Design of a Dual-energy High-energy-resolution Backscattering Monochromator for Ultraprecise Studies of Thermal Expansion

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Abstract

A dual-energy, double-bounce, high resolution x-ray monochromator was designed for ultra-precise backscattering studies of the thermal expansion of SiC-4H at low temperatures. SiC-4H is a technologically important polymorph of silicon carbide. Two Si backscattering Bragg reflections were chosen such that their d-spacings match those of two independent backscattering reflections in SiC-4H. Switching between the two backscattering energies requires crystal rotations in the range of a few degrees. Fine tuning of the photon energies is to be accomplished by high-precision scanning of the temperature of Si crystals. The Si crystals were cut with asymmetric geometry to increase the footprint of the incident x-ray beam on the crystal surface, which will help to minimize possible crystal deformations due to thermal x-ray loading. The working crystal surfaces were polished to mirror-like appearance using developed multi-step procedure in order to reduce damage to the crystal lattice from cutting. The crystallographic orientation of the surfaces was verified using Laue x-ray diffractometry. Final verification of the quality of the crystal surfaces will be performed using x-ray topography. Additional steps to improve the crystal quality could include chemical etching and further polishing.

1. Introduction

SiC is an important semiconductor material, but still not much is known about its thermal properties at low temperatures. One way to explore the thermal expansion of SiC is to use x-ray diffraction in backscattering using a high-energy-resolution energy-tunable monochromator [2,4,5]. Due to the hexagonal lattice structure of SiC, two backscattering reflections will need to be studied for the SiC-4H sample. Therefore, two Si backscattering Bragg reflections are chosen such that their d-spacings match two independent backscattering reflections in SiC-4H. In backscattering, the relative spectral resolution (dE/E) of the monochromator is comparable to the Darwin width of the studied reflections. Changing the temperature of the Si crystals in the monochromator will allow it to approach the appropriate energy levels. The double-crystal arrangement is used to preserve the original direction of the x-ray beam (i.e., in-line monochromator). Using a goniometer to adjust the orientation of the monochromator crystals in the azimuthal direction will allow it to switch between these two backscattering reflections. Since the thermal expansion of Si is known to a good precision, the photon energy scale of the monochromator can be calibrated with great accuracy. The Si crystals are cut asymmetrically to reduce temperature gradients due to heat load on the incident x-ray beam. One of the biggest considerations in crystal design is having a flat and strain-free surface to ensure the narrow bandwidth of the monochromator. Cutting and grinding the crystal to the desired crystal surface orientation typically leaves a damaged surface layer which has to be removed using a polishing and etching process [3]. The resulting surface quality can be characterized using reflection X-ray topography.

In this work, the asymmetric Si crystals for the low-temperature x-ray backscattering study of thermal expansion of SiC-4H were designed and fabricated. A prior attempt of polishing and etching asymmetric Si crystals was characterized using x-ray topography. The results were not found satisfactory. The new crystals were polished using the revised procedure. A good mirror-like working surface quality has been achieved. The etching step was skipped. Further progress to be demonstrated upon x-ray topography characterization.

2. Matching Backscattering Reflections of Si and SiC-4H

We found matching (in terms of d-spacing) backscattering reflections in Si and SiC-4H.
Eb_Si = 10398.896 eV h k l = 7 5 3 (cubic) (12 meV)
Eb_SiC-4H = 10398.537 eV h k l = 1 3 10 (hexagonal crystal lattice)
Both have d=.59 Angstroms and thus Eb=10398 eV

Eb_Si = 14794.586 eV h k l = 10 8 2 (5 meV)
Eb_SiC-4H = 14794.028 eV h k l = 6 0 8
Both have d= .42 Angstroms and thus Eb=14794 eV

SiC-4H lattice spacings are calculated at 100K, and those of Si are calculated at 300K. For Si thermal expansion data of Reeber (Phys. Stat. Sol. (a) 32, 321 (1975) were used. For SiC-4H low temperature extrapolations of thermal expansion data of Stockmeier et al, (J. App. Phys. 105, 033511 (2009) were used.

3. Schematics of the Monochromator

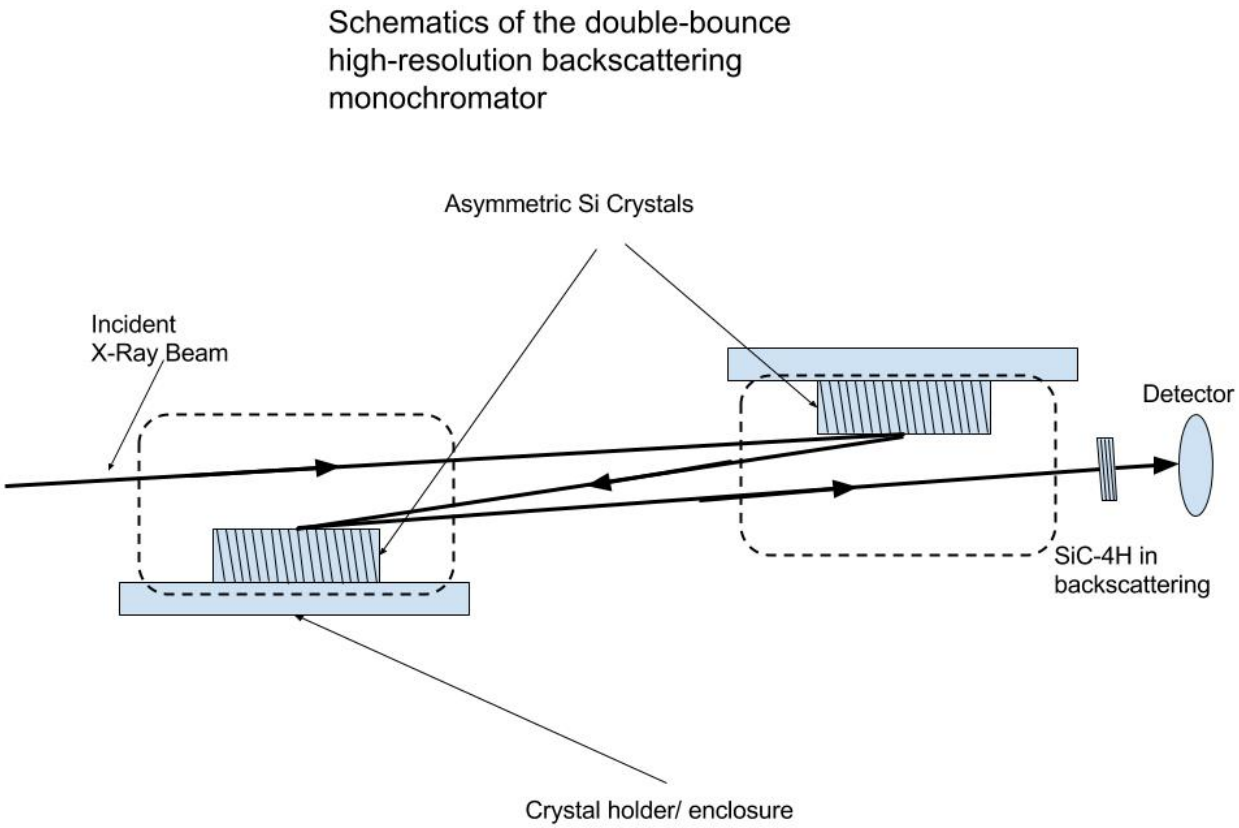

Figure 1: Schematic of the double-bounce, high-resolution backscattering Si monochromator. The monochromator acts as a filter for the energy of the incoming incident x-ray beam. With a narrow energy bandpass, we can analyze the thermal expansion coefficients of SiC-4H with greater detail. Switching between 7 5 3 and 10 8 2 backscattering reflections can be performed by small (3.1 deg) azimuthal rotation of the Si crystals.

