Diamond X-ray Rocking Curve and Topograph Measurements at CHESS

G. Yang[1], R.T. Jones[2], F. Klein[3]

[1] Department of Physics and Astronomy, University of Glasgow, Glasgow, UK G12 8QQ.
[2] University of Connecticut unit 3046, 2152 Hillside Rd., Storrs, CT, USA 06269-3046.
[3] Catholic University of America, 620 Michigan Ave., N.E. Washington, DC 20064.

Abstract:

X-ray rocking curve and topograph measurements were carried out at CHESS in Cornell University in November 2006. The purpose of this experiment was to check if the X-ray facility at CHESS is suitable to be used for GlueX in assessing diamond crystal quality and investigating radiation damage and surface deformation induced by milling. After modifying the C1 beam line by using an asymmetry silicon (111) monochromator and a channel cut silicon (220) monochromator, the x-ray beam was well suited to assess diamond for GlueX. The minimum rocking curve full width at half maximum (FWHM) we obtained is around 30μr. A pixel detector was used to record the topograph image. 2 D maps of diamond rocking curve width were generated, which tell precisely how the diamond quality is at each point across the crystal. The rocking curve peak positions vary dramatically over the measured regions, which gives evidence that the 20 microns thick diamond crystal is badly deformed. This indicates that the crystal thinning and the crystal mounting methods should be improved.

1. Introduction

The GlueX project requires a highly polarized high-energy photon beam, which will be created by the coherent bremsstrahlung process[1] which occurs when a beam of high-energy electrons passes through a carefully oriented single diamond crystal. The photon beam linear polarization should be as high as possible under the GlueX requirement[2]. Diamond is chosen as the radiator material because of its combination of low atomic number, high crystal packing density, and very high Debye temperature, all of which contribute to the efficiency of the coherent bremsstrahlung process[3].

The quality of diamond crystal has a vital effect on the polarization of the photon beam. Because diamond specimens always suffer from imperfections and the lattice regularity is disturbed by these imperfections, diamond crystal quality varies from sample to sample. Only those crystals that are of very high quality are suitable to be used as a photon radiator. These crystals must be of order 30-50 mm^2 in area but relatively thin, roughly 20μm or less under the GlueX requirement, to prevent multiple scattering from destroying the excellent emittance properties of the beam. Single-crystal diamond wafers of these dimensions are available from industry, but when they are first produced they roughly an order of magnitude thicker than this, and must be thinned after initial selection. Surface deformations may be induced during the process of thinning, but very little is known about the exact nature of this deformation. The diamond crystal quality can also be altered by radiation damage during the use of the diamond as a radiator. The nature of the defects generated by radiation damage and the rates at which they appear must also be understood, as it impacts the rate at which they must be replaced during the

running of the GlueX experiment. Therefore, we need a simple and efficient method to select diamond crystals and track their changes throughout their life-cycle. Our previous experiences at the Daresbury SRS proved that rocking curve and topograph measurements are particular useful for selecting diamond radiators. Unfortunately, the X-ray facility at SRS will be shut down in the near future and we need to find an alternative X-ray facility to do the diamond quality assessment. So we went to CHESS to see if their facility is suitable for GlueX to do the rocking curve and topograph measurements.

2. Experimental set up

There is no dedicated experimental set up for X-ray rocking curve and topograph measurements at CHESS. In order to let the current beam line to be suitable for diamond assessment, the following modifications were made to the C1 beam line.

First, an asymmetric crystal monochromator was used to reduce the vertical beam divergence. The vertical beam divergence is quite large in the C1 beam line, this is due to the short distance (14.5m) from the bending magnet to the Goniometer. The vertical beam size (HWFM) at the tangent point is ~1.39 mm; this gives an intrinsic beam divergence of ~96µr. Since the measured diamond rocking curve is the convolution of the crystal mosaic spread with the beam divergence, a very small crystal mosaic spread is difficult to resolve with the current beam divergence. On the other hand, a good diamond has a mosaic spread of the order of 10µr or less, which requires the beam divergence should be at least of the same level. Hence, we were forced to reduce the beam divergence. Obviously, it is not possible to improve the vertical beam divergence by increasing the distance. Fortunately, there is an alternative method to do that -- that is to use asymmetric crystal monochromators. The diffraction planes of an asymmetric crystal monochromator are not parallel to the crystal surface, while a symmetric crystal monochrometer has its diffraction planes parallel to the crystal surface. By using an asymmetric crystal monochromator, the X-ray beam divergence can be easily improved, and at the same time, the beam spot size also increased [4]. This is equivalent to moving the sample further downstream from the X-ray source. The available monochromator at CHESS C1 beamline is a double bounce silicon (111) monochromator. The angle between the crystal surface and the (111) bragg plane is 6 degrees for the first silicon crystal. When this monochromator was used, the vertical beam divergence is reduced by a factor of ~8.5.

