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Investigation of Glitches Induced by Single-crystal Diamond Compound Refractive Lenses Based on Crystal Orientation

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Abstract. Single-crystal diamond is considered as an ideal material for compound refractive lenses (usually abbreviated to "CRLs") mainly due to its stability when bathed in strong X-ray radiation. Since CRLs come into operation by a refractive mechanism, it is imperative to study the transmission spectrum carefully. Many factors may influence the spectrum, including experimental features (such as beam divergence) and crystal orientation in this case. To verify initial assumptions, two experimental setups were realized - the energy scan and ω-scan. Both at BM31 station, ESRF. The results show that a number of glitches appear between 16 keV and 18 keV and the largest drop of intensity can reach up to approximately 40%. In this paper, we find that both positions and strengths of the predicted glitches are very sensitive to orientational correction, but that we in principle are able to predict them accurately when the real orientation is obtained.

INTRODUCTION

Nowadays, the scientists are in pursue of even brighter X-ray radiation by building up the next generation source – X-ray free-electron laser (XFELs), which can produce coherent X-rays with brilliance 8~10 orders higher than the current 3rd synchrotron sources (SR). At the same time, X-ray instrumentation is also advancing rapidly to be compatible with the more brilliant source. As most X-ray relevant techniques, for instance, microimaging and microdiffraction techniques, require a demagnified focus with a gain in intensity, focusing optics is an important branch of X-ray optics. By now, several different kinds of X-ray focusing devices have been invented, such as K-B mirrors [1], compound refractive lenses [2], *etc*. This paper deals with compound refractive lenses experimentally and theoretically.

Since compound refractive lenses are operated by a refractive mechanism, it is thus necessary to investigate the transmission spectrum. It should be noted that the refractive phenomenon within X-ray range is different from visible light due to X-ray's high frequency. For X-rays, anomalous dispersion [3] takes place and becomes dominant afterwards, making the refractive index slightly smaller than unity. The refractive increment of most potential materials is so small (in the order of 10^{-9} when far from any absorption edge) that several lenses should be stacked together to achieve desirable focusing size and reasonable focal length. To achieve flexibility, a new device called "transfocator" [4] based on CRLs has been proposed.

This invention attracted worldwide attention and were used at almost all the synchrotron facilities, it is thus necessary to optimize the performance of CRLs in many aspects. A newly published article [5] reported to observe drops of intensity at some specific energies in the transmission spectrum of single-crystal diamond CRLs and called

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them “glitches” vividly. Essentially, glitches appearing here may be owing to many sources, such as “grooves” existing in the entrance and exit surfaces of the lenses, multiple diffraction, extinction and Bragg diffraction. Among all these possible factors, Bragg diffraction usually plays a dominant role, and is governed by the real crystal orientation. Provided that the accurate crystal orientation can be expressed quantitatively, it’s possible to predict the position of the glitches and estimate their corresponding “strengths”.

EXPERIMENTS

These experiments were set up at one of the two Swiss-Norwegian Beamlines (SNBL) - BM31. This is a bending magnet source at European Synchrotron Radiation Facility (ESRF). The reason why we choose BM31 is the monochromator setup is suitable for conducting continuous energy scans over a very wide range of energies, meanwhile, the orientational measurements can also be done via this setup due to the optimization of this beamline and its supplementary beamline optics.

Sample in the Experiment

The sample used in this experiment is 1D planar CRLs and consists of two sets of compound refractive lenses, denoted as CRL$_5$ (the upper one) and CRL$_2$ (the bottom one). Figure 1 is the photo of the sample and note that the subscript here only indicates the number of biconcave lenses. The experimental results in this paper are obtained by impinging X-ray radiation on one set of the lenses: CRL$_5$.

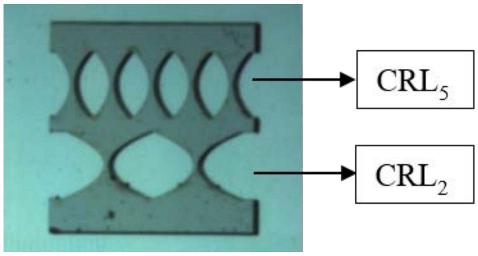

FIGURE 1. This optical photo of the planar lenses.