The energy bandwidth of the monochromator (dE/E) is comparable to the Darwin width of the studied reflections. The SiC-4H 1 3 10 darwin width is 17.5 meV. In order for dE/E to be narrow, surface crystal damage due to mechanical processing should be minimized. A specialized polishing procedure is required.

We want to scan energy of the monochromator by changing the temperature of the Si crystals [4,5]. Since the thermal expansion of Si is well known, we can measure the thermal expansion of other materials using Si data as reference.

3. Crystal Design

We used an asymmetric crystal geometry to reduce temperature gradients [1].
The monochromator is designed to be dual-energy to allow for a small azimuthal angular adjustment switch from 7 5 3 to 10 8 2.

A 5 degree adjustment angle is needed to provide optimal asymmetry angles.
Asymmetry angles should be in the range 70-88 degrees.
The 5 degree adjustment makes asymmetry angles for Si hkl 7 5 3 and 10 8 2 change from 70.77 and 81.13 to 75.77 and 86.13 degrees respectively.

Si hkl	Asymmetry Angle (degrees)	Backscattering Energy (eV)	Matching SiC-4H hkl	Si hkl Backscattering Bandwidth (meV)
7 5 3	75.77	10398.896	1 3 10	12
10 8 2	86.13	14794.028	6 0 8	5

Figure 2: Specifications of the monochromator crystals

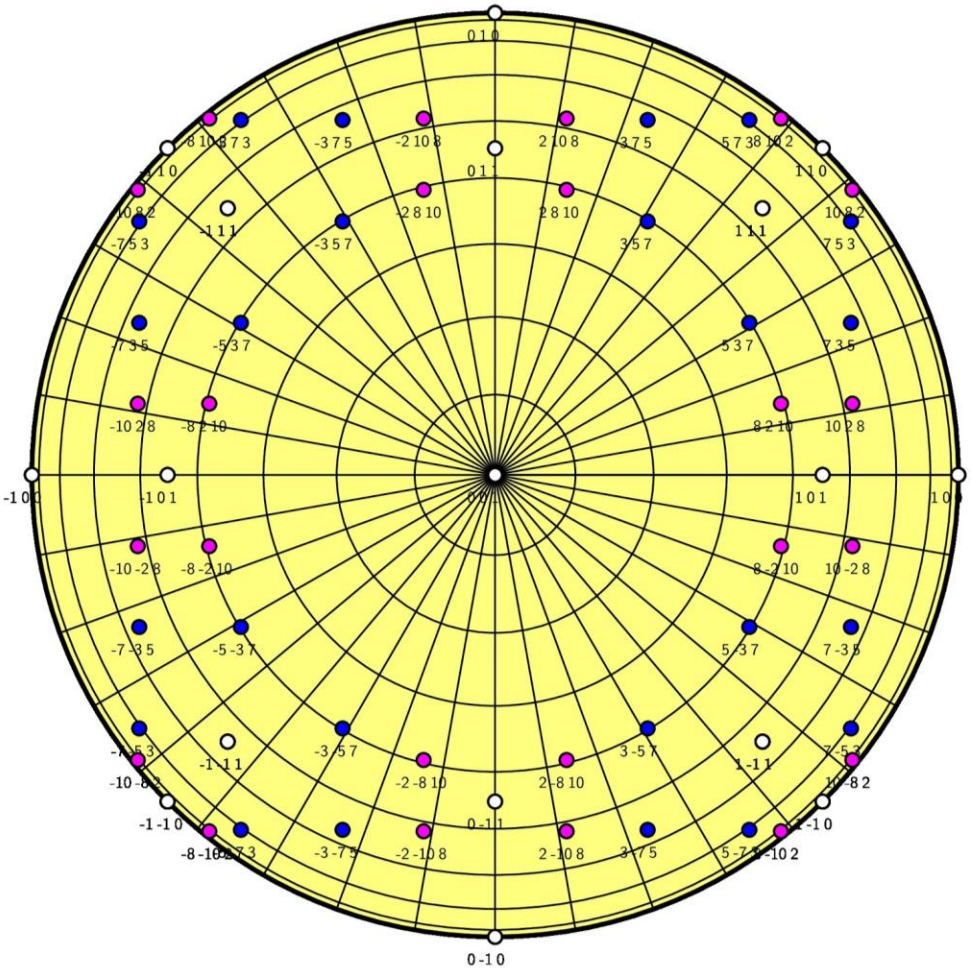

Figure 3: Reciprocal Space: A polar figure showing orientation of the 7 5 3 and 10 8 2 reciprocal vectors with respect to the main crystallographic directions 0 0 1 and 1 1 0 in a cubic crystal. The reciprocal vector 0 0 1 is normal to the drawing and is pointed outboard.

