Composites of Aluminum Alloy 7075 with Silver Nanoparticles Prepared by Mechanical Milling

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ABSTRACT

Aluminum alloy nanocomposites have been emerged due to their high mechanical properties and low densities, one of the routes of production is the mechanical milling due to its relatively easy way of processes it. During mechanical milling the different powder of the matrix are collisioning by milling media and are deforming, welding and fracturing repeatedly, during the process, the nanoparticles are tramped within the matrix powder. Conforms the powder is milled, its morphology change to an equiaxial form and the particle size is reduced conform the milling time increase. The nanoparticles within the matrix acts like second phase particles finely and homogeneously dispersed, which promote the reinforcement of the alloy breaking the dislocation motion. Nanocomposites base aluminum were prepared introducing carbon coated silver nanoparticles (Ag-C NP) into the matrix of the Al 7075 alloy (Al7075) by mechanical milling process in a high energy SPEX mill, the milling time was set at 5 and 10 h, hardened stell vials and balls were used as a milling media, the milling ball to powder ratio was set at 4:1, and methanol was added as a PCA. Silver nanoparticles modify the powder particle size as well as crystallite size acting as PCA, these avoid the dissolution of the MgZn₂ compound and increases the Vickers Hardness Number. It has been found that exist a saturation point where microhardness values starts to decay. The present work is focused in the improvement of the Al 7075 alloy introducing second phase particles with nanometer size into the aluminum matrix, by a solid state method like mechanical milling (MM). Microstructural and mechanical properties are discussed.

Palabras clave: Nanocomposites, Mechanical milling, Aluminum 7075.
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Abstract. 7075 aluminum alloy with silver nanoparticles coated with carbon were obtained by mechanical milling in a high energy shaker ball mill, the powders were compacted and sinterized in an argon atmosphere, and hot extruded into a die with circular aperture of 10 mm. Their analysis showed that silver nanoparticles are homogeneously dispersed in the aluminum matrix, the powder particle and crystallite size decreases with the increment of the milling time, as well as the nanoparticles content. Microhardness values of the nanocomposites show an important increment in relation with the 7075 aluminum alloy, in both the powder and the extruded bar conditions.

Introduction

Aluminum base alloys are widely used in industry due to their lightness and corrosion resistance; unfortunately these have low yield strengths, which limit their use in a wider range of applications. The main applications of aluminum alloys are window frames, door handles, metal pipes, barrels, boats, cooking pots, drink cans, combustion engines, power lines, car bodies and aircraft [1]. The 7XXX series Al alloys have received special attention in aerospace and automotive industries because they provide the highest strength among all aluminum alloys, in fact these alloys offer a wide range of strength and ductility [2]. The development of aluminum alloys with higher strength is of great interest for engineering applications. Composites are engineering materials made from two or more materials with physical or chemical properties significantly different, which remain separate and distinct on a microscopic level in the final structure [3, 4]. In the case of aluminum composites, fine particles can be introduced into the aluminum matrix, which act like second phase particle, retarding the dislocation movement of the material and increasing the mechanical properties [5]. If the particles have a nanometric size, the composite is known like nanocomposite [6], which is stable at medium-high temperatures [7].

Mechanical alloying (MA) and mechanical milling (MM) are processes that produce composite metal powders with a fine microstructure. MM is a powder processing technique that involves welding, excessive deformation and fracture of particles in a repetitive manner a large number of times. In this way the process enables the production of homogeneous materials from mixtures of metal powders [8]. The aim of this work was the microstructural and mechanical characterization of the nanocomposites prepared from a 7075 aluminum alloy reinforced with carbon coated silver nanoparticles.

Experimental

The 7075 aluminum alloy (Al7075) in the T6 condition and silver nanoparticles were the starting materials. The Al7075 was annealed for 24 hours at 415°C to remove the T6 heat treatment condition and then was machined to obtain metal burr with a size of 1000 to 7000 μm. Previous reports [9] show that silver nanoparticles are coated with carbon (Ag-C NP) and have a size of 10 to 20 nm. The Ag-C NP is a product of Nanotechnologies, Inc. (Austin, TX).
The Al7075 alloy was mechanically milled at different times to determine the microstructural evolution as a function of milling time. The composites were produced by mixing and milling the Al7075 alloy and Ag-C NP in different ratios, from 0.5 to 2.0 wt. %. The powders were generated in a high energy shaker ball mill SPEX 8000M at different milling times (5 to 25 h). The weight ratio of milling through sample weight was set at 4 to 1. The total sample weight was 10 g, and 0.25 ml of methanol was used as a process control agent (PCA).

