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Study of Al_2O_3 Nanofibers Dispersed in Al Matrix by EELS and Calculations Ab Initio

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The necessity for improving the mechanical properties of aluminum alloys has motivated the study of Al base composites [1]. Mechanical Alloying process is employed to produce hardened composites to introduce reinforcing particles; this process is able to produce several phases including supersaturated solid solutions, metastable phases, amorphous phases, as well as reinforced metal-particle composites [2]. Using techniques like Electron Energy Loss Spectroscopy (EELS) and Transmission Electron Microscopy, it is possible to characterize this kind of materials. Recently, very important advances have been achieved in the description of the crystalline solid electronic structure through numerical calculations. There is a great diversity of available codes for DFT calculations, among them are the CASTEP code (pseudopotentials) and WIEN2k code, two available commercial programs that offer the possibility of ELNES calculations. ELNES provides details of the atomic local environment, coordination, bond type and valence states. The ELNES calculation can be performed with an energy resolution much better than those obtained by experiment.
In this work, Al_2O_3 nanofibers were synthesized using Al powders (99 % pure) and C as raw materials. A mixture of Al powders at 75 wt. % and C powder at 25 wt. % was used to produce the compound. The mixture was mechanically processed in a high energy mill (Spex) for 4h and the product was compacted at ~950 MPa of pressure. The consolidated samples were sintered for 2h at 550°C. The characterization was carried out by Transmission electron microscopy (TEM) and electron energy loss spectroscopy (EELS).

A TEM analysis in the 0.75% C sintered composite revealed the presence of alumina nanofibers in the Al matrix. The alumina nanofibers show dimensions near to ~10-20 nm thickness and ~200-300 nm length (Figure 1). During the milling and the sintered processes, the alumina undergoes a phase transition when the graphite is present in the Al matrix [5]. Figure 2 shows the ELNES Al-K (experimental and calculated) in the as milled condition pure Al. In agreement with the analysis, the milled condition for pure Al reveals the presence of α-Al_2O_3 particles. However, the ELNES Al-K (Figure 3) shows that the Al-C composite in the milled condition, presents $\delta-Al_2O_3$ particles (metastable). On the other hand, The Al-C composite in the sintered condition reveals the presence of $\kappa-Al_2O_3$ (metastable). Apparently, the alumina particles undergo a phase transition and grow up during the sintered process until they take a shape of alumina nanofibers (See Figure 1).

With the Mechanical Milling technique is possible to produce hardened composites by dispersion of stable and metaestable phases. By using numeric calculations in the EELS spectra interpretations, is possible the characterizations of particles of nanometric size. It was found that the alumina is α in the milled pure Al, type δ in the Al-C powder composite and type κ in the sintered Al-C composite.

References

[1] G. Abouelmagd, J. Mat. Proc. Tech. 155–156 (2004) 1395–1401.

[2] M. Besterci1, O. Velgosová, L. Kováč, F. Lofaj, Mat. Sci. 9 (2003) 22-26.
[3] H.T. Son, T.S. Kim, C. Suryanarayana, B.S. Chun. Materials Science and Engineering. A348 (2003) 163-169.
[4] A. Santos et al. Journal of Alloys and Compounds (2007) 434-435.

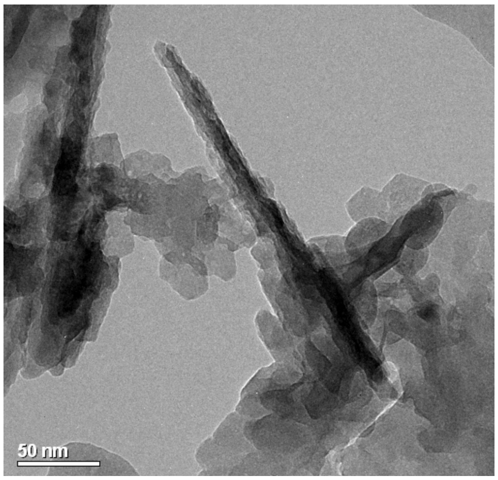

Figure 1. Alumina nanofibers of the Al-0.75%C composite.