4. Crystal Fabrication

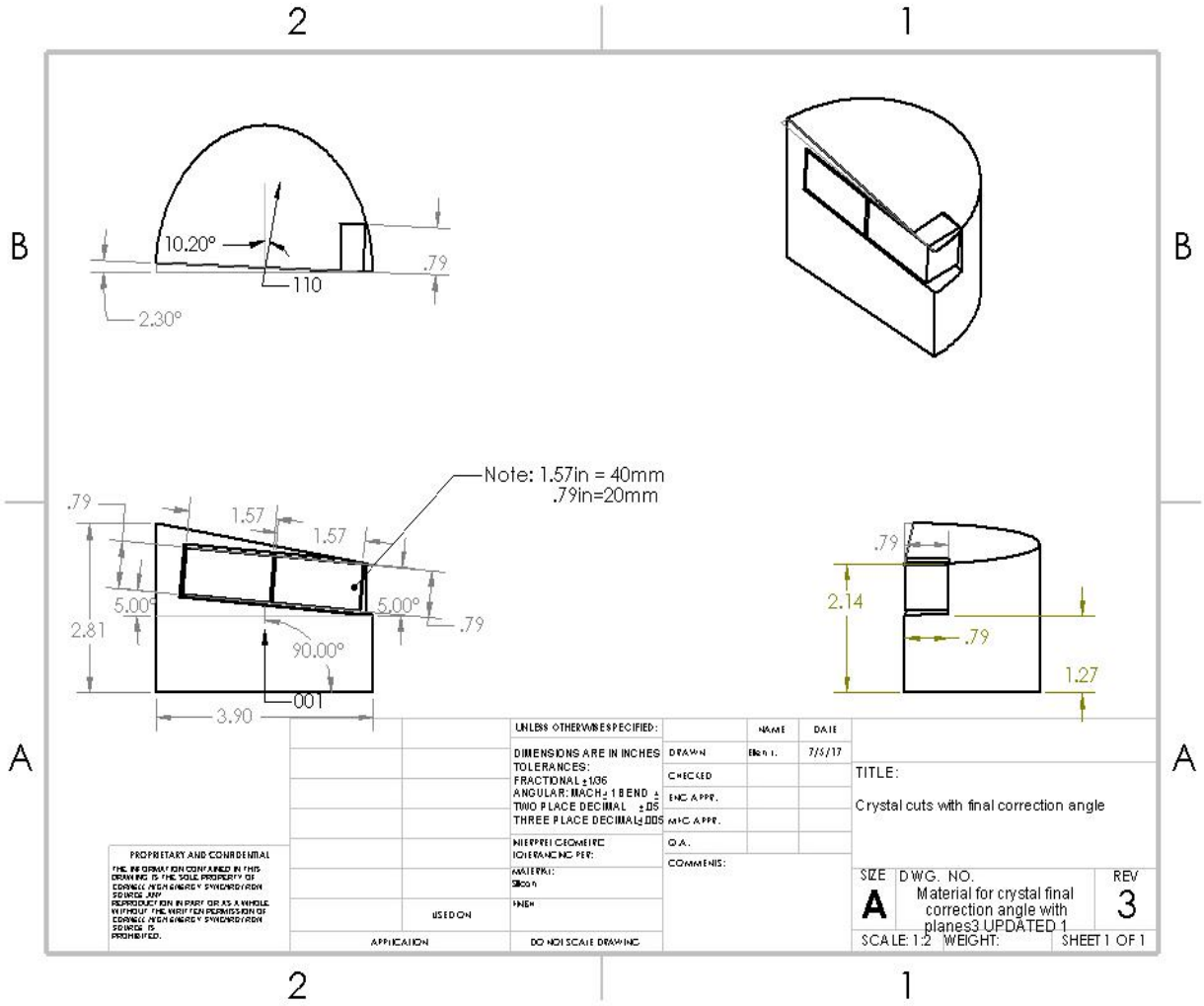

Figure 4: Drawing for cutting the Si crystals

A 2.3 degree adjustment is made to place the longer side in between the 7 5 0 and 10 8 0 planes.

A 5 degree correction angle is used to achieve the previously specified asymmetry angles.

