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High Spatial Resolution Quantification X-ray Microanalysis in a Field Emission Scanning Electron Microscope with an Annular Silicon Drift Detector

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The scanning electron microscope (SEM) was primary developed for imaging applications. With the introduction of the Si(Li) energy dispersive spectrometer (EDS), simultaneous imaging and x-ray microanalysis became possible. However, long working distance and high current were needed because the position and small solid angle of the EDS detector. SEM was initially and is still optimized for imaging applications, where the high spatial resolution is generally obtained at short working distance. This problem is still relevant today and unfortunately x-ray microanalysis is never performed in the best imaging conditions, i.e., not with the smallest probe size. With the introduction of an annular silicon drift detector (SDD) system, scanning electron microscopy is facing a revolution. This detector is inserted below the objective lens which gives a higher solid angle (up to 1.2 sr). In consequence, a lower working distance and probe current can be used. An improved spatial resolution becomes possible during x-ray microanalysis.

For quantification microanalysis where the absorption of the x-ray is important, the value of the takeoff angle is important. Lower value increases the absorption in the sample. Also current correction models, suppose a fix value of takeoff angle. Because of the geometry of the annular SDD, the mean takeoff angle is 33 degree with a minimum of 24 and maximum of 50 degree at the optimum detector distance. However, preliminary results indicate that this large takeoff angle range does not affect the correction model when the absorption effect is small or moderate, i.e., using the mean takeoff angle gives accurate composition. Because of the position of the annular detector, Mylar windows are used to prevent the backscattered electrons (BSEs) to damage the SDD segments. The shape of the background is strongly affected by the window absorption at low x-ray energy. For accurate quantitative analysis, the calculation of peak net intensity depends on the background subtraction method used. However, for energy greater than 1 keV, the current background subtraction method seem enough with this annular SDD. An example of x-ray elemental maps of an Al-Mg diffusion couple acquired with annular silicon drift detector (SDD) at low accelerating voltage is shown in Figure 1. The accuracy of the quantification with an annular SDD was evaluated with the standardless [1] and f-ratio method [2,3]. The effect of this detector geometry and position on the correction model is currently studied.

The annular SDD with is larger solid angle will clearly revolution the quantification microanalysis by moving from point analysis to quantitative micrograph with simultaneous electron imaging. Also, since the count rate can be as high as 1,500 kcps with our system, which lower significantly the detection limit of elements as well the minimum feature sizes of different phases that can be distinguished as shown in Figure 2.

References:

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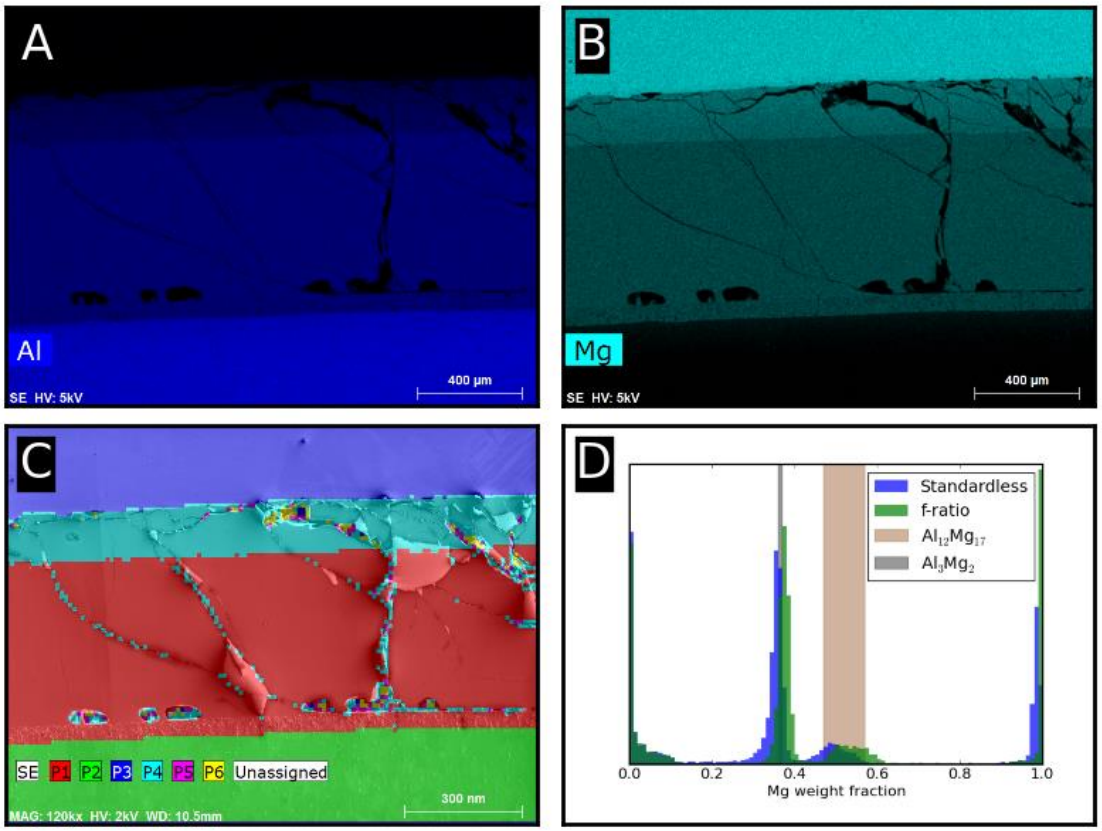

