A Novel Scheme to Fabricate High Performance X-ray Energy Analyzers

The spectrometer for inelastic x-ray scattering (IXS) experiments contains two essential and crucial optical elements: a monochromator and an analyzer. The monochromator delivers x-rays photons of specific energy and bandwidth to the sample, and the scattered x-rays photons from the sample are analyzed in the energy and momentum space by the x-ray analyzer. Such an analyzer consists of diffraction optics made from spherically bent perfect crystal wafers, such as silicon single crystal. Bending the wafer into a sphere provides semi-focusing of the diffracted x-ray into the detector; collection efficiency is enhanced but at the expense of momentum resolution. However, the strain produced by bending the crystal decreases energy resolution and reduces the intrinsic reflectivity of the analyzer. To improve energy resolution and reflectivity, we designed an analyzer in which the wafer is diced into blocks before being glued onto the spherical concave substrate. Maintaining the relative alignment of these individual blocks during the gluing process is the most critical part of the entire fabrication process. Based on the technique and experience of the ESRF group, we have developed a novel pressing technique for the fabrication of high-resolution crystal analyzers, and fabricated analyzers with satisfactory performance for the Taiwan IXS beamline (BL12XU) at SPring-8. In this article, we will discuss in more detail the design and the fabrication process of this novel technique.

In a typical high-resolution inelastic x-ray scattering spectrometer using the Rowland circle geometry,the sample is positioned at the source and the detector at the focus of the spherical crystal analyzer which operates at the near-backscattering energy of a high-order reflection. The energy resolution of the scattering spectrum depends on a number of factors, and is predominantly given by the bandwidth of the incident x-rays and the energy resolution of the crystal analyzer: $(\Delta E_{spectrometer})^2 = (\Delta E_{incident\ beam})^2 + (\Delta E_{analyzer})^2$. For crystal analyzers made from diced wafers, the energy resolution at the near-backscattering geometry, as discussed in details in Publications, is a function of the source size, the angular width of the impinging x-rays (i.e., the ratio of the individual block size to the bending radius), the lattice gradient induced by the bending strain, and the temperature variation of the analyzer. Our estimate gives a lower limit of about 50 meV for analyzers with block sizes of 0.5×0.5 mm^2 and a 2-m bending radius operating in ambient conditions. In principle, one can improve the energy resolution with smaller block size or larger bending radius. However, equally important is the concern for the efficiency of the analyzer, which depends on the effective diffracting area left after dicing, and the relative alignment of the individual blocks. Poor alignment broadens the energy bandwidth, reduces the reflectivity, and degrades the focusing quality of the analyzer. It is therefore extremely important that the relative alignment of the individual blocks is maintained during the fabrication, particularly during the press and curing processes when the wafer is glued to the spherical substrate. To meet these requirement, we have developed a novel pressing tool which applies uniform and constant pressure on the wafer during the curing process, thereby maintaining the relative alignment between the individual blocks. To preserve the reflecting area, a well-controlled final etching method has also been developed to remove just the back-wall of the wafer with minimum loss of the diffracting area.

In general, the essential procedures for fabricating high-resolution crystal analyzers include grooving, preliminary etching, gluing, curing and final etching. Our fabrication technique is based on the technique and experience of the ESRF group, with improvements made in the pressing device and the final etching method. The design details and the fabrication process are discussed in the following paragraphs.

To achieve good bonding between the wafer and the concave substrate, it is necessary to apply pressure in such a way that close contact between the glued parts is attained. Furthermore, the relative alignment of the diced crystal blocks must be maintained precisely during the curing process, which involves both heating and cooling. One could use mechanical means (e.g, clamps and bolts) to apply the necessary pressure, however, it is difficult to maintain a constant pressure due to the thermal expansion or contraction of the mechanical parts during the heating and cooling process. A uniform press on the wafer also requires a pressing piece with a convex surface precisely matched to

the concave substrate, which adds to the fabrication cost and introduces additional figure error. Using flexible materials as the pressing surface and regulated air pressure, we developed a novel pressing tool which applies uniform and constant pressure on the crystal wafer during the curing process. Fig. 1 shows the schematic design of the pressing tool. A regulator from the compressed air inlet controls the pressure used for the pressing, eliminating variation due to thermal expansion or contraction of the mechanical parts used in the device. The Kapton foil in the assembly transmits the air pressure uniformly on the wafer along the direction perpendicular to the curvature of the spherical substrate. It should be noted that the relative alignment of the individual blocks was maintained by the 0.2-mm thick back-wall of the wafer. The uniform, constant force of the pressing tool also helped to prevent breakage or dislocation of the wafer during the curing process. After curing, the whole assembly took 6 hours to cool to room temperature.

The final etching removes the back-wall of the analyzer crystal, leaving each crystal block glued individually on the substrate to relieve transverse strain. The isotropic etching solution was used to etch away the 0.2 mm thick back-wall. To preserve the largest possible diffracting area of the analyzer, ideally only the back-wall should be etched away. If the etching is not done uniformly throughout the whole wafer surface, the solution will penetrate through the first open area, and etch the crystal blocks in that area. If the block size is reduced by 0.1×0.1 mm^2, the entire diffracting area will be reduced by 20%. Therefore it is important to control the uniformity of the etching throughout the entire process.

During the etching process, the silicon was first oxidized by nitric acid and then the product is etched away by fluoric acid. Bubbles are usually formed in the reaction area. If they stay on the wafer surface, they will prevent the reaction from proceeding smoothly, and cause non-uniformity. Therefore, uniform etching requires uniform flow of etching solution and a proper way to remove the bubbles from the wafer surface. We developed a rotating etching apparatus to implement this idea. The silicon analyzer was rotated vertically to remove the bubbles and the etching solution was stirred to create uniform flow. The etching uniformity is examined by a precision profiler, ACCRETECH SURFCOM 130A, which has vertical resolution better than 0.1 μm.With this etching apparatus we have obtained etching uniformity about 1 μm, and no local dip in the edge was visible. The entire etching process took about two hours. Figure 3 shows the photo of a finished product.