Figures 5a-e: Crystal orientation using Laue diffractometry prior to cutting

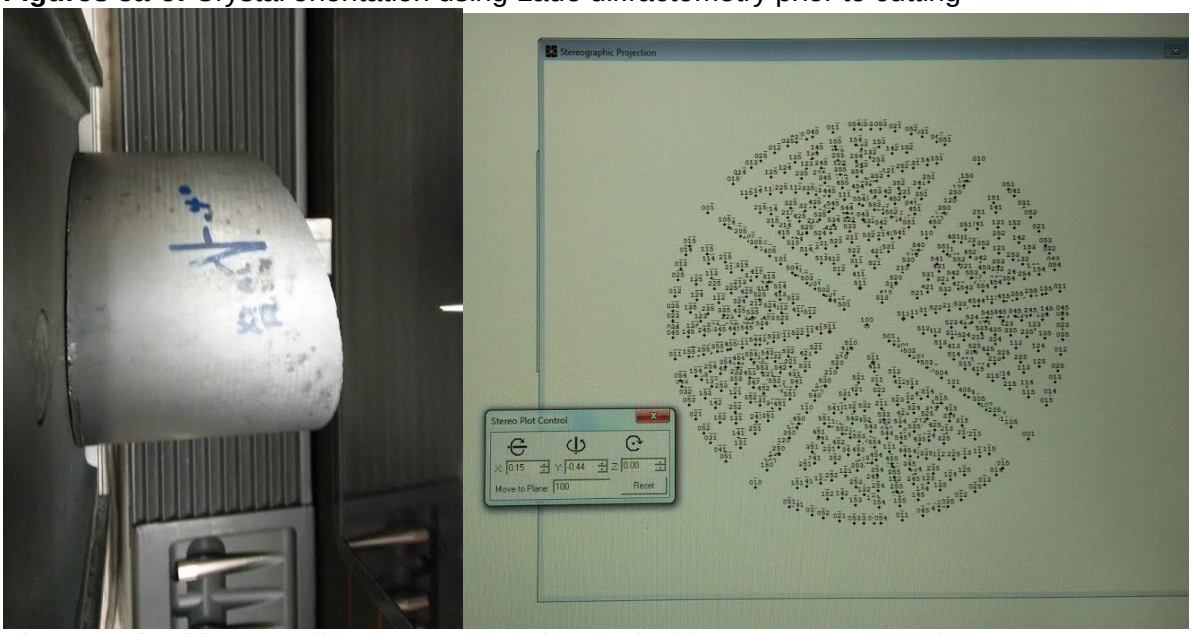

Figure 5 (a - b): Laue diffractometry of Si crystal with respect to 1 0 0 plane

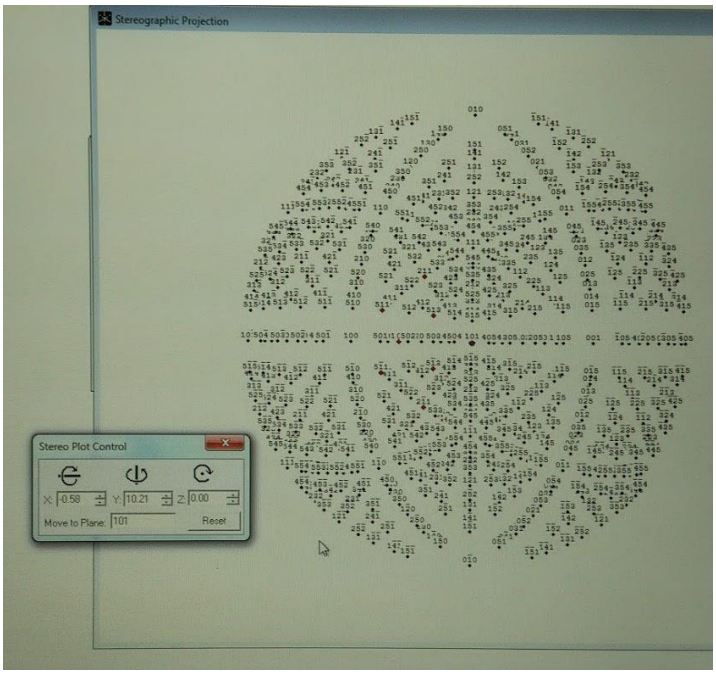

Figure 5(c): Laue diffractometry of Si crystal with respect to 1 0 1 plane

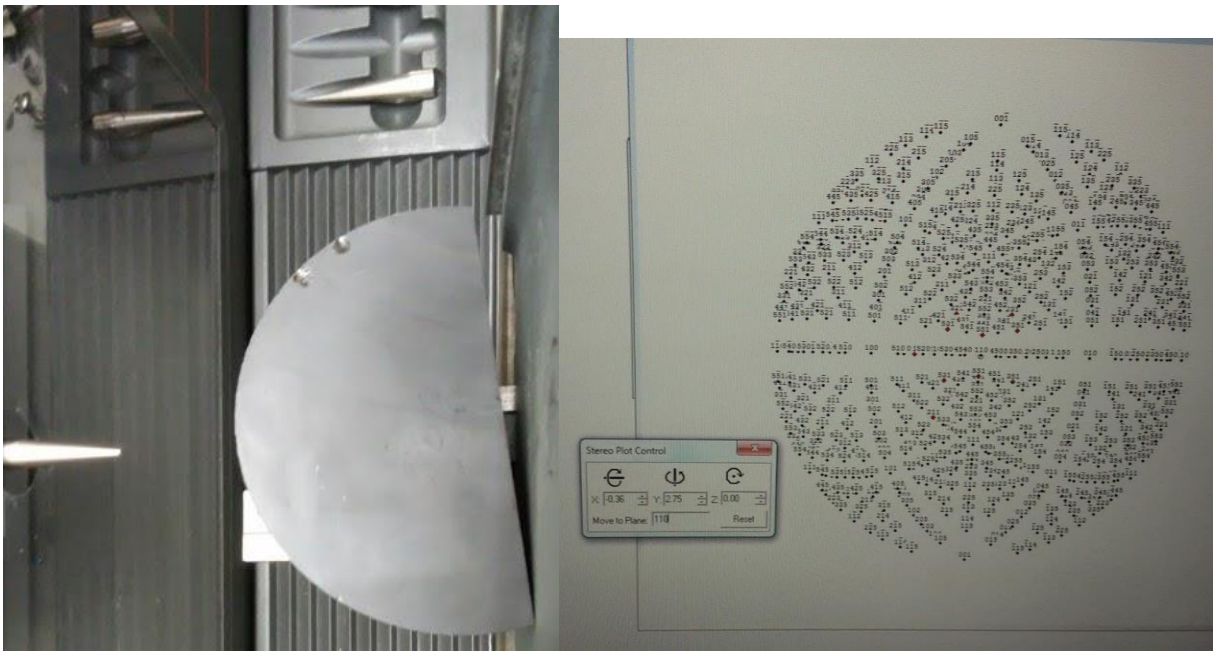

Figure 5(d - e): Laue diffractometry of Si crystal with respect to 1 1 0 plane (at about 10 degree angle to platform).

Laue diffractometry shows us where planes in the Si crystal lattice are located and how we needed to adjust to get to proper working, reflecting, and surface planes.
The top plane is about 10.25 degrees off of the 1 1 0 plane. Cuts were required to readjust the location of the plane.

Previously, two identical Si 2 2 0 asymmetric crystals were cut, ground, etched for ½ hour in solution of HF/HNO_3 acid and polished using the following procedure:

1. 3 um diamond lapping film (DLF)
50 rpm, 30 min
rotation + oscillation
2. 1 um DLF - 30 min
3. 0.3 um Al_2O_3 slurry on vibratory polisher 48 hours
4. 0.02 um colloidal silica
24 hours on vibratory polisher

Removing Sample:
1. Sonicate in Acetone 20 min
2. Hot Acetone 10 min
3. Hot TCE 10 min
4. Hot Methanol 10 min

The asymmetry angle was 10.5 deg.
X-ray topographs were taken in reflection geometry of 16.5 keV x-rays across the two Si 2 2 0 crystals. The experiment was performed at A2 station of CHESS using a double-crystal diamond crystal monochromator delivering a monochromatic beam of size of about 0.5 x 2.0 mm (vertical x horizontal).
Figure 6a shows individual topographs of the crystal #1 undergone the complete polishing procedure. Figure 6b shows topographs for the other crystal #2 processed similarly but skipping the last polishing step (0.02 um slurry).

