Focused Ion Beam Induced X-Ray Analysis

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Characteristic x-rays may be emitted as the result of ion-solid interactions and is the mechanism for particle induced x-ray analysis (PIXE). Conventional PIXE techniques use light ions or protons accelerated at energies in the MeV range [1]. However, x-ray emission has also been documented for heavy ions accelerated to a few keV [2]. The advantage to using heavy ions is that the collision cascade occurs close to the surface, and therefore, direct surface analysis and/or accurate depth profiling could be accomplished. In addition, since no continuum or Bremmstralung is generated by ion-solid interactions, ion beam induced x-ray analysis may be orders of magnitude more sensitive than electron beam induced x-ray analysis [2]. Previous results have also indicated that there is a characteristic cut-off energy which is ion and target dependent, above which no x-rays are emitted [2]. The collection of soft x-rays ($< \sim 2 \text{ keV}$) are most sensitive to PIXE, and thus, appropriate for light element analysis. Heavy ions (e.g., Ga⁺) in the keV energy range are readily available with commercial focused ion beam (FIB) instruments. Thus, the efficacy of FIB induced x-ray analysis (FIBIX) as an analytical technique is discussed below.

An FEI Nova NanoLab 600 DualBeam (FIB + scanning electron microscope (SEM)) equipped with an EDAX x-ray detector was used to collect x-ray energy dispersive spectra (XEDS) using 30 keV Ga⁺ ions at a beam current of 20 nA. Carbon coated NIST standard reference materials 1872 containing varying amounts of Ge, Pb, and O, were analyzed. The reference standards were mounted on a 45° pre-tilted holder and rotated such that their plane normal was equidistant between the FIB column and the XEDS detector.

FIG. 1 shows the superposition of XEDS profiles acquired in 100 s from sample K968 using either the FIB as the primary source (red) or the SEM as the primary source (blue). Note that the FIBIX spectrum drops to below 4 counts at ~ 4 keV which is much less than the incident 30 keV Ga⁺ ions as predicted by theory. The primary constituents of O, Ge, and Pb are evident in both the FIBIX and SEM spectra. A continuously decreasing Bremmstralung is noted in the FIBIX spectrum, and the large consistent Bremmstralung is noted in the SEM spectrum, particularly at the higher energies [3].

References

- [1] J.W. Martin, The Local Chemical Analysis of Materials, Elsevier, Amsterdam, (2003).
- [2] J.A. Cairns, Surface Science, 34 (1973), 638.
- [3] Special thanks to Mike Bernas, Trisha Rice, and Richard Young for helpful discussions.

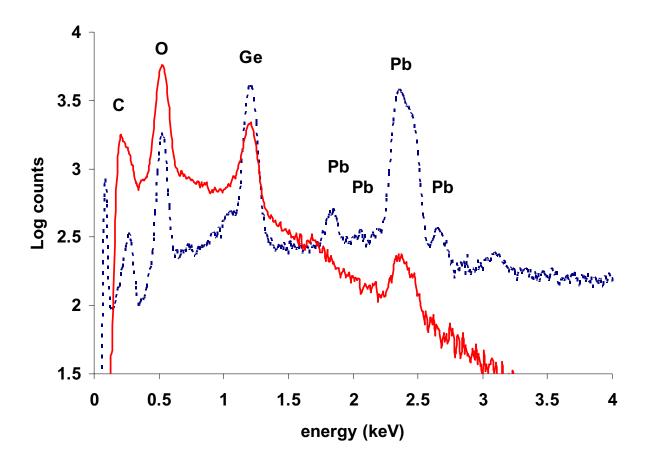


FIG 1. XEDS spectra obtained in 100 s using a FIB (red) or SEM (blue) as the primary source.