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Effect of Mg and SiC type on the processing of two-layer Al/SiC$_p$ composites by pressureless infiltration

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A great number of engineering applications demand variation of properties with position within the component. Functionally graded materials (FGM) are a class of materials designed with a gradual transition in composition and/or microstructure to reduce the effects of stress concentrations that may be caused by abrupt transitions in composition and properties [1].

Fabrication routes of functionally graded materials have been classified into two groups: constructive and transport based processes. Infiltration of a porous preform of the more refractory phase featuring a gradient in porosity by the liquid of the less refractory phase can be used to produce a FGM by a liquid/solid processing route. Moreover, the infiltration process can be accompanied by heat or mass transport to produce a functionally graded material [2]. Thus, functionally graded near-net shape metal matrix composites (MMC's) may be produced by pressure, vacuum assisted infiltration or by pressureless infiltration of ceramic preforms having a variation in the ceramic volume fraction and/or reinforcement characteristics.

The development of "pressureless infiltration" or "spontaneous infiltration" methods is highly desirable in order to abate processing costs. It is worth recalling however, that the term "spontaneous infiltration" does not refer to the kinetics of the process or even signify that infiltration occurs "instantaneously". Rather it means that process conditions are such that self-promoted infiltration is thermodynamically favorable [3].

The production of bilayer-graded Al/SiC$_p$ composites by a one-step pressureless infiltration process has been reported in a recent publication [4]. The effect of the following processing parameters on the production of bilayer-graded SiC$_p$ composites was investigated and quantified: temperature, infiltration time, SiC particle size, percentage porosity in the preform and preform layer height. It was found that the parameter that most significantly affects the height of infiltration of bilayer preforms is the interaction between porosity and preform layer height. This is followed by the infiltration temperature and the interaction between particle size and percentage porosity.

In spite of the efforts devoted to the optimization of processing parameters *via* pressureless infiltration, a number of difficulties still remain unresolved. With regard to the Al/SiC system, typical problems frequently reported are the presence of unwanted phases (Al_4C_3) and considerable levels of residual porosity in the composites. Unwanted phases are developed from the dissolution of the SiC reinforcement by the liquid aluminum and residual porosity is related to an inadequate or lack of wetting of silicon carbide by molten aluminum [5–8]. Both problems can be overcome by adequately controlling the processing parameters such as alloy chemistry, processing time and temperature, atmosphere, preform porosity, particle size, etc.

With regard to alloy chemistry, it has been reported in the literature that together with nitrogen in the atmosphere, magnesium in the system is one of the key ingredients in the non-assisted infiltration of ceramic compacts and powder beds by aluminum alloys [9, 10]. Its chemical interaction with nitrogen in the atmosphere leads to the formation of Mg_3N_2, a phase that plays an essential role in enhancing the wetting of the ceramic by liquid aluminum. In addition, depending on alloy composition, the level of Mg in the system has been associated with the formation of new phases (MgO or $MgAl_2O_4$) in the composites [11–13]. For instance, for the Al-Mg-Si system (Al-1% Mg, Al-5% Mg, A-356 and 6061 alloy), with artificially oxidized SiC particles, it is reported that $MgAl_2O_4$ crystals are mainly produced and that relatively high Mg contents (above 5%) favor formation of MgO in addition to $MgAl_2O_4$ [14]. It has also been reported that during the processing of aluminum-based MMCs, $MgAl_2O_4$ spinel is more likely to be formed at low Mg contents and at high temperatures, while the formation of the magnesium oxide (MgO) is enhanced at high Mg contents and low temperatures [15].

Within the variety of engineering approaches that have been utilized to prevent or retard the development of unwanted phases, the use of silicon either as an alloying element or as a coating on the SiC reinforcements has been successful [3, 16, 17]. Another important approach frequently reported in the recent literature is the presence of a layer silicon dioxide (SiO_2) on the SiC reinforcements procured via pre-oxidation treatments [18, 19]. Commercially, SiC powders are produced by the Acheson process in which two types of SiC are obtained, namely green and black SiC. While the high purity green SiC (GC-SiC) is produced at the core of the

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TABLE I Full factorial design 2^2 showing the parameters investigated

Trial	Mg (wt%)	SiC_p type
1	3	C
2	6	C
3	3	GC
4	6	GC
5	3	C
6	6	C
7	3	GC
8	6	GC

electrode, the black SiC (C-SiC) is formed at the outer layer. In the as-received condition, the commercially termed black SiC usually contains unreacted SiO_2 and C as impurities [20]. Moreover, C-SiC type costs less as compared with GC-SiC.

