



# Microstructural and mechanical characterization in 7075 aluminum alloy reinforced by silver nanoparticles dispersion

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## ABSTRACT

Composites of 7075 aluminum alloy ( $Al_{7075}$ ) with carbon-coated silver nanoparticles (Ag-C NP) were prepared by the mechanical milling process. It had been found that Ag-C NP have an effect in refining the powder and in the crystal size.  $MgZn_2$  phase presents in annealed samples was dissolved by milling process. The Vickers microhardness (HVN) values are higher at higher Ag-C NP contents. It has been found that a saturation point exists where microhardness does not present variation.

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## 1. Introduction

The development of high strength Al alloys for use at moderate temperatures is of great interest for engineering applications. Aluminum alloys with strength improvement can be obtained by the dispersion of fine and homogeneous nanoparticles (nanocomposites), which must be stabilized at medium–high temperatures [1]. During research to design such materials, the concept of composite materials was developed which can bring together the combined advantages of the constituent materials, something not possible when they are employed alone [2]. Metal matrix composites (MMCs) have emerged because of their high specific modulus, strength-to-weight ratio, fatigue strength, temperature stability and wear resistance [3]. Another important driving factor is the ability to tailor the mechanical and physical properties (such as the coefficient of thermal expansion) by selecting the reinforcement type and volume fraction, along with the matrix alloy. The term ‘nanocomposite’ is used when the dispersed phase is in the range of nanometers. Mechanical alloying and mechanical milling have emerged as alternative ways to produce nanocomposite materials, formed by nanoparticles dispersion into a metallic matrix [1,4–8]. The present study is focused on the

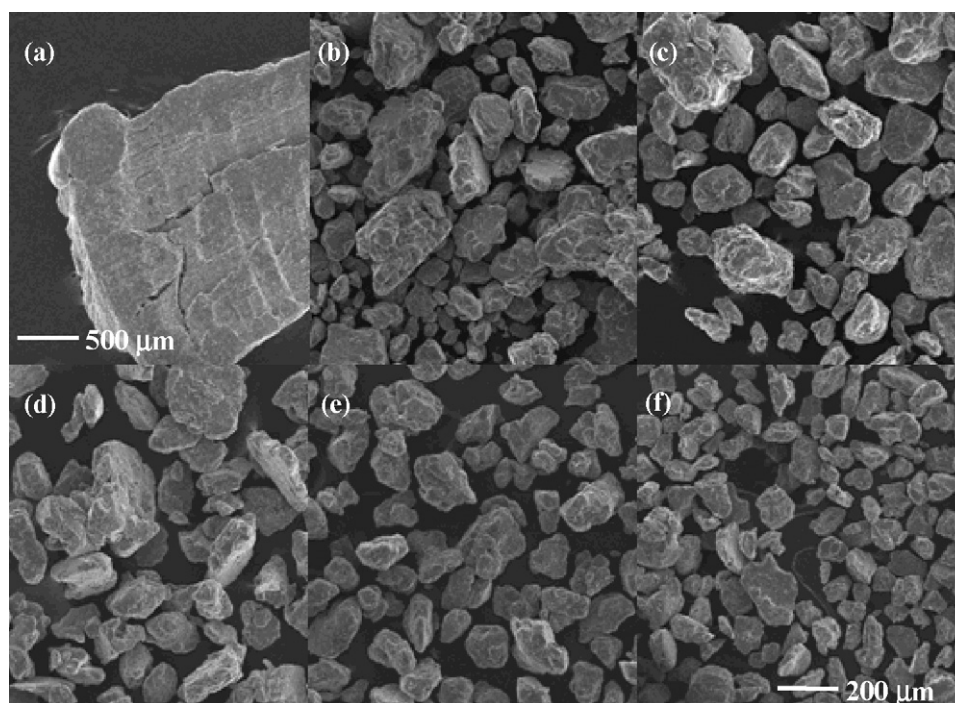
possibility of improving the properties of 7075 aluminum alloy by dispersing insoluble nanoparticles of silver recovered with a shield of carbon into the matrix by mechanical milling (MM). Microstructural and mechanical characterization is presented and discussed.

