ELNES of Al-Al4C3 Nanoparticles Produced By Mechanical Milling

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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 introducing 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]. The reinforcing particles Al4C3 have become an interesting reinforcing material because their high level of physical and mechanical properties, e.g. high temperature strength, thermal cyclic resistance, wear resistance and low linear expansion coefficient [3]. Therefore, the reinforcement of the aluminum using Al4C3 has recently become the subject of many studies and widely used for products and structures. Using techniques like Electron Energy Loss Spectroscopy (EELS) and Transmission Electron Microscopy, it is possible to characterize this kind of materials. The advantage of EELS is its very high spatial resolution down to the nanometer level, allowing the study of precipitates, grain boundaries, internal interfaces and other atomic interactions by analyzing the Near Edge Fine Structure (ELNES). Recently, very important advances have been achieved in the description of the crystalline solid electronic structure through numerical calculations. A great diversity of available codes for DFT calculations are available, 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, atomic coordination, bonding type and valence states. The ELNES calculations can be performed with an energy resolution higher than those obtained in other experimental procedures.

In this work, Al$_4$C$_3$ particles were synthesized using Al powders (99.5% pure) and C as raw materials. A mixture of Al powders at 75 wt. % and C powder at 25 wt. % was employed to produce the compound. The mixture was mechanically processed in a high energy mill (SPEX) for 4h. The mixture was compacted at ~ 200 MPa of pressure. The consolidated samples were sintered for 2h at 550°C. The characterization was carried out by X-ray diffractometry, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). EELS spectra were obtained with a PEELS spectrometer attached to the TEM. The experimental EELS spectra were compared with ab initio calculations using the Wien2k code to determine the Near Edge Fine Structure (ELNES) of Al$_4$C$_3$ nanoparticles processed in solid state by MM [4, 5].

X-ray diffraction pattern of the Al-C sintered powder is shown in Figure 1, the pattern shows the Al$_4$C$_3$ formation and the presence of Al$_2$O$_3$-$.\gamma$. Alumina type Al$_2$O$_3$-$.\alpha$ comes from the oxide layers of Al powders and it was introduced in the Al structure during the mechanical milling process, which is transformed to Al$_2$O$_3$-$.\gamma$ during the sintering processes [6]. A homogeneous composition is observed in the backscattered image from SEM in Figure 2 and a diffraction pattern SAD analysis in the Al-C sintered powder revealed a nanocrystalline state and the aluminum carbide formation
according with the indexed diffraction pattern (Figure 3). Figure 4 shows the ELNES C-K (experimental and calculated) of the Al-C sintered powder. From these results it is evident the presence of the Al and C elements. Additional information concerning details of the specific bonding can be found from EELS spectra near the ionization edge, as the ELNES features are substantially influenced by bonding states. In Figure 4 the measured fine structures of C-K ionization edges exhibits the presence of two characteristics peaks which correspond with the Al$_4$C$_3$ compound as can verified if compared from literature [7].

References


Figure 1. X-Ray diffraction pattern from the sintered mixture Al-C 4h MM and 2h sintering.

Figure 2. Backscattered image from the sintered mixture Al-C 4h MM and 2h sintering.

Figure 3. TEM bright field image and SADP from a particle of the sintered mixture Al-C.

Figure 4. Experimental and calculated C-K ionization edge.