The aim of this investigation was to gain further insight into the processing of functionally graded Al/SiC_p composites by pressureless infiltration. In this work, the effect of the type of SiC (GC-SiC and C-SiC) and Mg content in the alloy, on the degree of infiltration of bilayer SiC_p performs by aluminum alloys was investigated and quantified.

A full factorial experiment design of the kind 2^2 was used to investigate the effect of the parameters Mg content and SiC type and the interaction between both parameters on the degree of infiltration of bilayer SiC_p preforms. Factorial designs allow determination of the effect of a given factor in various levels on one or more response variable [21]. The contribution of each of the aforementioned parameters and their interactions on the variability of the degree of infiltration was investigated using analysis of variance (ANOVA) and the effect of each of the levels was determined using surface response analysis. In Table I, a standard 2^2 factorial design showing the established parameters and levels is shown.

Two-layer composites were fabricated starting from bilayer SiC_p preforms having 40% and 60% porosity on the top and at the bottom, respectively. In the bilayer preforms, particle size was varied from 75 to 20 μm from top to bottom. A schematic of the bilayer preform is shown in Fig. 1. Preforms were prepared by mixing thoroughly a predetermined amount of the SiC powders with 5% dextrin and distilled water. The mix was then compacted in a steel die to produce 3 cm $\times$ 4 cm $\times$ 0.5 cm slabs. The preforms were dried at 120 °C in a forced air drier for two hours, and then cured at 225 °C for two more hours. A preform was placed on top of a

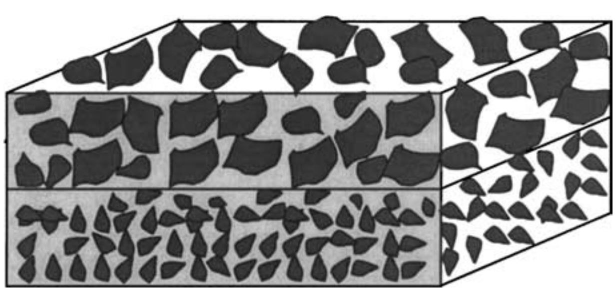

Figure 1 Schematic representation of a bilayer SiC_p preform.

TABLE II Chemical compositions (wt%) of the alloys used in the experiment

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

TABLE III Anova table for degree of infiltration

Source	Sum squares	D.F.	Mean square	F_o	P
(A) Mg content in Al Alloy	1120.889	1	1120.889	730.0407	0.000011*
(B) SiC type	677.455	1	677.455	441.2296	0.000030*
(AB)	334.965	1	334.965	218.1643	0.000122*
Error	6.142	4	1.535		
Total	2139.450	7			

*1% significance.

plate of the aluminum alloy (about 60 g) in a ceramic container that was previously coated with boron nitride. The chemical compositions of the alloys are shown in Table II.

Infiltration trials were performed in a horizontal tube furnace with a 6.5 cm diameter alumina tube provided with end-cap fittings to control 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 system was held isothermally for 60 min. After cooling to room temperature, composites were removed from the furnace and prepared for density evaluation using the Archimedes' principle and degree of infiltration determination. The degree of infiltration (%) in the preforms was determined knowing the value of the theoretical weight of the composite, assuming full infiltration, and the actual weight of the composite. In addition, calculation of the actual weight of the composite was based on the measured density of the alloy used for a given sample, in accordance with Table I.

Results from analysis of variance are shown in Table III. According to Table III, the degree of infiltration of the bilayer preforms is significantly affected within 1% significance by the three parameters investigated, namely, the Mg content, the type of SiC and the interaction between them.

