

Dual layer silica coatings of SiC particle reinforcements in aluminium matrix composites

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Abstract

The development of double layer silica coating on SiC particles is demonstrated as a method to create an active barrier which improves wetting and reduces interfacial damage during the manufacturing by a liquid route of aluminium matrix composite reinforced with SiC particles. Coatings tested have a dual structure formed by an inner partially crystallized silica layer obtained by SiC particle oxidation, and an outer one produced by a sol–gel method which forms an amorphous silica layer. Studies of thermal gravimetry/differential thermal analysis (TG/DTA) have allowed the determination of the reactivity grade of different duplex coatings obtained by controlling the densification level of the outer layer, using different heat treatments for the coated particles. Similar studies on uncoated and single coated particles (only oxidised or sol–gel treated ones) were also carried out for comparative purposes. Microstructural studies were also applied to determine the influence of the coating treatments on the composite microstructure. The resulting coating increases the reactivity of the reinforcement in the first stages of the composite manufacture while it better protects the inner SiC from the molten aluminium etching.

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1. Introduction

SiC particulate reinforced aluminium matrix composites are reference materials because of their wear and mechanical properties; they have a higher strength and increased wear resistance than unreinforced aluminium. Moreover, using high reinforcement concentrations, generally above 50%, can reduce the thermal expansion coefficient to a fifth of that of aluminium. These composite materials are compatible with the silicon technology and can be used as heat sinks directly attached to the silicon chips and in micro-electronic packaging, taking advantage of their high heat exchange coefficient [1–3].

When manufacturing high SiC reinforcement concentration aluminium composites several problems appear [4,5]. Low temperature casting is not valid because of the

reduced wettability of SiC by molten aluminium. At higher temperatures the reactivity between molten aluminium and SiC increases, so the wettability also does. Unfortunately, the interface reaction that takes place between composite constituents produces aluminium carbide, which expands in humid environments stressing and breaking the composite material while increasing its corrosion sensitivity.

Two main strategies have been used to avoid these problems. Silicon has been added to the aluminium alloy matrices because it reduces the aluminium carbide formation and simultaneously does so with the silicon carbide consumption; nevertheless this alternative limits the number of alloys that can be used as matrices. The other methods used consist in coating the particles with metals or ceramics to avoid the contact of unprotected SiC with the molten aluminium. Silica is one of the most successful coatings used. It can be obtained either through direct oxidation or via sol–gel. SiC particles can be oxidized in air at temperatures higher than 1100 °C to obtain a partially

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crystalline nanometric thick silica coating that goes under stress during cooling because of the volume change associated to the transformation [6,7]. Although this layer protects the reinforcement, its rapid consumption gives place to a preferential etching of SiC particles and to the formation of aluminium carbide in the oxidation layer cracks due to their stress state [8]. The sol–gel procedure gives place to a continuous micrometric amorphous silica coating that works out more properly, because of its wider thickness and continuous and free stress structure [9]. Moreover, it is also possible to reach a higher degree of design in its inner structure, by means of heat treatments, which allows controlling its reactivity [10].

The aims of this study is to design an active coating capable of a further reactivity, in relation with single sol–gel ones, to reach the following objectives: 1) a higher increase of wettability between molten aluminium and SiC particles; 2) a wider control in the final matrix composition (i.e. percentage of free silicon formed); 3) an enough protection against aluminium carbide formation. For it, the development of a dual silica layer around the particles, constituted by an inner oxidation layer surrounded by a sol–gel one, has been studied. Present paper compares the behaviour of SiC particles protected with these duplex coatings to those covered by single sol–gel or oxidation ones, as well as uncoated SiC reinforcements. Their reactivities, the resultant matrix microstructures and their relative wetting behaviours, considered as the influence of the coating nature and structure on the particle distribution, were analysed using: light microscopy (LM), scanning an transmission electron microscopies (SEM and TEM), X-ray diffraction (XRD), Thermal Gravimetry/Differential Thermal Analysis (TG/DTA) and specific surface measurement by the BET method.

2. Experimental procedures

2.1. Direct oxidation of SiC particles

The SiC particles used in this study were supplied by Navarro S.A. with the denomination F-360. Its average diameter is 26.2 μm , having a narrow size distribution. Their purity is 99.75% and the surface area specification of the particles is of 0.2 $\text{m}^2 \text{g}^{-1}$.

The particles were coated with a silica (SiO_2) layer by direct oxidation in an air-furnace at temperatures of 1100 and 1200 $^\circ\text{C}$ for 1 h. According to previous studies [8], this coatings should be partially microcrystalline with a thickness below 150 nm.

2.2. Sol–gel coating procedures

The starting sol was prepared from tetraethylortosilicate (TEOS) diluted in absolute ethanol and distilled water. After different trials the final process used for this work has a ratio ethanol/TEOS of 11. Acidulated water (HCl 0.1 M) was

added drop by drop under agitation to a final ratio water/TEOS of 5. The mixture hydrolysed for 2 h at room temperature under continuous stirring. Afterwards, particles were added to the mixture and were kept under agitation for 2 more hours, and for 30 min more without stirring. Coated particles were filtered and cleaned with ethanol and dried for 1 h at 120 $^\circ\text{C}$ in air to evaporate water and ethanol. Finally, a 1 h thermal treatment of 500, 600, 725 or 800 $^\circ\text{C}$ was given to the particles to control the porosity of the coating.

2.3. Duplex coating fabrication

The multilayer structure was used to add the advantages of both types of coatings. In order to obtain a duplex layer, SiC particles were firstly subjected to the oxidation procedure and, after a cleaning treatment with ethanol, they were immersed in the sol. Finally, the sol–gel coating procedure was carried out with the same stages and times used for the as received particles. Thermal treatments of 500, 600, 725 and 800 $^\circ\text{C}$ were also used to determine its effect on the composite material properties.

2.4. Composite material fabrication

Composite compacted pellets were made by mixing AA6061 aluminium powder from ALPOCO (Aluminium Powder Company, UK) with a 20% weight of the reinforcement. Different coated particles were used: oxidized, sol–gel and duplex coated (in both cases with different thermal treatments), as well as uncoated ones.

The samples were compacted with a pressure of 520 MPa to make 13 mm diameter and 1 g mass pellets. Fusion tests of the whole pellet were made in a high vacuum furnace ($4 \cdot 10^{-5}$ mbar) for temperatures ranging from 750 to 900 $^\circ\text{C}$ and maintenance times from 5 min to 1 h. This processes were also simulated by differential thermal analysis (DTA) using 90 mg pieces.

2.5. Extraction of the particles of the composite material

One of the tests carried out consisted in the observation of the surfaces of SiC particles after the composite manufacturing under the fusion conditions. To do it, particles were extracted from the composite material by diluting the aluminium matrix in an electrolytic procedure, using as reagent nitric acid/water solution (30% in volume) under different currents and voltages. This procedure dissolves the aluminium matrix and most of the reaction products formed at the interfaces, keeping unaltered the SiC surfaces which show the signal of the preferential etching, when it occurs. For it, extracted particles were observed by SEM.

2.6. Other tests

SEM observations were carried out with a Philips ESEM XL-30 under high vacuum conditions for conducting

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