Microstructural Study of Mechanically Alloyed Aluminium with Nanometer Size Silicon Carbide Powder

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Abstract: Dispersion of small ceramic particles is a common way to reinforce metal matrices with low melting points, such as aluminum. The size of the reinforcement has an important effect on the mechanical properties of the composite. In this report, aluminum particles of 40 μm in diameter were mechanically alloyed with 0 to 5% Si_xC_{1-x} ($x \approx 0.5$) nanometric powders (10-100 nm in diameter). These silicon carbide powders were obtained in a radiofrequency glow discharge of silane and methane mixtures. After mechanical alloying, the size of the alloyed powders (from 1 to around 200 μm) was found to be very dependent on the SiC content. These particles showed a peculiar microstructure. They had a highly porous appearance, as if they were formed by welded spheres of sizes around 100 nm. HIPing of these powders lead to a full dense material, and their hardness increased monotonically with the SiC content.

Introduction

Reinforced aluminum-matrix composites have been increasingly investigated as a suitable weight-saving material in the automotive and aerospace industries. Among several reinforcements, ceramic particles can lead to aluminum matrix composites which are of low cost and ease fabrication when compared to the corresponding fiber reinforced composites. SiC particles have been extensively studied as a reinforcement of aluminum because of its high stiffness and strength and because of the slow reaction rate between Al and SiC [1-3]. There is evidence that the small size of the reinforcement improves the mechanical properties of the composite (toughness and ductility)[2,4]. The extreme case of small size of the reinforcement corresponds to the precipitation-hardened alloys. In this case, the service temperature is limited by the fact that at relatively low ones the precipitates coarse. Nanometric SiC particles seem to be a good alternative because of their small size and high thermal stability.

This report deals with the investigation of the reinforcement of an aluminium matrix with nanometric SiC particles produced in a Plasma Enhanced Chemichal Vapor Deposition (PECVD) process from a gas mixture of silane and methane [5,6]. Besides their small size (diameter in the 10-

10^2 nm range), SiC particles obtained by PECVD have other features which may improve the

	Sample									
	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10
% wt. SiC	0	1.1	0	2.5	4.9	0	1.0*	2.1*	3.2*	4.5*
% wt.PCA	0	0	1	1	1	1	1	1	1	1
HV	-	-	-	-	-	91	87	115	118	120

Table I. Weight content of SiC and PCA in the samples investigated as well as the Vickers hardness in case it could be determined. (*) the SiC powder was annealed before mechanical alloying.

mechanical properties of the reinforced aluminum. First, their shape is almost spherical. These particles are amorphous and their shape presents no angular features. There are clear proofs that the rates of void iniciation and growth are lower for spheres [7]. Second, the chemichal composition of these particles can be completely controlled by the precursor gas mixture. It is well known that the extent of reaction between Al and SiC can be reduced by raising the level of Si in the alloy [1].

Concerning the fabrication of the alloy, a liquid phase processing of a MMC with nanometric particles is not convenient due to the segregation of particles as a result of particle pushing by an advancing solidification front. Therefore, aluminum and SiC powders have been mechanically alloyed in a planetary-ball mill. The microstructure of alloyed powders consisting of aluminum, with both as-deposited SiC and annealed SiC, has been investigated. Moreover, a series of samples with different SiC content was sintered in a Hot Isostatic Pressing (HIP) process. The hardness of the resulting material was determined.

Figure 1. TEM micrograph of as-deposited SiC nanometric powder

Experimental

Samples consisting of 2,5 gr of commercial grade Al powder (40 μm in diameter) and different contents of SiC nanometric particles (up to 5% wt.) were mechanically alloyed in a planetary ball mill (Retsch) for 10 hours. The Al-SiC powder mixture and the stainless-steel balls (the ball-to-powder weight ratio was 40/1) were introduced into a 125 ml steel container. Table I details the technological parameters of the samples. The microstructure of the alloyed powder was analyzed by SEM. In order to prepare suitable samples for planar-view SEM inspection, the powder was embedded in an epoxy matrix and then finely grinded and polished. A Kellers' solution was used in order to chemically etch the samples.