## 2. Experimental

7075 Aluminum alloy ( $Al_{7075}$ , henceforth) in T6 temper and silver nanoparticles were the starting materials.  $Al_{7075}$  was annealed during 24 h at 415 °C to remove the T6 temper and then was pulverized (metal shaving) by a machining tool process. Initial size in  $Al_{7075}$  varied from 1000 to 7000  $\mu m$ . Silver nanoparticles are carbon coated [4], henceforth Ag-C NP, and present a nanometric range size from 10 to 20 nm. Ag-C NP is a product obtained from Nanotechnologies, Inc. (Austin, TX).  $Al_{7075}$  alloy was mechanically milled at different times to screen the microstructural evolution as a function of milling time. Composites were produced by mixing and milling  $Al_{7075}$  and Ag-C NP in different proportions, from 0.5 to 2.0 wt.% NP. For milling runs, a high energy SPEX mill was used and the milling times ranged from 5 to 25 h. The milling ball-to-powder weight ratio was set at 4 to 1. The total sample weight, 10 g. 0.25 ml of methanol was used as a process control agent (PCA).

Morphology characterization was done in a scanning electron microscope (SEM) JEOL JSM 5800-LV, operated at 20 kV. Microstructural characterization was done by X-ray diffraction in a Siemens diffractometer, operated at 40 kV and 25 mA, in the  $2\theta$  range of 20–100°. Step and collection time were 0.05° and 5 s, respectively. The structural analysis was done by transmission electron microscopy (TEM) in a Philips CM 200, operated at 200 kV and the chemical analysis by characteristic X-ray energy dispersion spectroscopy (EDS) with a DX4 X-ray energy dispersive spectrometer coupled to TEM. Mechanical characterization in as-milled products was done by microhardness, in a Micro Hardness Tester (FM-07), using an indentation time of 10 s and a maximum load of 200 g.

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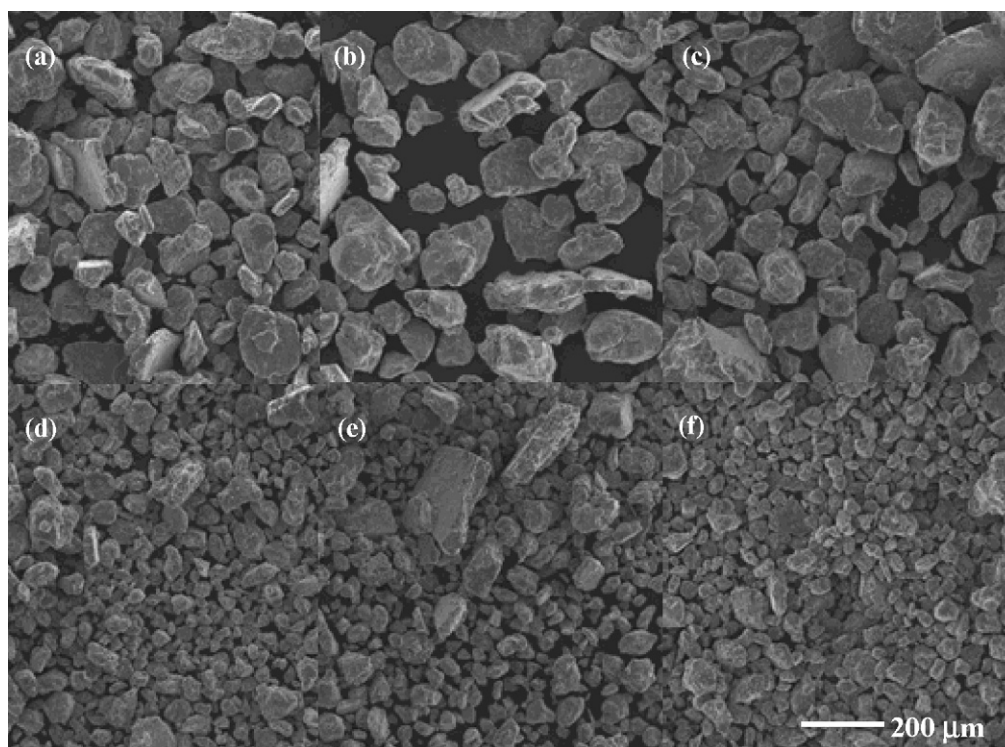


**Fig. 1.** SEM micrographs showing the particles morphology as a function of milling time. (a) 0 h, (b) 5 h, (c) 10 h, (d) 15 h, (e) 20 h, and (f) 25 h of milling.