The morphological characterization was performed in a scanning electron microscope (SEM) JEOL JSM 5800LV, operated at 20 kV, and in a JEOL JSM 7401F, operated at 5 kV. The microstructural characterization was done by X-ray diffraction (XRD) in a Siemens diffractometer operated at 40 kV and 25 mA, in the range 20 of 20 to 100 degrees; step and time of data collection were 0.05° and 5 s, respectively. The mechanical characterization was done by powder microhardness, in a microdurometer FM-07, using an indentation time of 10 s and a maximum load of 200 g.

Results

Structural analysis

Figure 1 shows the morphological evolution of the Al7075 Ag-C NP nanocomposites in the milled condition. Figure 1(a) presents the Al7075 alloy powders at various milling times: 0, 5, 10, 15, 20, and 25 h. Figures 1(b) and 1(c) show the Al7075 alloy with different concentrations of Ag-C NP milled for 5 h and 10 h, respectively. In figure 1(a) the particle size reduction is evident; as the milling time is increased, a smaller powder particle size is observed, as well as a powder morphological evolution. After 25 h of milling the powders are almost equiaxial with an average size of ~ 100 μm. These changes in the morphology and particle size as a result of MM in several systems are well documented [8]. In figures 1(b) and 1(c) it can be seen that as the content of Ag-C NP increases the particle size decreases for both milling times. Smaller particle size was obtained in composites with higher amounts of Ag-C NP and a higher milling time. The powder particle size is reduced at ~ 50 μm with 5 h of milling; after 10 h, the particle size is reduced to ~20 μm. This reduction supports that silver nanoparticles acts as a PCA, refining the particle during MM [10].

Figure 2 shows the microstructural morphology of the Al7075 nanocomposite with 10 h of milling in the hot extrusion condition. Figures 2(a) and 2(b) correspond to the alloy without silver nanoparticles. In figure 2(a) it can be seen an aluminum matrix generally free of particles, while in figure 2(b) it can be seen some second phase particles rich in copper (white particle) and magnesium particles (gray particles). Figures 2(c) and 2(d) show the nanocomposite with 1.5 wt. % of Ag-C NP. In figure 2(c) it can be seen an aluminum matrix with a significant number of particles well dispersed within the matrix; these particles correspond to the silver nanoparticles. In figure 2(d) it can be seen that the Ag-C NP have a particle size between 10 and 15 nm besides that this particles conserved their spherical shape after the milling, consolidation and hot extrusion processes.

Figure 3 shows the powder structural evolution during mechanical milling. Figure 3(a) corresponds to the powder without nanoparticles obtained at different milling times; diffraction peaks were broadening and their intensity decreased as the milling time increased, denoting a microstructure refinement; this behavior is typical of the mechanical milling process [8]. The sample in the annealed condition shows characteristic peaks of the MgZn$_2$ phase, which were not observed in the T6 temper condition, indicating that this phase precipitated during the annealing process. This was apparently dissolved (or fragment) as the milling time was increased without affecting the network parameter values. Figures 3(b) and 3(c) show the XRD patterns of the Al7075 nanocomposites milled for 5 and 10 h, respectively. Comparing the 5 h spectra in figure 3(a) and 3(b), and the 10 h spectra in figures 3(a) and 3(c), it is evident that the Ag-C NP have an effect on the sequence of the MgZn$_2$ phase precipitation. The characteristic reflections of this phase are present in all the patterns. Because of the low nanoparticles concentration, the characteristic peaks of Ag do not appear in the diffraction patterns. An important aspect found in figure 3 is that the spectra’s full width at half maximum (FWHM) is higher as the Ag-C NP concentration is increased, indicating a crystal refinement.

Figures 4(a) and 4(b) present the XRD spectra of the extruded Al7075–Ag-C NP nanocomposites. It can be observed the structural variation as a function of nanoparticles content for 5 and 10 h of milling. In both figures, the peaks from aluminum matrix are observed; peaks
corresponding to MgZn$_2$, MgO and Al$_4$C$_3$ phases are also observed, whose intensity increases as the Ag-C NP content increases. The presence of MgO is because this oxide has the lower free energy of formation, as reported in Hellingan diagrams [11], and it was probably formed during the handling of powders in the experimental step. The Al$_4$C$_3$ was formed most likely from two carbon sources: the PCA and the graphite shell that covers the nanoparticles. Because the Al$_4$C$_3$ peaks are more defined in composites with 1.5 wt % of Ag-C NP, we expect that the main graphite source is from nanoparticles shell. However, this phenomenon was not observed before in similar works [9, 12]. The MgZn$_2$ phase precipitates from saturated aluminum solid solution during the nanocomposites heat treatment, and it is expected that nanoparticles favor its nucleation. However, further work to elucidate this hypothesis is necessary. The presence of these phases has a favorable effect on the nanocomposites strengthening and it is expected that an additional heat treatment (i.e. T6) could yield a nanocomposite stronger than a conventional Al7075 alloy in the same condition.

