A bent Laue analyzer detection system for dilute fluorescence XAFS

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Introduction

The intense beams produced by undulators at the Advanced Photon Source (APS) pose difficult challenges for x-ray absorption fine structure (XAFS) studies of dilute species in biological and environmental samples. A major source of noise in such cases is statistical fluctuation in the number of detected photons, most of which are elastically scattered or fluorescence from elements in the samples other than the ones of interest. Although conventional solid-state detectors do a good job of separating the desired fluorescence photons from the scattered background, they have maximum count rates of several hundred kilohertz per channel. The high flux of the full undulator beam often produces so much scatter the detector becomes saturated.

In anticipation of this problem, G. Bunker proposed the concept of a multilayer analyzer array in the early '90s. A device of this type has been developed by K. Zhang, *et al.* [1] of Bio-CAT. This analyzer works well, but it loses efficiency at high energies. Because of this limitation, G. Bunker and D. Chapman have proposed an alternative device optimized for high energies, which is based on an array of asymmetric bent Laue crystals. This concept has been further developed in this collaboration. Here, we report tests of this idea.

Methods and Materials

Initial experiments were carried out on the MR-CAT beamline 10-ID at Argonne National Laboratory, with a 100 mm vertically oriented slit defining the incident beam onto the sample. Ag foil, 25 microns thick, was used as a source of "signal" fluorescence and 25 micron Pd foil behind the Ag foil was used to simulate "background" fluorescence. The experimental setup is shown in Figure 1.

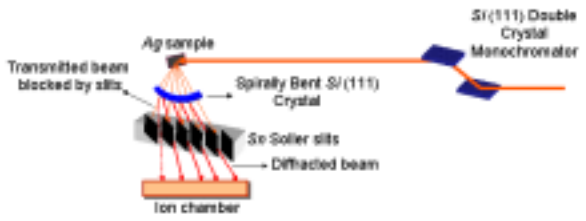

Figure 1: Experimental setup.

In order for the Bragg condition to be satisfied over the surface of the crystal, the 0.2 mm thick Si(111) crystal was bent to a logarithmic spiral shape by constraining the crystal to the surface of an aluminum form numerically machined to the correct shape. In polar coordinates (r, q), the correct logarithmic spiral shape is given by equation 1 [2].

$$r = \rho_0 \cos(\chi - \theta_B)\exp(\tan(\chi - \theta_B)\ \theta) \quad (1)$$

where ρ_0 is the bend radius at the center of the crystal $\theta = 0$, θ_B is the Bragg angle, and χ is the crystal asymmetry angle.

The diffracted beam is deflected in angle by $2\theta_B$, but the nondiffracted beam, which contains elastic scatter and/or other undesired contributions, passes directly through the crystal. Tests were performed by alternately blocking the transmitted and diffracted beams.

Since the diffracted (signal) and nondiffracted (scatter) contributions coexist in the same region of space, but have distinct directions, they can be separated using slits. Soller slits were fabricated to block the direct beam, but allow the diffracted beam to pass through unattenuated. Tin foils were chosen for this purpose, because the K-edge (29.2 keV) of Sn is higher than most of the elements targeted (e.g., Mo, Ru, Pd, Ag, and Cd), and hence would not be excited by the scattered x-rays. Also, the L-edges of Sn are sufficiently low in energy (<4.5 keV) that their fluorescence is readily attenuated by windows or thin absorbers. Tin can also be obtained in pure foils at a reasonable price, but they are difficult to work with. The slits have the same angular divergence as the nondiffracted beam, but when aligned are rotated by $2\theta_B$.

Results

Tests were performed in which 1 cm-wide slits were placed in front of the analyzer crystal, while the diffracted and nondiffracted beams were alternately blocked. Rocking curves were measured with the diffracted beam blocked, and as shown by the depth of the dip in the extinction curve (Figure 2a), the reflection efficiency is approximately 10%. The background rejection can be estimated by blocking the nondiffracted beam when the analyzer is detuned from the Bragg condition. The background-to-signal reduction ratio was approximately 20 under the test conditions.
Tests were also done using the dual Ag/Pd foils to simulate an element embedded in a sample with a high concentration of a slightly lower atomic number element (Figures 2b–d). Use of the Soller slits reduced the background transmitted through the crystal by a factor of 32 (.95/.03), which is encouraging. However, misalignment of the slits also caused a substantial loss of signal, and a definitive assessment of slit performance will require further experiments. The edge in Figure 2c is inverted because the Ag foil modulates the beam intensity impinging on the Pd foil. This effect is diluted when the analyzer is tuned on peak. In a realistic case, the concentration of the element of interest would

likely be much lower and this effect most likely would not be significant.