By using this technique, we expected the X-ray source at CHESS should be well suited to assess the diamond crystal quality for GlueX. But during the experiment, we found the crystal rocking curve width was still too large. We realized that the silicon (111) crystal has very large band-width (10^{-4}), and the difference between the reflection angle of the monochromator 111 plane and the reflection angle of the diamond 220 reflection plane is too large; all of these will broaden the final rocking curve width. To further reduce the instrumental broadening, we inserted a channel cut silicon (220) monochromator in the x-ray beam and it reduces the measured crystal rocking curve width dramatically. In figure 1, rocking curves measured with (top graph) and without (bottom graph) the silicon (220) monochromator are shown. It can be see that the bottom one is much narrower than the top one. One side effect of using the second monochromator is that it reduces the beam vertical size to ~2mm. Because the diamond

samples have a size of around 4 by 4 mm^2, this made it impossible to measure the whole crystal at one time.
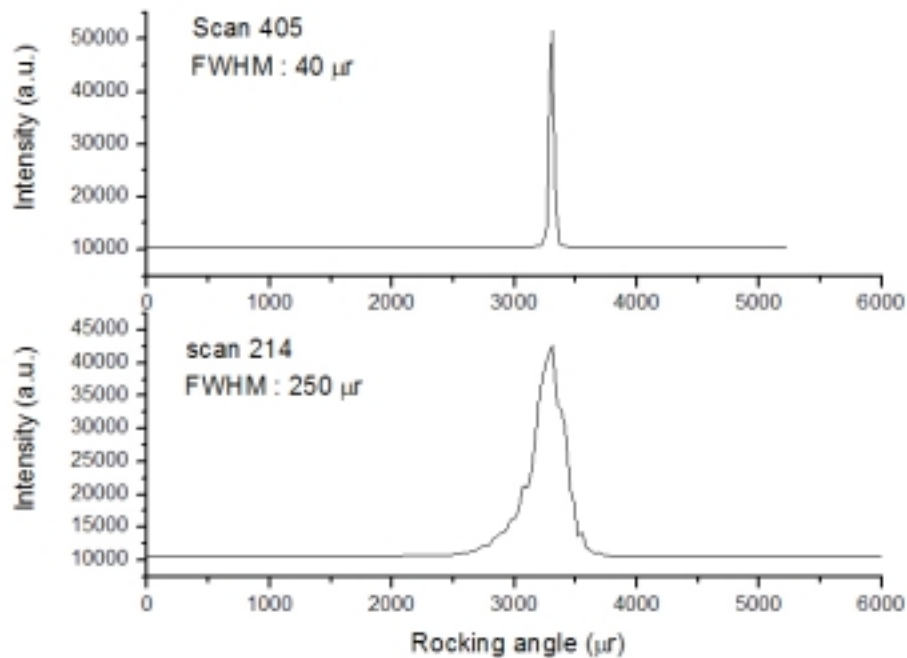

Figure 1, Rocking curves measured with (top) and without (bottom) the second monochromator.

Second, a 4-circle goniometer was used during the experiment to facilitate the crystal alignment.

In order to probe the entire thickness of diamond samples, transmission geometry was used. When using transmission geometry, it is difficult to align the crystal. This is because the diffraction plane for symmetric transmission geometry is perpendicular to the crystal surface, and there is no suitable reference surface to guide the alignment. Furthermore, the diffraction plane may not be strictly perpendicular to the crystal surface depending on how the crystal was polished. Hence, a 4-circle goniometer is needed to facilitate the crystal alignment.

The last one, a pixel detector was used to improve the performance.

