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TEM and EELS Characterization in Al-C-Cu-Al_2O_3 Composites

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Introduction

Aluminum-based metal matrix composites (MMC) reinforced with ceramic particles are demanded because of their low density and high specific stiffness . Dispersion strengthened materials belong to the group of composite materials, which are made mainly by the techniques of Powder Metallurgy (PM). Their microstructure is composed by a polycrystalline matrix, in which dispersed particles are incorporated (mainly oxides, carbides and nitrides). The Mechanical Alloying (MA) and Mechanical Milling (MM) processes have been widely recognized as alternative routes for the formation of metastable phases for selected applications.

On the other hand, the interactions between the hardening particles and the matrix include atomic-level effects that are responsible for the new and novel properties of composites. By using Electron Energy Loss Spectroscopy (EELS), it is possible to characterize the materials from the electronic point of view.

The raw materials were Al powders (99.5 % purity, mesh -325) and pre-milled graphite with Cu. The mixtures of pure Al, Al-C, Al-Cu and Al-C-Cu were employed to produce the composites. Each one was mechanically processed in a high energy SPEX mill for 4 h. Argon was used as the milling atmosphere. Consolidated samples were pressure-less sintered for 1 h at 823 K under vacuum (~ 1 Torr).

Figure 1 shows a typical HRTEM micrograph of the Al-C-Cu composite in the as milled condition. Graphite nanoparticles are present in the Al matrix whose dimensions are ~ 2 nm in thickness and ~10-15 nm in length. Figure 2 shows typical nanofibers found in the Al-C-Cu composite in the as-sintered condition. Nanofibers show asymmetrical shape, irregular surface and the dimensions are ~20-40 nm in thickness and ~200-300 nm in length. During the sintering process Al react with O and form Al_2O_3 which present a fiber shape.

Additionally to HRTEM characterization, EELS analyses were carried out for all Al-based composites. Figures 3 and 4 show the Al-K ionization edge for metallic Al, Al-C and Al-C-Cu composites in the as-milled and sintered condition respectively. Figure 3(b) and (c) present changes with respect to metallic Al, 3(a). It is expected C have two routes of reaction during milling and sintering process, one of them is a preferential reaction with oxidized shell in aluminum powders and the second one is the reaction with aluminum matrix to crystallize Al_3C_4. By comparing Figures 4(a) and 3(a), was found that Figure 4(a) presents Al_2O_3 presence which is not perceptible in Figure 4(a), this means Al and oxygen still react during sintering process to crystallize Al_2O_3. Analyzing Figure 3(b) and 4(b) were found differences in the Al-K ionization edge of Al-C components, these differences could be the result of changes in Al_2O_3 structures. An analysis by HRTEM, developed on the alumina nanofibers in the as-sintered samples revealed that alumina is Al_2O_3-κ type. We assume that the alumina formed during the milling is Al_2O_3-α and it is transformed in the metastable Al_2O_3-κ.

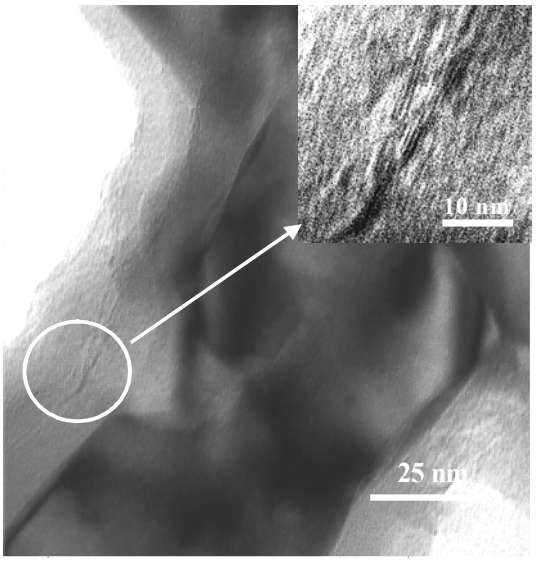

Figure 1. HRTEM image from an Al-C-Cu composite in the as-milled condition showing the shape of carbon nanoparticles.

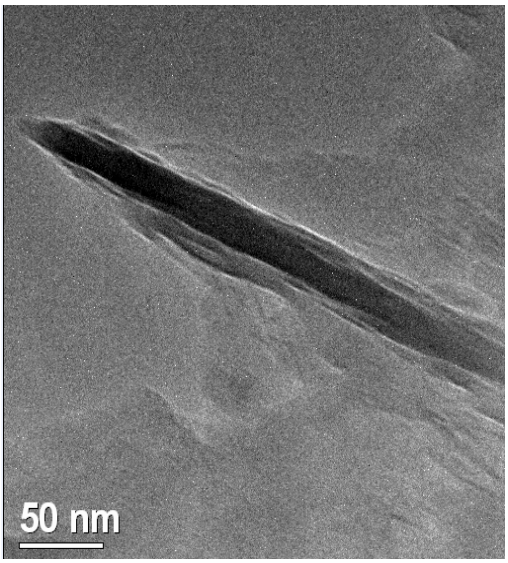

Figure 2. TEM image from an Al-based Composite showing the distribution and shape of Al_2O_3 nanofibers.

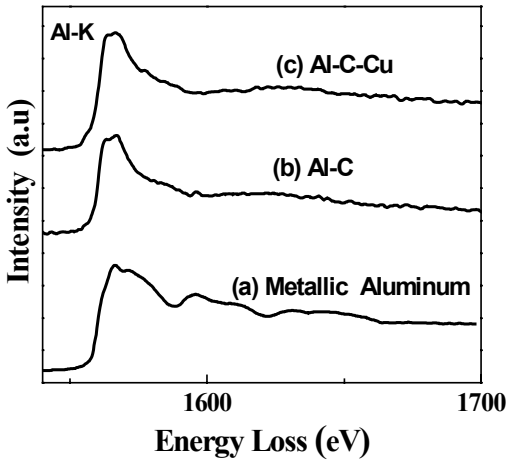

Figure 3. EELS spectra from Al-K Ionization edges in Al-C, Al-C-Cu composites and metallic aluminum in the as-milled condition.

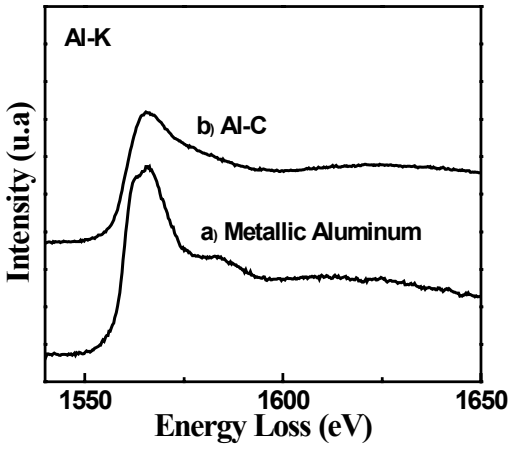

Figure 4. EELS spectra from Al-K Ionization edges in Al-C composites, metallic aluminum in the as sintered condition.

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