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PERFORMANCE OF SPHERICALLY FOCUSING Ge(444) BACKSCATTERING ANALYZERS FOR INELASTIC X-RAY SCATTERING

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Abstract

A spectrometer designed to use an undulator source and having targeted resolutions of 0.01 eV in one mode of use and 0.2 eV in another will operate at the APS. We report here on analyzers that we have constructed for use on this spectrometer for 0.2-eV resolution. We have tested them at NSLS beamline X21 using focused wiggler radiation and at CHESS using radiation from the CHESS-ANL undulator. Analyzers were constructed by gluing and pressing 90-mm-diameter, (111) oriented Ge wafers into concave glass forms having a radius near 1 m. An overall inelastic scattering resolution of 0.3 eV using the (444) reflection was demonstrated at CHESS. Recent results at X21 revealed a useful diameter of 74 mm at an 87° Bragg angle.

1. Introduction

Since the early 1980's, inelastic x-ray scattering (IXS) has been considered both a desirable and achievable investigative tool with which to attack some of the fundamental problems in condensed matter physics^{1,2,3,4}. Unlike inelastic neutron scattering, IXS is almost completely unhampered by the kinematic constraints arising from conservation of energy and momentum during the scattering process, and, unlike inelastic electron scattering, x-rays penetrate the bulk sufficiently to rule out anomalies due to surface properties. However, cross sections for IXS are extremely small (e.g., 10^{-27} cm²/eV for the double differential cross section of the plasmon in aluminum⁵), which implies that synchrotron radiation sources should be employed to obtain useful data. With the advent of the APS,⁶ which is a third-generation synchrotron source, we expect that sufficiently great incident photon fluxes can be delivered that spectra of inelastically scattered photons with reasonably good statistics for a wide range of samples can be obtained in scans lasting several hours.

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The beamline optical layout will consist of a high-heat-load monochromator followed by a high-resolution monochromator. The diffuse scattering from a sample will be collected by a spherically focusing crystal analyzer.

II. Backscattering

Backscattering for the analyzer is a propitious geometry because 1) angular Darwin widths become very large, and 2) the derivative of the energy with respect to angle in Bragg's law goes to zero.⁷ The consequence of 1) is that the angular acceptance of analyzers increases, and the consequence of 2) is that the resolution becomes less dependent on angular divergence. Because of absorption in the sample, it is favorable to work at as high an energy as possible. Backscattering from silicon at higher energies occurs from high order reflections (e.g., (777)), which leads to resolutions of several milli-eV. A spectrometer with a 3-m-long 2-theta arm designed to employ high order reflections from a silicon analyzer is under construction on sector 3 of the Synchrotron Radiation Instrumentation (SRI) Collaborative Access Team (CAT) at the APS. A resolution of several meV is useful for the study of phonons. However, it is not needed for the study of electronic excitations and results in an unnecessary bandpass collection penalty in that case.

III. Ge(444) Analyzer

We have achieved a coarse energy resolution of several hundred meV by using a lower order reflection (i.e., (444)) from an analyzer made of Ge with a focusing distance of 1 meter. At this radius, neither the intrinsic energy width nor the efficiency of a bent Ge wafer is significantly reduced compared to the unbent case. This is demonstrated in Fig.1 which is the result of a dynamical diffraction simulation.⁸ A calculated analyzer resolution of 94 meV FWHM was obtained. Just as in the unbent case, the Darwin width in energy is almost independent of Bragg angle near backscattering.

The most successful procedure we have found to construct an analyzer is : 1) first prepare a two-component epoxy mixture by pumping the air out of the mixture in a bell jar, 2) spread the epoxy over a concave form and then place the wafer over the epoxy in as dust free an environment as possible, then 3) press the wafer into the form while the epoxy is exuded. The epoxy we have used had a viscosity of 100 cps, and the curing was done at room temperature.

The Ge wafer was sliced from a 90-mm-diameter, (111) oriented boule for which we measured a value of 0.068 millirad FWHM for the

Ge(333)xGe(333) double-crystal ($\pm$) rocking curve using CuK α radiation. Since the theoretical double crystal rocking curve FWHM is 0.032 millirad, a value of 0.042 millirad is deduced by quadrature subtraction for the mosaic broadening for each crystal (assumed to be identical). The ratio of the intensity diffracted by the second crystal to that incident on it in this same double-crystal arrangement was 65%.

Slope errors for the analyzer were obtained by measuring the change in Bragg angle for the Ge(111) reflection as the analyzer was translated across the beam.⁹ For the best analyzer to date, analyzer #6 shown in Fig. 2, a value of 0.29 millirad for the rms slope error (compared to a perfect sphere) over 34 mm translation along a radius was measured. We also measured an rms value of 0.28 mrad over 33 mm translation in the opposite direction (i.e., along one diameter). Given an analyzer position such that the angular acceptance of the analyzer is comparable to these slope errors, we estimate from these measurements that the analyzer should have a useful diameter of ~ 67 mm.

We have also characterized analyzers using synchrotron radiation. We tested analyzer #6 at NSLS using 7.603 keV radiation at a Bragg angle of 87°. The dynamical Darwin width is 0.21 millirad at this Bragg angle. The analyzer was arranged to diffract the beam coming from the monochromator with the incident beam traversing the sample position but with no sample present. A two-theta scan around the sample position was made from which the useful diameter of the analyzer was obtained. The FWHM of the scanned profile was 74 mm. The complete profile drops to zero very rapidly outside this diameter. We note that the measured result is close to the expected value as gauged from slope error measurements.

IV. Inelastic Scattering Results

During undulator experiments¹⁰ at the Cornell high energy synchrotron source (CHESS), we measured the IXS resolution function. The experimental arrangement is shown in Fig. 3. The resolution function is a convolution of the monochromator spectral reflectivity function with that for the analyzer. (We used an earlier generation of the analyzer for these experiments.) The beam from the undulator source had a vertical divergence of only 0.035 millirad FWHM. The monochromator optics were designed to match this divergence and have a spectral FWHM of 0.15 eV. We measured a value of 0.30 ± 0.07 eV for the elastic peak from a powdered C₆₀ sample for a Ge(444) analyzer set to Bragg diffract at 89.2°. Projecting these results to the APS where we expect to have only 0.025 millirad vertical divergence, we expect to be able to reach the desired resolution of 0.2 eV.

We have obtained IXS data for single crystal 6H-SiC using analyzer #6 at beamline X21 at NSLS.¹¹ The results are shown in Fig. 4 and clearly reveal the presence of a plasmon.¹² These data were obtained in reflection at 7.60 keV, and we obtained a value of 1.05 eV for the width of the elastic peak from the SiC. The IXS data were obtained by scanning the monochromator energy and collecting at a fixed analyzer energy. The ordinate was converted to an equivalent count rate for 200-mA current in the storage ring. Error bars ($\pm$ one standard deviation) are for a Poisson distribution.

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Figure Captions

- 1) Reflectivity as calculated using a dynamical matrix simulation method.
- 2) Analyzer #6.
- 3) Schematic layout of IXS experiments at CHESS.
- 4) IXS data for single crystal 6H-SiC showing the plasmon peak.

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