The detectors we used in the measurements were a high-resolution homemade pixel detector provided by CHESS staff and ion chambers. We used ion chambers with fast readout to align the crystal for rocking curve measurements, and then use a pixel detector to scan through the rocking curve peak and image the crystal with around 20 μm spatial resolution. The benefit of using a pixel detector is that it produces a two-dimensional rocking map in a short time period, compared with using a single detector and scanning the whole crystal using a pin-hole beam [5]. This two-dimensional map measures precisely the diamond quality at each point across the crystal.

It was also found during the experiment that the crystal vibration could dramatically broaden the diamond rocking curve. Possibly, this vibration is caused by the air movement. Effort was made to eliminate this vibration by isolating the crystal from the environment by using mylar films, and it turns out to be useful.
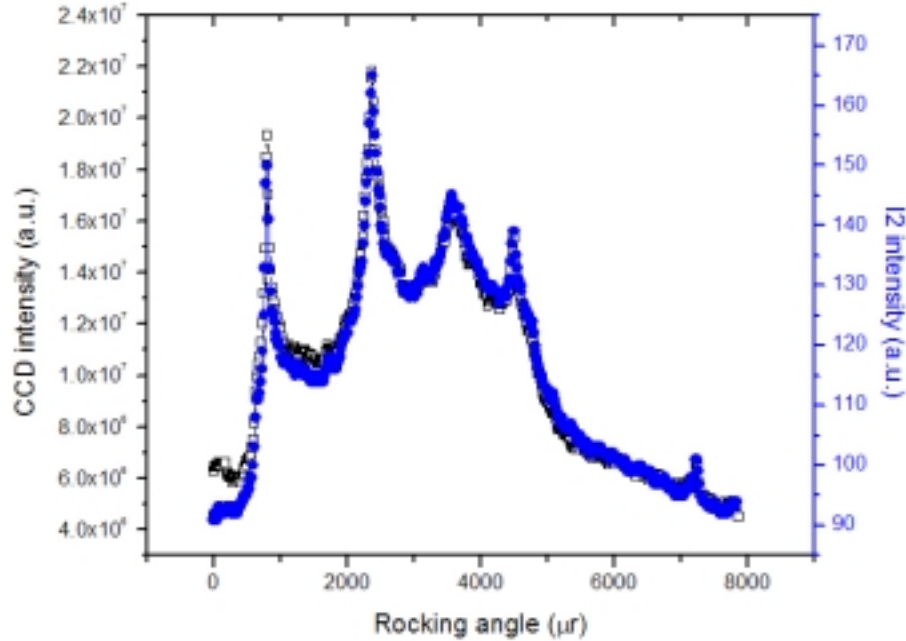
Figure 2. Rocking curves measured by ion chamber (blue) and CCD camera (black).

3. Results and discussions

Five diamond crystals were measured during this experiment. Most of them show very broad rocking curves, which indicates that the crystal quality is poor; only the 20 microns thick diamond radiator and one of the 50 microns thick diamond radiators show narrow rocking curves. The following are some interesting results.

In figure 2, rocking curves measured by using ion chamber and by using the CCD camera are shown. The diamond crystal is the 20 microns thick one. When using the CCD camera, although each pixel of the CCD camera probes only a small region of the diamond crystal. The sum of all the pixels of the CCD camera gives the same information as that obtained from the ion chamber. The almost identical curves from the ion chamber and the CCD camera in figure 2 confirm that the data obtained from the CCD camera is as reliable as the data obtained from the ion chamber.

In figure 3, typical rocking curves measured from isolated pixels of the CCD camera are shown. The pixel size of the CCD camera is roughly 20 microns by 20 microns. It was found that most of the rocking curves show a single peak structure and a Gaussian type peak shape. Only at some special regions, such as the radiation-damaged region, rocking curves with multiple peaks were observed. The minimum rocking curve width we obtained is around 30µr. It should be point out that the measured rocking curve width is larger than the true rocking curve width of the diamond crystal due to instrumental broadening. The X-ray beam divergence and the additional divergence deriving from geometry setting of the monochromator crystal and the diamond crystal are two main

