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Optimal azimuthal orientation for Si(111) double-crystal monochromators to achieve the least amount of glitches in the hard X-ray region

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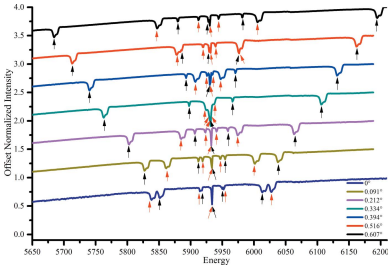
Simulations of the periods, split regularities and mirror symmetries of the glitch pattern of a Si(111) crystal along with the azimuthal angles are presented. The glitch patterns of Si(111) double-crystal monochromators (DCMs) are found to be the superposition of the two sets of glitch patterns from the two crystals. The optimal azimuthal orientation $\varphi_{1,2} = [(2n+1)\pi]/6$ ($n = 0, \pm1, \pm2\ldots$) for Si(111) DCMs to achieve the least amount of glitches in the hard X-ray region has been suggested.

1. Introduction

Double-crystal monochromators (DCMs) based on two-beam Bragg diffraction have been widely used to monochromatize white synchrotron radiation beams. During energy scanning of a DCM, the X-ray multiple-beam diffraction (MBD) effect occurs when a single crystal is so oriented that two or more sets of atomic planes of the crystal simultaneously satisfy Bragg's law in diffracting the incident beam. In reciprocal space, this corresponds to the situation where two or more reciprocal lattice points are located on the Ewald sphere simultaneously (Yang *et al.*, 2000; Chang, 2004). For DCMs, the MBD process can significantly reduce the diffraction efficiency of the primary reflection and induce extra undesirable/harmful features in the diffraction pattern, namely glitches that refer to the output intensity variation at particular energy positions (Konigsberger & Prins, 1987; Bunker, 2010; Huang *et al.*, 2012). Glitches are detrimental to experiments. In X-ray absorption fine structure (XAFS) spectroscopy, for instance, glitches may distort the spectra, especially for non-uniform or dilute samples, thus decreasing the signal-to-noise ratio and making it difficult to resolve the real local structure of the samples.

Generally, varying the glitch pattern by adjusting the crystal orientations of the DCMs to avoid producing glitches is the most practical way to reduce the number of glitches. For the glitch pattern, a general expression for the glitch positions for one silicon crystal has been derived theoretically, and a proper choice for the azimuthal orientation of the crystal to achieve a large glitch-free region in the soft X-ray region had also been suggested (Van der laan & Thole, 1988). In the hard X-ray region, however, the glitch patterns of the DCMs may be more sophisticated because of the higher glitch density and overlapping of two sets of glitch patterns from each of the two crystals (Arthur, 1989), so that it is difficult to achieve a large glitch-free region in the hard X-ray region. In this paper, the



approach stated above will be extended to find the optimal azimuthal orientations for the Si(111) DCMs in order to achieve the least amount of glitches in the hard X-ray region.

2. Simulation and experiment

2.1. Periods, split regularities and mirror symmetries of glitch patterns for one Si(111) crystal

For the periods of the glitch patterns, calculations for three azimuthal angles with $\varphi = 0$, $\varphi = \pi/3$ and $\varphi = -\pi/3$ in the energy range 5–6.3 keV have been made. During the calculations, equations (S1) and (S2) and the coordinate system shown in Fig. S1 have been employed (see supporting information). The results, given in Table 1, show that the glitch positions remain fixed for the three azimuthal angles, *i.e.* the period of the glitch patterns for one Si(111) crystal was $\pi/3$ which is consistent with the symmetry of the rotation axis. Changing the azimuthal angle means rotating around the [111] axis. Because the symmetry of the [111] axis of silicon crystal is sixfold, the glitch patterns repeat every 60° during the rotation.

Table 1
Calculated glitch positions for three azimuthal angles $\varphi = 0$, $\varphi = \pi/3$ and $\varphi = -\pi/3$ in the energy range 5–6.3 keV.

The first row presents the three azimuthal angles. For each angle there are two columns: the first column is the index of operative and cooperative diffraction planes, *i.e.* $g/(h-g)$; the second column is the glitch position. Energy units are eV.

