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Aluminum–graphite composite produced by mechanical milling and hot extrusion

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Abstract

Aluminum–graphite composites were produced by mechanical milling followed by hot extrusion. Graphite content was varied between 0 and 1 wt.%. Al–graphite mixtures were initially mixed in a shaker mill without ball, followed by mechanical milling in a High-energy simoloyer mill for 2 h under argon atmosphere. Milled powders were subsequently pressed at ~950 MPa for 2 min, and next sintered under vacuum for 3 h at 823 K. Finally, sintered products were held for 0.5 h at 823 K and hot extruded using indirect extrusion. Tension and compression tests were carried out to determine the yield stress and maximum stress of the materials. We found that the mechanical resistance increased as the graphite content increased. Microstructural characterization was done by transmission electron microscopy. Al–O–C nanofibers and graphite nanoparticles were observed in extruded samples by transmission electron microscopy. These nanoparticles and nanofibers seemed to be responsible of the reinforcement phenomenon.

Keywords: Nanostructured materials; Metal matrix composites; Mechanical properties

1. Introduction

Aluminum alloys have a great diversity of industrial applications because of their low density and good workability, but the use of these alloys is limited due to their relatively low yield stress. Recently, the interest to increase aluminum strength for applications in the aerospace and aeronautic industries has motivated the study of aluminum matrix composites (AMC), which can present better mechanical properties at both, medium (473 K) and room temperatures. Additionally, one of their most important characteristics is its high specific stiffness while maintaining a low density [1–4].

The main reasons to produce AMC are to increase the strength, stiffness and wear resistance of aluminum or aluminum alloys, but this is usually achieved at the expense of other properties such as ductility. Aluminum and aluminum alloys can be strengthened by dispersing hard particles like carbides, oxides or nitrides into the aluminum matrix by using solid or liquid state techniques [5–7]. The reinforcement can be done by adding continuous or discontinuous fibers, particles, or whiskers. The last three are usually ceramic materials such as alumina, silicon carbide, or silicon nitride.

AMC can also be fabricated in the solid state through Powder Metallurgy (PM) techniques, which are used due to their great versatility and low cost of production. The process of fabrication consists in mixing the hardening particles with the metallic powders followed by consolidation and sintering. Additionally, by using mechanical milling (MM) it is possible to produce a fine and homogeneous distribution of the hardening particles. In some cases, it is possible to synthesize them in situ, generally by reaction milling and also during the sintering process. Therefore, the MM process is a useful tool for the development of novel materials by using PM techniques.

Even though graphite acts as an excellent lubricating agent under conditions of friction, graphite dispersion in aluminum or aluminum alloys by MM process has not been deeply investigated. In this work, we present the mechanical properties and structural analysis of aluminum–graphite composites produced by MM and followed by hot extrusion.

2. Experimental procedure

The raw powder materials used were Al (99.5% purity, −325 mesh in size) and graphite nanoparticles. The selected graphite content was 0.0, 0.25, 0.50,

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Table 1
Identification in samples, composition and experimental conditions

Identification	Composition	Milled	Extruded
Al_P	Pure aluminum	–	X
Al_M	Pure aluminum	X	X
Al_{25}	Al–0.25% C	X	X
Al_{50}	Al–0.50% C	X	X
Al_{75}	Al–0.75% C	X	X
Al_{100}	Al–1.00% C	X	X

0.75, and 1.0 wt.%. Each mixture was blended (mixed) during 0.5 h in a shaker mill without ball, seeking to have a homogeneous mixture, and then mechanically milled in a high-energy simoloyer mill for 2 h in an argon atmosphere. The milling device and the milling media used were of hardened steel. The milling ball-to-powder weight ratio was set at 20, and the total sample weight was 50 g for all the samples. No process control agent was used due to the short milling time. Consolidated bulk products (40 mm of diameter Ø) were prepared by pressing the milled powder at ∼950 MPa for 2 min under uniaxial load. Pressed samples were next sintered under vacuum for 3 h at 823 K with a heating rate of 50 K/min. Sintered products were held for 0.5 h at 823 K and hot extruded into a rod of 10 mm of diameter by using indirect extrusion with an extrusion ratio of 16. Also for comparison, not-milled pure aluminum in the as-extruded condition was tested. Table 1 shows the sample identification, composition and experimental conditions used.