sources of the instrumental broadening. For an isolated pixel, the beam divergence is
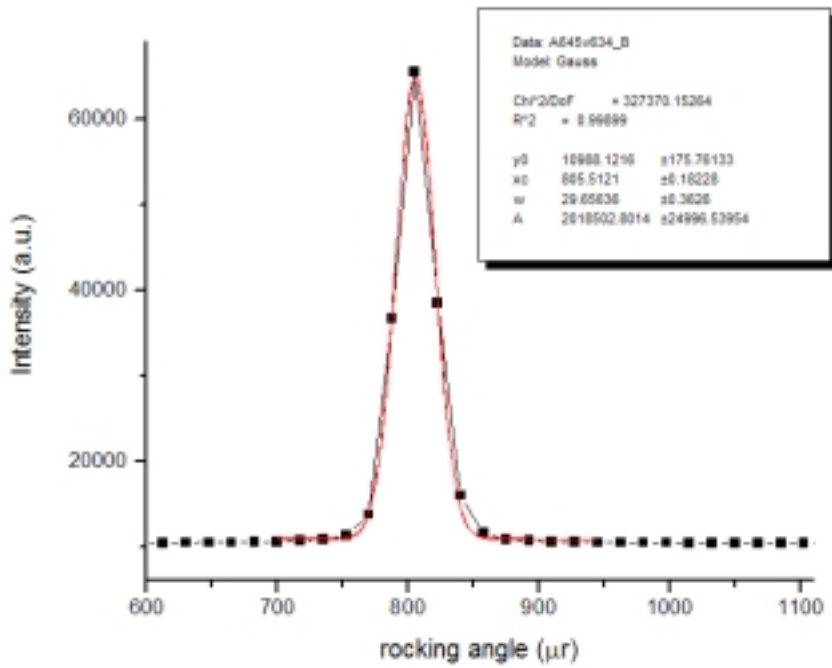