

Spectromicroscopy at the XM-1

G. Denbeaux,^{1,2} L.E. Johnson¹, and W. Meyer-Ilse¹

¹Center for X-ray Optics, Ernest Orlando Lawrence Berkeley National Laboratory,
University of California, Berkeley, California 94720, USA

²Physics Department, Duke University, Durham NC 27708, USA

INTRODUCTION

The XM-1 x-ray microscope has been shown to have a spatial resolution of 43nm¹. In addition to this high spatial resolution, the XM-1 also has a good spectral resolution. Work is underway to characterize the spectral resolution of the XM-1 microscope. The aim is to use the XM-1 to image samples with 43nm spatial resolution and at the same time to use the high spectral resolution to distinguish different elements and even different chemical states within a sample. It has been shown that the energy resolution of the XM-1, $E/\Delta E$, is better than 750. We have shown that with this energy resolution we can distinguish between different chemical states of a particular element. We can see spectra with adequate signal to noise even for individual 42nm pixels. With this, we expect to soon begin work on various experiments in which we will image a sample and distinguish different chemical species of specific elements with 43nm resolution.

EXPERIMENT

The XM-1 X-ray Microscope is a full field imaging microscope using a condenser zone plate to illuminate the sample and objective zone plate to image the sample. The spectral resolution is determined by a linear monochromator which includes the condenser zone plate and a pinhole. The energy is chosen by changing the distance between the condenser zone plate and the pinhole. The bandwidth is determined by the size of the pinhole, which is usually less than 10 microns. To obtain a spectral (energy) scan of a sample, a series of full field images are acquired by the CCD camera, with each image at a different energy. A series of images taken at 100 different energies each covering a field of view of 10 microns and with a spatial resolution of about 43nm takes about 20 minutes.

RESULTS

We have determined the spectral resolution of the XM-1. We scanned the energy of the XM-1 around the L-edge absorption energy of Calcium and measured the absorption in a sample of CaSO₄. The graph of this absorption is shown in Figure 1. The measured width of the absorption peak of Calcium from the XM-1 data is 0.5 eV. For comparison, the absorption in the same sample was measured at the Calibration and Standards Beamline, 6.3.2, which has an energy resolution, $E/\Delta E$, greater than 5000.² The measured width of the absorption peak at that beamline was 0.4 eV. The broadening of the absorption feature is due to the finite spectral width of the XM-1. From this, an early estimate of the spectral resolution of the XM-1 is $E/\Delta E$ greater than 750.

This resolution is fine enough to be able to distinguish between different chemical states of specific elements. The absorption peak energy shift due to the different oxidation states is larger than the broadening due to the finite spectral resolution. We performed spectral scans of Chromium metal, hexavalent Chromium in K₂Cr₂O₇, and trivalent Chromium in CrO_x. The

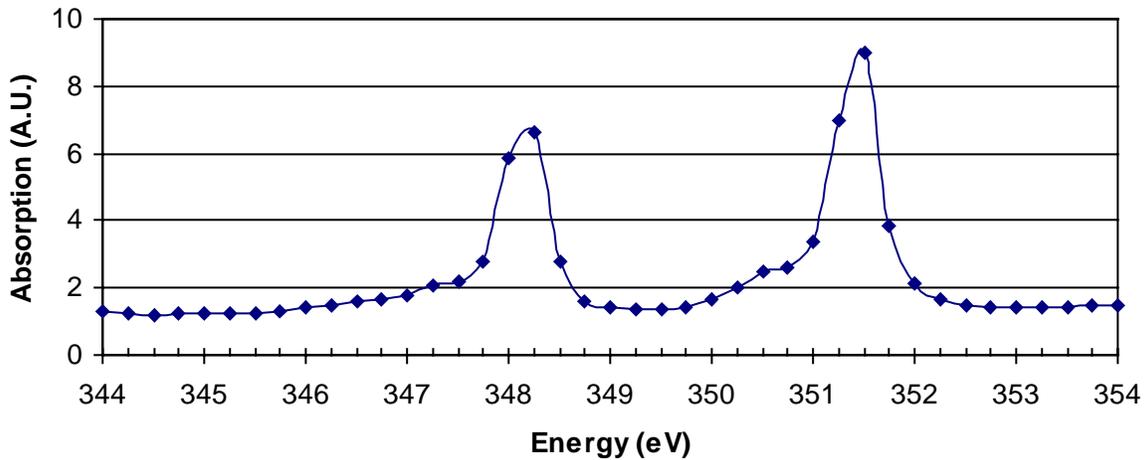


Figure 1. Absorption of CaSO_4 from the XM-1. The FWHM of the peaks are 0.5eV .

results are shown in Figure 2. It is clear that the different oxidation states of the Chromium cause a shift in the absorption spectra that is large enough for us to distinguish the different states.