### 3. Results and discussion

Fig. 1a–f shows the morphology evolution in as-milled powder of the  $\text{Al}_{7075}$  at several milling times: 0, 5, 10, 15, 20 and 25 h. Under milling conditions employed, the powder comminution is evident; as the milling time increased a lower powder size

was observed. Fig. 1a, corresponds to initial alloy (metal shaving, 0 h of milling), shows an irregular morphology, flakes-like. Fig. 1b–f show the morphology evolution as a function of the milling time. Longer milling times resulted in a more equiaxed shape in milled products. After 5 h of milling, the morphology changed to an irregular shape, with an average size of  $\sim 300 \mu\text{m}$ .



**Fig. 2.** SEM micrographs showing the morphology of particles as a function of Ag-C NP content. (a) 0%, (b) 0.5%, (c) 1.0%, (d) 1.5%, (e) 2.0%, and (f) 5.0% of Ag-C NP, milled during 5 h.

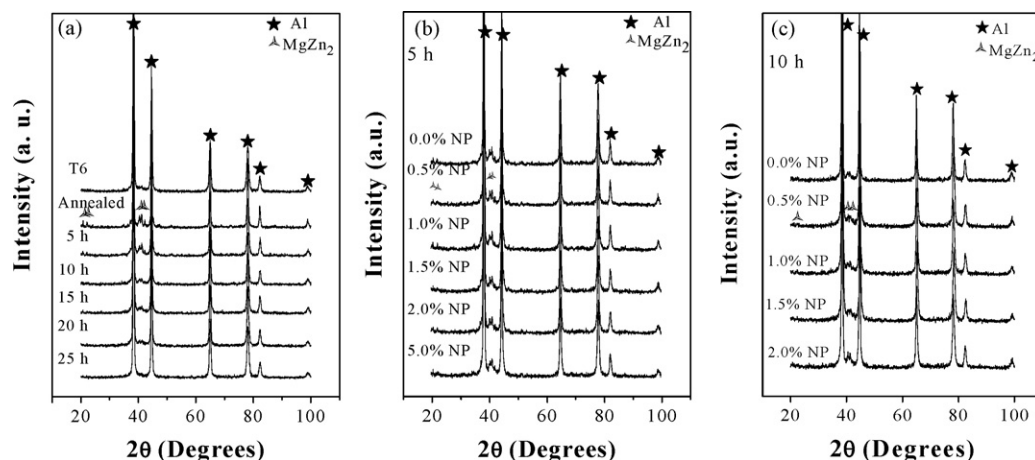


Fig. 3. XRD spectra in (a)  $\text{Al}_{7075}$  milled at different times, (b)  $\text{Al}_{7075}$ -Ag-C NP composites milled for 5 h, and (c)  $\text{Al}_{7075}$ -Ag-C NP composites milled for 10 h.

After 25 h of milling almost equiaxed powders with average size of  $\sim 100 \mu\text{m}$  were obtained. The changes in morphology and powder size as a product of MM in several systems are well documented [9].

Fig. 2 shows  $\text{Al}_{7075}$  with different Ag-C NP concentrations milled for 5 h. As the Ag-C NP content is increased, the particle size decreased. Lower particle sizes were obtained in composites with higher Ag-C NP amounts (Fig. 2f, 5 wt.% Ag-C NP). From Fig. 2 it can be seen the existence of an abrupt change in particle size refining with contents above 1.5 wt.% Ag-C NP. With 0, 0.5 and 1.0 wt.% the particle size changed slowly; at a higher content the change is faster up to a particle size of  $50 \mu\text{m}$ . Apparently NP work as a PCA, refining the particle size during milling. Particle size refining by nanoparticles dispersion during MM was not found to be reported in the reviewed literature.

Fig. 3 shows the structural evolution during milling in powder mixtures, Fig. 3a shows the effect of the milling time on XRD spectra, diffraction peaks were broadened and shortening as the milling time was increased, denoting a microstructure refining; this behavior is typical in the mechanical milling process. However, peaks shift to lower–higher angles was not observed, indicating no variations in aluminum lattice parameter. The sample in annealing

condition presents characteristic peaks from  $\text{MgZn}_2$ , which were not observed in the sample with T6 temper, denoting precipitation of  $\text{MgZn}_2$  phase during annealing. This phase was apparently dissolved (or fragmented) as the milling time increase, with no effect on the lattice parameter values. Fig. 3b shows XRD spectra from  $\text{Al}_{7075}$ -Ag-C NP composites milled for 5 h. By comparing milling time in Fig. 3a (5 h) and 3b, it was evident that Ag-C NP has an effect on the dissolution of the  $\text{MgZn}_2$  phase.

