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Improving EDS For Low Energy X-Rays Under 1000eV Using an Attachable Detector Optic

David O'Hara, Greg Brown, Eric Lochner
Parallax Research, Inc., Tallahassee, FL
donkloos@earthlink.net

Introduction

Although considerable advances have been made in Energy Dispersive Detectors for microanalysis, low energy analysis under 1000eV is still relatively poor due to detector response and inefficient production of low energy x-rays. X-ray optics fabrication methods by O'Hara and measurements by McCarthy et. al.[1] indicated that it should be possible to fabricate x-ray optics that could be used to significantly increase the low energy x-ray flux seen by an EDS detector without increasing the beam current. Such an optic would be useful to increase low energy counts without moving the detector closer, which would simply increase the high energy counts and dead time.

X-Ray Optic Design and Fabrication

An optic for testing was designed from a simple approximation to an x-ray compound elliptical concentrator. Such an optic collects x-rays diverging into a large solid angle and reduces the divergence so the x-rays fall onto the surface of a detector increasing the x-ray flux to the detector. This optic has no focal points although it is designed for optimum performance at a specific location. The optic was designed for the most common EDS detector size of 10 mm^2 with the detector face normally located at a distance of 35 mm from the sample. It should be noted that this is the position of the detector face, not the distance to the detector x-ray collimator/electron trap. Calculations indicated large energy dependent flux gains for energies below 1 KeV with gain falling to near unity and remaining at unity after 1.5 KeV. The optic should not alter the spectrum above 1.5 KeV, thus not interfering with 'high energy' analysis.

Fig. 1. Parts of the Low Energy EDS Optic. L. to R. Removable pointer for alignment, gimbaling optic assembly that fits into the electron trap, and the electron trap. Top left inset shows how the optic is used. Bottom right inset shows the electron trap with the conventional graphite collimator.

Gain measurements were to be made with the detector at the same location both with and without the optic, so an electron trap/optic holder was designed into which either a conventional carbon collimator could be placed or upon which the optic could be mounted. Both the optic mount and collimator mount were keyed so as to fit in only one position for repeatability. Although alignment is not nearly as critical as with most x-ray optics, the optic axis must pass through the x-ray emitting point so the optic had to be made adjustable in an angle coaxial with the EDS "snout". The possibility of breaking the EDS detector window was a major concern, so the optic mount was made in such a way that the optic or holder could not reach or touch the window. The optic was designed for a working distance of 4 mm from the x-ray emitting sample to the optic

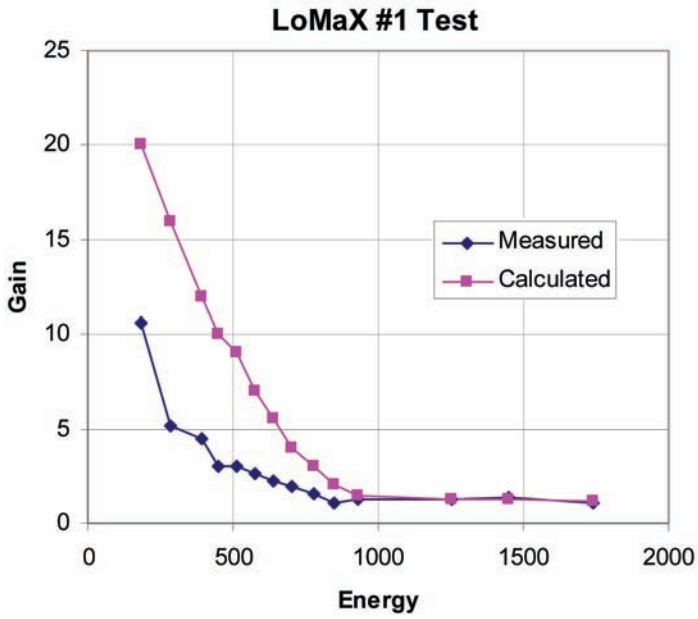


Fig. 2. Test of Optic #1 made from mandrel #2, which did not have optimum shape.

