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EELS Investigation of Al_2O_3 at 30 keV and below; First Results of Alumina's Structural Sensitivity to a Low-Energy Electron Beam

Takeshi Sunaoshi[1], Manabu Shirai[1], Satoshi Okada[2], Kazutoshi Kaji[2] and Edgar Voelkl[3]

[1.] Hitachi High-Technologies Corp., Application Development Department, Ibaraki, Japan.
[2.] Hitachi High-Technologies Corp., Electron Microscope Systems Design 1st Dept., Ibaraki, Japan.
[3.] Hitachi High Technologies America, NSD, Clarksburg, Maryland, USA.

Alumina or Al_2O_3 is a material used in many applications. Its typical occurrence is polymorphic α-Al_2O_3, but its cubic γ- variety has important technical applications. Because alumina is inherently an insulator, chemical and electronic modifications occur due to radiation effects especially at high-energy defect structures like grain boundaries [1-3]. Because of the many varieties of Alumina (γ, η, θ, am phases), Alumina can have many properties. One way of distinguishing its many phases is to investigate its ELNES features as EELS is sensitive to the arrangement and bonding of the nearest neighbors.

We selected for our investigation γ-Al_2O_3, following up earlier work described in [3]. In this work, the Al-L_{23} edge was investigated with 300keV electrons, where the nominally Al^{3+} cation is surrounded by nearest neighbor Oxygen anions within a tetrahedral (*T*) or octahedral (*O*) configuration leading to the location of their EELS peaks at 78.2 eV and 79.8 eV respectively both verified experimentally and via first principle calculations [4]. The location of the α-Al_2O_3 L_{23} peak at 79.1 eV was not seen as a significant contribution in our initial data sets.

We now wanted to determine if the radiation damage can be reduced by lowering the energy of the electron beam to 30 keV and below, while maintaining an energy spread of the electron beam $<<$ 1 eV. This was possible by using Hitachi's low-voltage STEM/SEM microscope equipped with (also a Hitachi) EELS (Electron Energy Loss Spectrometer) [5], with a total energy spread of 0.37 eV, see Figure 2, left.

The initial electron energy used was 30 keV and we investigated the appearance of the Al-L_{23} edge as a function of the relative thickness t/λ (with λ the effective mean free path) of the sample. The BF-STEM image shown in Figure 1, left, indicates the three spots selected for EELS acquisition. The corresponding EELS data are shown in in Figure 1, right, and, unsurprisingly, indicate that a good thickness range would be $t/\lambda \leq 0.5$ also for 30 keV electrons. We expect to find the same behaviour for even lower electron energies.

In order to track the impact of the electron beam on alumina we observed the Al-L_{23} edge over time at a new location with a relative thickness of $t/\lambda = 0.26$. Clear damage to the sample area could be observed for both the octagonal (*O*) as well as the tetragonal (*T*) configuration around the exited Al^{3+} within less than 1 s. Results at even lower electron energies will follow, hoping to reach an experimental setup that causes little to no damage to Alumina.

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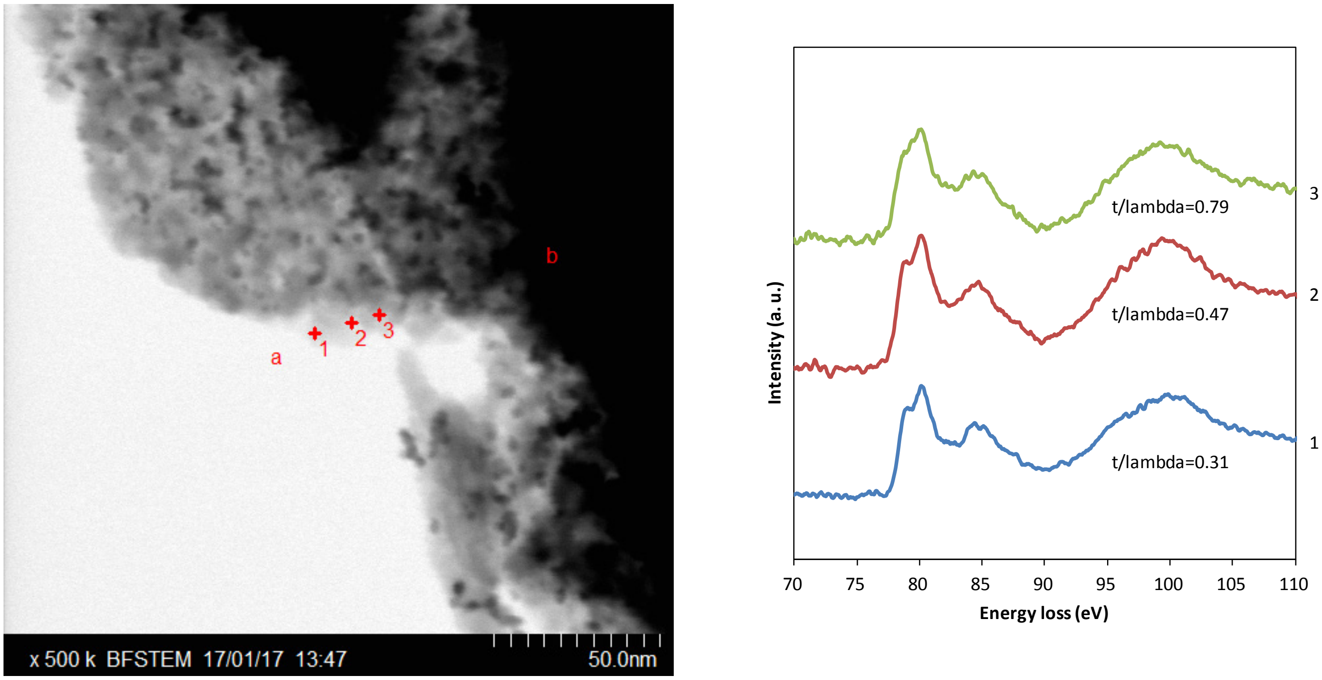