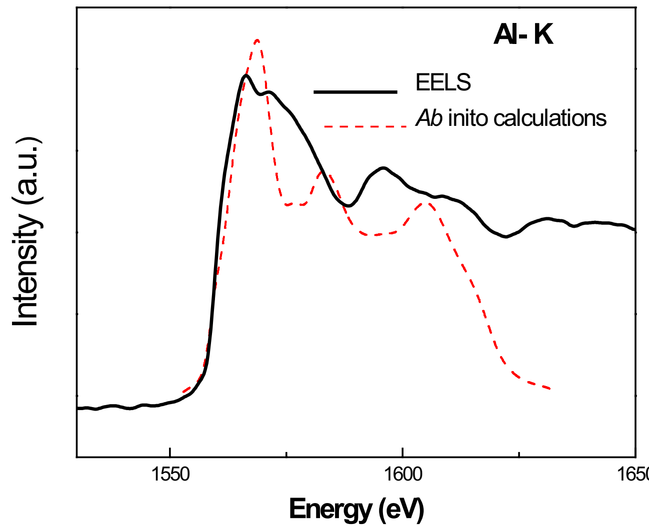

Figure 2. Experimental and calculated Al–K ionization edge of pure Al (Al2O3 -α)

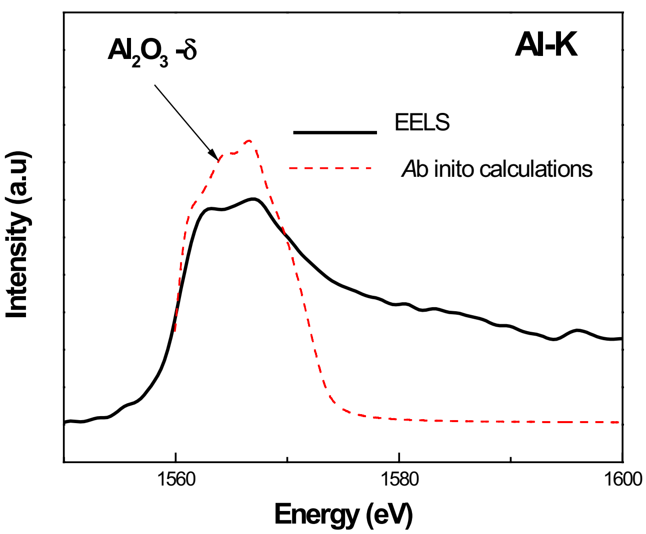

Figure 3. Experimental and calculated Al–K K ionization edge non sintered Al-C (Al_2O_3–δ)

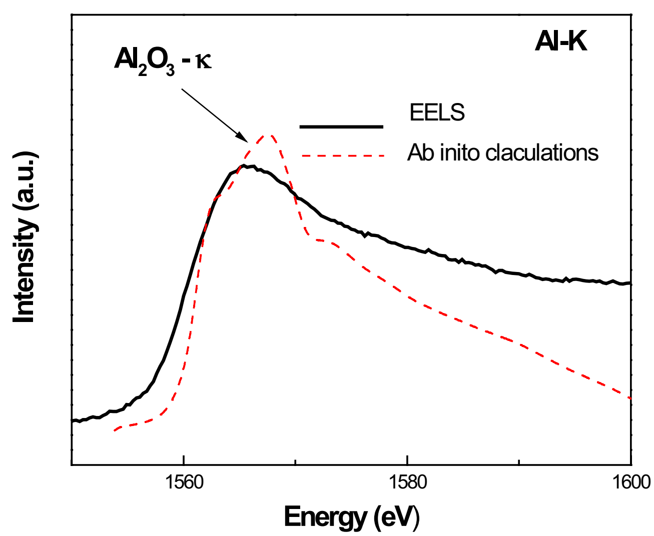

Figure 4. Experimental and calculated Al– ionization edge sintered Al-C (Al_2O_3 -κ)