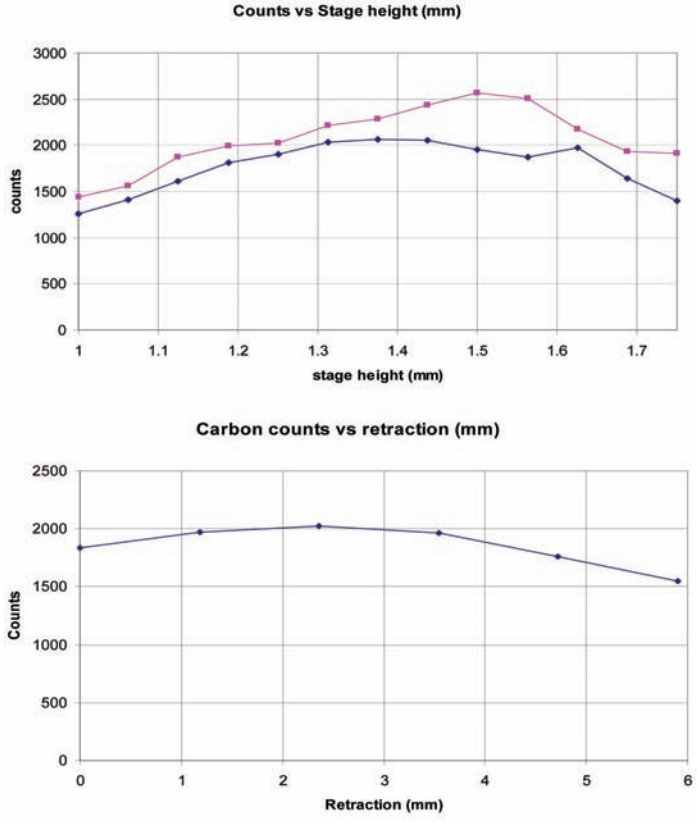
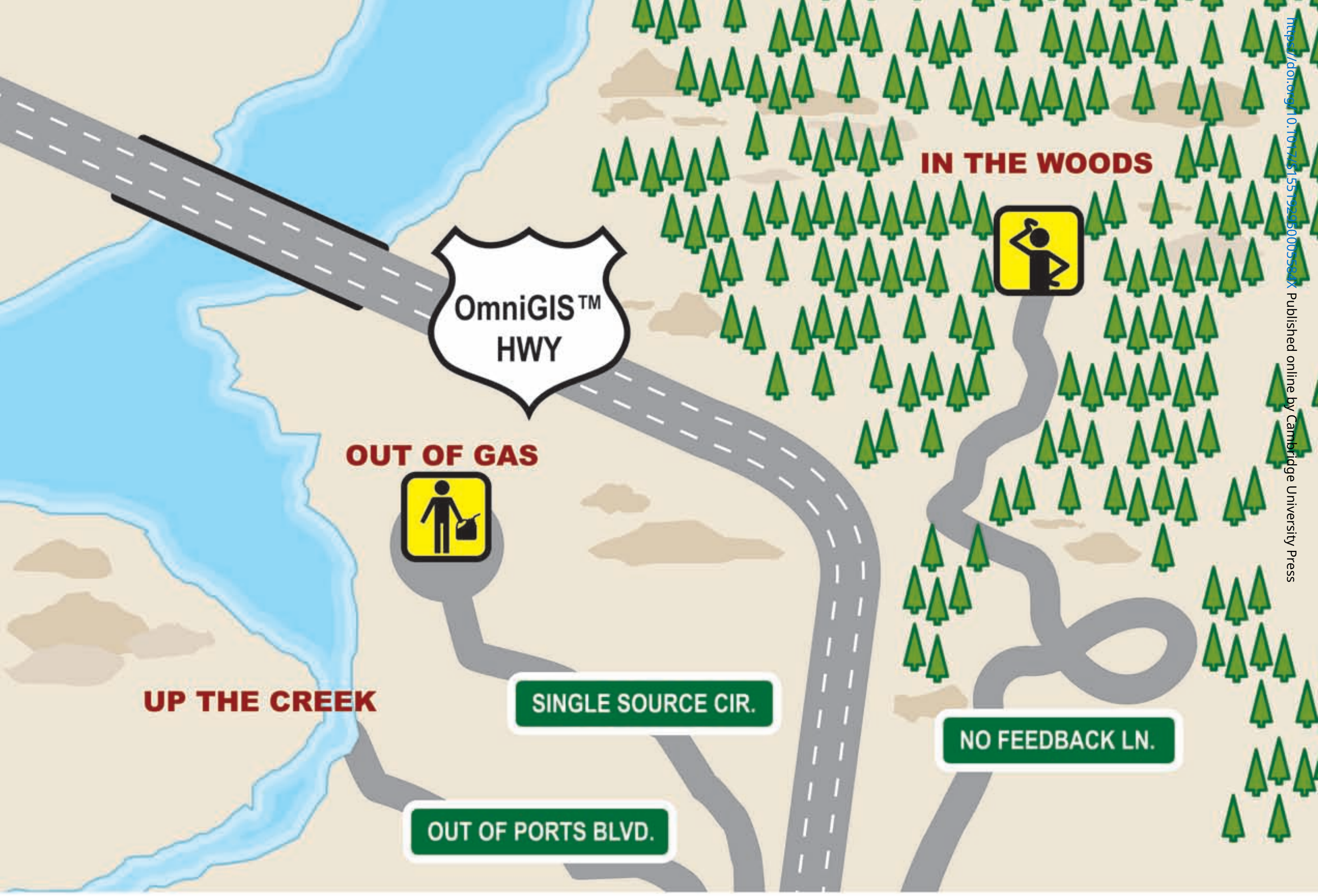