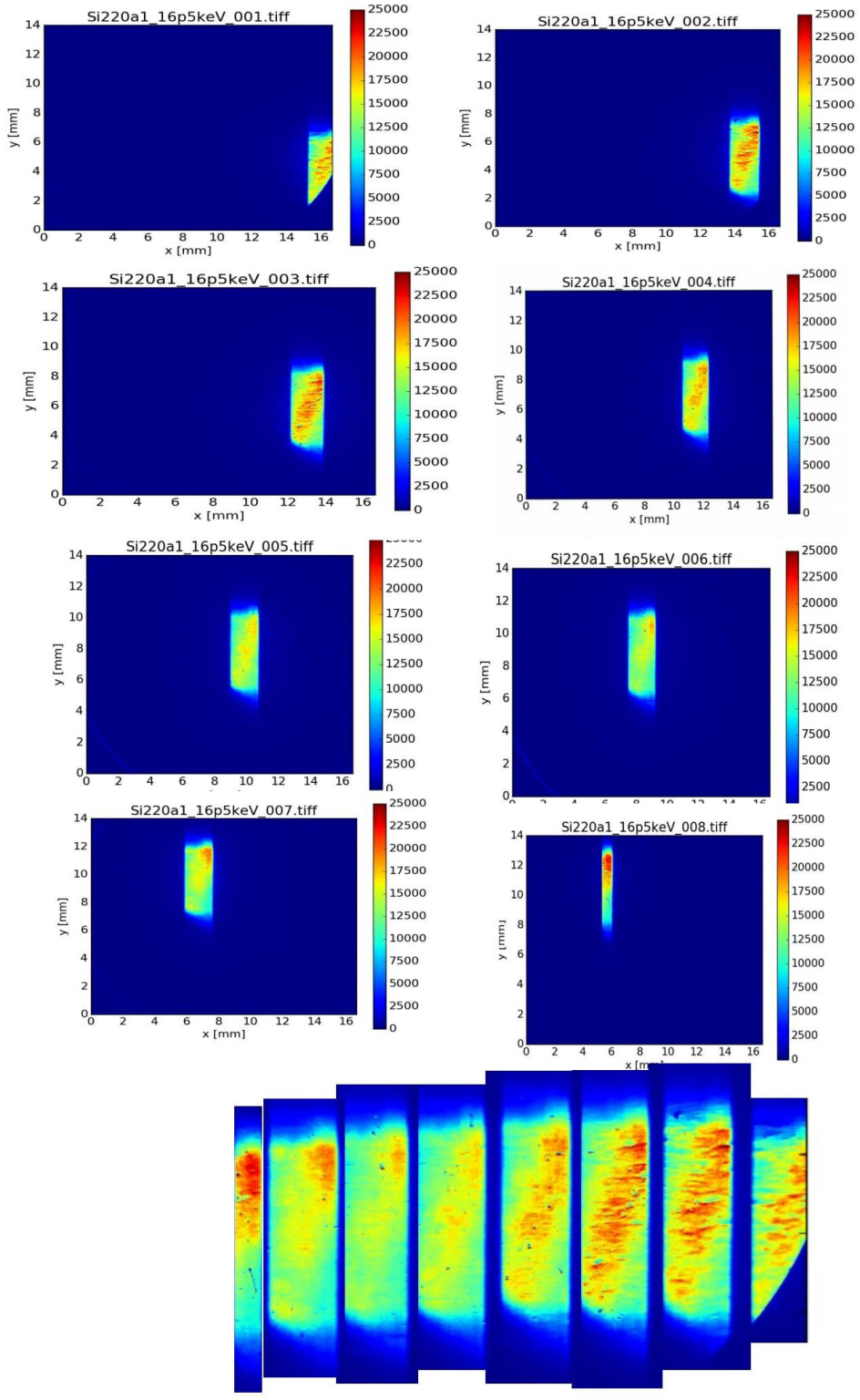

Figure 6 (a): X-ray topographs of the Asymmetric Si 2 2 0 Crystal #1

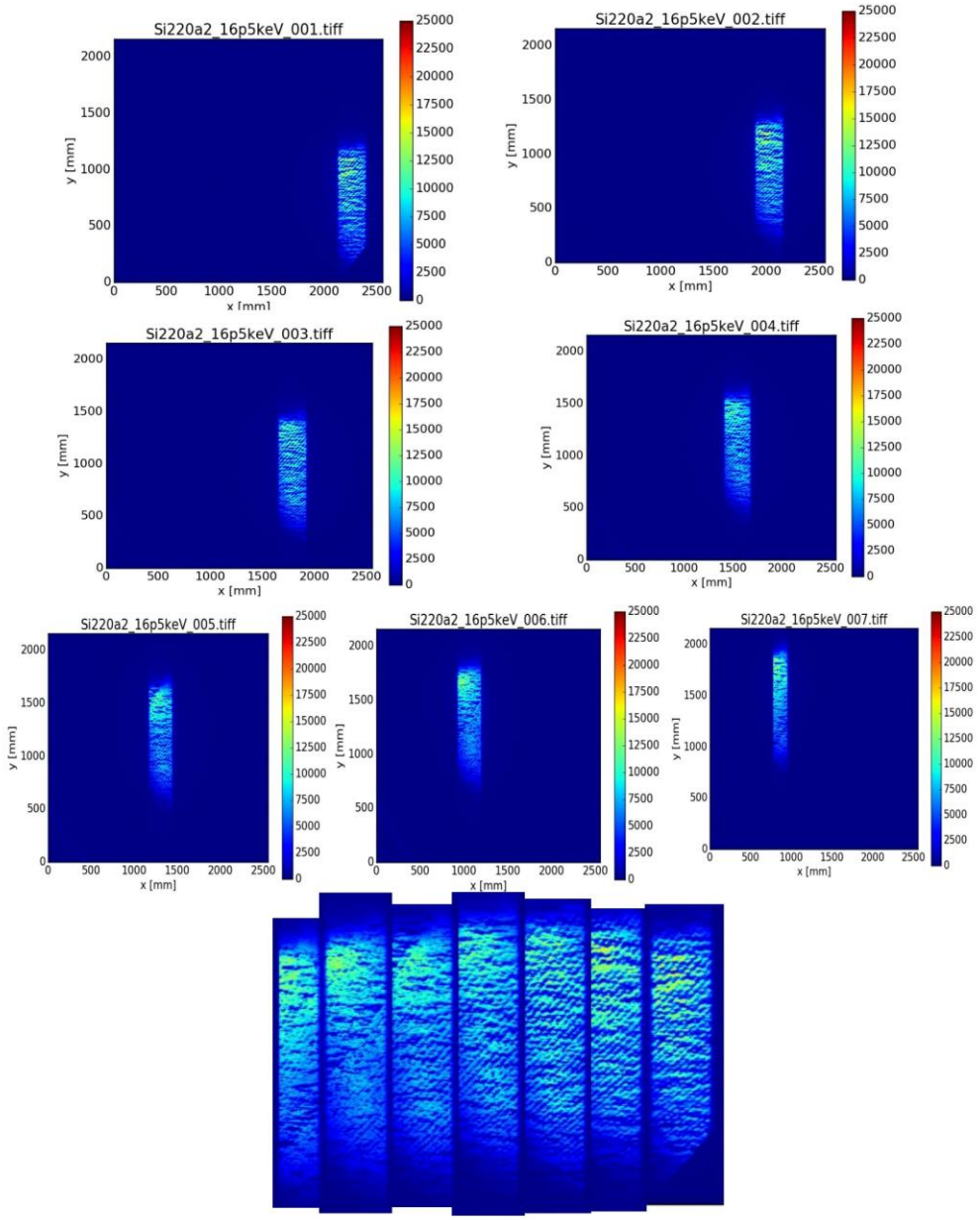

Figure 6 (b): X-ray topographs of the Asymmetric Si 2 2 0 Crystal #2

The topographs have shown presence of strain on crystal surfaces. For crystal #2 traces of the cutting wheel were not removed by the etching/polishing process. A revised polishing procedure was attempted for the asymmetric crystals of the dual-energy monochromator.
It was decided to skip the etching step and polish the crystals to a mirror-like appearance.

Steps used for polishing:

First crystal:
1. 2 hours of polishing on polishing wheel using 3 um diamond film at 50 rpm
   rotation + oscillation
2. 2 hours of polishing on polishing wheel using 3 um diamond film at 100 rpm
   rotation + oscillation
   (1st hour rotating clockwise
   2nd hour rotating counterclockwise)
3. 30 min rotating counterclockwise on 1 um diamond film at 100 rpm
   rotation + oscillation

Second crystal:
1. 1 hour 9 um SiC film 50 rpm
   just rotation
2. 2 hour 3 um film diamond film 50 rpm
   just rotation
3. 3.5 hour diamond film 100 rpm
   rotation + oscillation
4. 30 min 1 um film 100 rpm clockwise
   rotation + oscillation
5. 30 min 1 um film 100 rpm counterclockwise
   rotation + oscillation

Both crystals then subject to vibratory polishing:
1. 48 hours of vibratory polishing using the 0.3 um Al_2O_3 slurry
2. 24 hours of 0.02 um colloidal silica slurry

Remove crystal bond compound from the samples:
1. Sonicate in Acetone for 20min
2. Hot Acetone for 10min
3. Wash in Methanol, wipe off residual Acetone

Monochromator Si crystal pieces at different points in polishing process:

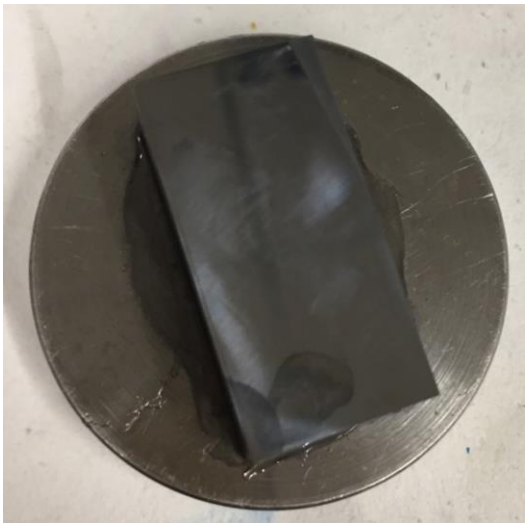

Figure 7(a): Asymmetric Si 7 5 3 Crystal 1 after polishing wheel
Facets present, some scratches present