The alloyed powder was sintered in a Hot Isostatic

Pressing experiment. The powder was encapsulated in a stainless steel container, then it was degased at 550°C during 4h under vacuum, and finally the HIPing process was performed at 528°C and 150 MPa during 4h. A conventional Vickers indenter, installed in an optical microscope, was used to evaluate the effect of the SiC content on the hardness of the sintered samples.

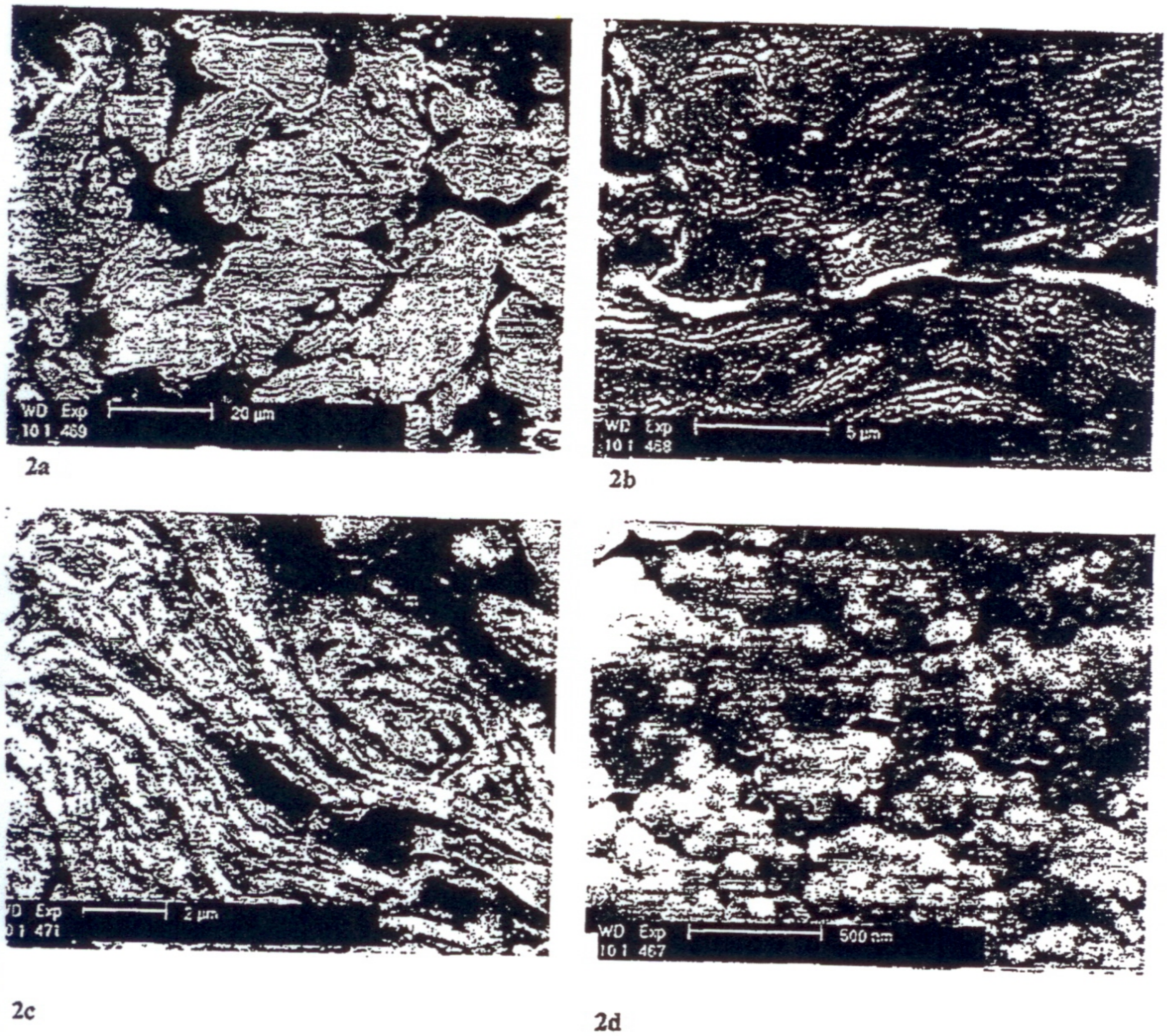

Figure 2. SEM micrographs at increasing magnification of planar views of sample S5 after mechanical alloying. The alloy powder was finely grinded, polished and chemically etched.