Fig. 3, top. Counts vs. stage height for optic #1, top line is for Oxygen in SiO_2 and bottom is Carbon.

Fig. 4, bottom. Counts vs. optic retraction along the EDS linear slide for optic #1.



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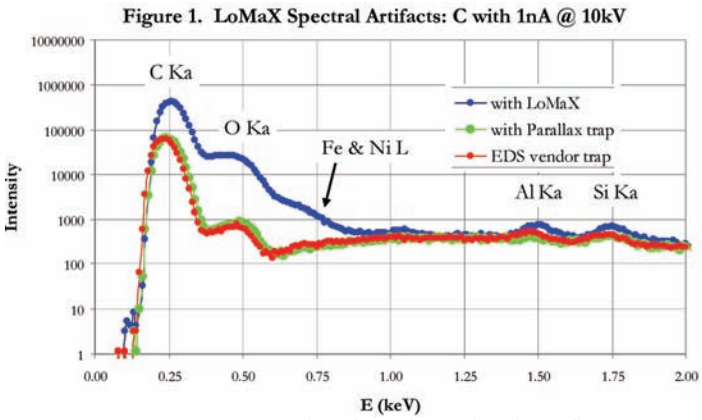

Fig. 5. Low energy spectral artifacts of Ni and Fe due to fluorescence from the optic surface.

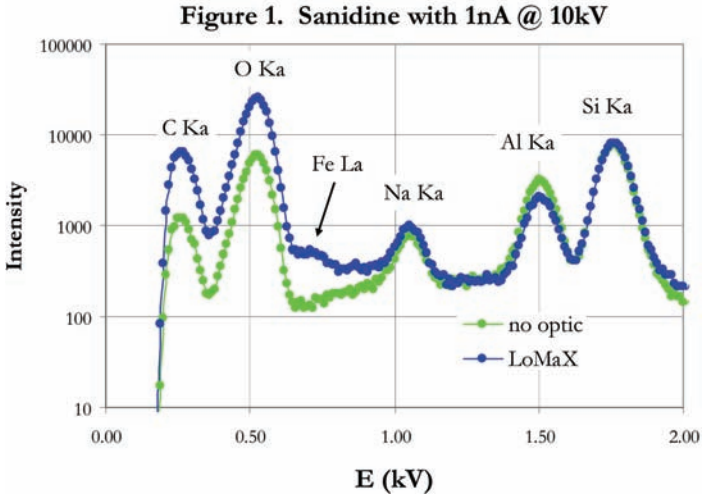

Fig. 6. A scan of Sanidine using optic #1 showing several lines enhanced by use of the optic. And showing the nearly unity gain above 1 KeV.

entrance aperture. Interference with the magnetic field in the SEM was a concern so the optic was fabricated of a very thin reflecting film of Ni followed by a thick shell of Cu. Finally, it had to be aligned easily so a detachable "pointer" was fabricated that fits over end of the optic and indicates the correct alignment relative to the sample. In Fig. 1, the collimator/electron trap can be seen along with the optic in its adjustable mount, the demountable alignment tip and the carbon collimator that can be placed into the collimator/electron trap when the optic is removed.

