HIGH ANGULAR RESOLUTION MEASUREMENTS OF K SHELL X-RAY EMISSION CREATED BY ELECTRON CHANNELING IN THE ANALYTICAL ELECTRON MICROSCOPE*

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HIGH ANGULAR RESOLUTION MEASUREMENTS OF K SHELL X-RAY EMISSION CREATED BY ELECTRON CHANNELING IN THE ANALYTICAL ELECTRON MICROSCOPE

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Since the original observations by Duncumb in 1962, a number of studies have been conducted on the effects of electron channeling on characteristic x-ray emission and microanalysis. Most of the recent studies have concentrated upon using the phenomenon to perform site specific distributions of impurity elements in ordered compounds using the ALCHEMI methodology. Very few studies have attempted to accurately measure the effect as a function of orientation and compare these results to theory. In this study, two dimensional high angular resolution studies of channeling enhance x-ray emission were performed and herein the results are compared to theoretical calculations of Allen et al.

All experimental measurements presented here were conducted on a Philips EM 420T analytical electron microscope. The instrument was operated in the TEM mode, at 120 kV using an LaB6 electron source. The characteristic x-ray emission was measured using an EDAX ultra thin window Si(Li) detector having a FWHM of ~145 eV at Mn Kα. Nominal probe sizes used during the study were 200-500 nm with beam convergence half angle defined by the Condenser apertures. Control of the relative orientation of the incident probe was accomplished via direct computer control of the beam tilt coils, after the specimen was first manually oriented to an appropriate zone axis using the specimen tilt stage. Two dimensional measurements were carried out using a 128 x 100 pixel scan corresponding to an angular range of ~100 by 80 mR using customized computer program running on a EDAX 9900 microanalyzer system. Careful alignment and manual optimization/adjustments of beam tilt pivot coils, minimized probe wobble during data acquisition. The effects of this were additionally mitigated due to the relative uniformity of the specimen thickness in the analyzed zone. Typical acquisition times for a complete two dimensional scan were 18-24 hours. Essential to the success of these measurements was the stability of the probe current, minimal specimen drift, and absence of hydrocarbon contamination. The latter being accomplished using argon plasma processing of the specimen (MgAl2O4) which was initially prepared by mechanical tripod polishing.

In figure 1 we show line traces through a portion of one 2D data set along a <400> direction between the {012} and {013} zones axes for two difference convergence half angles for the Al Kα line. Figure 2 plots the scaled relative intensities of the O, Mg, and Al Kα lines at that same orientation. Figures 3 and 4 show corresponding theoretical calculations of these signals taken from the work of Allen et al. From figure 1 we can see that the beam convergence angle, as expected, makes a significant difference to the recorded angular profiles and only under the highest angular resolutions do the experimental measurements approach the theoretical calculations. By comparison of figures 2 and 4, we see that under moderate beam convergences (2.6 mR), typical of ALCHEMI conditions, the salient features are present, however, the fine structure is not faithfully reproduced.

It is expected that high angular resolution measurements, such as these, may provide an method to study elemental redistribution in ordered compounds caused by irradiation produced defects and additional work is in progress to investigate this application.

References:
Figure 1.) Experimental Variation in the Al Kα X-ray Emission as a function of orientation and convergence half angle (1.3, 5.2 mR)

Figure 2.) Experimental Variation in the O, Mg, Al Kα X-ray Emission as a function of orientation. Convergence half angle = 2.6 mR
Note: Vertical scales optimized for clarity.

Figure 3.) Theoretical Calculation of Al Kα X-ray Emission as a function of orientation (after Allen etal). Compare with data of Figure 1.

Figure 4.) Theoretical Calculation of O, Mg, Al Kα X-ray Emission as a function of orientation (after Allen etal). Note: Vertical scales optimized for clarity.

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