Experimental Setups

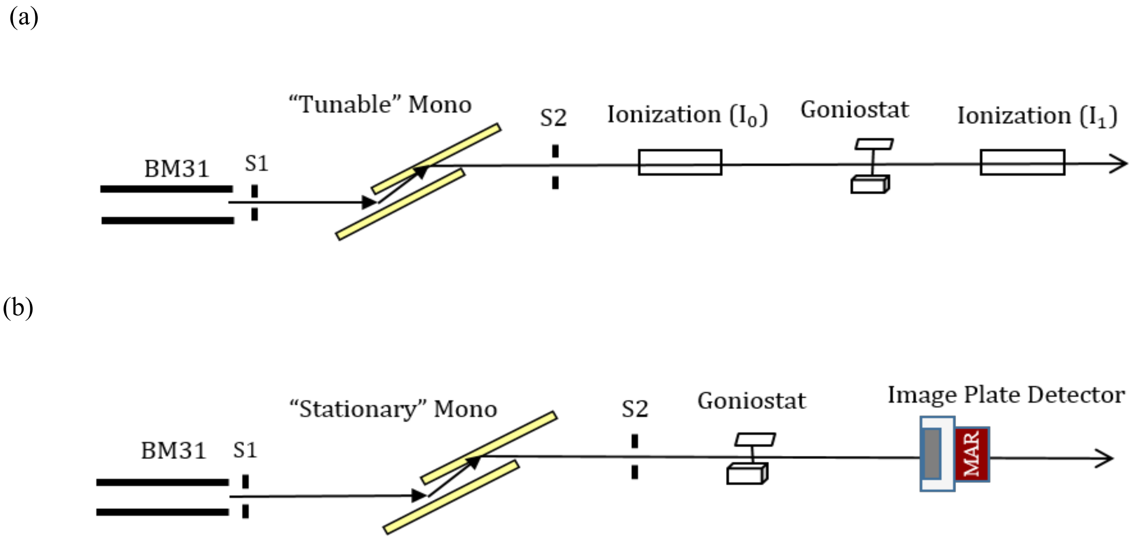

FIGURE 2. These are two experimental setups at BM31, SNBL. (a) depicts schematically the energy scan, (b) is the fixed wavelength ω-scan setup which is used for orientational measurements.

Figure 2(a) and 2(b) schematically show the two experimental setups at BM31, ESRF. The arrows in both figures specify the propagation of X-ray beam. Figure 2(a) shows a sketch of the energy scan experiment with transmission geometry, two gas-filled ionization chambers were thus installed right before and after the sample to measure the intensity, the chamber upstream of the sample is denoted as $\mathrm{I_0}$, while the other one placed downstream of the sample is denoted as $\mathrm{I_1}$. The three-cradle goniostat was mounted so that the beam hit the center of all three circles in Eulerian geometry. From Fig. 2(b), it can be seen that the second setup replaced two ion chambers with an image plate detector to record diffraction spots induced by the lenses. This will provide necessary information required for determining the orientation matrix.

Data Processing of the Energy Scan Setup

It is worth noting that the glitches recorded in the $\mathrm{I_1}$ chamber are caused by both the monochromator (single-crystal silicon) and the sample. Therefore, an appropriate method was chosen so that the transmission spectrum after processing retained the glitches induced only by single-crystal diamond. To start with, we divided $\mathrm{I_1}$ by $\mathrm{I_0}$ ($\mathrm{I_1} / \mathrm{I_0}$) as a function of the incident energy and masked all the glitches in this data column. Then low order polynomial (1 or 2, max 3) fitting through the remaining $\mathrm{I_1} / \mathrm{I_0}$ line was applied. The purpose of this fit was to mark the 100% reference line and the dips of the glitches were then given in percent with respect to the 100% baseline. With this method, the glitches produced by the monochromator could be removed. Finally, we could plot the transmitted intensity with the energy by importing raw dataset into the software – Origin [6].

Predicting the Glitches via ω-scan Setup

The so-called "orientation matrix" can be computed in two different cases [7]: one is we have three accessible independent reflections and the other is we have two independent reflections with known lattice parameters. For this given setup, only two more independent reflections are needed. The procedure is shown in Fig. 3:

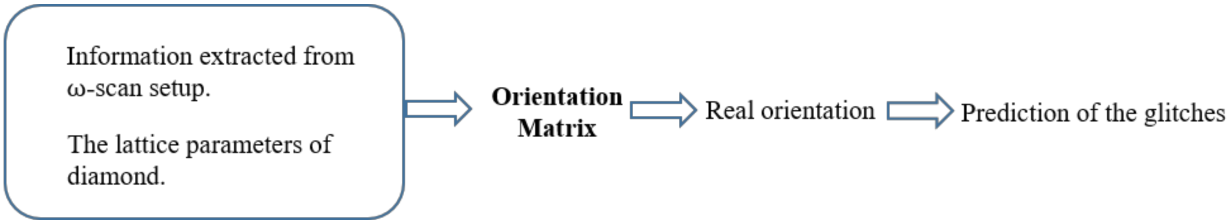

FIGURE 3. The procedure of predicting the glitches.