Tension and compression tests were carried out in the longitudinal (extrusion) direction at room temperature in an Instron testing machine; the displacement rate was of 0.016 mm/s. The yield stress (σ_y) was evaluated at the elastic limit. For tension test, bone shaped samples was used in accord with ASTM B557M standard. For compression test, two height-to-diameter ratios (h:Ø) were used (0.8 and 2.0) in accordance with the ASTM E9 standard.

Microstructural characterization was done by transmission electron microscopy (TEM) in a Philips CM 200 operated at 200 kV and equipped with a DX4 X-ray energy dispersive spectrometer (EDS) and a parallel electron energy loss spectrometer 666 digi-PEELS. Thin foils samples for TEM observations were prepared from extruded samples by electro-polishing using a mixture of 26 vol.% nitric acid in methanol at around 248 K and 20 VDC.

3. Results and discussion

3.1. Mechanical properties

Fig. 1 shows the tension curves σ versus ε of the aluminum graphite microcomposites samples tested in the extrusion direction as a function of graphite content. All samples follow a milling–sintering–extrusion sequence, with exception of Al_p sample, which was only sintered and extruded. From this figure it is evident the increment of the resistance upholding the ductility in all the samples. Figs. 2 and 3 present σ_y and σ_{max} values found in the tension test; it is manifest the important effect of graphite content on the mechanical properties. Both, σ_y and σ_{max} present a positive slope as a function of graphite content. σ_y increased from 111.8 to 148.1 MPa which correspond to an increment of about 25.5%; and for σ_{max} the increment was around 30%. This is an important enhancement of the mechanical resistance, considering in addition that the ductility of the samples was kept, as it is shown in Fig. 1. The increment in the mechanical properties can also be pointed out remarking that σ_y in sample Al_{100} is approximately the same than σ_{max} found for Al_M (milled sample), and higher than σ_{max} found for Al_P (see Figs. 2 and 3). Fig. 4 shows the variation of σ_y for compression as a function of graphite content. It is clear that as the graphite content increases, σ_y increases as well; the exception was sample with 0.5 wt.% C, which showed blistering on the surface, this affected the mechanical properties, also observed in tension test results (see Figs. 1–3). In this test σ_{max} was arbitrarily evaluated at a strain of $\varepsilon = 0.1$ for all microcomposites, due to the fact that the maximum resistance in tension were found around this ε. Comparing σ_y and σ_{max} values found in both, tension and compression test (Figs. 2–5), it is observed that compression values

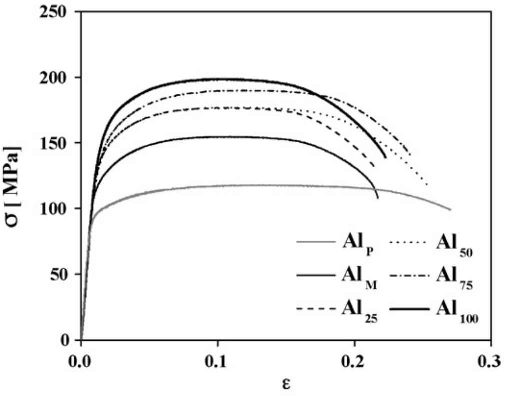

Fig. 1. Tension curves σ vs. ε for all samples tested in the extrusion direction.

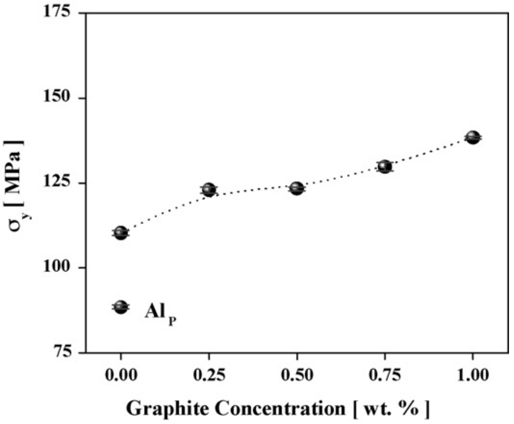

Fig. 2. σ_y as a function of graphite contents.