The characteristic reflections of  $\text{MgZn}_2$  phase decreased as the Ag-C NP concentration increased (Fig. 3b). However, no variations in lattice parameter were observed. Fig. 3c shows XRD spectra from  $\text{Al}_{7075}$ -Ag-C NP composites milled for 10 h. By comparing milling time in Fig. 3a (10 h) and c, it was evident, again, that Ag-C NP have an effect on the dissolution sequence of the  $\text{MgZn}_2$  phase, only a very few characteristic reflections of  $\text{MgZn}_2$  phase are showed in the XRD spectra. Because of the low Ag-C NP concentration used, characteristic peaks from Ag-C NP were not evident in the XRD spectra (Fig. 3b and c). An important aspect found in Fig. 3b and c was the spectra's full-width at half-maximum (FWHM), at higher concentrations of Ag-C NP the FWHM was higher, denoting a crystal refining as a consequence of Ag-C NP concentrations.

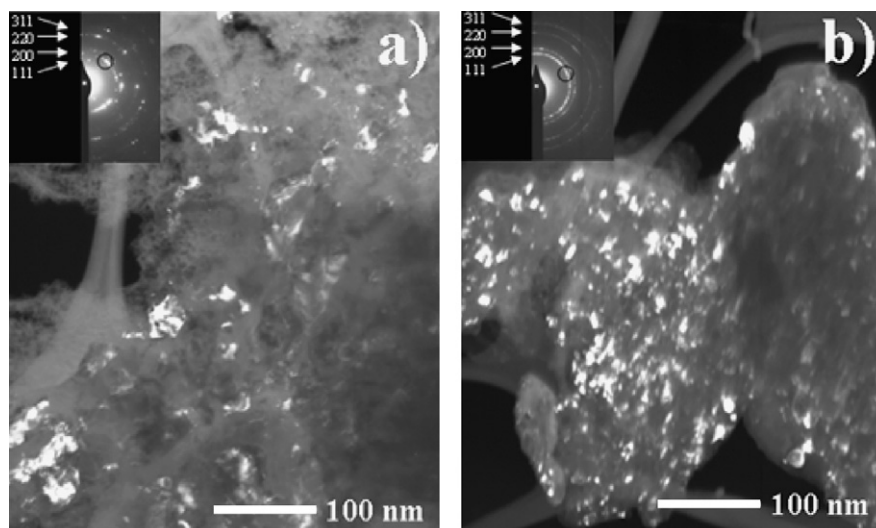


Fig. 4. TEM micrograph from as-milled products, (a)  $\text{Al}_{7075}$  milled for 10 h and (b)  $\text{Al}_{7075}$ -2.0 wt.% Ag-C NP composite milled for 5 h.



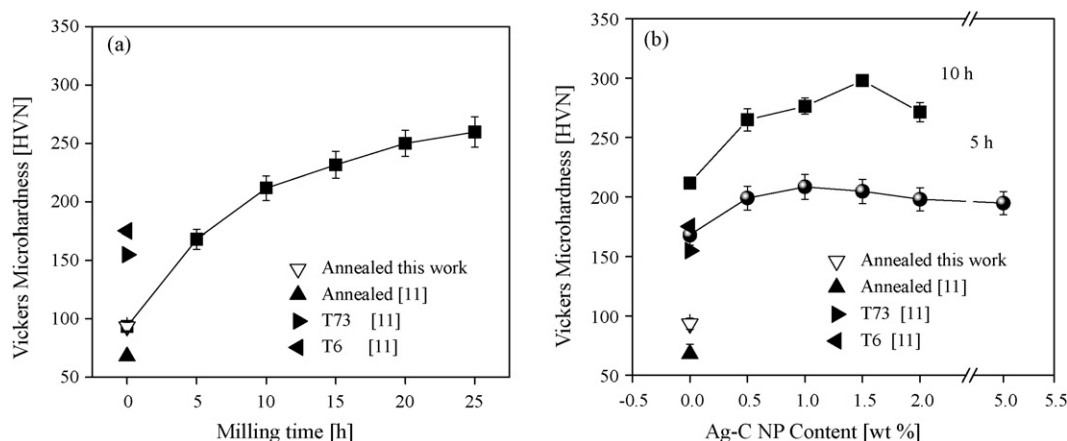


Fig. 5. Microhardness results as a function of (a) milling time and (b) Ag-C NP content milled during 5 h and 10 h.

