

OXIDATION STATE DETERMINATION USING XANES WITHOUT A SYNCHROTRON

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Oxidation state is an important feature in materials characterization. Fortunately x-ray absorption measurements are a convenient method for characterizing the oxidation state of atoms. X-ray absorption fine structure (XAFS) is an x-ray absorption measurement based on how x-rays are absorbed by atoms. The absorption of the x-ray photon is based on the physical and chemical state of the atom. The x-ray absorption spectra are sensitive to oxidation state, coordination chemistry, and the distances and coordination number and species of atoms immediately surrounding the specific atom of interest. XAFS measurements of a specific atom provide an easy way to determine both the chemical state and the local atomic structure of an atom. These measurements are relatively straightforward and are dependent upon having a source of intense and energy tunable x-rays. This typically requires the use of a synchrotron as the source.

The XAFS spectrum has 2 regimes, the X-ray near edge absorption spectroscopy (XANES) and extended X-ray absorption fine-structure spectroscopy (EXAFS). XANES is sensitive to oxidation state and coordination number while EXAFS provides information on the local atomic structure. There are hundreds of publications on XANES and EXAFS of numerous atoms in a variety of applications utilizing synchrotron excitation.

This work demonstrates the use of a doubly curved crystal (DCC) in obtaining XANES spectra using a conventional laboratory X-ray tube. The significance of this achievement is the capability of obtaining XANES and EXAFS spectra in a laboratory environment. A breadboard system was constructed using a 50 W X-ray tube, a DCC and an energy dispersive X-ray detector. The appropriate x-ray fluorescence line was detected while the relevant absorption edge was scanned. XANES spectra for several iron, chromium, praseodymium and europium compounds were obtained using both $K\alpha$ and $L\alpha$ absorption edges for excitation and fluorescence lines for detection.