The bent Laue analyzer is a potentially important tool for fluorescence XAFS of dilute species under high-flux conditions that may saturate solid-state detectors. Initial results suggest this is a viable approach. Further optimization, testing, and engineering design will be needed for this approach to achieve its potential.

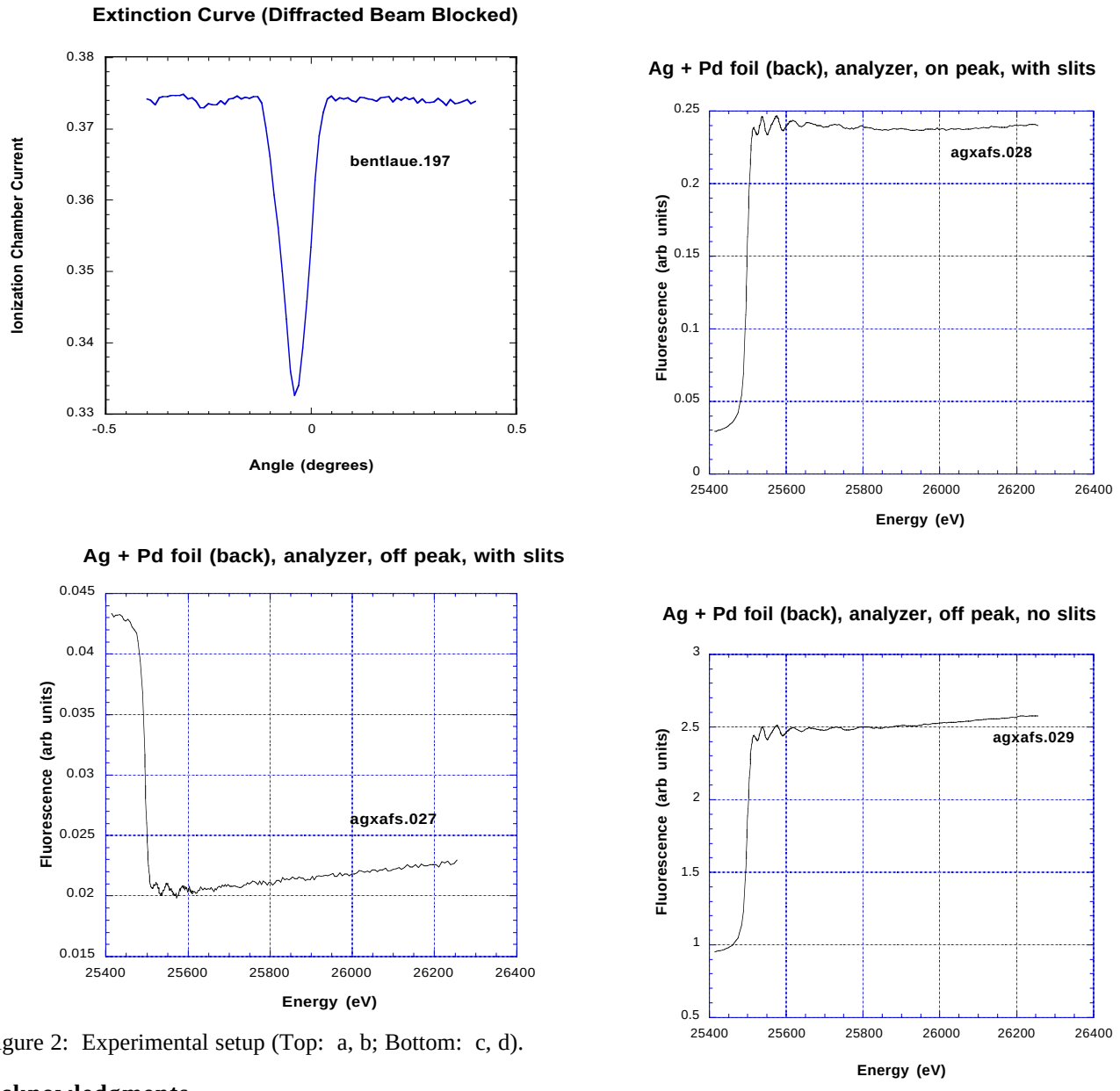

Figure 2: Experimental setup (Top: a, b; Bottom: c, d).

Acknowledgments

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References

[1] K. Zhang, G. Rosenbaum, and G. Bunker, *J. Synchrotron Rad.* **6**, 220–221 (1999).

[2] Y. Sakayanagi , *Jap. J. Appl. Phys.* **21**, L225–L226 (1982).

[3] Z. Zhong, D. Chapman, B.A. Bunker, G.B. Bunker, R. Fischetti, and C.U. Segre, *J. Synchrotron Rad.* **6**, 212–214 (1999).