$\varphi = -\pi/3$		$\varphi = 0$		$\varphi = \pi/3$	
Index $g/(h-g)$	Energy	Index $g/(h-g)$	Energy	Index $g/(h-g)$	Energy
$(2\bar{2}\bar{2})/(\bar{1}33)$	5282.7	$(33\bar{1})/(\bar{2}\bar{2}2)$	5282.7	$(\bar{2}\bar{2}2)/(3\bar{1}3)$	5282.7
$(4\bar{2}0)/(\bar{3}31)$ $(40\bar{2})/(\bar{3}13)$	5412.7	$(31\bar{3})/(\bar{2}04)$ $(13\bar{3})/(0\bar{2}4)$	5412.7	$(04\bar{2})/(1\bar{3}3)$ $(\bar{2}40)/(\bar{3}31)$	5412.7
$(202)/(\bar{1}1\bar{1})$ $(3\bar{3}1)/(\bar{2}42)$ $(5\bar{1}1)/(\bar{4}22)$ $(4\bar{2}\bar{2})/(\bar{3}33)$ $(31\bar{3})/(\bar{2}24)$ $(220)/(\bar{1}\bar{1}1)$	5937.3	$(1\bar{1}\bar{1})/(022)$ $(42\bar{2})/(\bar{3}13)$ $(224)/(\bar{1}\bar{1}5)$ $(33\bar{3})/(\bar{2}\bar{2}4)$ $(24\bar{2})/(\bar{1}33)$ $(\bar{1}\bar{1}1)/(202)$	5937.3	$(220)/(\bar{1}\bar{1}1)$ $(\bar{1}33)/(\bar{2}\bar{2}4)$ $(\bar{1}5\bar{1})/(\bar{2}42)$ $(\bar{2}4\bar{2})/(\bar{3}\bar{3}3)$ $(\bar{3}3\bar{1})/(\bar{4}\bar{2}2)$ $(022)/(1\bar{1}\bar{1})$	5937.3

In addition, for the split regularities and mirror symmetries of the glitch patterns, similar calculations for the three azimuthal angles $\varphi = -1°$, $\varphi = 0°$ and $\varphi = 1°$ in the energy range 5–6.3 keV have also been made. The results, given in Table 2, show, first of all, that a small deviation of the azimuthal angle from $\varphi = 0°$ will cause a quick glitch position split, and the split rate of the glitch, $\Delta E/\Delta\varphi$, varies with the index of the corresponding operative diffraction planes. A high split rate means a high sensitivity to the azimuthal angle variation, and *vice versa*. Secondly, it is obvious that the glitch positions for $\varphi = -1°$ and $\varphi = 1°$ remain fixed while the operative diffraction planes show a series of mirror symmetries. The series of mirror symmetries in the glitch patterns depends on the symmetry of the rotation vector. Because the [111] axis of silicon crystal is sixfold, and taking into account the entering and exiting of the Ewald sphere, the mirror symmetries take place around $\varphi = n\pi/6$ ($n = 0, \pm1, \pm2\ldots$) which is consistent with the simulation results.

Table 2
Calculated glitch positions for three azimuthal angles $\varphi = -1°$, $\varphi = 0°$ and $\varphi = 1°$ in the energy range 5–6.3 keV.

The first row presents the three azimuthal angles and the split rate. For each angle there are two columns: the first column is the index of operative and cooperative diffraction planes, *i.e.* $g/(h-g)$; the second column is the glitch position. Energy units are eV.

$\varphi = -1°$		$\varphi = 0°$		$\varphi = 1°$		$\Delta E/\Delta\varphi$ (eV/°)
Index $g/(h-g)$	Energy	Index $g/(h-g)$	Energy	Index $g/(h-g)$	Energy	
$(33\bar{1})/(\bar{2}\bar{2}2)$	5283.4	$(33\bar{1})/(\bar{2}\bar{2}2)$	5282.7	$(33\bar{1})/(\bar{2}\bar{2}2)$	5283.4	0.7
$(31\bar{3})/(\bar{2}04)$ $(13\bar{3})/(0\bar{2}4)$	5385.3 5442.0	$(31\bar{3})/(\bar{2}04)$ $(13\bar{3})/(0\bar{2}4)$	5412.7	$(13\bar{3})/(0\bar{2}4)$ $(31\bar{3})/(\bar{2}04)$	5385.3 5442.0	−27.4 29.3
$(1\bar{1}\bar{1})/(022)$ $(42\bar{2})/(\bar{3}13)$ $(224)/(\bar{1}\bar{1}5)$ $(33\bar{3})/(\bar{2}\bar{2}4)$ $(24\bar{2})/(\bar{1}33)$ $(\bar{1}\bar{1}1)/(202)$	5783.4 5906.4 5938.1 5970.2 6102.9	$(1\bar{1}\bar{1})/(022)$ $(42\bar{2})/(\bar{3}13)$ $(224)/(\bar{1}\bar{1}5)$ $(33\bar{3})/(\bar{2}\bar{2}4)$ $(24\bar{2})/(\bar{1}33)$ $(\bar{1}\bar{1}1)/(202)$	5937.3	$(\bar{1}\bar{1}1)/(202)$ $(24\bar{2})/(\bar{1}33)$ $(33\bar{3})/(\bar{2}\bar{2}4)$ $(224)/(\bar{1}\bar{1}5)$ $(42\bar{2})/(\bar{3}13)$ $(1\bar{1}\bar{1})/(022)$	5783.4 5906.4 5938.1 5970.2 6102.9	−153.9 −30.9 0.8 32.9 165.6