Some useful parameters, including some calibration data for the specific setup, the pixel positions of the bright diffraction spots and their corresponding ω-angles were extracted from the ω-scan setup. With the availability of these parameters, we are able to calculate the orientation matrix with the following equation:

$$\mathrm{E[keV]} = \frac{12.398}{\lambda[\text{Å}]} = 12.398 \times \left(\frac{-2\mathbf{g}\cdot\mathbf{G}^{-1}\cdot\mathbf{h}}{\sqrt{\mathbf{g}\cdot\mathbf{G}^{-1}\cdot\mathbf{g}\cdot\mathbf{h}\cdot\mathbf{G}^{-1}\cdot\mathbf{h}}} \right)^{-1} \tag{1}$$

Where $\mathbf{G}^{-1}$ is the reciprocal metric tensor, $\mathbf{g}$ is the reciprocal lattice vector (alignment of the incident wavevector along it), $\mathbf{h}$ characterizes the lattice planes with three Miller indices $\{h, k, l\}$.

However, we still need to place some constraints on the final solutions. It's known to all that diamond possesses face-centered cubic structure (FCC) and the intensity of the scattered wave is zero if h, k, l are mixed odd, even; or h, k, l are all even and $h + k + l = 4n$ (n is an integer). They are so-called "forbidden reflections" based on diffraction selection rules and should be excluded from our predictions. Moreover, negative energy solutions ought to be avoided. Followed by that, we are able to visualize the "strengths" of the glitches by squaring the "kinematical" structure factors associated with the actual excited reflections.

RESULTS AND DISCUSSIONS

Through processing the experimental data from the energy scan, we can readily retrieve the transmission spectrum in Fig. 4 and visualize these glitches with different dips.

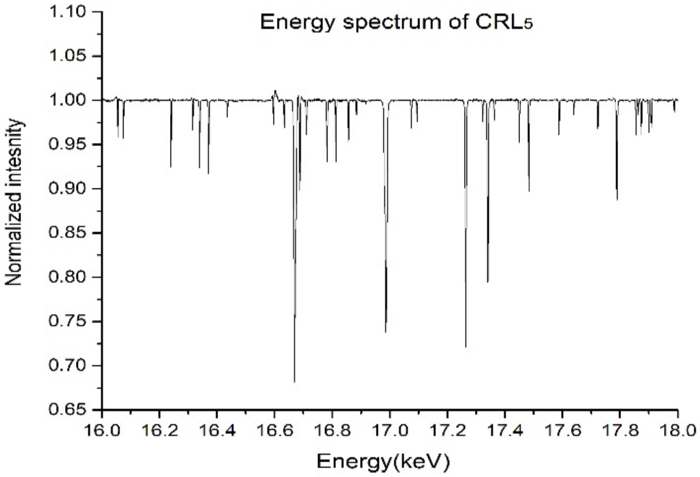

FIGURE 4. The transmission spectrum of CRL5 from the energy scan.

To start with, let us justify the sensitivity of the squared "kinematical" structure factors ${F_{hkl}}^2$ versus small misalignment (denoted as δ) from assumed $\langle 100\rangle$ alignment, shown in Fig. 5. The horizontal axis is user-defined energy interval (10-20 keV) and the vertical axis is the total scattering intensity at specific energy from the point of X-ray's kinematical theory. Since several planes may satisfy Bragg condition simultaneously, their corresponding squared structure factors should be summed up, *n* characterizes the number of planes satisfying Bragg's law. In other words, the "strengths" of the glitches are characterized by their heights.