Figure 7(b): Asymmetric Si 7 5 3 Crystal 2 after polishing wheel,
Reflective except for left edge, some scratches present

Figure 8: Asymmetric Si 7 5 3 Crystal 1 and 2 after Al_2O_3 slurry
Removed facets on second crystal, some scratch removal on both crystals

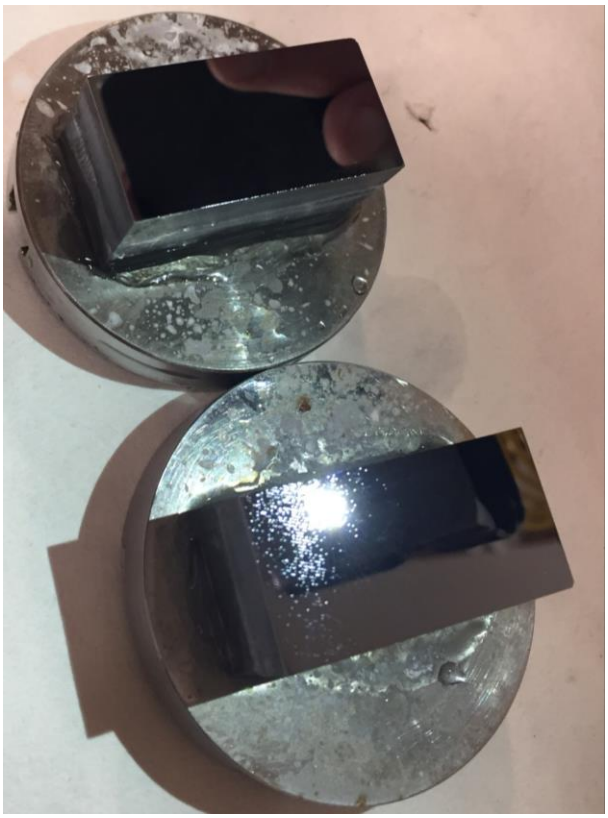

Figure 9: Asymmetric Si 7 5 3 Crystal 1 and 2 after Silica slurry, Small scratch removal

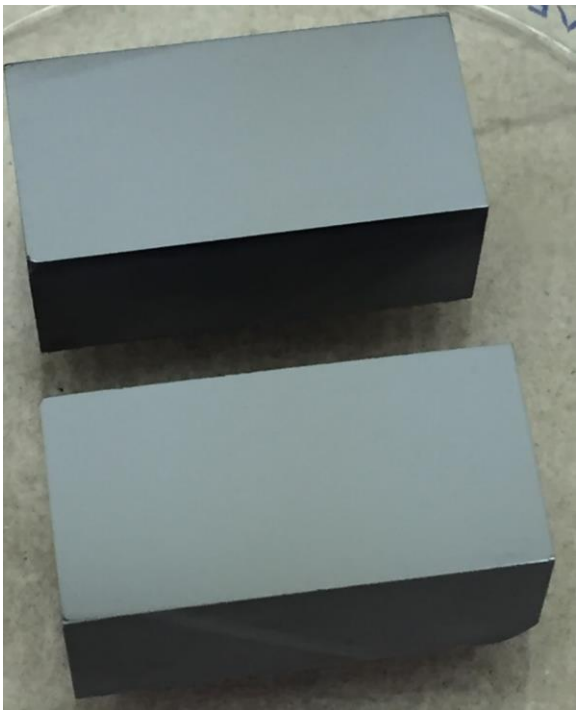

Figure 10: Asymmetric Si 7 5 3 Crystal 1 and 2 after cleaning with acetone and methanol Pieces appear very reflective, Mirror-like appearance of the working surfaces except some remaining cutting grooves on crystal 1 about ⅓ length from the edge (still about ⅔ of the crystal have mirror-like appearance)

5. Evaluation of Crystal Quality:

1. It has to look mirror-like and have minimal scratches.
2. Should have flat, planar top surface. (Facets should not be present)
3. X-ray topography allows detailed analysis of crystal quality

Both crystals now appear to have flat planar working surface and are mirror-like.
To verify locations of any defects, x-ray topography will need to done since they are difficult to see with the naked eye.

6. Verification of Crystal Orientation Using Laue Diffractometry

Figure 11(a): Side view of Asymmetric Si 7 5 3 Crystal 1 during Laue diffractometry

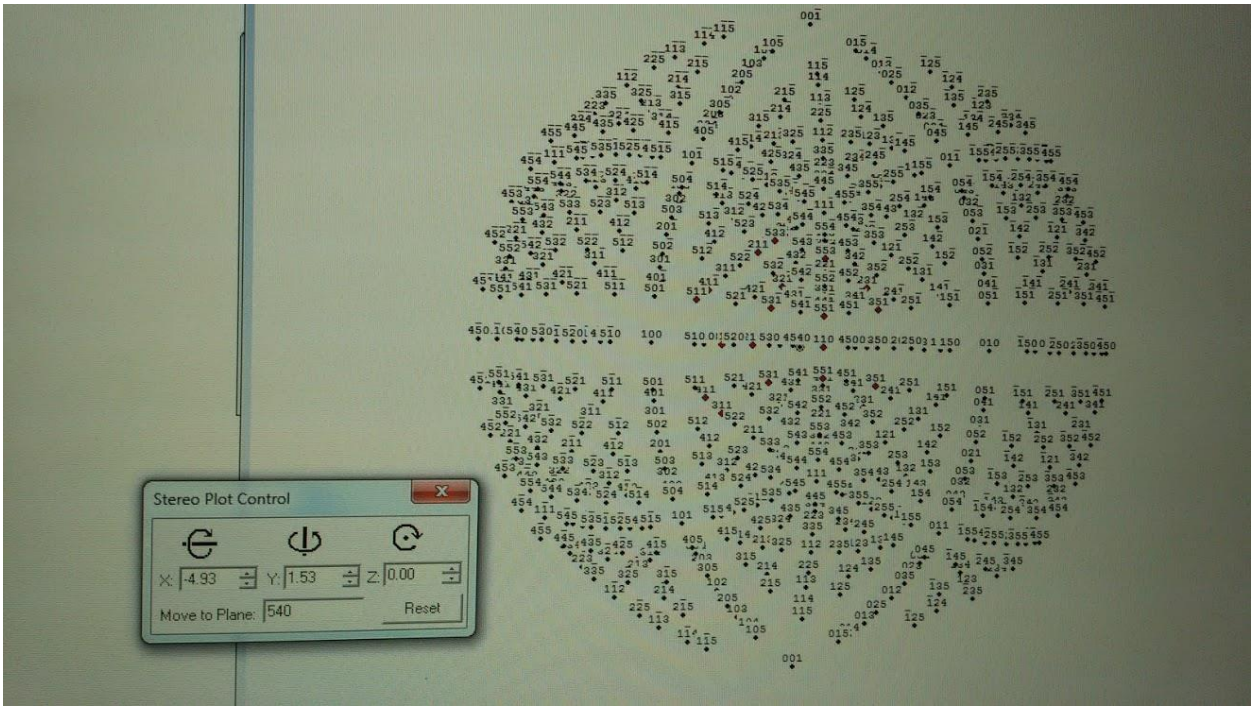

Figure 11(b): Laue diffractometry of Asymmetric Si 7 5 3 Crystal 1 with respect to 5 4 0 plane