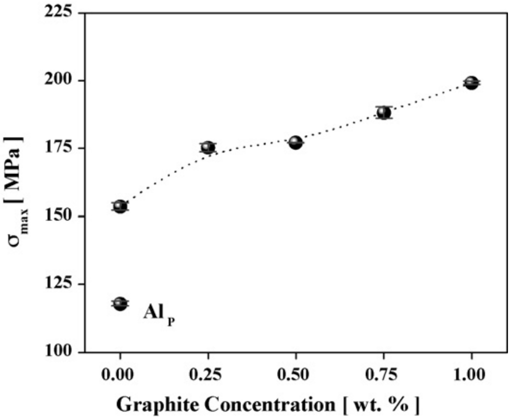

Fig. 3. σ_{max} as a function of graphite contents.

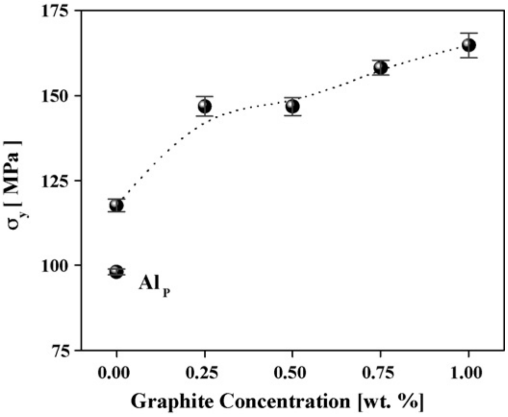

Fig. 4. σ_y as a function of graphite contents, for compression test.

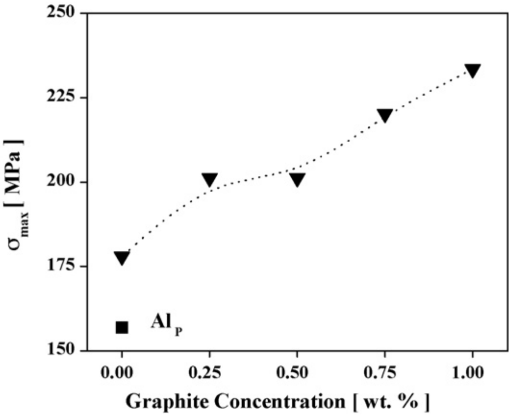

Fig. 5. σ_{max} as a function of graphite contents, σ_{max} arbitrarily taken at $\varepsilon = 0.1$ from compression results.

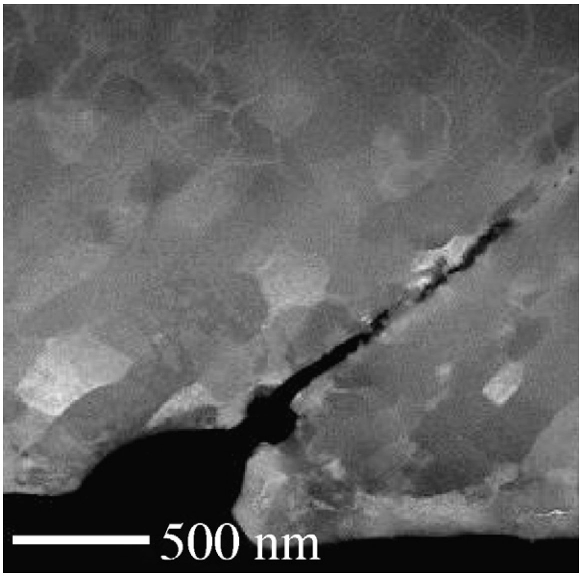

Fig. 6. TEM micrograph from pure aluminum (Al$_p$ sample). Notice the partial recrystallization.

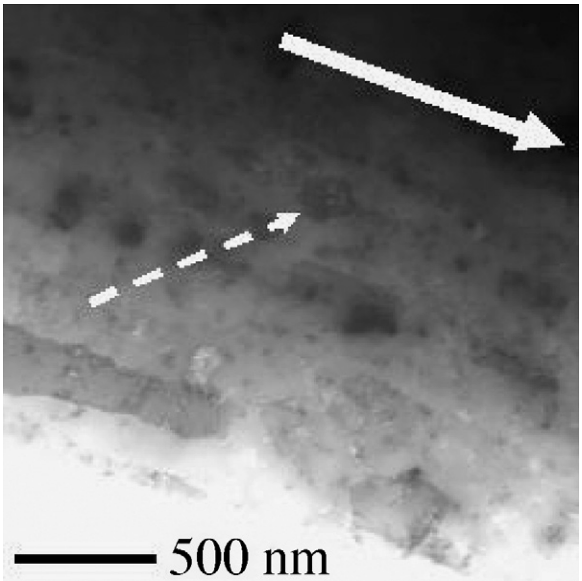

Fig. 7. TEM micrograph from Al$_{75}$ composite. An incipient recrystallization is beginning in boundaries of deformed grains.

are 10–15% higher than those found in tension test; this could be explained by the positive variation of transversal area during compression test.

