DOI: 10.1017/S1431927605507232
Microsc Microanal 11(Suppl 2), 2005

Improve X-ray Microanalysis in Variable-Pressure SEMs with Polycapillary X-ray Optics

Ning Gao[a], Brian Gallagher[a], David Ackland[b], and Dale Newbury[c]

[a] X-Ray Optical Systems, Inc., 15 Tech Valley Drive, East Greenbush, New York 12061 USA
[b] Center for Advanced Materials and Nanotechnology, Lehigh University, Bethlehem, Pennsylvania, 18015 USA
[c] Surface and Microanalysis Science Division, National Institute of Standard and Technology, Gaithersburg, Maryland 20899 USA

One of the problems to perform x-ray microanalysis at elevated sample chamber pressure in a variable-pressure SEM is the electron beam skirting due to the presence of the gas[1]. The electrons in the skirt may strike the area millimeters away from the center probe area, generating x-ray fluorescence signals that interfere with the useful signal from the center area and cannot be distinguished by the energy dispersive (EDS) x-ray detector[2]. This will greatly reduce the detection sensitivity for very small samples, or degrade the image quality when performing an elemental mapping.

The effect of electron skirting on x-ray microanalysis can be greatly reduced by using a polycapillary x-ray optic[3] between the specimen and the EDS detector. The optic is capable of collecting a large solid angle of x rays from a well-defined small area and redirecting them to the EDS detector. The x rays from the outer area of the specimen that is struck by the electron skirt will not be "seen" by the detector system. The approach will greatly improve the detection sensitivity and spatial resolution of x-ray microanalysis in a variable-pressure SEM system. The focusing feature of the polycapillary optic allows a reasonable working space between the optic and the specimen, which is critical for the sample protection and ease of operation.

The polycapillary focusing optic used in our work has an input focal distance of 10 mm and a field-of-view of about 0.2 mm for 1 keV x-rays. The optic was attached to a 30 mm^2 EDS detector in a FEI XL-30 environmental SEM system (Figure 1). The detector-mounting flange was modified to allow a fine adjustment of the detector position relative to the specimen. The optic was aligned so that the focus overlapped with the center of the electron beam at the working distance of 10 mm. X-ray analysis was performed on a specimen made of a 0.5 mm gold wire embedded in silver-loaded epoxy that filled a 3.5 mm hole of a brass disk 25 mm in diameter (Figure 2). X-ray spectra were taken from the center of the gold area with and without the optic attached to the detector. Figure 3 shows the comparison of the spectra at different chamber pressures. It is clearly seen that the silver signal excited by the spreading electrons outside the gold disc area was completely eliminated when using the optic. The higher the chamber pressure, the better improvement will be obtained by using the optic. The effective collecting solid angle when using the optic was equivalent to that when the detector was placed 40 mm from the sample (no optic), which was about 0.02 sr. Therefore the improvement of x-ray image quality is not at the cost of collection efficiency.

References:

1. G. D. Danilatos, Scanning Microscopy International, Supplement 7 (1993) 57-80
2. R. B. Bolon, Microbeam Analysis (1991) 199-200

3. N. Gao, I. Yu. Ponomarev, Q.F. Xiao, W. M. Gibson, D. A. Carpenter, Appl. Phys. Lett., **69** (1996) 1529-1531
4. This work was supported by the Department of Commerce under contract SB1341-02-C-0018.