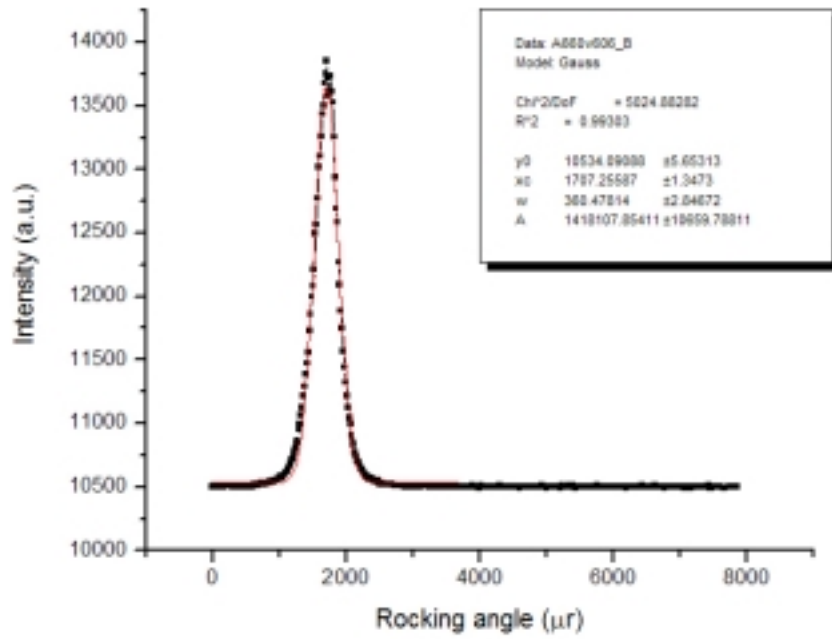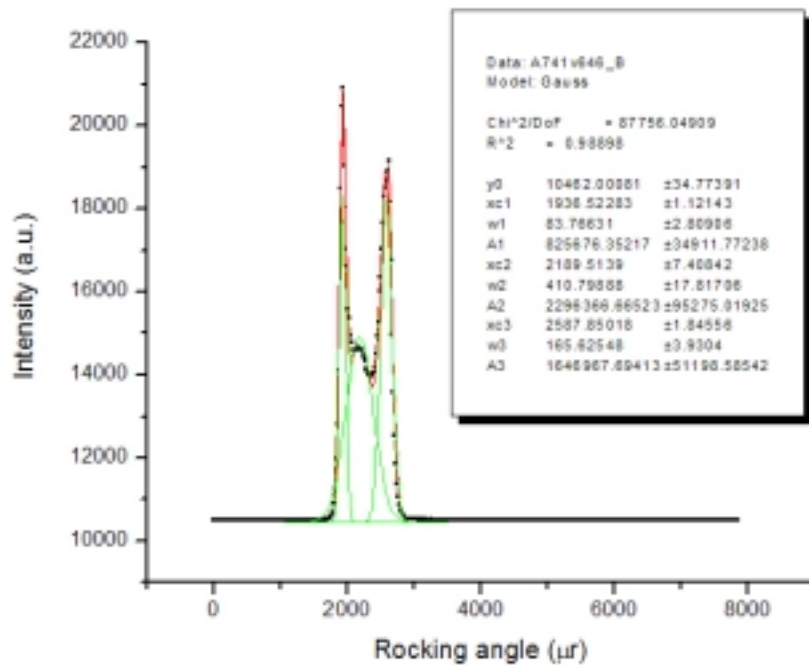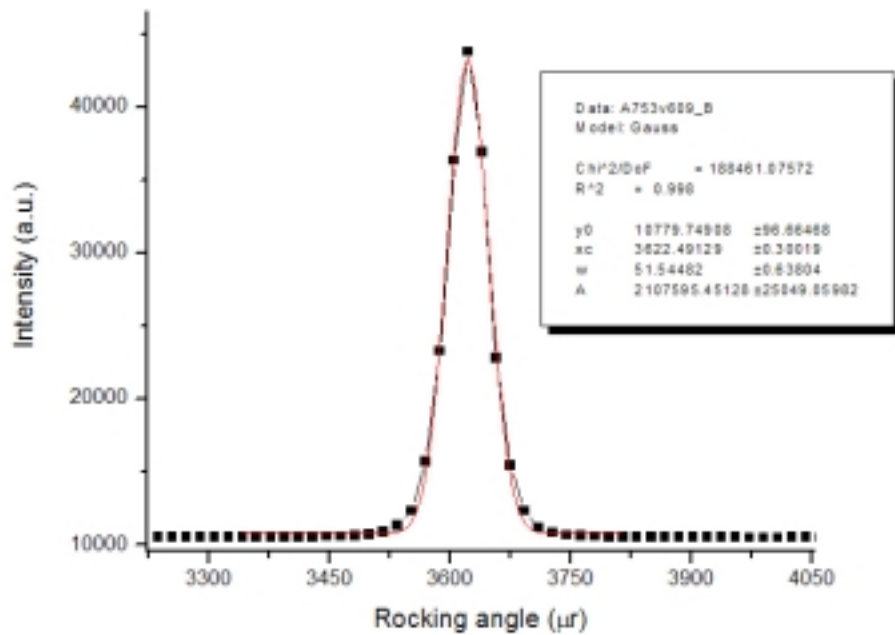

