EELS Analysis of Bulk Plasmon Harmonics of Aluminium at 30 keV

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EELS is especially useful for the detection of light elements, since these elements have ionization edges of low energy[1,2]. High-energy STEM-EELS has been used for the positive identification of lithium in lithium-copper precipitates in Al-2099, as a complement to low energy back-scattered electron (BSE) microscopy and X-ray energy dispersive spectroscopy (EDS)[3].

This paper shows how plasmon resonances of a precipitate-rich aluminium alloy (AL-2099), have been studied using a low-energy scanning transmission electron microscope (STEM), a Hitachi SU-9000EA, operating at a beam energy of 30 keV. The microscope is equipped with a dual mode electron energy loss spectrometer (EELS). The first mode is a 1024 bin CCD array, for recording conventional EELS spectra. The second mode consists of three diodes, each covering a broad area of the EELS spectrum. This second mode is used to gather energy filtered images (EF-STEM) of the sample.

The inelastic mean free path of an electron traveling through matter is inversely dependent on the electron's kinetic energy[1]. Therefore, the intensity of the plasmon peak of the EELS spectrum of a material will increase with lower beam energy. Beyond a certain thickness, individual electrons will lose energy due to multiple events (plural scattering). This is most easily seen as a repeating peak in the low-loss region of the EELS spectra. These repeating peaks are also known as harmonics of the plasmon peak[1]. Figure 2 and 3 are good examples of such Plasmon harmonics in thick sections of aluminium alloy.

In aluminium, the bulk plasmon peak has an energy of roughly 15 eV, while a surface plasmon is expected to be found at energies between 4 and 7 eV, near the edges of specimens[4]. By placing the EF-STEM windows in a manner so that the zero-loss peak (ZLP), the surface plasmons and the bulk plasmon each occupy their own energy window, fast, high resolution, high contrast images are obtained. Figure 3 shows four images simultaneously acquired using, the high angle annular dark field detector in the case of a) and the EF-STEM diodes for b), c) and d). Each of the EF-STEM images emphasise different kinds of precipitates. The surface plasmon mas enhances the contrast of the δ ' precipitates, while the bulk plasmon map enhances the contrast of T1 precipitates. The advantage of this technique is the speed at which these EF-STEM images can be acquired. Figure 3 was acquired in under 15 seconds. The alternative method for producing such images in a STEM is through EELS mapping. STEM-EELS require the acquisition of a full spectrum per pixel, which, for a 512 by 512 images, is both time and data consuming.

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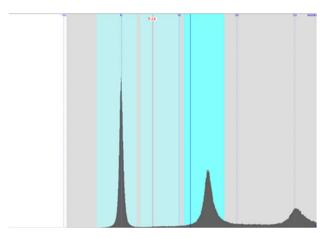


Figure 1. Spectrometer condition used for the acquisition of the images in Figure 2, with the strong bulk plasmon at 15 eV, and the less intense harmonic at 30 eV of energy loss.

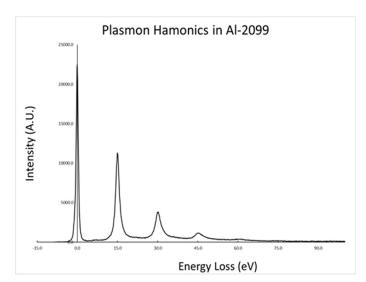


Figure 2. EELS spectra of Al-2099 in a thick region, showing up to the fourth plasmon harmonic, at 60 eV.

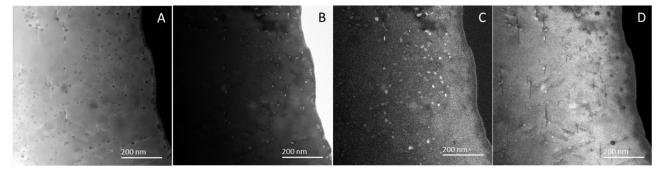


Figure 3. Images of Al-2099 acquired at 30 keV, showing a) high angle annular dark field, b) the ZLP intensity c) the surface plasmon and d) the bulk plasmon peak.