Figure 1. Left: BF-STEM image at 30 keV with markers indicating locations for EELS acquisition. Right: Al-L_{23} edge shows a clear distinction between the tetragonal and octagonal γ-Alumina due to the low energy spread of 0.37 eV due to the cold FEG. The current on the sample was 320 pA.

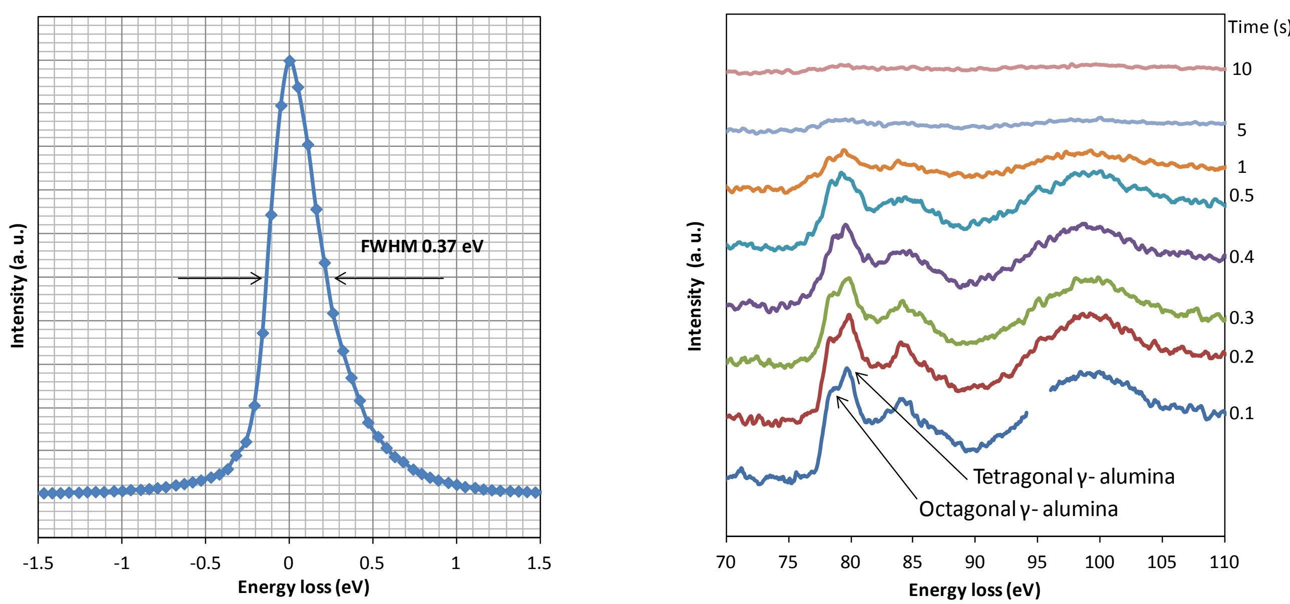

Figure 2. Left: energy width of the ZLP peak was measured at 0.37 eV at 30 keV and 320 pA. Right: EELS spectrum of γ-Al_2O_3 indicating the octagonal (78.2 eV) and tetragonal (79.8 eV) configuration around theAl^{3+}. Also apparent is the rapid disintegration of structure under the electron beam over time.

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