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Effect of process parameters on micro and macro-properties of an Al-based nanocomposite prepared by means of mechanical milling

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ABSTRACT

In this research, some Al-based composites reinforced with silica nanoparticles were fabricated using mechanical milling and processing by powder metallurgy. Density, morphology evolution, hardness and strength of the composites were studied as a function of milling time and additive concentration. Microstructural evidence reveals that milling conditions generated a uniform dispersion of nanoparticles in the Al matrix. Noticeable matrix grain refinement was induced by the milling process. An optimum mechanical response was reached with small amounts of reinforcement nanoparticles complemented with medium milling intensities. These manufacturing conditions allowed to achieve an improvement of mechanical properties compared with the reference material (pure milled Al and un-milled composites processed under the same conditions). On the other hand, an adverse effect was observed with longer milling time and higher additive concentration.

1. Introduction

Discontinuously reinforced metal matrix composites (MMCs) [1] are the object of extensive research due to their high specific stiffness, strength and high wear resistance in comparison with corresponding unfilled matrix alloys [2]. Aluminum metal matrix composites (Al-MMCs) are assuring materials for wear and structural applications because of their low density and improved mechanical response, mainly caused by a good incorporation of the particles in the metal matrix [3]. Al-MMC can be produced by dispersing of the second phase into the vortex of the molten metal in the liquid route [4], or by infiltration of molten metal into a porous fiber preform under pressure [5,6]. Unfortunately, the homogeneous integration between the matrix (metal) and hard particles (ceramic) is often difficult because of the differences in chemical and physical properties, interphase reactions [7], wettability problems and the formation of new and unwanted phases [8]. The use of high temperature and its associated problems is avoided with a solid route, which is based on a high-energy mechanical milling (MM). In this technique, powder particles are energetically milled in a vial using ball collisions, obtaining a very fine grain size with homogeneous structure [9]. The impact energy related with the use of different types of milling media on the material is an important parameter to be taken into consideration when choosing the method of MM. In order to obtain homogeneous structures, the ball milling bodies are preferable [10]. Many authors have used high-energy ball milling to obtain nanometer or sub-micrometer sized ceramic particles distributed in the matrix by solid-state reaction at low temperature [11–13]. The most important process parameters are the correct amount of powders, velocity and process duration [14]. MM not only provides better distribution of reinforcement particles in matrix, but can also result in significant improvement in mechanical properties of final products due to deformation and refinement of matrix crystalline structure [9,12,14,15]. Difficulties can occur during the dispersion of ceramic particles even with the use of this technique, mainly because of inherent agglomeration problems [16]. Thus, the proper dispersion of reinforcement particles is one of the most important issues to defeat in order to obtain a full strengthening in synthetized composites. The nanocomposite preparation involves the use and application of nanometric reinforcements, where the internal structure determines the properties and performance of the bulk. In this particular type of materials, the reinforcement/matrix interface plays a key role leading the overall performance. The challenge is to increase the bonding strength of the interphase by controlling manufacture process [17] and some particle features as size, morphology and volume fraction [2]. The use of ceramics as reinforcement agents is getting relevant because of their hardness, mechanical strength, chemical inertness and suitable physical properties [18], the typically used reinforcing particles are SiC [18,19], TiC [20] and Al_2O_3 [21]. However, for many applications, these materials are not appropriate due to the high cost of additives and complicated processing modifications required during their incorporation into the metallic matrix [3]. In our study, silica was used for Al matrix reinforcing. This couple can be suitable for

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composite preparation due to combination of amphoteric properties of aluminum (Al) and the acid character of silica (SiO_2), indeed, there are some papers, devoted to mechanical properties of such composites [7,22,23]. Silica nanoparticles (SNPs) can be fabricated from a pre-hydrolyzed solution of the silica precursor using the organic sol–gel route [23–25] at low processing temperatures [18]. Additionally, SNP can be subjected to surface modifications e.g. by the incorporation of organic functional groups, which provide strong affinity between the matrix and the nanoparticles [26]. As a complement of these studies, a research based on the preparation and characterization of some Al-MMC with SNP addition could be interesting and innovative. In this research, some Al–SNP composites were prepared using a solid-state route complemented with PM techniques. The structural characterization of nanocomposites, evaluation of the influence of milling process on the SNP distribution in the matrix and their effect on mechanical response (strength and hardness) of composite materials are presented.