Figure 1. Quantitative x-ray maps of an Al-Mg diffusion couple sample at 5 kV. **A** and **B** shown the net intensity (with background subtraction) of Al and Mg, respectively. **C** Phase map obtained from the standardless quantification results. P3 is pure Mg, P4 $Al_{12}Mg_{17}$ phase, P1 Al_3Mg_2 phase, and P2 pure Al. The other identified components are from cracks filled with mounting resin. **D** Mg weight fraction pixel distribution calculated with standardless [1] and f-ratio [2,3] quantification methods. The phase diagram composition range of the two phases are also shown.

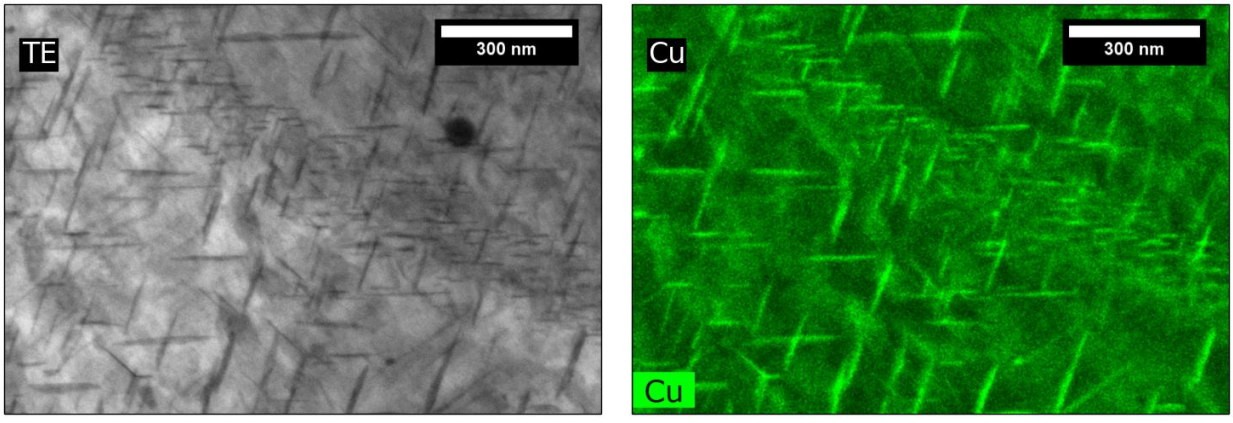

Figure 2. High spatial resolution x-ray elemental map of an Al-Li 2099 alloy at 20 kV in SEM/STEM mode. The T_1 (Al_2CuLi) palettes observed in the bright-field micrograph (TE) are clearly visible in the Cu map.

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