Results

The microstructural characteristics of the SiC nanometric powder produced by PECVD can be found elsewhere [5,6]. Basically, the particle size ranges from 20 to 200 nm, although the majority of them are around 50 nm (figure 1). The as-deposited powder is amorphous, it has a very high hydrogen content (. 40% at.) and oxidizes rapidly in the atmosphere. It is necessary to handle it under vacuum or inert atmosphere. In order to degas the SiC powder, it can be thermally tretated at 900°C in an inert atmosphere. This treatment removes the hydrogen content, stabilizes the nanometric particles against spontaneous oxidation and enhances the short-range order of its amorphous structure. SiC powders produced by PECVD remain amorphous after this low-temperature thermal treatment. Powder alloys, with both as-deposited and thermally treated SiC particles, were obtained

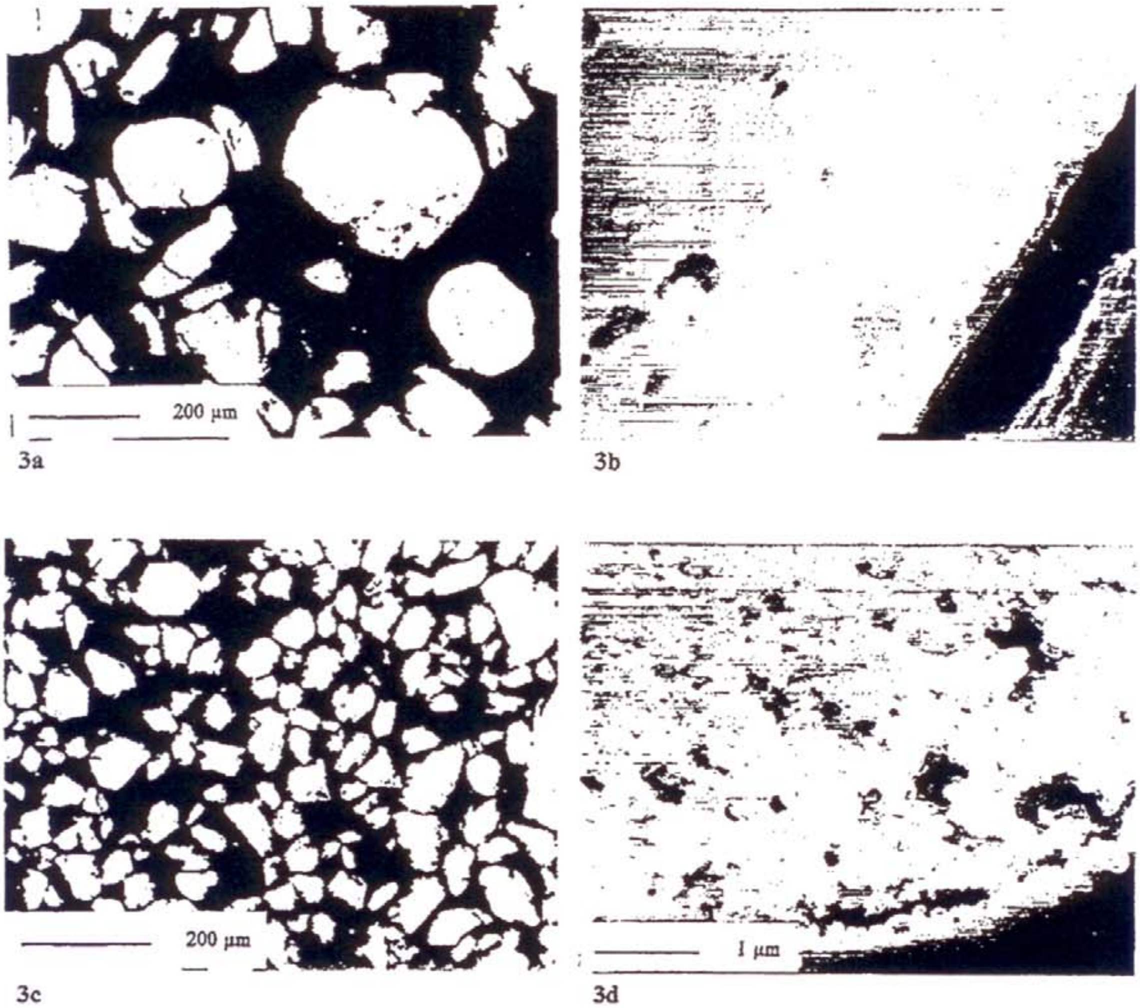

Figure 3. SEM micrographs of samples S6 (3a and 3b) with 0% SiC; and S10 (3c and 3d) with 4.5% SiC. The SiC content caused a higher porosity, a decrease in the particle size and a submicrometer grain structure.

