Preliminary Comparison of Monolithic and Aperture Optics for MXRF

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Abstract

Comparisons between standard aperture optics and a custom designed monolithic capillary x-ray optic for the Kevex Omicron are presented. The results demonstrate the feasibility of retrofitting an Omicron with a monolithic capillary. Increased flux is observed especially at lower energies, which results in an increase in sensitivity and a potential increase in spatial resolution. Alignment is a critical factor in achieving optimal performance of the monolithic capillary. Further improvements in flux output, spot size and overall sensitivity are expected with better alignment.

Introduction

Capillary optics for micro-x-ray spectrometry have developed rapidly within the last 10 years [1-4]. Most applications have centered on research instruments, rotating anodes, synchrotrons or new commercial instruments. The most obvious application would be to retrofit an Omicron to both increase x-ray flux at the sample and increase spatial resolution. The Omicron instrument manufactured by Kevex, is based on aperture optics and suffers from loss of flux as smaller apertures are inserted in front of the x-ray tube. This paper presents preliminary comparisons of a monolithic capillary designed for the Omicron geometry with the instrument aperture optics and some of the difficulties associated with retrofitting. The monolithic capillary is best suited for retrofit applications due to the constrained geometry of the Omicron. The monolithic capillary consists of an assembly of glass capillaries formed into an ellipse. This allows a compact, high efficiency x-ray lens to collect and focus x-rays and provide an increase in x-ray flux at the sample relative to using an aperture.

Experimental

A Kevex Omicron (Valencia, CA) equipped with a 50 W (50 kV, 1 mA maximum) rhodium x-ray tube and apertures with diameters from 3 mm to 50 μm is used in this work. A monolithic capillary (X-ray Optical Systems (XOS), Albany, NY) was designed specifically for the Omicron geometry. Figure 1 is a diagram of the optic with the appropriate dimensions. The advantages of the XOS monolithic capillary are determined by the collection distance, 25.4 mm, and the focal distance, 6 mm. The total distance from x-ray tube to sample within the Omicron optical kernel is just under 61 mm. The body of the optic is 29 mm long encased in a stainless steel tube with the ends sealed with beryllium windows. The predicted focal spot size is about 23 μm when properly aligned.

Results and Discussion
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The capillary was placed within the Omicron at a fixed distance from the x-ray tube. No effort was made to change this distance since the primary goal of this work was to determine flux increase. The adjustment of the capillary to x-ray tube distance mainly affects the focal spot size. A crude measurement of the spot size was accomplished using an iron wire 250 μm wide. The plot in Figure 2 shows the movement of the beam over the iron wire in 28 μm steps as the intensity of the iron Kα line was monitored. The beam spot size measured at FWHM is about 300 μm. This is about an order of magnitude greater than the expected spot size of 23 μm. Clearly the capillary-to-x-ray tube distance needs to be optimized. However this does demonstrate the potential for a continuously changing spot size with proper mechanical engineering within the instrument and control of the capillary-tube distance.

Figure 1. Schematic of monolithic polycapillary optic with appropriate dimensions. Expected focal spot is about 23 micrometers.

Figure 2. Linewidth scan of capillary spot over a 250 μm diameter iron wire. Measured FWHM of beam diameter is about 300 μm.
The primary objective of using the capillary optic is to increase the x-ray flux at the sample by focusing the x-rays from the x-ray tube. Figure 3 shows a comparison of the tube spectrum scattered by a Plexiglas target at 20 kV and 1 mA. The aperture spectrum from a 300 μm aperture is multiplied by a factor of 10 in order to move it above the baseline of the plot. The striking feature is the increase in the capillary x-ray flux between 1 and 7 keV. The large peak around 2.7 keV corresponds to the rhodium Lα and Lβ Rayleigh and Compton scatter. The capillary spectrum shows an increase of over one order of magnitude in low energy excitation. The tail-off of the higher energy profile above 8 keV is more pronounced than the aperture and has significant implications in terms of alignment and excitation, which will be discussed later.

The effect of the increased low energy excitation flux is illustrated in Figure 4. The sample is an aluminum alloy consisting of several percent copper. The aperture has a count rate of about 1300 cps for the aluminum Kα line. The aluminum intensity obtained with the capillary excitation is almost 6000 cps, an increase of about a factor of 5. This has significant implications for improving light element analysis. However, this signal enhancement by the capillary is not observed for the case of the higher energy copper line. While the aperture has a copper intensity in excess of almost 13,000 cps, only 5,550 cps is observed when using the capillary. This lower copper intensity and decline in the excitation above 8 keV noted in the Plexiglas spectrum is due to misalignment between the capillary and the x-ray tube. The acceptance angle dependence of the different energies at the input to the capillary means the capillary must be precisely aligned with the input beam to allow the full energy spectrum to be transmitted through the capillary. However, alignment of the capillary within the Omicron is not a trivial operation. The basic procedure involves the following steps: 1) insert capillary, 2) put sample in place, 3) close lid, 4)
observe count rate, 5) open lid, 6) move capillary by hand, 7) repeat steps 2 through 6 until count rate is maximized. Without live feedback of the count rate, the process becomes quite tedious. Small movements in the x and y positions of the capillary done by hand are also a hindrance in achieving the proper alignment. This is another mechanical engineering aspect, which must be addressed for optimal performance. However, these alignment difficulties do not preclude the use of a monolithic capillary within the Omicron.

Figure 4. Comparison of spectra from an aluminum alloy for the monolithic capillary (dashed line ---) and 300 μm aperture (solid line —) in air.

Figure 5 shows the potential for increased sensitivity in quantitative analysis. The

Figure 5. Spectrum of 50 microliter dried spot of a 10 ng/mL standard excited by the monolithic capillary.
spectrum in Figure 5 is from a 50 μL drop of a 10 ng/mL standard solution, which was allowed to
dry on a polypropylene substrate. The calculated limit of detection for the iron in the sample is
less than 0.1 ng/mL. Since there is less than 0.5 ng of iron in the dried spot, the estimated amount
of iron in the excitation region approaches less than 0.05 ng. The sensitivity calculated for
aperture excitation of the same sample using a 300 μm aperture is slightly greater than 1 ng/mL.
Thus, the capillary exhibits better than an order of magnitude improvement in sensitivity.

Conclusions

Monolithic capillary optics are feasible for retrofit in Omicron instruments. The
enhancement in flux improves the analytical sensitivity of both qualitative and quantitative
analysis. The major drawback is the critical alignment necessary to achieve both flux throughput
and complete energy distribution. The need for fine motion control of x, y and z axes to optimize
the flux and energy distribution would make the retrofit implementation of the monolithic capillary
easier and maximize its effectiveness. The potential for even further gains in analytical sensitivity
when the capillary is coupled with a higher power x-ray tube will be explored in the future.
Further experiments in alignment within the constraints of the standard Omicron geometry as well
as exploring potential design changes will also be investigated.

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References

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