Crystal size refinement by Ag-C NP dispersion was corroborated by TEM. Fig. 4 shows a dark field image from as-milled  $\text{Al}_{7075}$  powders after 10 h of milling (Fig. 4a) and  $\text{Al}_{7075}$  with 0.5 wt.% of Ag-C NP, after 5 h of milling (Fig. 4b). The inset squares show the corresponding selected area diffraction pattern (SADP) composed of rings and spots associated to the Al matrix. Circle in the SADP shows the reflection used to obtain a dark field image. From SADP and images, the sample in Fig. 4b presents a lower crystal size. After 10 h of milling, the  $\text{Al}_{7075}$  sample (Fig. 4a) shows a crystal size of  $\sim 50$  nm; in half the milling time, the composite material (Fig. 4b) presents a smaller crystal size in the range of 10–15 nm. The pictures in Fig. 4a and b support the XRD analyses referred to above. From Figs. 2 and 4, a new characteristic of NP dispersion by MM is opened, NP have an important effect on the particle and crystal size refinement during milling, additional to the well known effect, blockage of dislocations movement [10].

Fig. 5 shows the microhardness results in as-milled products. Fig. 5a shows the microhardness variation as a function of milling time. Work hardening in milled products is evident, increments about 200% were obtained, for milling times higher than 5 h, microhardness values found were higher than those reported for the same alloy in the T73 and T6 temper [11]. Fig. 5b illustrates the microhardness variation as a function of Ag-C NP content keeping constant the milling time (5 and 10 h). The same as in Fig. 5a, all the composites show microhardness values higher than those reported for the aluminum 7075 alloy in the T73 and T6 temper. It is significant to notice that the most important increment was observed with lower NP content; higher NP contents apparently have no effect on hardness values. An apparent saturation point is observed at 1.5 wt.% of Ag-C NP. For 5 h of milling time, 1.5 wt.% presents an increment of about 100 VHN, compared with the partner alloy, and 35 VHN more than the powder without Ag-C NP. For 10 h of milling time, higher microhardness values were found. An apparent saturation point was observed at 1.5 wt.% of Ag-C NP, which represents almost an increment of  $\sim 40\%$  with respect to the  $\text{Al}_{7075}$  alloy, and 93 VHN more than the same composite milling during 5 h. The higher value in microhardness was obtained with 1.5 wt.% Ag-C NP and 10 h of milling time (Fig. 5b).

The important increment on hardness of  $\text{Al}_{7075}$  alloy is achieved through the uniform dispersion of second phase nanoparticles into the original aluminum phase matrix. The enhancement in strength is highly dependent on the type, distribution and size of precipitate particles present [2,4]. From Fig. 5, it is concluded that by combining nanoparticle dispersion and MM process, it is possible to produce stronger nanocomposites than the parent alloy.

It has been previously reported that stronger matrix alloys tend to produce stronger composites, although the increase in strength due to reinforcement tends to be lower when higher strength matrix alloys are used, and in the case of lower strength matrix alloys, the strengthening effect of the reinforcement is higher [2]. Even though  $\text{Al}_{7075}$  alloy is considered a strong alloy and is expected to have a low level of reinforcement by dispersion, 75 U in Vickers scale represents more than twice that pure of aluminum hardness, or a similar value to several aluminum alloys [11].

Apparently, Ag-C NP concentrations are not important at higher contents than 1.5 wt.%. Hot extrusion and tensile tests are currently being done to answer the questions regarding what is happening with ductile properties. Similar work in pure aluminum reported no increment in mechanical properties at saturation concentration of dispersed graphite nanoparticles, but an important decrement in ductility was present [5].

#### 4. Conclusions

Al based nanocomposites have been produced by dispersing Ag-C NP by mechanical milling. Dissolution of  $\text{MgZn}_2$  during milling is affected by milling time and apparently by Ag-C NP presence. Ag-C NP have an important effect on particle and crystal size refinement. Microhardness increases as Ag-C NP content increases. Microhardness increases in the nanocomposite as the milling time increases. By combining nanoparticles dispersion and MM process it is possible to obtain better properties than those reached by T73 and T6 tempers in  $\text{Al}_{7075}$  alloy. Concentrations higher than 2 wt.% do not have an important effect on microhardness. Ag-C NP dispersion strengthening has important potential applications in aluminum alloys.

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