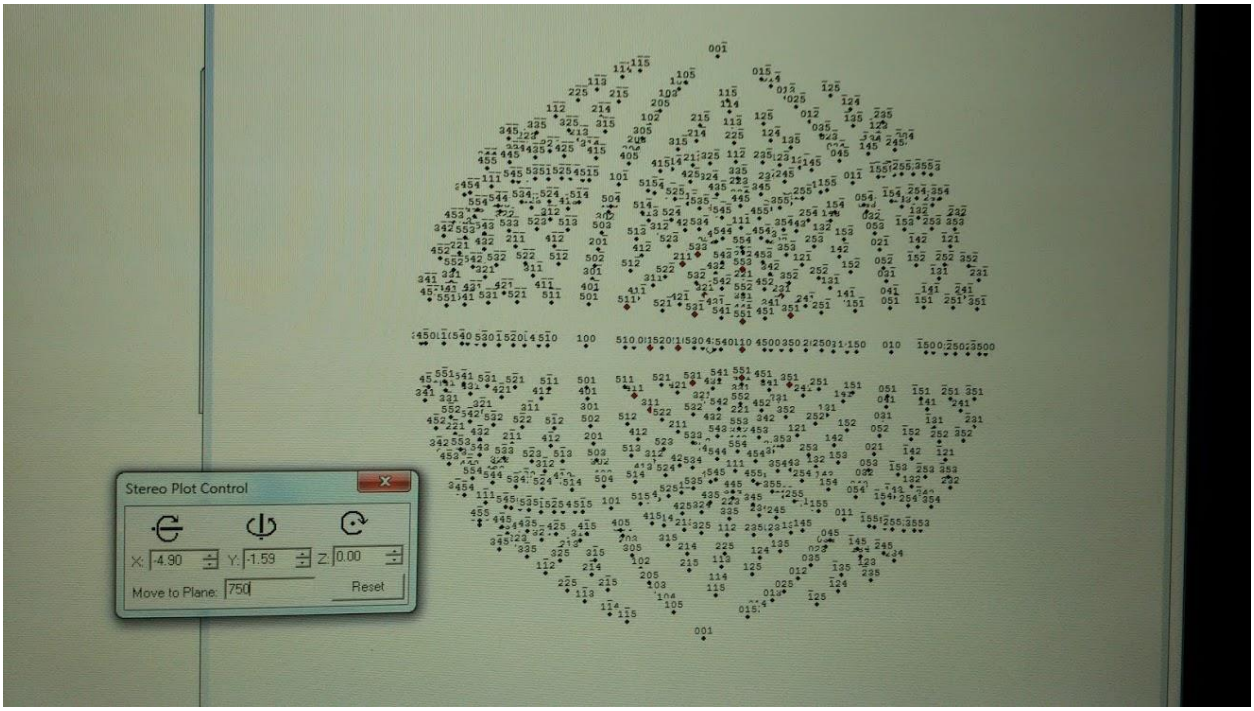

Figure 11(c): Laue diffractometry of Asymmetric Si 7 5 3 Crystal 1 with respect to 7 5 0 plane

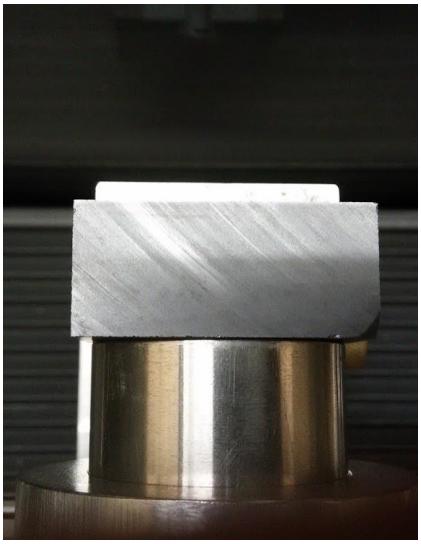

Figure 12(a): Top view of Asymmetric Si 7 5 3 Crystal 1 during Laue diffractometry

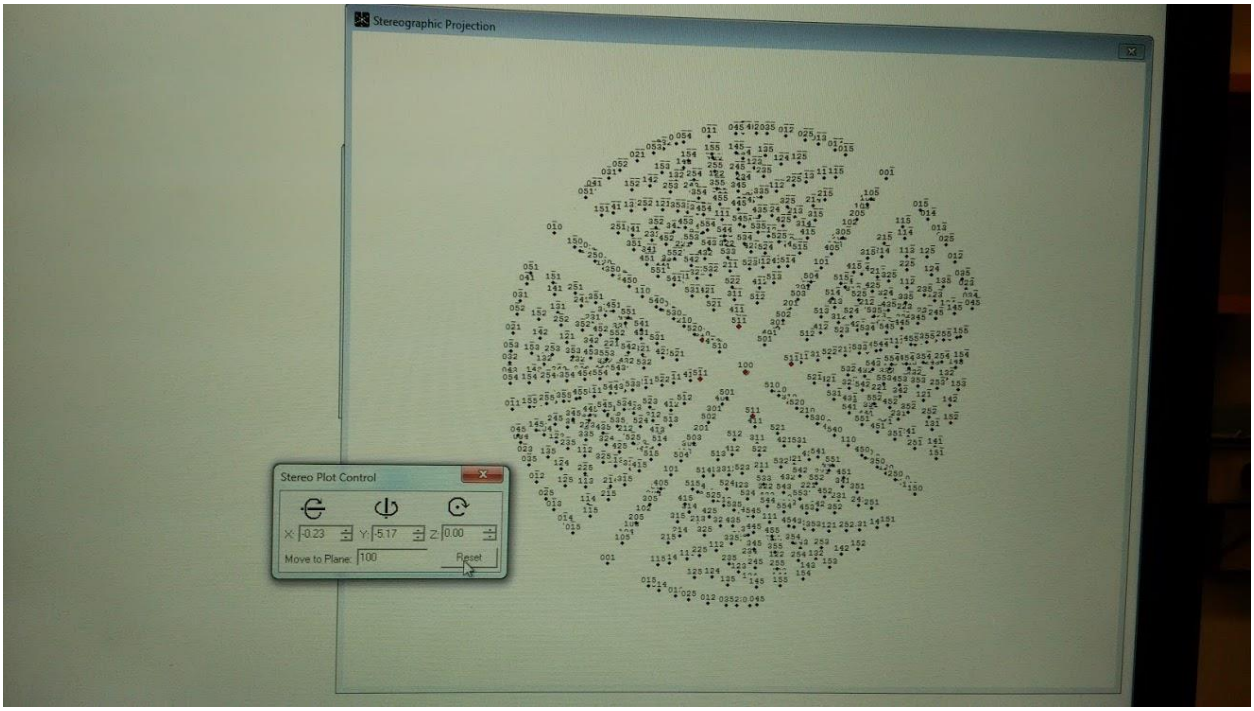

Figure 12(b): Laue diffractometry of Asymmetric Si 7 5 3 Crystal 1 with respect to 1 0 0 plane

