A Silicon Bent-Crystal Analyzer as an Energy Bandpass Filter for Fluorescence XAFS

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Introduction

Two of the primary limitations on using x-ray absorption spectroscopy for many interesting environmental and biological problems are the presence of a high level of background radiation from a variety of sources, and interfering absorption edges that limit the data range. A highly bent silicon x-ray analyzer in the Laue geometry has some notable advantages compared to other detection methods and fills a significant gap in the arsenal of detectors available. We have demonstrated that this geometry may be used to resolve the L fluorescence x-rays of neighboring actinide elements, permitting measurements that would be very difficult by other techniques.

Methods and Materials

The design of the bent crystal analyzer has been discussed previously.[1,2] The essential property of such an analyzer is that bending the crystal solves the problem of the mismatch between the narrow acceptance angle of a perfect crystal and the large divergence of fluorescence x-rays from the sample.[1] The shape required to preserve the correct angle between the x-rays originating from the sample and the diffraction planes is a logarithmic spiral.[3,4] In addition, bending the crystal increases the energy bandwidth, making the analyzer a nearly ideal bandpass filter, with the exception of the somewhat low reflection efficiency.

The samples (ca. 100 μg) contained a mixture of actinide elements in the approximate ratios of 10:30:1 for U:Np:Pu. The geometry of this experiment was such that the analyzer rotated about an axis parallel to and directly above the beam. The beam was masked to produce an adequate source point; however, the energy resolution was significantly more sensitive to the horizontal slit size than to the vertical size (because of the limited analyzer aperture).

Results and Discussion

For samples containing mixed actinide elements, very good energy resolution is required, as the $L\alpha$ fluorescence lines for U, Np, and Pu are only separated from each other by ~330 eV. Furthermore, the $L\alpha_1$ line of one element is separated by less than 150 eV from the $L\alpha_2$ of the neighboring element. Solid-state detectors are not capable of resolving these lines at useful count rates (i.e., short pulse shaping times). Even the best detectors in combination with longer shaping times (>2 μs) would achieve a resolution no better than 210 eV at 14 keV.

Figure 1 shows the intensity vs. energy (by scanning the analyzer angle) around the actinide $L\alpha$ lines. A 0.1 mm χ 0.1 mm source point provides sufficient resolution to separate the $L\alpha$ lines and show evidence for $L\alpha_1/L\alpha_2$ splitting. The full-width at half-maximum is about 155 eV.

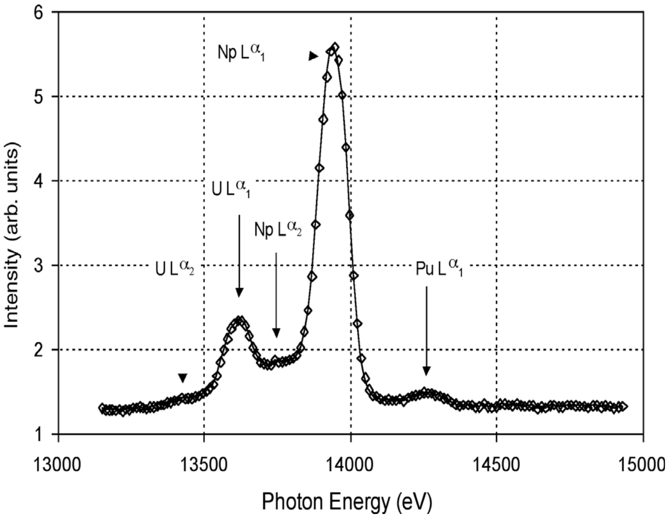

FIG. 1. Intensity as a function of energy (incident energy, 18.4 keV; beamsize: 0.1 mm χ 0.1 mm).

The reflectivity of the analyzer was measured by comparing the signal due to the yttrium $K\alpha$ fluorescence line from a mixed yttrium oxide/strontium oxide sample with and without the analyzer present. The measured reflectivity of 14% compares reasonably well to the calculated value of about 20%.[1]

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