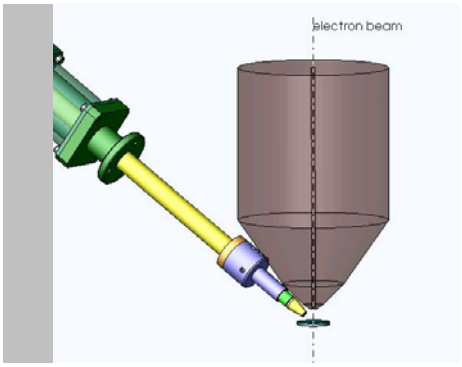

Figure 1. Polycapillary optic attached to the EDS detector in a SEM system

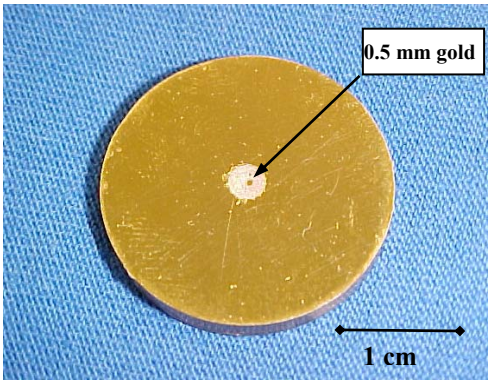

Figure 2. Photograph of the sample used for system testing

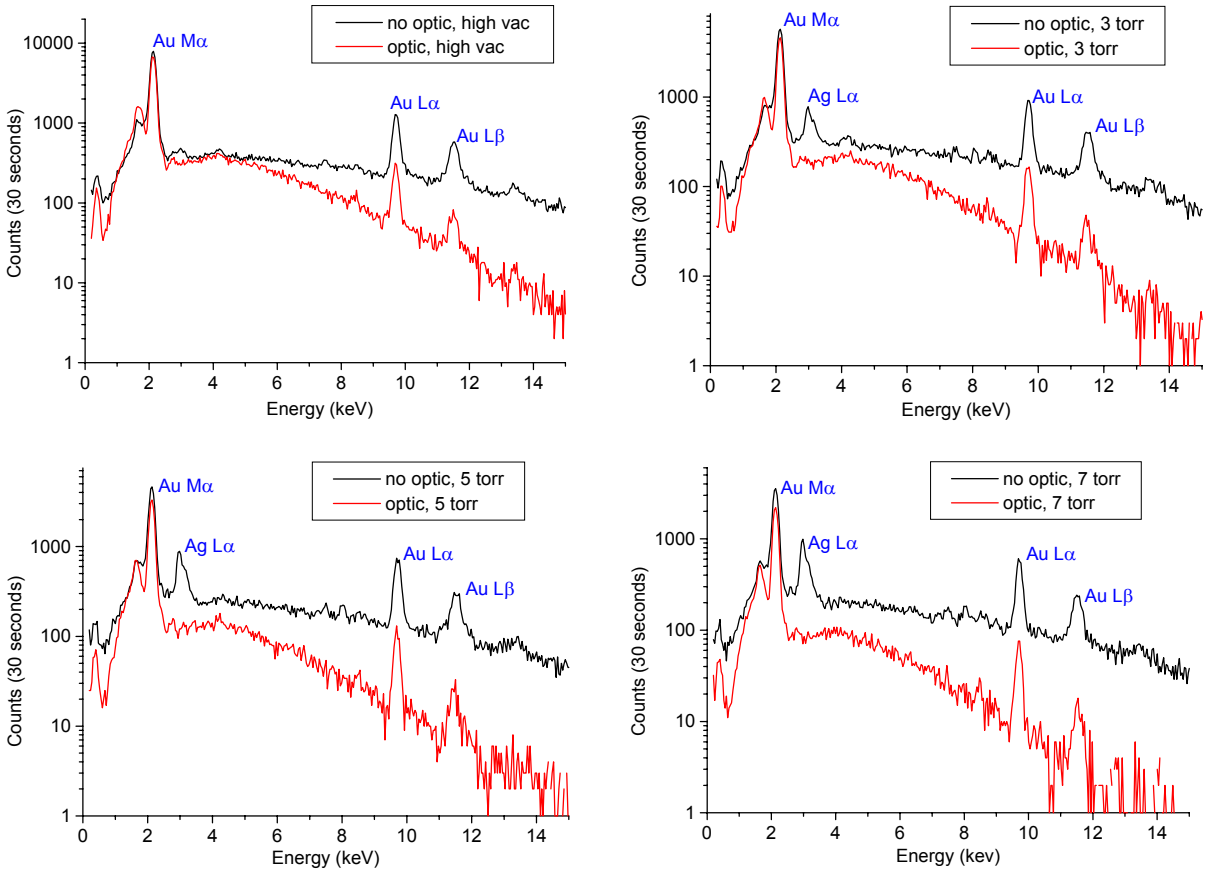

Figure 3. Spectra comparison with and without using the polycapillary at different chamber pressures

https://doi.org/10.1017/S1431927605507232 Published online by Cambridge University Press