Figure 2 (to be continued)

Figure 3, Rocking curves taken by using isolated pixel of the CCD camera. (continued)

Estimated to be ~15 µr. A (+,-) crystal set-up for the second monochromator crystal and the diamond crystal was used. The diffraction angles for the monochromator crystal and the diamond crystal are different, so this setting is not truly non dispersive. The additional

rocking curve width from dispersion is estimated to be ~ 9 μr. Taking the above two contributions into account, the diamond rocking curve width should be less than ~24 μr.
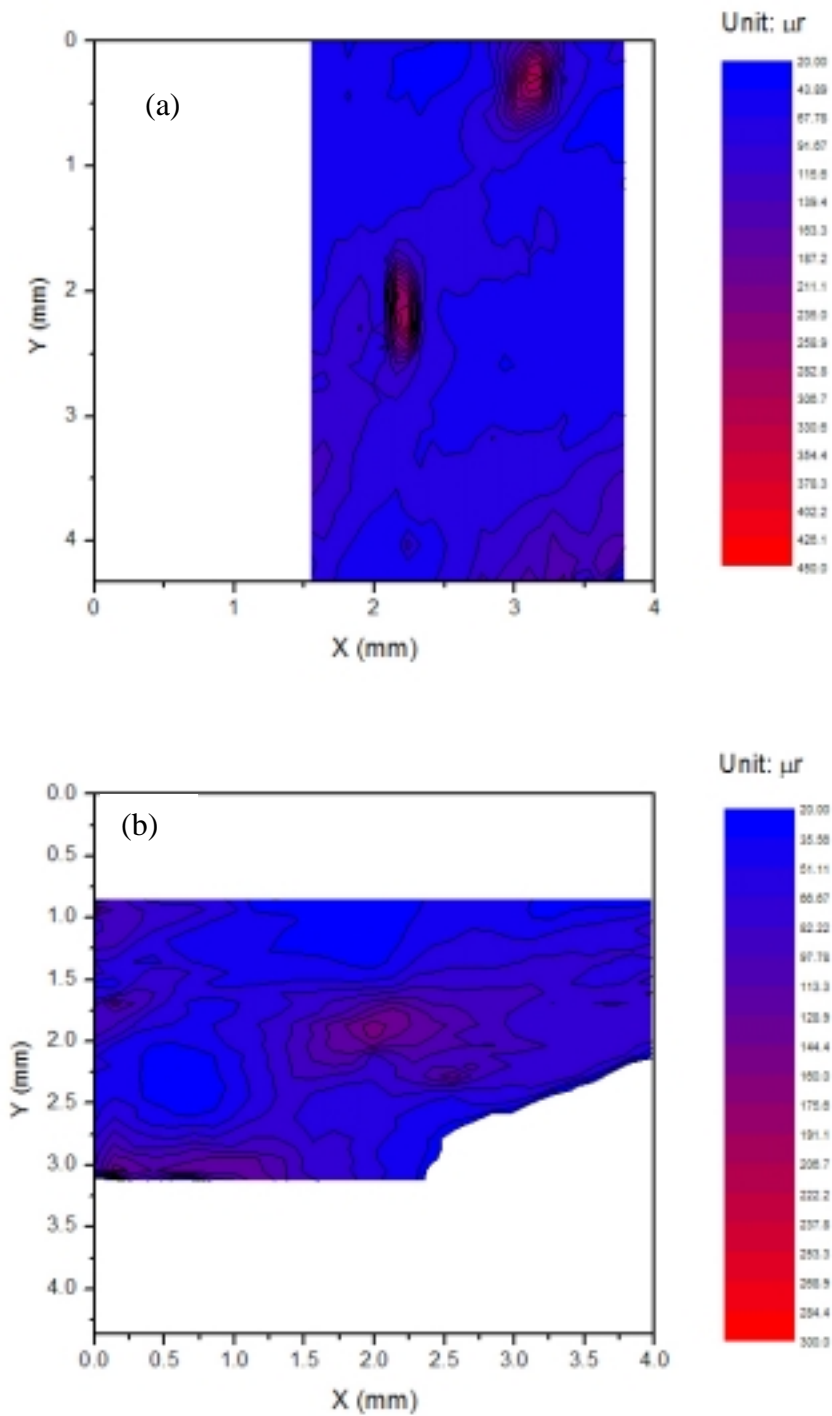

Figure 4. Contour map of rocking curve width over the measured regions for the 20 micron thick diamond crystal (Top: diffraction plane: (-2 2 0), bottom, diffraction plane: (2 2 0)).

Rocking curves were measured for the 20 microns thick diamond for both (220) and (-220) bragg plane. Because the vertical size of the X-ray beam is smaller than the crystal size, only part of the crystal were measured. Contour maps of rocking curve width over the measured regions are shown in figure 4. The picture frames roughly show the size of the diamond crystal, while the colour contour map is corresponding to the measured regions. It can be seen that, the rocking curve widths are quite small and uniform across the measured part, except at some small regions where the rocking curve widths increase up to 450µr. Comparing the features on the rocking curve contour map with that on the crystal, we found that the region showing large rocking curve width on the top right corner of the rocking curve map figure 4 (a) corresponds to an ink mark position on the crystal, while the futures on the centre of figure 4(a) correspond to a radiation damaged region.