2. Experimental procedure

Commercial pure Al powder (99.5% purity −325 meshes in size) and SNP were used as raw materials for composite fabrication. For SNP preparation, the Stober [27] synthesis method was used. It involves ammonia-catalyzed reactions of hydrolysis and condensation of tetraethylorthosilicate with water in the presence of ethanol. The obtained gel was dispersed, washed (to remove the unreacted components) and dried out without a calcination step in order to maintain an organic layer over the particles surface, after this procedure spherical particles with an average size of 83.3 ± 1.5 nm (40 measurements) were obtained. Composite preparations were done from mixtures of silica and Al with 0.1, 0.5 and 1.0 (wt.% SNP), which were processed with a high energy SPEX 8000M mill under an inert argon atmosphere. Hardened steel balls (13 mm in diameter) were used as milling media; a ball–sample ratio of 5:1 was kept for all experimental runs. Selected milling intensities were 0, 1, 2 and 4 h. X-ray diffraction analysis of powders was carried out using a Broker D8-Advance diffractometer under the following conditions: 15–90°, 0.033° step and 25 s/step. The diffraction patterns were analyzed using standard XRD procedures to determine micro strain and crystallite size of milled composites. Morphology, size and particle distribution were examined using a JSM-7201F scanning electron microscope (SEM); cross section samples were mounted and prepared using standard metallographic techniques. Consolidated products were obtained by pressing the powder samples in a circular die at 900 MPa under uniaxial load and sintered for 3 h at 723 K in order to carry out the mechanical tests. Hardness tests were carried out in a Wilson Rockwell device and compressive tests were done using an Instron universal testing machine at room temperature. Transmission electron microscopy (TEM) studies on sintered and deformed probes were carried out in a JEM2200-FS microscope, thin foils for TEM investigations were prepared using a JEM9320-FIB. Pure Al and un-milled composites processed under the same conditions were used as reference materials.

3. Results and discussion

3.1. Microstructural characterization

Fig. 1 shows the effect of milling on the general morphology in an Al–1%SNP composite after processing. The as-mixed sample (Fig. 1a) presents semi-spherical particles, typical for gas-atomized powders. It is important to notice the homogeneous distribution of SNP on a single Al particle and their relative size (in the square). Milled products exhibit higher particles sizes compared with unprocessed sample (0 h) as Suryanarayana [28] describes it due to the ductile behavior of Al. After 1 h of milling (Fig. 1b), the metal particles are plastically deformed due to the collision between milling media and powder. The particle morphology changes from spheroidal to irregular with the presence of large aggregates formed at the expenses of crushed small particles [29]. Forge hardening of the large deformed particles reached a critical value, which led to fracture process activation (Fig. 1c). Agglomerates broke into small pieces and flake-like particles appeared (Fig. 1d) with further processing.

Fig. 2 shows some micrographs in cross section of powders processed from 1 to 4 h. At the beginning of MM, ductile Al particles were trapped between colliding milling media and get cold-welded and flattened. Meanwhile, small silica particles were trapped and encapsulated between new interfacial Al welding boundaries. This behavior is observed in Fig. 2a, where a strong agglomeration of SNP among the coarse convoluted lamellae microstructure is revealed. The lamellae spacing became smaller with further processing, forming structures sub-micrometric in size (Fig. 2b). After 4 h of milling, spacing became so fine that it was not longer evident (Fig. 2c). This phenomenon facilitates the homogeneous particle distribution of the strengthening phase in the metal matrix during milling [7]. The presence of tiny particles embedded between matrix layers with the shape of dark dots, which correspond to silica particles distributed on the surface of the sample, was evident.

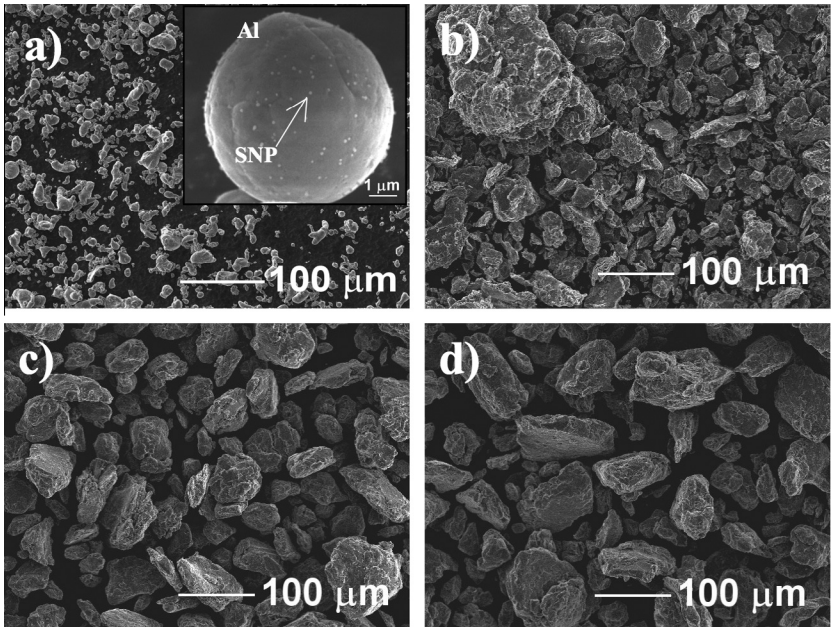

Fig. 1. SEM micrograph (250×) of Al–1%SNP mixture after (a) 0, (b) 1, (c) 2 and (d) 4 h of milling.