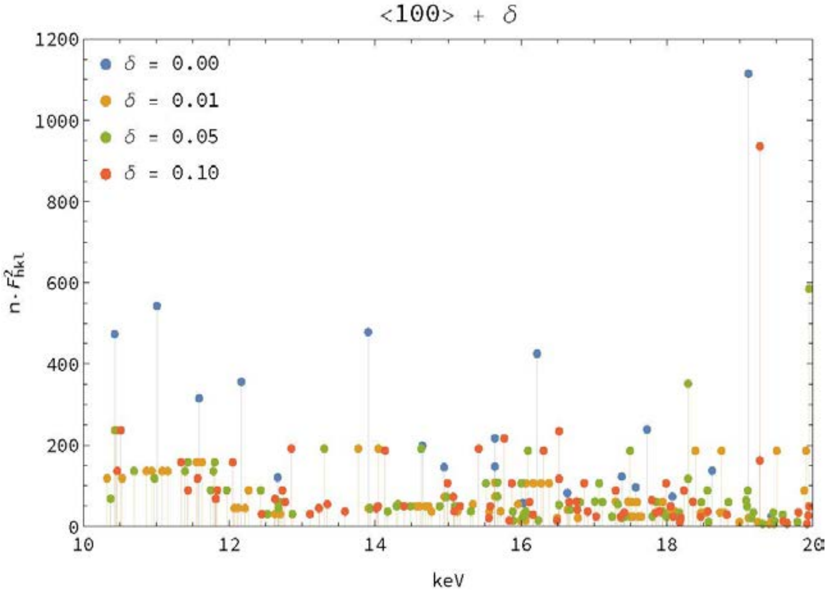

FIGURE 5. Predicted glitch patterns with different deviations from the assumed alignment.

To illustrate this issue, the experimental results, as well as theoretical predictions (with and without correction) were plot in Fig. 6 for comparison. The black curve is obtained from the real experimental setup, while the red and blue vertical lines represent the squared kinematical structure factors without and with orientational correction, respectively.

It can be found that only 6 diffraction events appear within this selected interval (16 keV-18 keV) without correction (red lines). In comparison, by using the corrected alignment, the predictions show better agreement with the experimental results: the predicted spectrum with correction can reproduce most occurrences of the glitches. In spite of this, there are still some discrepancies between the experiment and theoretical predictions. This is mainly attributed to the uncertainty associated with the actual orientation matrix, for instance, the pixel positions read out from the diffraction patterns may introduce uncertainties owing to the blurring and broadening of the diffraction spots; and the ω-angles introduce 1° angular uncertainty because the angular interval between adjacent diffraction images is 1°.

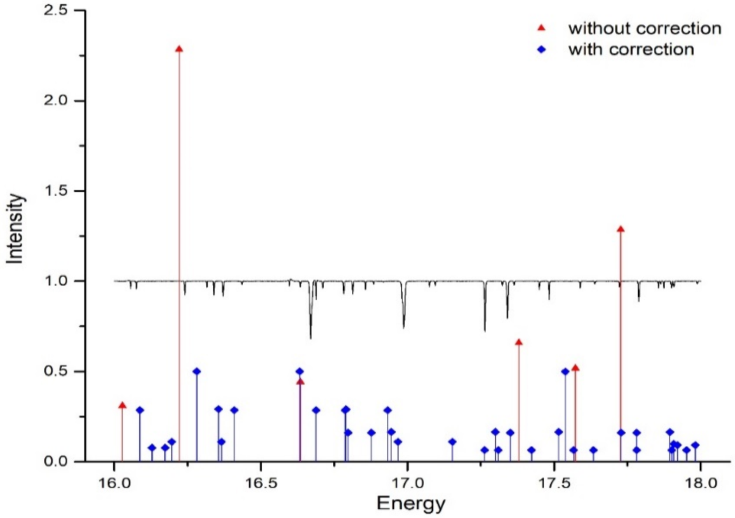

FIGURE 6. Predicted glitch patterns with different deviations from the ideal alignment.

CONCLUSIONS

In summary, glitches appearing in the transmission spectrum mean the loss of intensity and thus should be dealt with seriously. The ultimate purpose of this paper is to show that the glitch positions (and corresponding strengths) may be predicted via the orientation matrix. The simulation results clearly show that the positions and strengths are very sensitive to the orientational correction. It is meaningful to determine the real orientation of the sample.

For this given setup, some mismatches still exist, but it has been shown that introducing the correction provides a better prediction. In order to obtain good positional precision, the accuracy of the orientation matrix is crucial. It's thus absolutely necessary to further improve the precision, *i.e.*, the 1° angular uncertainty can be reduced by replacing the image plate detector with a point detector within the angular interval where sharp Bragg reflections appear.

It is worth mentioning that the method described in this paper is not only restricted to single-crystal diamond, but can also be extended to other single-crystal transmission devices.

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