In figure 5, two 3 D graphs show how the rocking curve peak positions vary over the measured regions. It can be seen that the rocking curve peak position varies dramatically from one position to another position. From one side of the crystal to anther side, the total variation rocking peak position is as high as around 8000µr. Since for a perfect stress free crystal, the rocking curve peak position does not change with changing positions, the big difference in the rocking peak position means that the diamond crystal is severely deformed by stress.   The possible stress sources are the surface deformation induced by milling and the adhesive used to glue the metal wire to the crystal. Because the diamond crystal thickness is only 20 microns, these stresses can easily make the crystal curved. So the normal direction of the diffraction plane is different at the different part of the crystal and the measured rocking curve peak position is different. Large stress can also change d spacing of the crystal, which also affects the rocking curve peak position, but its effect is small in most of the places.

If we assume the curvature of the crystal is the only reason that is responsible to the variation rocking curve peak position. We can use the information of peak position to reconstruct the shape of diamond crystal. The following graph in figure 6 shows a reconstructed diamond crystal shape. It can be seen that the crystal is heavily deformed. A small concave structure can be seen at the left part of the crystal, which corresponds to the ink mark position on the diamond crystal.

The proposed electron beam spot size for the GlueX project is 1.5 mm by 0.5mm. For such large a beam spot size, the additional rocking curve resulting from the crystal curvature is on the order of 1000µr. Such large rocking curve widths could spoil the linear polarization the generated