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Effect of Specimen Geometry on Quantitative EDS Analysis with Four-Quadrant Super-X Detectors

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In recent years, X-ray signal collection efficiency in energy dispersive X-ray spectrometry (EDS) has been significantly improved by incorporating state-of-the-art four-quadrant Super-X detectors in aberration-corrected microscopes [1, 2] and has enabled routine atomic-resolution EDS elemental mapping, as shown in Figure 1a. The breakthrough in detector technology has sparked interest in quantifying the chemical information directly from atomically resolved EDS maps [3-5]. In order to obtain an atomic-resolution EDS map, the specimen however must be tilted to a low index zone axis, resulting in different orientation with respect to each detector. This will inevitably result in different X-ray signals arriving at each detector due to their different absorption distances and effective take-off angles. Such a variation could largely affect the ability to accurately quantify the overall EDS spectrum from four Super-X detectors without precautions as evidenced in this work. Therefore, understanding the X-ray signal variation from each detector under tilted specimen condition becomes both experimentally and theoretically important as a starting point for atomic-resolution EDS quantification.

In this talk, we will show that the specimen geometry can have a strong effect of on quantitative EDS when using a four-quadrant Super-X detector configuration. Ni_3Al is an ideal prototype material to investigate geometry effects as the Ni and Al characteristic X-ray peaks are well separated and strong absorption of Al-K occurs by Ni [6]. As shown in Figure 1b, a wedge-polished Ni_3Al sample was studied using a Titan G2 S/TEM with the sample thin region facing to detectors 3 and 4 in an azimuth angle of 45 degrees. Figure 2a-c show the total intensity of Al-K, Ni-K and Ni-L X-ray signals received from each detector in the same thickness region, respectively. Special care was made to minimize the channeling effect from the specimen. As seen, X-ray peaks obtained from each detector not only vary with the tilt angle, but also systematically shift about 5 degrees towards the positive tilt angle. More importantly, a large deviation of intensity ratios of Al-K/Ni-K and Ni-L/Ni-K peaks from different detectors was observed with same specimen tilt as seen in Figure 2d-f. Furthermore, the ratio deviation also varies with the degree of specimen x-tilt, and appears to be non-symmetric, which is correlated with the wedge shape of the specimen. Although the deviation of X-ray signal from four Super-X detectors may be averaged out in the overall spectrum, significant change of the overall intensity ratio occurs at a higher tilt angle even larger than 10-15 degrees. In view of the large quantification uncertainty with the tilt sample, the correction of X-ray signal from overall spectrum for all Super-X detectors is essential and will be discussed. The limitation on tilt angle regarding the absorption, spurious X-ray and fluorescence effect in the Super-X detector configuration will also be presented. The effect of sample shape on the detected X-ray signals from Super-X detectors will be further compared for wedge, FIB and twin-jet polished specimens [7].

References:

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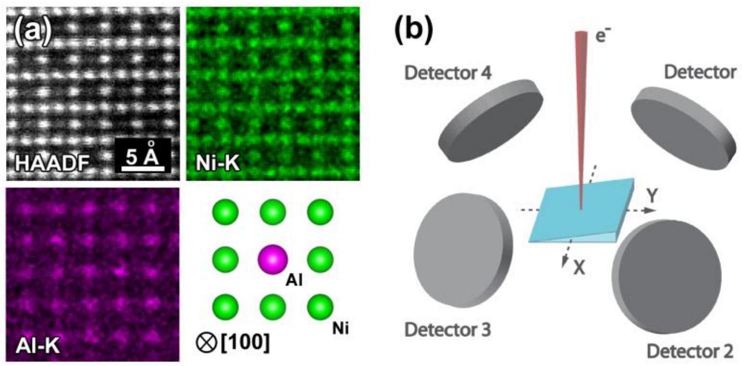

Figure 1. (a) HAADF image and the corresponding atomic-resolution EDS maps (Ni-K and Al-K) for Ni_3Al oriented along a [100] direction. The EDS maps are filtered using a 3 points average. (b) Schematic illustration of the geometric orientation relationship between the wedge-shape specimen and four-quadrant Super-X detectors.

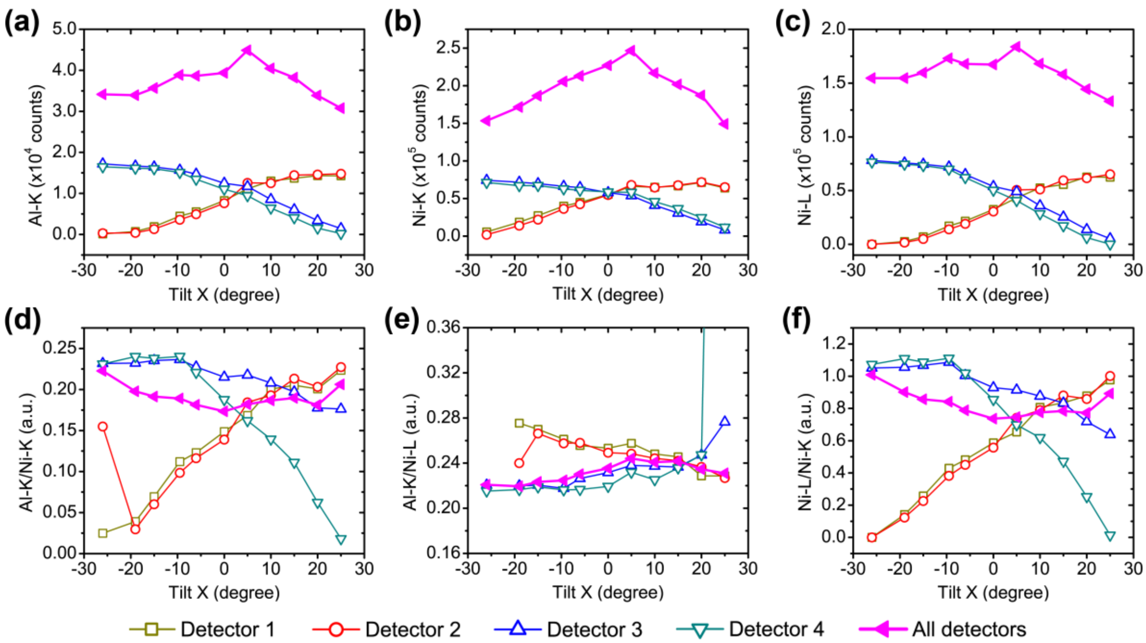

Figure 2. Deviation of X-ray signals from Super-X detectors in terms of (a) Al-K, (b) Ni-L and (c) Ni-K intensities, and their intensity ratio of (d) Al-K/Ni-K, (e) Al-K/Ni-L and (f) Ni-L/Ni-K. Such deviation also varies with the specimen x-tilt, and appears to be non-symmetric. All spectra were obtained from a wedge-polished Ni_3Al specimen in the same thickness region (0.5 mean free path from EELS) and under the same microscope condition (probe-corrected STEM with a 0.11nA and 14 mrad probe). The orientation relationship between the specimen and Super-X detectors is shown in Figure 1b.