Experimental Procedures and Results

The optics are made via electroforming replication from shaped and polished mandrels. Two mandrels were made and profiled. Mandrel #1 was found to have a shape close to theoretical, but was destroyed in processing. Mandrel #2 was found to have significant angular errors that would reduce the measured gain but it was decided to use it anyway to make optics for testing and research purposes.

Alignment was achieved by bringing the Faraday Cup to the center of the screen, venting and placing the optic in the collimator/electron trap with the pointer on its small end. The EDS detector is then moved toward the sample until the tip is adjacent to the Faraday Cup. A mirror was used to view the position of the pointer relative to the cup and adjustments were made to the positioning screws on the front of the optic holder to align the pointer with the cup. The SEM was pumped down and the image of the tip was viewed relative to the cup at 20X; the SEM vented, another adjustment made, SEM pumped down and viewed again. Three venting cycles were necessary to obtain alignment and then the alignment tip was removed. In all it took about 45 minutes to complete installation and alignment of the optic. No subsequent alignments were necessary even after de-mounting and re-mounting the optic.

Measurements were made on standards of BN, C, SiO_2, Mg and Al for K lines and the L(a) lines of transition elements - both with and without the optic. Both measured and calculated gains are shown in Fig. 2.

Since x-ray optics used for Parallel Beam WDS are known to be sensitive to stage height, it is useful to know the sensitivity for this one. For the prototype optic, measurements for the effects of stage height were made for C and O (SiO_2) with data shown in Fig. 3 indicating that for O, a change of 0.1 mm in stage height will change the counts by 10%. Calculations indicated that motions along the optical axis should produce less effect than changes in stage height and this effect is seen in Fig. 4, which shows that considerably larger sample position changes can be tolerated. This is relatively insensitive to stage height, position changes, and sample topography compared to other fixed x-ray optics.

It was expected that the optic would produce spectral artifacts due to fluorescence of elements in the optic surface and this effect was investigated by observing spectra from a pure C surface as seen in Fig. 5. A significant artifact of Fe and Ni L lines is observed and this is verified by observing their corresponding K lines for these elements with and without the optic.

To determine how well the optic would work in actual practice, the mineral Sanidine ($KAlSi3O_8$) was examined as seen in Fig. 6. Considerable increase in count rates can be observed for both C and O. The Fe(L) line was also seen, but this may be a spectral artifact, although the standard is known to have roughly 0.14% Fe.