To determine the effect of the levels of each factor on the degree of infiltration of SiC_p preforms, surface response plots were constructed. In Fig. 2 a surface response plot for the interaction between SiC type and Mg content in the aluminum alloy is shown. According to Fig. 2, the highest degree of infiltration (close to 100%) can be obtained by using an aluminum alloy 6%. Mg with GC-SiC. However, the difference between using GC-SiC and C-SiC is negligible. On the other hand, when the alloy used contains 3% Mg, the difference between using GC-SiC and C-SiC is considerable.

Fig. 3 shows a two-dimensional surface response plot for degree of infiltration. Fig. 3 suggests that

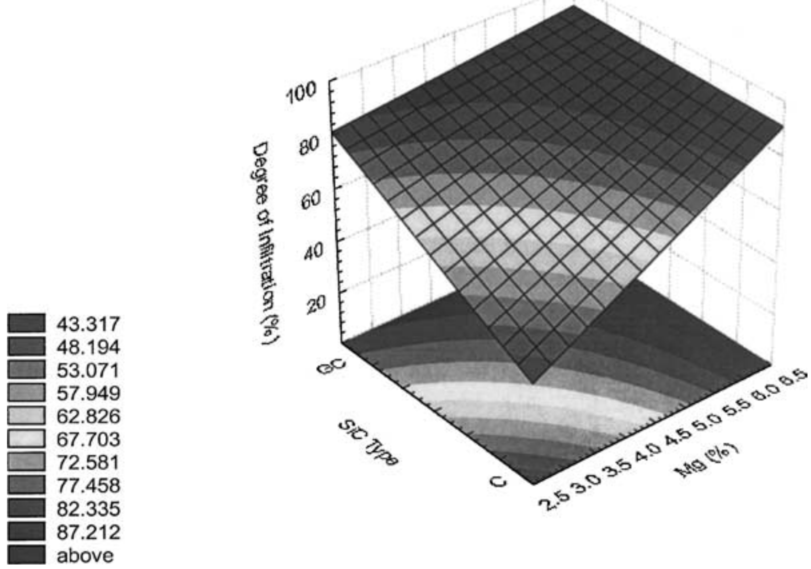

Figure 2 Surface response plot for the interaction between SiC type and Mg content in the aluminum alloy.

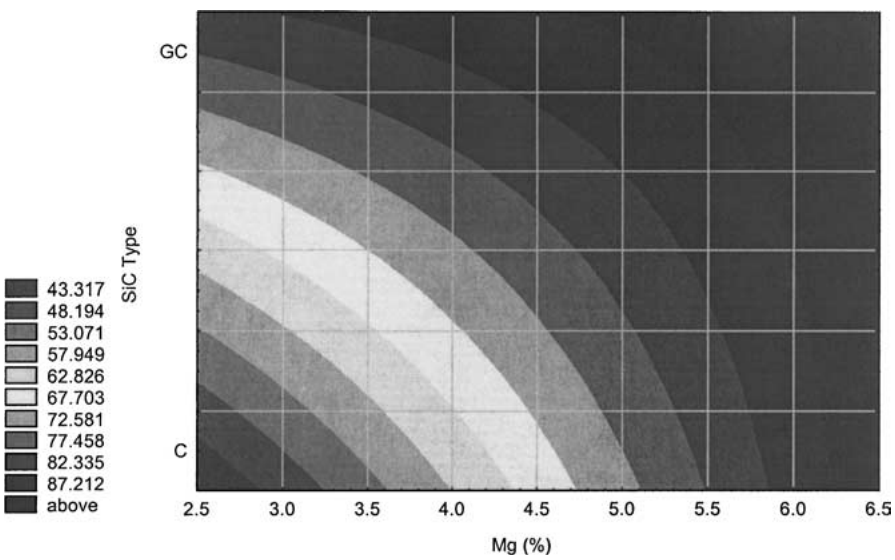

Figure 3 Bidimensional projection of Fig. 2 for the interaction between SiC type and Mg content in the aluminum alloy.

the best degree of infiltration is obtained with GC-SiC and 6% Mg. However, the same or similar results can be obtained when using Mg in the range 5–6.5%.

Considering only the parameters and interactions included in Table I and the levels tested, the maximum degree of infiltration can be obtained by using a bilayer ceramic preform of GC-SiC powders and an aluminum alloy with 6 wt% Mg.

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