**Mechanical properties**

Figure 5 shows the microhardness results of the nanocomposites in the powder condition. Figure 5(a) shows the variation of microhardness as a function of milling time. It is evident the work hardening of the powders during MM, increments of ~ 200% were obtained for milling times of 5 h with respect to the annealed condition of the alloy; this value increases with higher milling time, and the microhardness values were higher than those reported in the literature for the alloy in the T6 and T73 conditions [13]. Figure 5(b) illustrates the variation of microhardness in the nanocomposites milled for 5 h and 10 h as a function of the Ag-C NP content. In the same way as for figure 5(a), all nanocomposites show higher microhardness values with respect to the alloy in the T6 and T73 conditions. It is important to note that the most significant increment was observed with low contents of nanoparticles. An apparent saturation is observed at 1.5 wt. % of Ag-C NP for both milling times. At 5 h of milling time, 1.5 wt. % shows an increment of about 100 HVN as compared to the base alloy, and 35 VHN higher than the powder without nanoparticles. At 10 h of milling and 1.5 wt. % of Ag-C NP, the nanocomposite hardness increases 40% compared to the Al7075 alloy in T6 temper, and 93 VHN higher than the same nanocomposite milled for 5 h. The significant increase of microhardness in the Al7075 alloy is achieved due to the uniform dispersion of second phase particles of extremely small size in the aluminum matrix. The increment in resistance is highly dependent on the type, distribution and shape of precipitated particles present [9, 14].

Figure 6 presents the microhardness results of the nanocomposites in the extruded condition. From this figure, it is evident the elimination of the powder work hardening during the sintering and hot extrusion processes. Nevertheless, the microhardness value still remains at higher values with respect to the Al7075 alloy in the annealed condition. This increment in properties is due to the homogeneous distribution of the silver nanoparticles into the aluminum matrix. With 5 h and 10 h of milling the nanocomposite remains 20 HVN units and 25 HVN units, respectively, more than Al7075 alloy in the annealed condition. At 1.0 wt. % of Ag-C NP, the nanocomposites present the higher microhardness values for both times 5 and 10 h of milling; this value is almost 80 % higher than that of the Al7075 alloy, with higher concentrations of nanoparticles, the microhardness value has a small decrement, indicating a saturation point in the aluminum matrix with Ag-C NP. This saturation occurs probably due to the agglomeration of the silver nanoparticles, and this effect produces a decrement in properties; the agglomeration of Ag-C NP produces a bigger particle, and this delimits the number and distribution of second phase particles into the matrix, decreasing their strengthening effect [5, 14].

Due to the production process, the extruded nanocomposites are in the annealed condition; in this condition the nanocomposite has better properties than those of the Al7075 alloy in the same condition, this is, if the nanocomposites are age hardened (T6 temper), higher properties are expected; at the moment T6 heat treatment is carry out to clarified this phenomenon. From figures 5 and 6, we conclude that combining nanoparticle dispersion and mechanical milling, production of nanocomposites stronger than the base alloy is possible.

**Conclusions**

Nanocomposites of the Al7075 have been produced by dispersion of Ag-C NP using mechanical milling and hot extrusion processes. It was found that mechanical milling process and silver nanoparticles dispersion have an important effect on the mechanical properties enhancement; the
best results were found at lower silver nanoparticles concentrations. The MgZn$_2$ phase is affected by the presence of silver nanoparticles.

References


Figure captions

Figure 1. SEM micrographs showing the powder particle morphology as a function of (a) the milling time, (b) the Ag-C NP content with 5 h of milling, and (c) the Ag-C NP content with 10 h of milling.

Figure 2. SEM micrographs obtained from the Al 7075 Ag-C NP nanocomposite milled for 10 h. (a) and (b) without nanoparticles, (c) and (d) with 1.5 wt. % of nanoparticles.

Figure 3. XRD spectra of (a) Al7075 alloy milled at different times, (b) Al7075 Ag-C NP nanocomposites milled for 5 h and (c) Al7075 Ag-C NP nanocomposites milled for 10 h.

Figure 4. XRD spectra of the Al7075 Ag-C NP nanocomposites milled for (a) 5 h and (b) 10 h.

Figure 5. Microhardness results of the nanocomposites in the powder condition as a function of (a) milling time, (b) Ag-C NP content, milled for 5 h and 10 h.

Figure 6. Microhardness results of the nanocomposites in the extruded condition as a function of the Ag-C NP content, milled for 5 h and 10 h.
Figure 1.

Figure 2.
Figure 3.

Figure 4.

Figure 5.

Figure 6.