

## Variations in Peak Position and Energy Resolution for a Microcalorimeter EDS by Temporal X-ray Spectroscopy

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The superior energy resolution of the current generation microcalorimeter energy-dispersive x-ray spectrometer ( $\mu$ EDS, 15 eV FWHM at 1.5 keV) allows the separation of closely spaced spectral features that would overlap with a conventional semiconductor EDS (cEDS). One application of this better energy resolution is the x-ray mapping of elements with closely spaced peaks. One disadvantage of the  $\mu$ EDS is the low throughput of x-rays detection (a maximum of 300 counts per second). To obtain a meaningful map of low concentration elements a long acquisition time is required. An acquisition time of 4 to 10 hours is typical for low concentrations. The detector stability, over this long acquisition time, impacts the accuracy of such map.

We have studied the effect of acquisition times on peak position and energy resolution. A temporal spectrum, measurement of energy and detection time for each x-ray, for 8 hours (30000 s) from a composite specimen made of titanium, aluminum tape and silver paint was acquired at 10 kV with the ORNL low-voltage microprobe (JEOL 6500F) that is equipped with an EDAX Polaris  $\mu$ EDS. The spectrum can be split into multiple smaller data sets with shorter acquisition times (600 s or 10 minutes). From these spectra, the peak position and energy resolution (full width half maximum) are extracted by peak deconvolution [1].

Figures 1 and 2 show variation of Al  $K_{\alpha}$  (1486 eV) and Ti  $K_{\alpha}$  (4508 eV) peaks position with increasing acquisition time. An abrupt change in the peak position can be seen for both peaks. The magnitude of the change is 4.5 eV for Al  $K_{\alpha}$  and 19 eV for Ti  $K_{\alpha}$ . The exact explanation for this change is not known, but we suspect that the transition-edge sensor (TES)/SQUID detector could have caused this apparent gain change. The x-ray energy is related to the voltage measured by the TES/SQUID by a linear factor. A small change in the measured voltage shifts the peak position of a detected x-ray by an amount proportional to its energy. This behavior would explain the different peak shifts between the two peaks. Also a small variation in the peak position without correlation in time (random) is noticed in figures 1 and 2,  $\pm 1$  eV for Al  $K_{\alpha}$  peak and  $\pm 3$  eV for Ti  $K_{\alpha}$  peak. Again the difference shift for the two peaks is explained by the linear factor in the x-ray energy calculation.

Figures 3 and 4 show the evolution of the energy resolution (FWHM) with increasing acquisition time. No trend is seen with an increase of the acquisition time. A variation of  $\pm 1$  eV for Al  $K_{\alpha}$  in the FWHM value can be explain by the change in the peak position (increase the width) and error in the deconvolution process. For Ti  $K_{\alpha}$  peak a larger variation of  $\pm 10$  eV from the mean value is noticed. The effect of the separation of  $K_{\alpha 1}$  and  $K_{\alpha 2}$  of 6 eV on the error in the deconvolution process and the linear factor are possible explanations for this larger variation. The linear factor effect would broaden the peaks with higher energy more than peaks with lower energy from small, random changes in the gain.

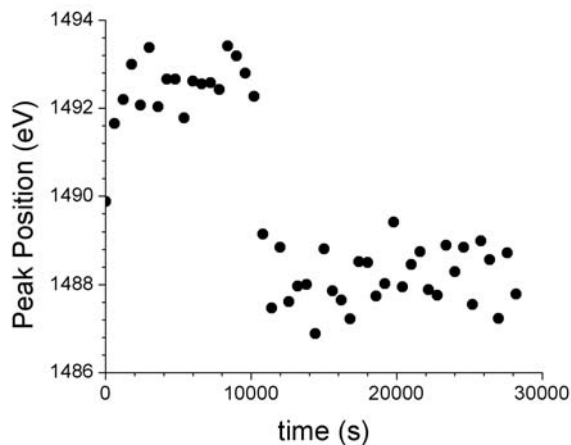


Fig. 1. Evolution of the aluminum  $K_{\alpha}$  peak position with acquisition time.

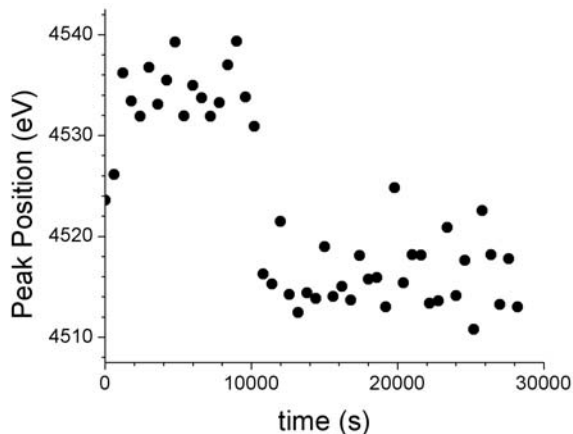


Fig. 2. Evolution of the titanium  $K_{\alpha}$  peak position with acquisition time.

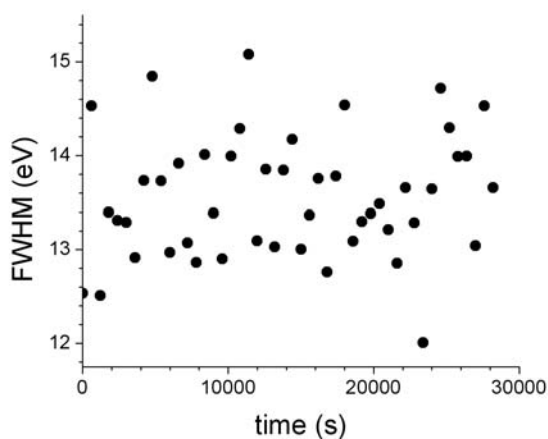


Fig. 3. Evolution of the aluminum  $K_{\alpha}$  peak energy resolution (FWHM) with acquisition time.

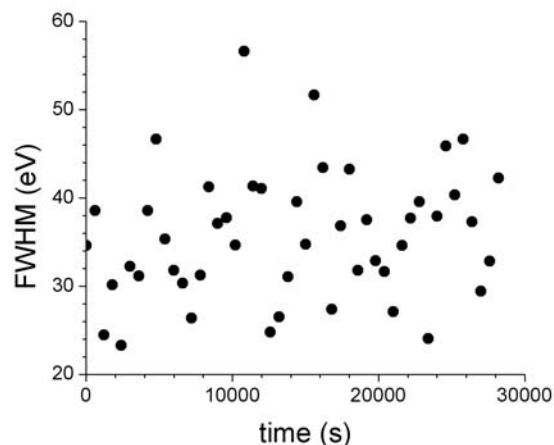


Fig. 4. Evolution of the titanium  $K_{\alpha}$  peak energy resolution (FWHM) with acquisition time.

## References

- [1] H. Demers, R. Gauvin, I. M. Anderson, D. C. Joy, and E. A. Kenik, *Microscopy and Microanalysis* (2005), 11: 464-465.
- [2] Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Office of Basic Energy Sciences, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.