Sapphire analyzers for high-resolution X-ray spectroscopy

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Abstract

We present a sapphire (Al\textsubscript{2}O\textsubscript{3}) analyzer for high-resolution X-ray spectroscopy with 31-meV energy resolution. The analyzer is designed for resonant inelastic X-ray scattering (RIXS) measurements at the CuK\textsubscript{\alpha} absorption edge near 8990 eV. The performance of the analyzer is demonstrated by measuring phonon excitations in beryllium because of its known dynamical structure and high counting rates.

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1. Background

Inelastic X-ray scattering (IXS) has proven to be very useful in studying collective excitations in condensed matter systems in various fields [1–3]. The technique is bulk sensitive, and finite momentum transfer helps to identify the localized or dispersed nature of the excitations. Furthermore, with the help of microfocusing mirrors, small samples can be studied at high pressures or in magnetic fields.

Since the very first days of X-ray spectroscopy, lack of intensity has been one of the biggest challenges [4]. The advent of synchrotron radiation has significantly improved this situation by making intense monochromatic beams more readily achievable. The current difficulty is primarily in achieving high efficiency in crystal analyzers. Unlike the monochromator, the analyzer has to accept radiation spherically scattered from a point-like sample. The problem stems from the difficulty with efficiently collecting photons of a narrow bandwidth scattered from a point source.

Spherically bent analyzers are used to satisfy the same Bragg condition at every point on the analyzer surface [5,6]. Thus, all the rays diffracting from the analyzer are focused to a point. A good focus can be achieved on a detector spot when the sample, the analyzer, and the detector are placed on a Rowland circle, the diameter of which is equal to the radius of curvature of the analyzer. Bending a crystal wafer introduces stress due to elastic deformation of lattice planes, which broadens the band-width [7–9]. In order to avoid this effect and keep the lattice periodicity undisturbed, crystal wafers need to be diced and mounted on a spherical surface so that stress-free flat crystal pixels make a mosaic spherical surface. The resolution of the analyzer would then vary with the size of the pixel because the Bragg angles on two extreme points of a single flat pixel differ by an amount that depends on the size of the pixel and its distance from the scattering center [5]. The tolerance to this variation is proportional to the intrinsic angular acceptance of a crystal and is larger when
the Bragg angle is very close to 90°. This makes it advantageous to design an analyzer that operates at or very close to backscattering. Another advantage of being in a backscattering geometry is negligible demagnification. Geometrical contributions to the energy resolution become smaller [10] and focusing becomes less sensitive to deviations from the Rowland circle condition.

2. Analyzer design

The commonly used materials for high resolution analyzer, silicon and germanium have a single type of atom in a cubic unit cell. As a possible alternative, Al2O3 has a hexagonal unit cell with two different types of atoms. This less symmetric, multiple atom arrangement yields a backscattering geometry with unique energies to be more than an order of magnitude more possible backscattering planes to match with unique energies to be studied [11,10]. Serendipitously, Al2O3 (0 4 14) has an edge resonant inelastic X-ray scattering (RIXS) measurements.

As the backscattering energy is already very close to the energy at which RIXS measurements are performed, the analyzer can be designed to take advantage of this near backscattering condition. Our analyzer operates at an 89.87° Bragg angle. The angular acceptance for Al2O3 (0 4 14) lattice reflection is then 1800 μrad. This allows us to make 1.8-mm pixels along the scattering plane for a quasi-backscattering analyzer located 1 m away from the scattering center without any loss of the intrinsic features of the crystal.

Grinding and cutting of the Al2O3 crystal introduces surface damage. These damaged layers are removed with an etchant composed of a 1:1 mixture of 95% sulfuric acid (H2SO4) and 85% phosphoric acid (H3PO4) at 300 °C [12]. The analyzer needs a special bonding technique that can tolerate the concentrated acids at the high temperatures involved in the etching process. The technique used here is metallic diffusion bonding, which involves two layers of metal, i.e., Cu and Au, pressed against each other in vacuum at a temperature close to their melting point [13,14].

Here is a brief summary of the steps for preparing the sapphire analyzer. Sapphire wafers are etched for 4 h initially. Grooves 1 mm deep and 200 μm wide are made on one side of a 1.5-mm-thick sapphire disk with surface normal oriented along the (0 4 14) lattice direction. After bonding this side to another flat sapphire wafer (300 μm thick) with an orientation different than the previous one, the remaining grooves are cut, leaving isolated pixels bonded to the sapphire supporting wafer. This two-sided cutting technique is to prevent scratching the supporting wafer, because any possible scratch may cause the wafer to crack during bending. After this last cutting step, the analyzer is etched for approximately 20 min. The whole assembly is then glued with epoxy resin (EPO-TEK® 301-2) to a blank concave sapphire lens with 1 m radius. Using the same material for the lens, substrate, and pixels helps prevent possible stress due to different thermal expansion coefficients.

3. Experimental details

Experiments were carried out at beamlines 3-1D and 30-ID of the Advanced Photon Source. Synchrotron radiation was first filtered by a diamond (1 1 1) premonochromator to a level of 600 meV, then further monochromatized by a four-reflection Si (4 4 4) high-resolution monochromator to 22 meV. Monochromatic beam was scattered from a Be sample and analyzed by the Al2O3 (0 4 14) analyzer placed at an angle that defines the momentum transfer. The analyzed beam is focused back to an Amptek® CZT-PIN photodiode detector. The analyzer was 20 mm in diameter and placed 1 m away from the sample point. This geometry gives a solid angle approximately 3.24 × 10⁻⁴ steradian which determines the momentum resolution to be 0.16 Å⁻¹.

Energy scanning can be performed by either keeping the incident energy fixed and varying the analyzer energy or vice versa. The analyzer energy can be scanned by either changing the Bragg angle or the d-spacing by varying the temperature. Fig. 1 shows the spectrometer resolution curve measured by scanning the incident energy as well as measured by scanning the temperature of the analyzer when the incident beam energy was fixed. They are identical within experimental error, and the overall resolution is measured to be 38 meV. The reason to consider temperature scanning is that for RIXS measurements with specific incident energy requirements the analyzer energy has to be scanned to measure the spectrum.
of the sample. Changing the Bragg angle of the analyzer to scan the energy would push the analyzer far away from its 90° Bragg angle and sacrifice the advantages of back-reflection. The thermal expansion coefficient of sapphire is 4.9×10⁻⁶ K⁻¹ at room temperature. Backscattering energies for the Al₂O₃ (0 4 14) lattice reflection at 100 K and 500 K are 9001.0 and 8977.61 eV, respectively, which gives more than 20-eV scanning range.

In order to assess the efficiency of the spectrometer, we measured phonon excitations in beryllium at the first and second Brillouin zones along (0 0 ζ). Since there is no resonance effect expected around 9000 eV for Be measurements, spectra were collected by scanning the incident energy. Phonon excitations at energies as low as 28 meV were observable as a demonstration of the resolving power of the spectrometer. Fig. 2 shows our measurements compared with data taken previously [15,16].

4. Conclusion

It is shown that alternative crystals can be considered to meet specific requirements for RIXS measurements. For copper Kα edge experiments, the Al₂O₃ (0 4 14) lattice reflection appears to be a good match with regard to resolution, angular acceptance, and reflectivity. The novel feature of this spectrometer over other RIXS instruments is that the analyzer operates very close to backscattering. This enhances efficiency without sacrificing resolution. As an example, Be phonon dispersion has been measured and compared with data reported by others [15,16]. It may be possible to further improve collection capacity by making larger analyzers.

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