2.2. Glitch number distribution of one period for one Si(111) crystal

In order to obtain the proper azimuthal orientation for achieving the least amount of glitches in the hard X-ray region, a simulation of the glitch number distribution of one period for one Si(111) crystal has been made. For the simulation the energy range was chosen as 4–27 keV, which is the conventional energy range for X-ray absorption spectroscopy experiments, and the azimuthal angle step was 0.1°. The result of the simulation is given in Fig. 1. It can be seen from Fig. 1 that an obvious decrease in the glitch number takes place at mirror symmetry positions, *i.e.* $\varphi = n\pi/6$ ($n = 0, \pm1, \pm2\ldots$), which confirms the split regularities and mirror symmetries discussed in §2.1. Furthermore, it is clear to see that $\varphi = (2n+1)\pi/6$ ($n = 0, \pm1, \pm2\ldots$) are the best azimuthal angles for one Si(111) crystal to achieve the least amount of glitches in the hard X-ray region.

2.3. Azimuthal angle adjustment experiment

The discussion above only focuses on one Si(111) crystal; however, Si(111) DCMs for X-ray absorption spectroscopy beams usually consist of double flat perfect silicon crystals. In order to reveal the glitch patterns of the Si(111) DCMs, an azimuthal angle adjustment experiment was carried out on the

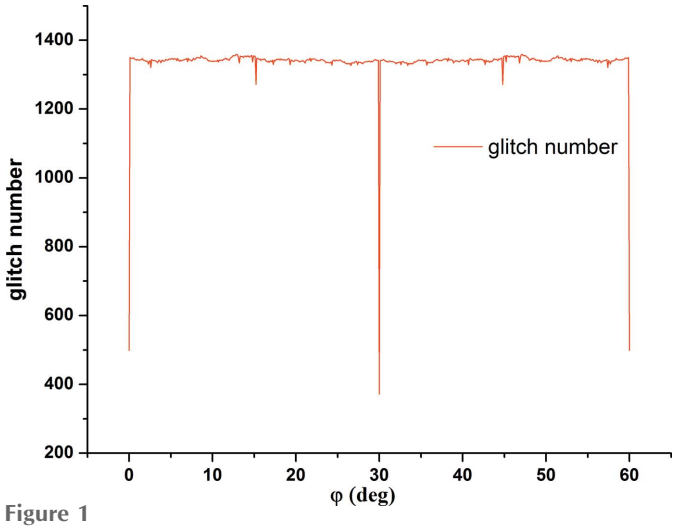


Figure 1
Simulated glitch number distribution of one period for one Si(111) crystal. The abscissa is the azimuthal angle and the ordinate is the glitch number. During the simulation the energy range was chosen as 4–27 keV, which is the conventional energy range for X-ray absorption spectroscopy experiments.

1W1B station of Beijing Synchrotron Radiation Facility (BSRF). During the experiment, the storage ring was working at 2.5 GeV with a maximum electron current of about 250 mA. An ion chamber detector was used to detect the output beam intensity of the Si(111) DCM and the experiment energy range was chosen as 5.5–6.3 keV. A stepper motor was used to adjust the azimuthal angle. The two crystals were adjusted as a unit because their relative azimuthal angle was fixed.

The results of the experiment, given in Fig. 2, show, firstly, that one set of glitches, indicated by the red arrows, merge from five glitches to one and then split into five, while another set, indicated by the black arrows, keep splitting far away from the centre during the azimuthal angle adjustment. Secondly, a mirror symmetry can also be observed during the merging and splitting process. According to the the simulation in §2.1, the azimuthal angle of one crystal varying from a non-zero position to the zero position and then back to a non-zero position is responsible for the merging/splitting process and mirror symmetries of that set of glitches. On the contrary, due to a smaller deviation of the azimuthal angle of the other crystal from the zero position to a larger one, another set of glitches keeps splitting far away the centre. All the features discussed above are consistent with the theoretical simulation. Therefore, the conclusion that the glitch patterns of the Si(111) DCMs are the superposition of two sets of glitch patterns from the first and second crystals can be drawn, which is identical to Arthur's report (Arthur, 1989). In addition, due to the mirror symmetries and high angular sensitivity of the split rates of the glitches, different combinations of the relative azimuthal angles of the two crystals will lead to complicated glitch patterns. As stated above, the optimal azimuthal orientation for one Si(111) crystal is $\varphi = (2n + 1)\pi/6$ ($n = 0, \pm1, \pm2\ldots$). Therefore, the conclusion that the optimal azimuthal orientations for Si(111) DCMs for achieving the least amount of glitches in the hard X-ray region are $\varphi_{1,2} = (2n + 1)\pi/6$ ($n = 0, \pm1, \pm2\ldots$) can be drawn. Here, φ_1 and φ_2 represent the azimuthal angles of the two crystals.

