Improving EDS for Light Elements using an attachable X-ray Optics with an SDD in SEM

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The silicon drift detector (SDD) technology has reached good resolution at low X-ray energies. But the analysis of light elements is still poor due to the insufficient production of low energy X-rays. To get more signal of the light elements, the beam current might be increased or the detector could be moved closer to the sample. But both would increase also the high energy counts and the dead time. Reducing the accelerating voltage one can only afford if no high energy X-ray lines are to be stimulated.

In this paper an X-ray optics is described, which significantly increases the detection sensitivity for low energy X-rays. The spectrum above 1.5 keV is not affected and the settings of the SEM will not change. The experiments are carried out with a detector optics LoMAX from Parallax Research Inc. attached to an e2v SDD equipped with an AP3.3 Moxtek window and a digital pulse processor from XIA LLC.

The optics works by total reflection, similarly to a shaped mono-capillary. It is a grazing incident X-ray optics, using grazing angles from about 6 down to 2 degrees. X-rays diverging into a large solid angle were captured by reducing that angle and thus directed towards the detector. The optics that I use has three reflecting surfaces in the shape of cones of revolution with different opening angles. X-rays, going from the sample into a large angle with respect to the detector axis hit the optics near the small entrance while those emitted into smaller angles hit the optics near the detector. But due to the different grazing angles within the optics they all fall onto the surface of the detector. The reflecting surface is polished nickel which is a very good reflecting material for the examined energy range.

The optics is attached to the SDD with a ring clamp adaptor (Fig. 1, 2). Alignment has to be done carefully so that the optics axis passes through the X-ray emitting point. A deviation of 200 μ m leads to a drop down of the X-ray signal. Therefore the precise adjustment of the working distance of the SEM is necessary. The optics is designed for a distance of 4 mm from the X-ray emitting sample to the optics entrance aperture. Motions along the optical axis produce less effect than changes in stage height.

Fig. 3 shows the measurements of a manganese standard sample with the detector at the same location both with and without the optics. The intensity of the Mn-K line is 8% lower with the optics attached due to the difficult adjustment of the working distance. But the gain of the optics at the Mn-L lines is clearly visible and there are no spectrum artefacts. The measurements on a standard of CaCO₃ with the optics in the right position result in a unity gain for the X-rays of the Ca-K line which are passed directly through the optics without reflection, and a gain as high as 8 to 10 for the low energy X-rays of Oxygen and Carbon (Fig. 4). The measurement of an oxidized Beryllium foil shows maximum gain at the lowest energy detectable with an EDS detector (Fig. 5). The intensity of the Beryllium line is strong even at high accelerating voltage of 20 kV (Fig. 6). Thus it is possible to detect low and high energy X-ray lines with similar sensitivity.

To work with these X-ray optics with high gain at low energies it is important to use an EDS detector and pulse processor with short shaping time. Otherwise the pile-up effects will disturb the spectrum, since no pile-up rejector works in the low energy region.

The described X-ray optics is an appropriate tool to increase low energy counts. The current drawbacks are the sensitivity to exact working distance and the position of the X-ray optics very close to the sample surface.





Figure 1. X-ray optics inside the SEM chamber

Figure 2. optics with ring clamp adaptor

Fig. 3-5: The orange graphs are measured with, the black graphs without the optics.