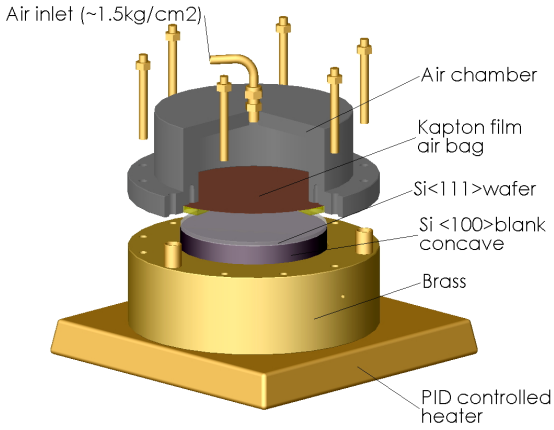

Fig. 1: *The uniform pressing tool that exerts constant pneumatic force during curing.*

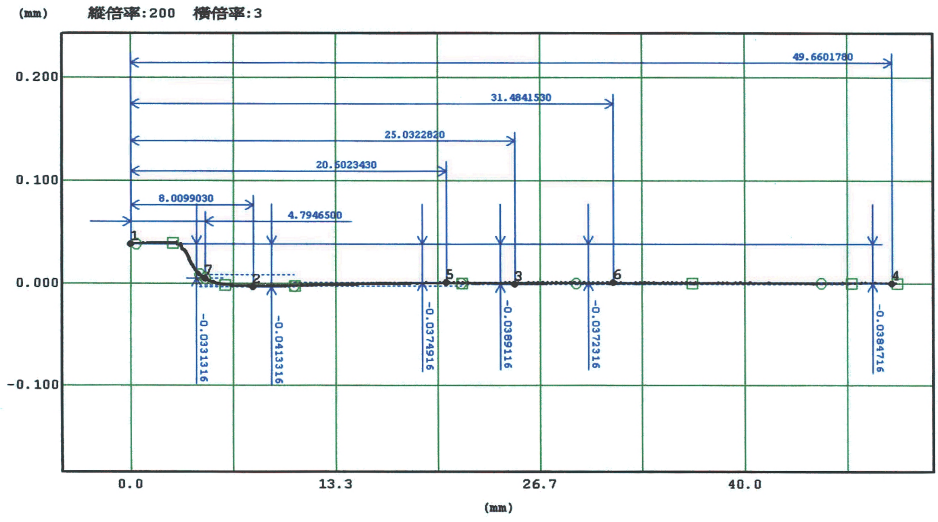

Fig. 2: *Final etching with micron uniformity.*

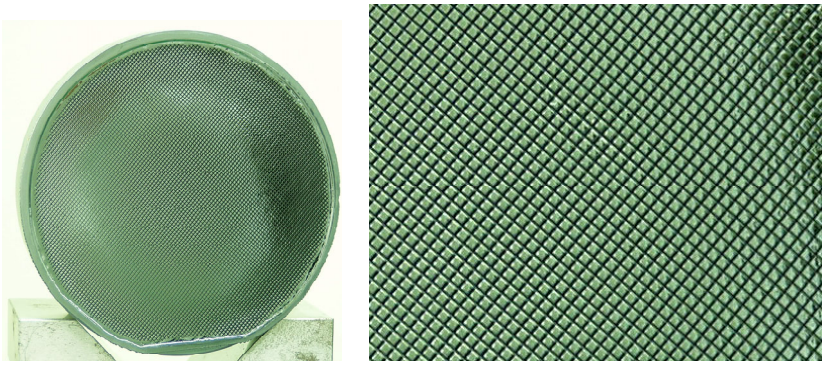

Fig. 3: A 2 m radius diced analyzer (left) and uniform crystals with 600 microns width (right)

Table 1. Summary of the test results of various Si (555) analyzers.

Analyzer	Analyzer Resolution (meV)
2-m Diamond-saw diced	49
2-m DRIE diced	157
2-m Continuous bent	225

We have made and tested several analyzers on the Taiwan inelastic x-ray scattering beamline BL12XU at SPring-8. The test results are summarized in Table 1. All tests were done at the near-backscattering energy of the Si(555) reflection at 9.886 keV. The incident beam was delivered from a Si(333) 4-bounce channel-cut monochromator, giving a 50 meV energy width at 9.886 keV. The sample was a 2 mm thick Plexiglas. The sample to detector distance was 55 mm. Each analyzer tested has a 2 m radius with a mask about 80% area opening in front of the analyzer. The total spectrometer resolution is obtained by measuring the FWHM of the quasi-elastic peak from the plastic sample. The analyzer resolution is then calculated by de-convoluting the incident beam energy bandwidth from the total spectrometer resolution. It is clear that analyzers made by the diamond saw dicing and subsequent chemical etching give the best energy resolution of 49 meV, which approaches the estimated limit achievable for a 2-m radius analyzer. The continuously bent analyzer (non-dicing and glued) has energy resolution > 200 meV because of bending strain. Details of the DRIE analyzer can be found. Some strain was relieved by the DRIE dicing which leads to an intermediate energy resolution.

In summary, we have presented a reliable way to fabricate high performance x-ray analyzers. In the fabrication process a novel uniform pressing tool is used for the curing and a well-controlled final etching process is applied to ensure uniform etching.

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