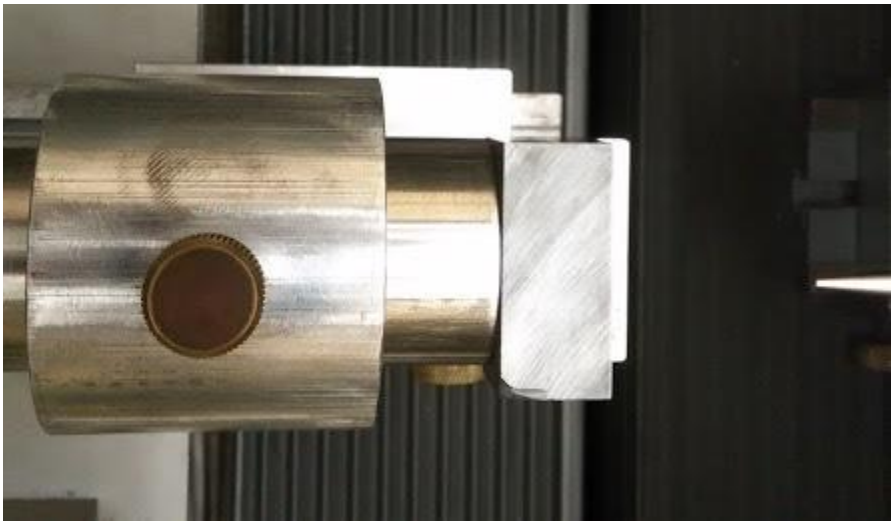

Figure 13(a): Top view of Asymmetric Si 7 5 3 Crystal 2 during Laue diffractometry

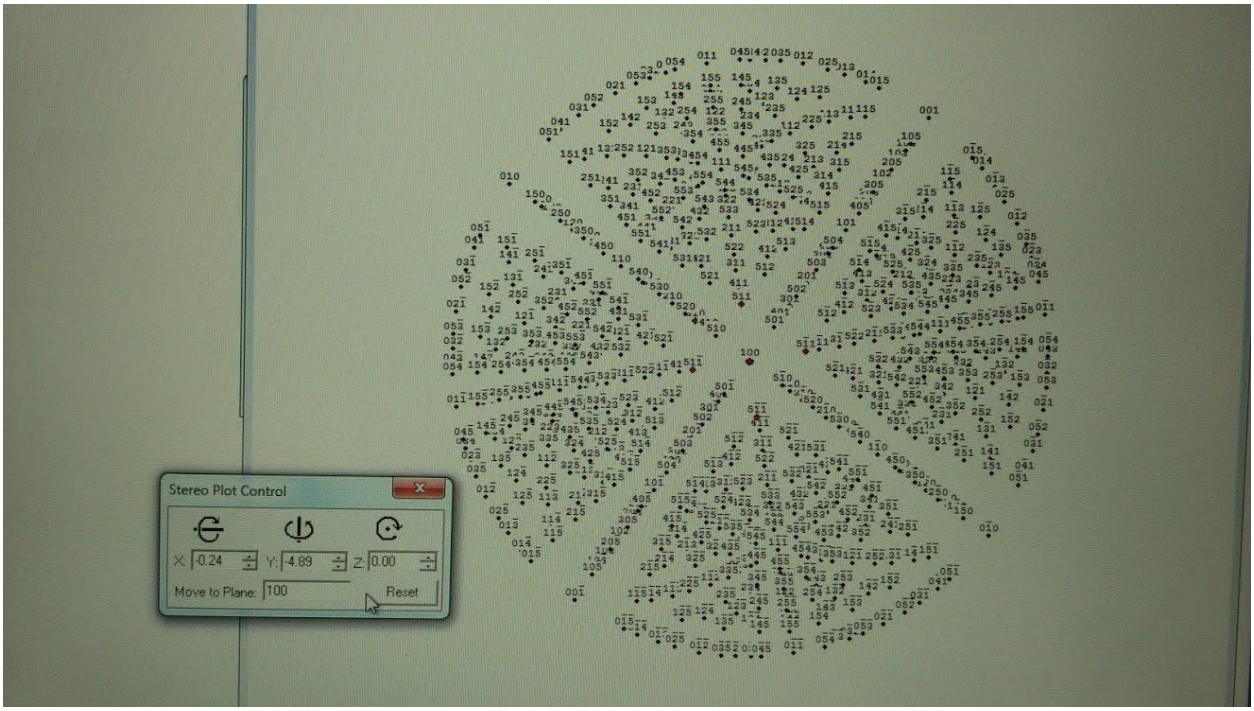

Figure 13(b): Laue diffractometry of Asymmetric Si 7 5 3 Crystal 2 with respect to 1 0 0 plane

7. Preliminary Design of the Monochromator Crystal Holder :

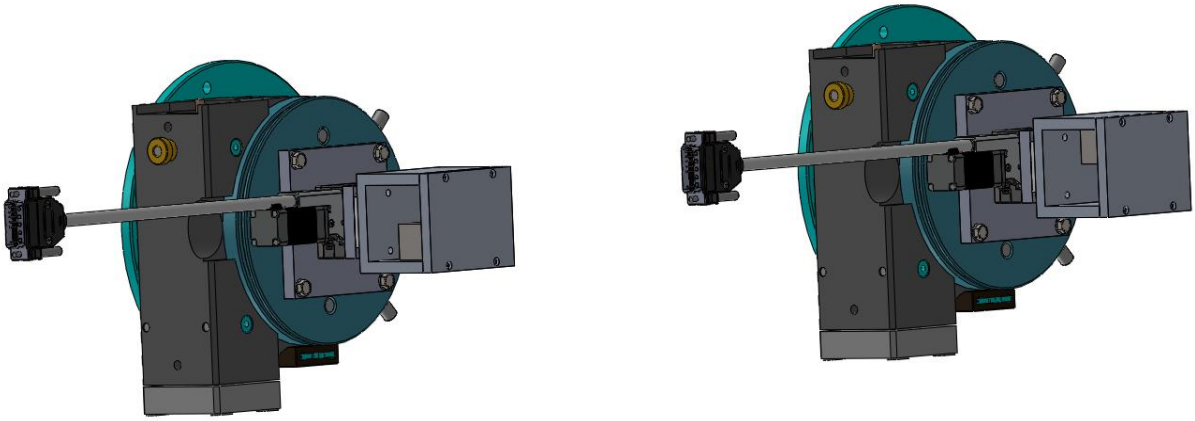

Figure 14: Preliminary Design of Monochromator Crystal Holder

Goniometer adjusts appropriate angle for crystal. Thermal insulating box filled with He gas will allow us to minimize temperature gradients to control Si crystal temperature with high accuracy.

8. Summary

- Monochromator crystals were designed taking into account the crystal structure of Si
- A section of Si boule machined to design specifications to produce two rectangular 20x20x40 mm^3 specimens
- The working surface is polished to mirror-like appearance using a developed multi-step procedure
- Crystal orientation is verified using Laue diffractometry

References

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Acknowledgments

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