Development of a novel sub eV, High Throughput, High Spatial Resolution, Laboratory X-Ray Absorption Spectrometer for XANES and EXAFS measurements

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X-ray Absorption Spectroscopy (XAS) is a widely-used technique for determining chemical states such as oxidation state by XANES (x-ray absorption near edge structure), interatomic distances and coordination number by EXAFS (extended x-ray absorption fine structure) in materials. For example, EXAFS and XANES combined are used extensively in the study of catalysts in fuel cells, semiconductor quantum structures, magnetic semiconductor structures, nanotechnology, materials for energy storage and conversion. Currently, XAS is predominantly performed at synchrotron facilities due to their high brightness and tunable energy x-ray beams. However, there are several major limitations that include difficulty in timely access (typically requiring several months in advance to apply for beamtime), limited beamtime and challenging logistic issues such as traveling and transportation of any equipment/instruments or transportation of certain kinds of samples. Hence, there is significant need for a high throughput and high performance laboratory-based XAFS system that enables academic and industrial researchers perform routine measurements and analyses on various samples.

Up to now, performance of laboratory XAS systems has been largely limited by combination of many factors, including low brightness of the Bremsstrahlung radiation of laboratory x-ray sources, low diffraction efficiency of the crystal analyzer associated with the use of high index reflections, and non-optimal spectrometer design. Those limitations result in unacceptably long acquisition times (~tens of hours) and/or poor energy resolution. As a consequence, there are few laboratory XAS systems being used with adequate performance. Recent development in laboratory XAS system uses spherically bent crystal analyzers to achieve sufficiently high energy resolution for XANES measurement [1]. However, that system has several limitations. First, the x-ray energy range for each crystal is typically limited to single element for XANES measurement. As a result, a large number of crystal analyzers are required if multiple elements are to be analyzed. Second, throughput is relatively low (data collection time was several hours).

We present a novel, compact, laboratory-based XAS system operating in the 4.5-12KeV energy range with the following major capabilities: sub-eV energy resolution over a fairly large energy range, short data collection time (seconds to minutes), and high spatial resolution (~10 micrometers) for small spot analysis or mapping. The performance of the system is achieved in part with a patented high brightness x-ray source, a novel system design, and the use of the latest x-ray crystal analyzers and spatially resolved detector. In this talk, we shall discuss the basic principles behind the design of the instrument and its performance.

References

1. Seidler, G. T., et al. "A modern laboratory XAFS cookbook." Journal of Physics: Conference Series. Vol. 712. No. 1. IOP Publishing, 2016.