Samples S1 and S2 consist of Aluminum powder with 0 and 1% as-deposited SiC respectively. During mechanical alloying the powder agglomerated and the final particles were almost spherical with diameters of around 0.5 mm, clearly unsuitable for a later compaction process. Besides that, a fraction of around 50-60% of the alloyed powder remained stuck to the steel balls or to the container. In order to prevent the agglomeration of the powder, some stearic acid (PCA) was added to the powder mixture before milling. The experiments showed that a minimum of 1% wt. of PCA was required to obtain an alloyed powder with diameters below 10^2 μm.

Samples S3 to S5 correspond to mixtures of aluminum with as-deposited SiC (from 0 to 5% wt.) and 1% wt. PCA. It was observed that after mechanical alloying the microstructure of the powder could be described at two levels (Figures 2a-2d). At the micrometer range (figures 2a and 2b), the average size of the powder was dependent on the SiC content. Indeed, it decreased from 300 μm to 20 μm for 0 to 5% SiC respectively. The planar views of the chemically etched specimens obtained by SEM revealed plied layers of material in a non-dense arrangement. Higher magnification pictures of these layers (figures 2c and 2d) showed a substructure of the powder in the range of the SiC particle size (50 nm).

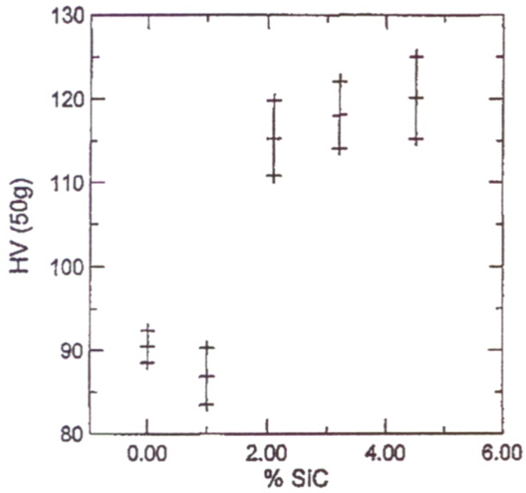

Figure 4. Dependence of Vickers hardness of the alloyed powder on the SiC weight content.

These results indicate that the SiC powder prevented the agglomeration of the powder during the milling process, thereby reducing the final particle size. Moreover, the SiC powder induced a particular structure which seems promising with a view to obtain a sintered material with submicrometer grain size. However, these powders could not be densified satisfactorily in a HIP process.

Further experiments were performed with SiC nanoparticles, which had been previously annealed at 900°C. Samples S6 to S10 were produced by mechanical alloying of aluminum powder, 1% PCA and 0 to 4.5 % of annealed SiC powder. Figures 3a-3d allows us to compare the microstructure of the alloyed powders with 0% and 5 % wt. of SiC. First, and similarly to samples S3 to S5, the particle size diminished with increasing SiC content. Second, a higher SiC content induced a richer substructure characterized by a higher porosity and a more clear submicrometric grain structure. The sample with 0% SiC appears almost homogeneous, whereas the nanometric grain substructure is clearly evident in the sample with 5% SiC (figures 3b and 3d).

The latter samples were satisfactorily sintered in a HIP process, and their microstructure showed a full densification. Then the Vickers-hardness was measured in the sintered samples. The results are shown in figure 4. The addition of SiC powder to aluminum increases its hardness monotonically. It is worth to note that, in spite of the low SiC content, the hardening slows down as the SiC content increases

Conclusion and Perspectives

The results presented in this paper show that the hardness of Al can be enhanced significantly by the addition of submicrometer SiC powders. Further microstructural investigations by HRTEM in order to determine the grain size after the HIP process, as well as to analyze the Al/SiC interface, are in course. Besides that, the mechanical properties of the compacted alloy at higher temperatures should be also studied.

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