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HIGH RESOLUTION EXAFS-XANES SPECTROMETER

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Introduction

A double crystal (n,+n asymmetric) spectrometer with high resolution (0.1-0.2 ev) for analysing near edge XANES and EXAFS spectra is described. The spectra obtained on this instrument using a rotating anode source compare favorably with those obtained at a synchrotron.

Discussion and results

This spectrometer is a double crystal in the (n,+n) dispersive mode using two asymmetric reflections of Silicon (111). The standard way to obtain high energy resolution is with double crystals in the (n,+n) dispersive mode. The primary controlling factor in energy resolution is the diffraction line widths (Darwin) of the exit beam from the first crystal and the incident acceptance width of the second crystal. This can be understood by reference to the Dumond diagrams shown in Fig.1, 2. The wave length (and hence energy) resolution is given by the size of the overlap or window region a-b-c-d- shown in the figure for a particular setting of the two crystals. Rotating one of the crystals is equivalent to sliding its curve (band or window) horizontally and in this way the transmitted band moves up and down the (energy) scale. It is obvious that to obtain better energy resolution the a-b-c-d- window must be reduced. This can be accomplished by using higher order reflections to obtain a narrower line width but at the cost of some intensity. For this reason it was decided to use asymmetric reflections of (111) silicon as shown in Fig. 3.

In this arrangement the narrow (Darwin width) asymmetric exit beam of the first crystal feeds into the narrow incident asymmetric reflection of the second crystal. This reduces the overlap region a-b-c-d- as shown in Fig.2. In addition the wider acceptance angle of the first crystal means that more energy is channelled into the system (input energy to window Fig. 2). This has the advantage of theoretically increasing both the intensity and resolution over symmetric reflections by a factor of 5 to 10 times, depending on the degree of asymmetry. A measured increase in intensity of 7 times was obtained.

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The mechanical arrangement of the spectrometer involves elastic torsion bearings with torsion couplings. To provide $\theta$, $2\theta$, $3\theta$, $4\theta$ motions for the crystals, sample and counters with only one stepping motor. The automatization of our system and the data acquisition is obtained by a computer.
The spectrometer functions reasonably well in the laboratory with a rotating anode source. However, even with the seven fold increase in intensity due to the asymmetric crystal arrangement, the counting times are long. It takes 2 1/2 days for an EXAFS spectrum and one day for an XANES spectrum. A synchrotron source would give faster and better data. This would require the addition of counter weights for operation of the spectrometer in the vertical plane at the synchrotron.
The question of the actual spectrometer resolution compared to the calculated is difficult to answer. Two methods, one involving a Mossbauer source and the other involving double crystal reflection are under consideration to actually measure the spectrometer resolution experimentally.

References