photon beam. Therefore this crystal is not suitable for using in GlueX project. It should be point out that before this crystal was thinned to 20 micron thick, the original crystal shows a very narrow rocking curve width of ~30µr even when the whole crystal was probed. Possibly, we can used chemical etch method to reduce the stress induced by milling, and by using non stress glue to eliminate the stress generated by crystal mounting. At this moment, we still do not know which one dominates the crystal deformation. Hence, further study is required on both the milling and mounting method.
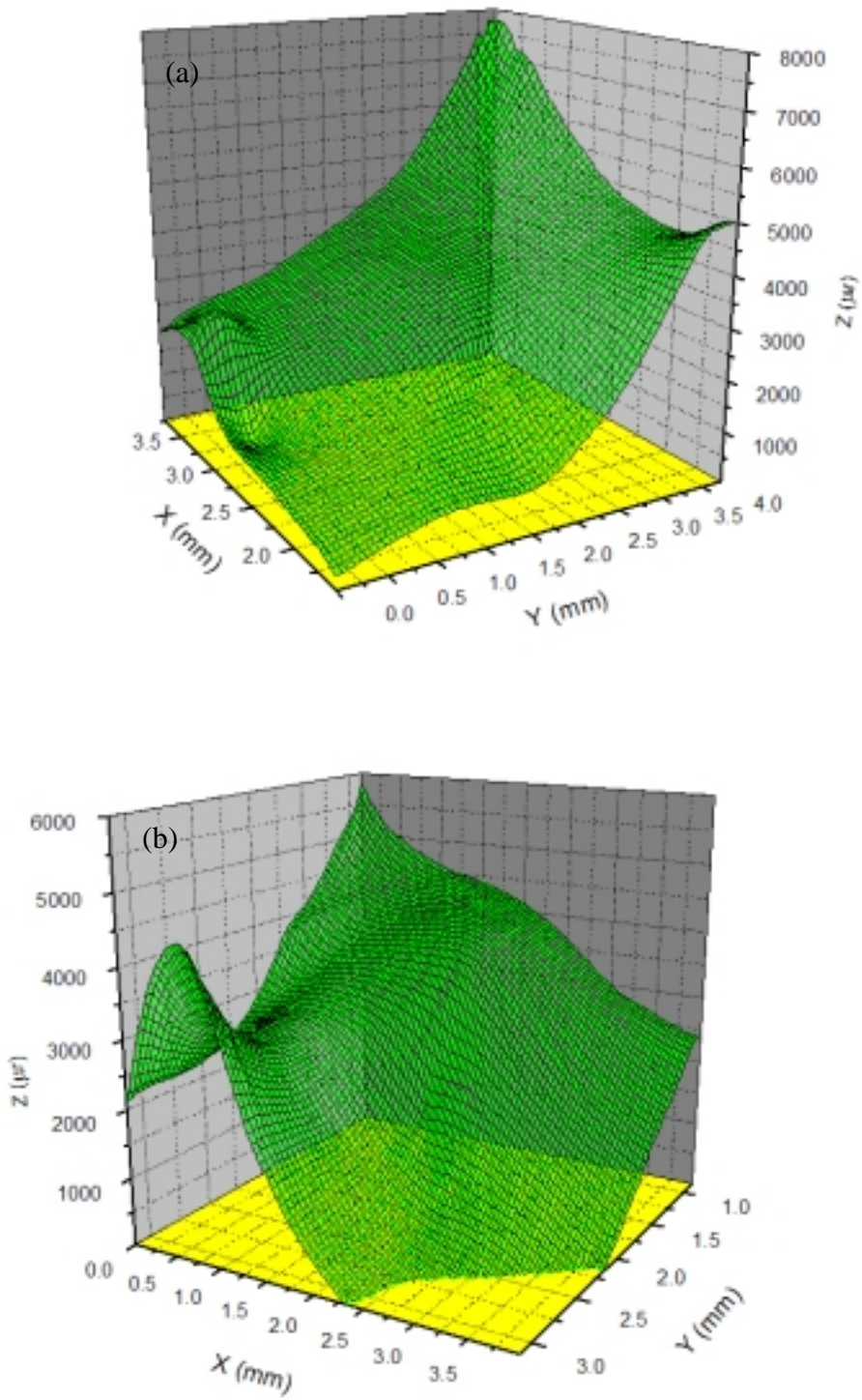
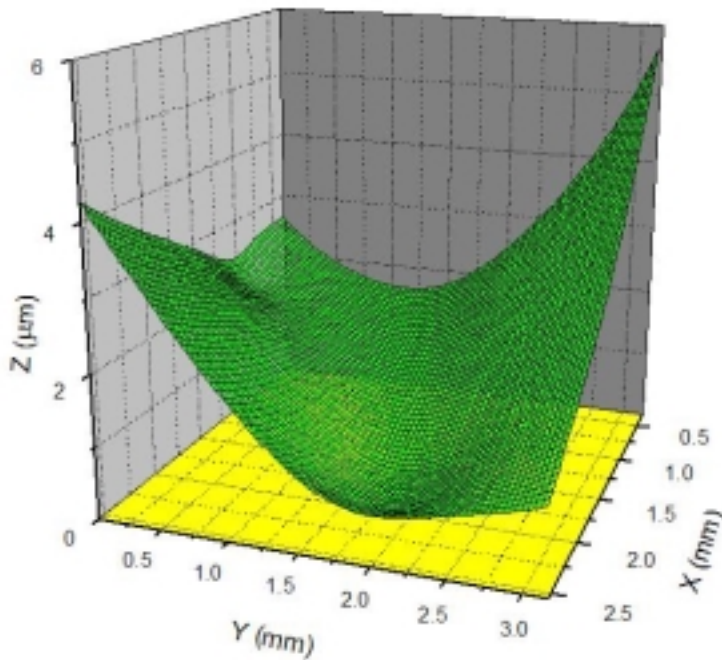Figure 5, rocking curve peak position over the measured regions.

Figure 6, crystal shape calculated by using the rocking curve peak position of each pixels.

4. Conclusion

Rocking curve and topograph measurements were carried out at CHESS in Cornell University. The results show that the X-ray facility at CHESS C1 beam line is well suited to carry out diamond crystal quality assessment for GlueX. Pixel detector has been used for the first time for GlueX diamond assessment. From the measured rocking curve width and peak position map, we found the 20 microns thick diamond crystal is severely deformed by stress. Further study on crystal thinning and mounting method is required.

We want to thank Ken and other members of the CHESS staff for the helpful discussions that we had during our visit to CHESS.

References
[1] RT Jones, Jefferson Lab Hall D Conceptual Design Report, v4.0 September 25 2002.
[2] RT Jones, JLab Hall D note, GlueX-doc-646-v5.
[3] U. Timm, Fortschr. Phys. 17 (1969) 765.
[4] Moreton Moore, Synchrotron x-ray topography, Radiat. Phys. Chem. Vol 45, No. 3 (1995) 427-444.
[5] T. Albert Macrander, Szczesny Krasnicki, Yucheng Zhong, et al. Appl. Phys. Lett. 87(2005) 194113.