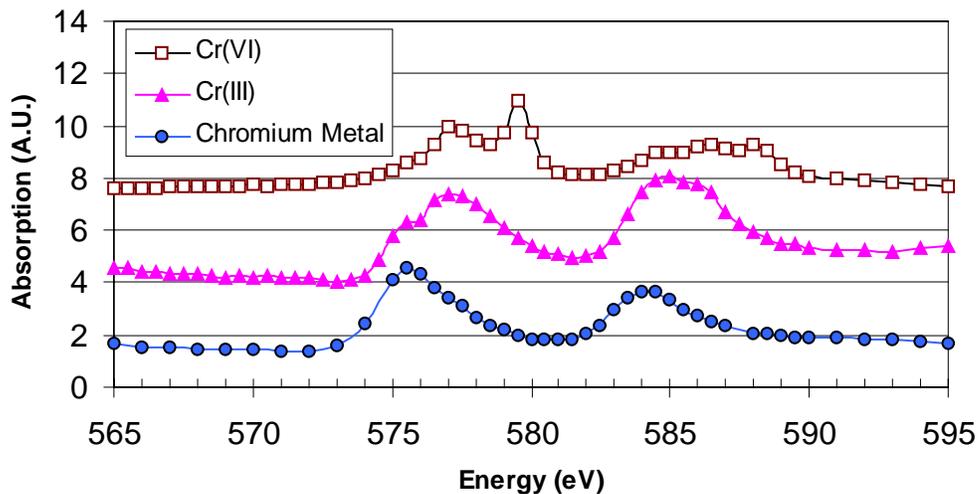


Figure 2. Absorption of Chromium in three different oxidation states.

In order to reap the full benefits of this spectral resolution on the XM-1 microscope, the spectral resolution must be available on the microscopic scale approaching the 43nm resolution of the microscope. Since there are fewer photons passing through small areas on the sample, the photon noise is larger and the spectra have decreased signal to noise. With reasonable exposure times which do not destroy the sample, at least for a dry sample, then the signal to noise is clearly good enough that shifts in absorption peaks of a few eV corresponding to oxidation state changes would clearly be visible. Figure 3 shows sample spectra from four single 42nm by 42nm pixels.

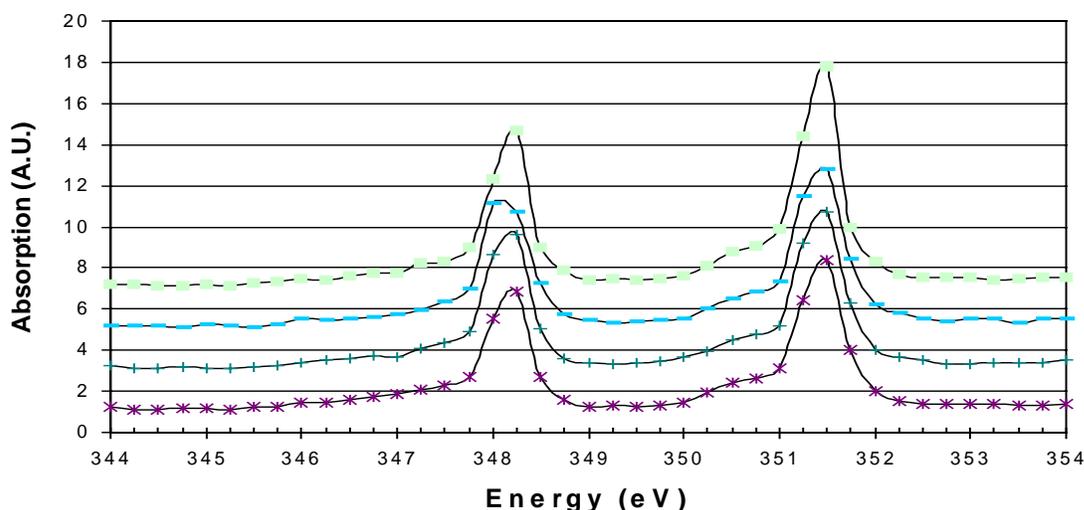


Figure 3. Absorption spectra for four different single 42nm pixels from a sample of CaSO_4 .

CONCLUSIONS

With this ability to distinguish between different elements and different chemical states of these elements on a 43nm scale, a variety of interesting experiments are now accessible for future research at the XM-1. One interesting experiment that has been started looks at bacteria that are known to reduce hexavalent Chromium into trivalent Chromium. It is important to understand the mechanism by which these bacteria reduce the Chromium. We hope to be able to map the distribution of the different Chromium species within and around the bacteria.

REFERENCES

1. John M. Heck and David T. Attwood, "Resolution determination in X-ray microscopy: an analysis of the effects of partial coherence and illumination spectrum," *Journal of X-Ray Science and Technology*, 8 (1998), 95-104.
2. J.H.Underwood, E.M.Gullikson, "High-resolution, high-flux, user friendly VLS beamline at the ALS for the 50-1300 eV energy region," *Journal of Electron Spectroscopy and Related Phenomena*, 92 (1998), 265-272.

This work was supported by:

U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

U.S. Army Research Office Grant DAAH04-96-1-0246,

U.S. Navy, Office of Naval Research Grant N00014-94-1-0818,

Air Force Office of Scientific Research

Principal investigator: Werner Meyer-Ilse, Center for X-ray Optics, Ernest Orlando Lawrence Berkeley National Laboratory. Email: w_meyer-ilse@lbl.gov. Telephone: 510-486-6892.