High Performance X-ray Energy Analyzers

A spectrometer for inelastic X-ray scattering (IXS) experiments involves two essential and crucial optical elements: a monochromator and an analyzer. The monochromator delivers X-rays with specific energy and resolution to the sample, and the analyzer analyses the scattered X-rays from the sample in the energy and momentum space by rocking the analyzer crystal at various scattering angles. In inelastic X-ray scattering, the process is incoherent and the scattering cross-section is low. In order to increase the counting efficiency the scattered X-rays are usually collected over a solid angle and then focused to a detector. Such an analyzer is usually fabricated by spherically bending a perfect single crystal. However, the strain induced by the bending is harmful to the energy resolution of the analyzer. It is therefore a challenge to fabricate analyzers with the desired energy resolution suitable for high-resolution inelastic X-ray scattering experiments. We have fabricated such high-resolution analyzers for use in inelastic X-ray scattering experiments on beamline BL12XU. In this report, we briefly discuss various methods for fabricating these analyzers and their performance.

Fig. 1 shows a schematic layout of the X-ray spectrometer. The spherical crystal analyzer shown is composed of well-aligned diced square crystals. The sample (signal source), spherical crystal analyzer and detector are positioned on the Rowland circle to satisfy the focusing condition. The spherical crystal analyzer acts like an energy filter, diffracting inelastically scattered photons only within a certain energy range (energy resolution of the analyzer) into the detector. The detector is placed close to the sample to satisfy the backscattering condition for the optimum resolution. This geometry is crucial in determining the energy resolution. The larger the radius of the analyzer R is, the closer the set-up is to the backscattering configuration. In addition to the use of a larger radius R, the reduction of the block size C and the source size S can also result in a better energy resolution. However, reducing the block size C sacrifices the efficiency of the analyzer because there is only less area available for diffraction as the size of the blocks is reduced. Hence, the size of every block of the diced crystal cannot be too small. The compromise is that c/R should be on the order of the Darwin width (intrinsic value) for the diffraction from the perfect crystal blocks, which sets the block size to about 0.5 mm.

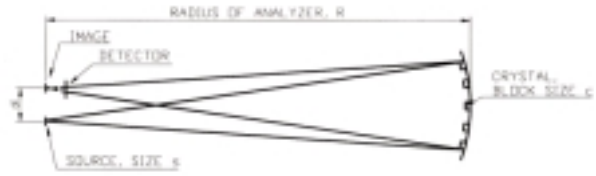

Fig. 1: Schematic layout of X-ray spectrometer used in inelastic X-ray scattering.

The energy resolution of a spectrometer is not only determined by its configuration, but also on the perfection of the analyzer crystal. Experimentally, a higher resolution is achieved by using higher order reflections in the backscattering mode. A continuously bent crystal has the maximum active area, but also inherits substantial bending strain that broadens the resolution. It is therefore necessary to eliminate all strain-induced effects within the crystal in fabricating analyzers

for the ultrahigh resolution (1 meV) measurements. Bonding small flat crystal blocks onto a concave spherical substrate to form an energy analyzer can effectively reduce the elastic bending strain, but requires very sophisticated technical skill.

Many methods for fabricating the energy analyzer have been proposed and realized to achieve ultrahigh resolution for the study of lattice excitations. One of the most reliable ways is to dice many individual blocks on the wafer by using a diamond saw. The sawing (grooving) is not done all the way through the wafer, but leaves a thin back-wall that holds the blocks in alignment. More than 9000 pieces (0.9 mm square blocks) of silicon crystals are formed after dicing with a diamond saw. Subsequently, the blocks are glued to a spherical concave substrate and the strained back-wall layer is etched away. All the crystals should have the same orientation to optimize analyzer efficiency. This method has been employed in making analyzers for use in nearly backscattering geometry to achieve ultra-high energy resolutions below 1 meV using high order reflections.

For the studies of non-resonant scattering from electron excitations and resonant Raman scattering, energy analyzers with resolutions of about 100 meV are appropriate. In this resolution regime, a lower order reflection is used, and hence the perfection requirement of the crystal is less strict than that in the ultrahigh resolution case. Un-diced bent wafers can be used to make analyzers with maximum active area (no loss of diffracting area due to the grooving widths), while keeping some tolerable strain-induced effects. Thus the key issue for fabricating the high-resolution analyzer is how to bend a thin crystal wafer and glue it uniformly onto a spherical concave substrate. Therefore, grooving, preliminary etching, gluing, curing and final etching are the required procedures for fabricating ultra-high resolution crystal analyzers, whereas only gluing and curing are needed for high-resolution bent crystal analyzers.

Based on the above considerations, we have developed various setups and procedures at the SRRC for the fabrication of spherically bent crystal analyzers with various energy resolutions, including a robotic system for gluing diced wafers, a unique pressing device to ensure uniformity at curing, and setups for performing chemical etching. The technical details will be reported elsewhere. Several such analyzers have been fabricated for use in the commissioning of BL12XU. These analyzers were tested on the SRI-CAT sector 3 beamline at the Advanced Photon Source. The elastic scattering from a Plexiglas using an incident beam of 9.885 KeV was analyzed to determine the energy resolution. Each analyzer tested had a 2-m radius with a mask covering about 20% area on the outer rim of the analyzer. These measurements were all performed using the Si (555) reflection. The results are summarized in Table 1. It is clear that analyzers made by diamond saw dicing and subsequent chemical etching have the best resolution of 105 meV, close to the calculated value of 94 meV. The spherically bent analyzer (un-diced and glued) has energy resolution > 200 meV because of the bending strain as expected. The prototype analyzer fabricated by the DRIE (deep reactive ion etching) method has energy resolution close to 200 meV, which is higher than that obtained from the diamond saw diced analyzer because the back wall was bent and glued to the substrate.

Table 1: Summary of the test results of various analyzers.

Analyzer #	Measured △E (meV) Direct Beam	*Calculated △E (meV)	Measured △E (meV) Plexiglas	Description
A	200	94	263	Spherically bent, non-diced, glued
B	151	94	190	DRIE diced (0.5x0.5 mm), 0.5 mm thick
C	198	94	238	Same as A
D	100	94	105	Diamond-saw diced and chemical etched

*The calculated value is determined from a convolution of incident beam width and back-reflection analyzer width.

In summary, we have discussed the methods currently in use for fabricating crystal analyzers for inelastic X-ray scattering experiments. Analyzers fabricated using these methods have been tested and shown to give energy resolutions in the range of 100 - 200 meV at 10 keV. New techniques, particularly those used in silicon nano-fabrication processes (e.g., deep reactive ion etching or DRIE) are now being explored to improve the dicing process, which offer the flexibility of selecting the block size, with finely controlled groove width (i.e., minimal loss of material), and hence the possibility of controlling the energy resolution of the analyzer.

Authors:

D. J. Wang, S.Y. Perng, C. K. Kuan, Y.Q. Cai and P. Chow

Synchrotron Radiation Research Center, Hsinchu, Taiwan