3.2. Microstructure

Fig. 6 shows a representative microstructure found in Al$_p$ sample in the as-extruded condition. It was observed the pres-

Fig. 8. Nanoparticles found in Al$_{100}$ composites. Particles size is lower than 100 nm.

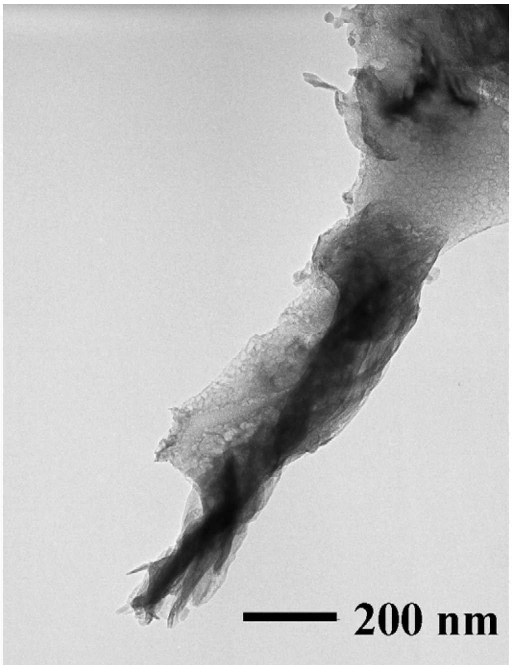

Fig. 9. Al–O–C nanofibers found in Al_{100} composite. Nanofibers present amorphous structure.

ence of both, equiaxial and deformed grains, due to partial recrystallization during hot extrusion. Fig. 7 shows a characteristic view of Al_{75} sample in the as-extruded condition. It can be observed the existence of deformed grains in the extrusion direction (white arrow). Additionally, an incipient recrystallization is beginning in boundaries of deformed grains, shown by dashed arrow. An analysis of Figs. 6 and 7 suggests that graphite additions increase the recrystallization temperature (Tx); a deep characterization in Tx variation as a function of graphite concentration is being carried out. Fig. 8 shows a typical view of reinforcement nanoparticles, which present sizes around to 50 nm and distributed at random. H.T. Son reported before [8] the existence of a bad adhesion between graphite micrometric particles and aluminum matrix. However, because of the nanosize of the particles used in the present work, the adhesion of the particles was excellent; any evidence of an additional phase was not found. As well to nanoparticles, TEM observations detected the existence of nanofibers (Fig. 9); microanalysis on these nanofibers showed the presence of Al, O and C. Diffraction analysis has shown that nanofibers present amorphous structure. A characteristic nanofiber of about 800 nm long and diameter lower than 200 nm found in Al_{100} composite is presented in Fig. 9.

From these results, it is evident that mechanical milling process is an excellent tool to disperse and incorporate nanoparticles into metallic matrix. Furthermore, compression forces during hot extrusion and thermal contraction during cooling helps the nanoparticles adhesion into the aluminum matrix. Graphite nanoparticles are minimally intrusive and offer the promise of get better mechanical properties without sacrificing structural integrity. Aluminum–C composite could therefore advance the state-of-art in the field of composite materials. These nanoparticles and nanofibers are the responsible of the excellent mechanical properties found in composites Al–C. It is known that the size of the particle (or fiber) plays a very important role on the reinforcement mechanism; smaller particles provide greater strength.

4. Conclusions

Aluminum–graphite composites prepared by mechanical milling followed by hot extrusion showed excellent mechanical properties. The mechanical resistance of the composite increased by more than 30% as the graphite content increased only 1 wt.%. It is worthwhile to notice that the increment of the resistance take place preserving the ductility of all the samples. The nanoparticles and nanofibers observed in extruded samples by transmission electron microscopy seemed to be responsible of the reinforcement phenomenon. Also, TEM results suggest that Al–C composites showed a higher recrystallization temperature than pure aluminum. Finally, these outcomes also establish that mechanical milling process is an excellent tool to disperse and incorporate nanoparticles into metallic matrix.

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