantly affects residual porosity; nonetheless, a composite with C SiC exhibits the minimum residual porosity. Using 75  $\mu$ m powders, variation in type of SiC, significantly impacts residual porosity. Regardless of the SiC type, variation in Mg content does not significantly affect the residual porosity. At a given particle size, variation in Mg content does the highest porosity. Accordingly, the optimum parameters for minimum residual porosity are: SiC type C, 20 $\mu$ m particle size and an alloy 6 % Mg.

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