# Microstructural characterization in AI-C-AI<sub>2</sub>O<sub>3</sub> Composites Produced by Mechanical Milling.

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#### Abstract

Aluminum-graphite-copper (AI-C-Cu) novel micro-composites have been produced using the Mechanical Alloying process (MA). The mechanical properties of the obtained composites have been evaluated. Yield strength ( $\sigma_y$ ) values reached in the composites are considerably higher than those reported for pure aluminum. There is a direct relationship between  $\sigma_y$  and final graphite content in the composite.  $\sigma_y$  values increase as the nominal C content increases as well. We found that the most important hardening element was the graphite. We found that the optimal ratio Cu/C correspond to 0.33% for different volume fractions of graphite and cooper. There is an apparent synergy effect in  $\sigma_y$  between Cu and C. Results of TEM analysis have shown the presence of alumina particles in fiber shape from oxide surface of powder. Presence of alumina fibers in the composite improves the mechanical properties.

Keywords: Aluminum-based composites, AI-C, mechanical properties.

#### 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 because their relative low yields stress. Recently, the need of increase the aluminum strength for applications in aerospace and aeronautics industries, has motivated the study of



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Aluminum matrix composites which present excellent mechanical properties at both, elevated (473 K) and room temperatures, being one of the most important characteristic the specific stiffness, but maintaining the low density [1-4]. The alloys can be strengthened by dispersing hard particles like carbides, oxides or nitrides into the aluminum matrix by using techniques in solid or liquid state [5-7]. The aluminum composite can be fabricated in solid state through the Powder Metallurgy (PM). The PM is employed due their great versatility and low cost of production. The process of fabrication with the metallic powders followed for the consolidation and sintering processes. Another way to produce composite materials is by using Mechanical Alloying (MA) or Mechanical Milling (MM) process before the consolidation and sintering processes. By using MA process it is possible to produce amorphous phases, quasicrystals, nanocrystals, and supersaturated solid solution among others [8-16]. Therefore, the process is expected to be useful for development of novel materials.

In this work we have studied the effect on the mechanical properties of microcomposites produced by the addition of graphite (previously milled with Cu) into and aluminum matrix. Several composited of C-Cu and different ratios of AI:C-Cu were used in the preparation of aluminum-base composites. An analysis of the microstructure and yield stress ( $\sigma_y$ ) variation is presented and discussed as a function of chemical composition.

#### Experimental

The raw materials were powders (99.5% purity) and pre-milled metal-coated graphite. Several mixtures AI:C-Cu were employed to produce the composites. Each one was mechanically milled in a high energy Spex mill during 1 h. Argon was used as



milling atmosphere. Devices and milling media used were made from hardened steel. The milling ball to powder weight ratio was 5 to 1. The sample weight was 5 g. Because of the short milling times employed; the use of process control agent was not necessary.

Consolidated products were obtained by pressing during two minutes at ~950 MPa in a uniaxial load. Consolidated samples were pressure-less sintered during 3h at 823 K under vacuum. Microstructural observations by using optical microscopy and scanning (SEM) and transmission electron microscopy (TEM) were performed. Compression stress of the composites was measured using an Instron testing machine at room temperature at a constant displacement rate of 0.008 mm/s. Yield stress was measured at elastic limit. The ratio h: $\Phi$  (height to diameter) used was 2.0 in accordance with ASTM standards.

#### **Results and Discussion**

Mechanical properties:

Several microcomposites with different compositions were evaluated on their mechanical properties. Figure 1 shows the results of the compression test after sintering. This Figure shows the variation of  $\sigma_y$  as a function of the graphite content keeping constant the copper content. In the majority of the cases an important graphite hardening effect was evident; this behavior has been reported before [17-18]. Additionally, an important effect in  $\sigma_y$  produced by copper content was not observed. Samples having high graphite content (0.3 wt %C) show brittle behavior as evidenced by the compression test. However, in the group of samples having the graphite content



constant (Figure 2), and increase in the mechanical properties was observed as well as the ratio Cu:C was increased. The most important hardening effect was found at Cu:C ratio of 0.33.



Figure 1. Yield stress as a function of nominal graphite concentration in base composites. Figure 2. Yield stress as a function of ratio Cu:C in base composites.

Apparently, low content of copper increase  $\sigma_y$  keeping in some cases the ductility of the composite. For the majority of the results the strengthening effect of the copper is because the copper is dissolved into the aluminum matrix and its effect is by solid solution.

We suppose that C is present in both solid solution and dispersed into the aluminum matrix, but at this moment is not confirmed yet. Also, the presence of low content of aluminum carbides probably was produced during the sintering process; for this, a deep characterization is course to search the graphite and copper distribution and incorporation into the aluminum matrix.

**Micrstructure:** 



The as-milled powders showed a heterogeneous particles size distribution (from ~20 to ~300µm), as shown in Figure 3 (milled and compacted sample, not sintered). Typical ductile behavior was observed in the as-milled powders.

This figure also shows a representative view of the microstructure of aluminumbased composite in the as-sintered condition; after the heat treatment, recrystallization and grain growth are evident; additionally, the lamellar structure has disappeared. Figure 4 shows the presence of small pores, typically present in sintered products; however the size of the pores is lower than 10 µm,





**Figure. 3.** SEM micrograph of as-milled powders, showing particle size distribution and a typical ductile behavior.

**Figure 4.** SEM micrograph of as-sintered based composite, showing the grain size and the presence of small flaws.

During TEM characterization the presence of Al<sub>2</sub>O<sub>3</sub> nanofibers was observed as evidenced by EELs analysis [19]). Figure 5 shows a representative view of the alumina nanofibers found in the based microcomposites produced in the present work. Nanofibers show asymmetrical shape and irregular surface, and dimensions of ~19-20 nm in thickness and ~200-300 nm in length.





Figure 5. TEM Micrograph of as-sintered powders showing the alumina nanofibers.

 $AI_2O_3$  nanofibers from the oxidized surface of raw powders are present and contribute to the increase of the mechanical properties of the matrix [20]. We expect that the amount of  $AI_2O_3$  fibers is constant in all samples, therefore the effect on mechanical properties is the same in all composites.

## Conclusions

By using Mechanical Milling we have produced AI-C-Cu-Al<sub>2</sub>O<sub>3</sub> composites. There is a combined hardening effect between copper and graphite; however, only the graphite has a positive effect on the mechanical properties in all composited studied. The most important hardening effect was found at Cu:C ratio of 0.33. The main hardening element in these composited was graphite. Apparently Al<sub>2</sub>O<sub>3</sub> nanofibers have a constant effect on the mechanical properties.

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