Optics of this type are expected to be useful for mapping because

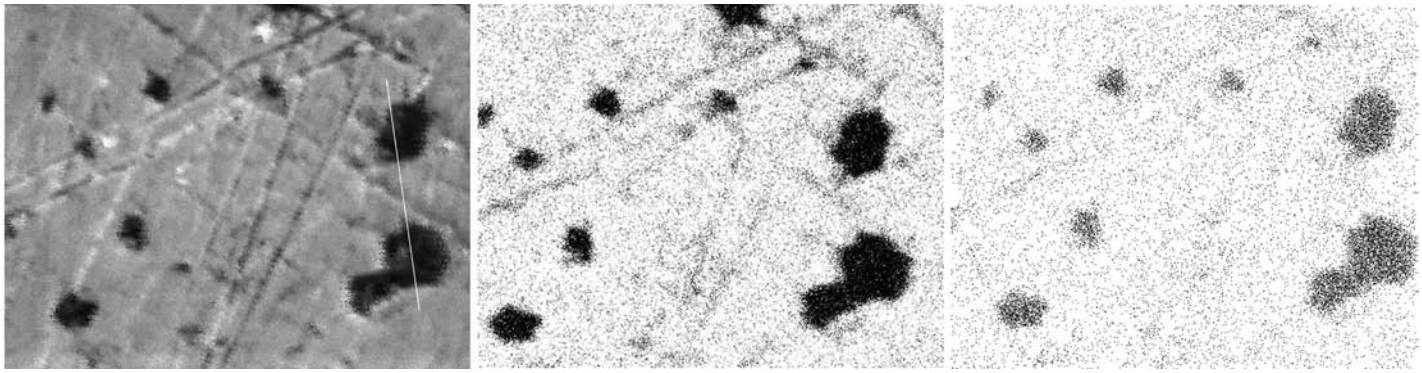

FIG. 7a. Secondary electron image and spectral maps at C Kα with and without the optic, respectively, for a mild steel sample (L to R). The white line on the right of the SEI designates the line scan shown in Fig. 7b.

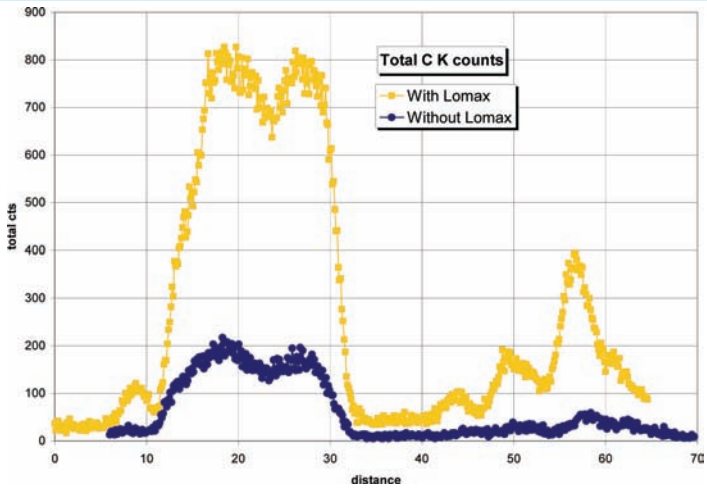

FIG. 7b. Line scan of C Kα intensity, acceleration potential of 5 KV, 0.4 nA of current, characteristic time of 25.6 μs, 200 ms dwell time.

they will considerably reduce the total time for a useable image when light elements are being observed. Fig. 7a and 7b show a wrought iron sample with C inclusions mapped both with and without the optic and the data for the optic shows structure in the inclusions that cannot be seen without the optic.

Further Work

The concepts here can be applied to a 30 mm^2 detector as well, but the gain will be less than with a 10 mm^2 detector. For example, whereas a gain of about 8X can be achieved for an optic of this type for B with a 10 mm^2 detector, a similar optic for a 30 mm^2 detector would give a gain of 4X. In general, an optic designed for a 30 mm^2 detector will give about half the gain it will give for a 10 mm^2 detector.

Such an optic can also be configured as a 'low pass filter' to minimize dead time and preferentially increase low energy counts by blocking the direct x-ray path to the detector and only allow reflected x-rays through. Increasing the beam current then will not cause high energy counts to increase but will only increase the desired low energy counts.

Additionally, for energies below 200 eV, Palladium is a better x-ray reflector than Nickel so that an optic can be made that will give another 2.5X gain compared to the Ni -coated optic. This would be applicable only for B and Be. Again, by blocking x-rays direct from the sample, such an optic would be a powerful tool for measuring B in Silicon wafers by blocking the direct Si x-rays. If it was necessary to measure low concentrations of Be, a gain of over 50X could be achieved by using a Pd coated optic with larger grazing angles.

Conclusion

The measured gains for this first optic were substantial and in the range of 10X for B and 3X for oxygen even though the optic's physical profile did not quite match the desired theoretical shape. It is expected that an additional improvement will be realized when the actual shape matches the theoretical. Further refinements and tests are pending. ■

Reference

[1] J. McCarthy and J. McMillan., "Applications of X-ray Optics to Energy Dispersive Spectroscopy", Microscopy and Micro-Analysis, Vol. 4, pp 632-641, Cambridge University Press, 1998