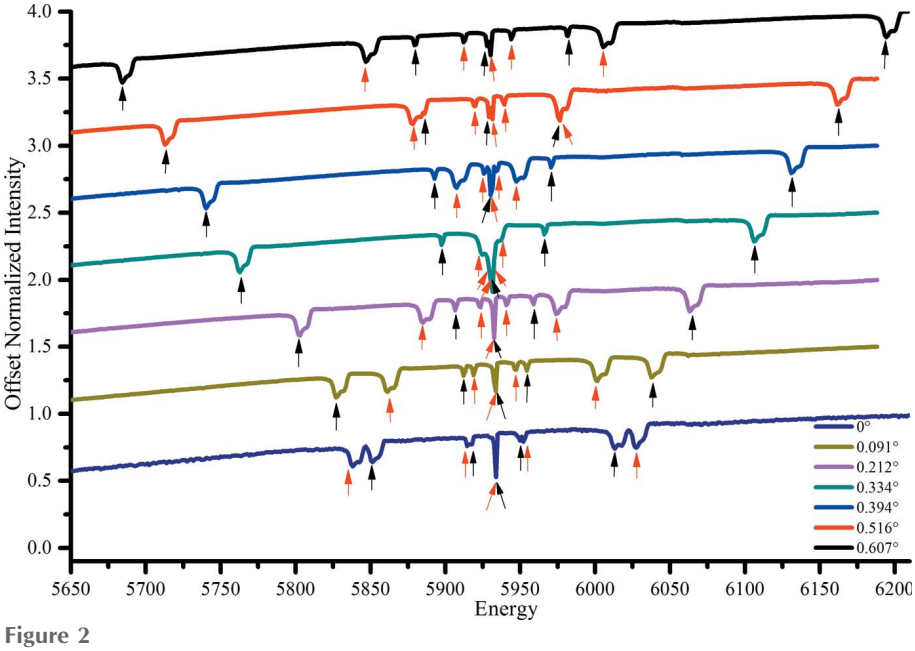


Figure 2
Movement of the glitch positions for different azimuthal angles. The labels show the variation of the angles during the experiment, which are calculated geometrically. The intensities of the X-rays are normalized to 1 and the offset of each line is 0.5. The black and red arrows indicate each set of glitch patterns belonging to the first and second crystals, respectively.

3. Conclusions and discussion

In summary, for one Si(111) crystal the period of the glitch pattern is $\pi/3$ and a series of mirror symmetries takes place at $\varphi = n\pi/6$ ($n = 0, \pm1, \pm2\ldots$). A quick glitch position split occurs when the azimuthal angles deviate from the center of mirror symmetries, and a simulation of the glitch number distribution of one period shows that the optimal azimuthal orientation for one Si(111) crystal is $\varphi = (2n + 1)\pi/6$ ($n = 0, \pm1, \pm2\ldots$). For the Si(111) DCMs, the glitch patterns are the superposition of two sets of glitch patterns caused by the two crystals. Therefore, the optimal azimuthal orientations for the Si(111) DCMs for achieving the least amount of glitches in the hard X-ray region are $\varphi_{1,2} = (2n + 1)\pi/6$ ($n = 0, \pm1, \pm2\ldots$).

From the simulations and experiment results stated above for the Si(111) DCMs, because of the mirror symmetries and high angular sensitivity of the split rates of the glitch patterns the relative difference of the azimuthal angles of the two crystals is the key parameter for reducing the amount of glitches in the hard X-ray region. In

practical design, cutting and setting up of the Si(111) DCMs, however, little attention had been paid to this because it has no influence on the intensity of the output beam (see §S2 of the supporting information). During the crystal-cutting procedure, for instance, we often focus on the primary diffraction plane of the crystal while ignoring the other two planes which are perpendicular to it. In addition, during the setting-up procedure, an adjustment of the relative azimuthal angle has also been ignored, so that sophisticated glitch patterns appear. Therefore, in order to reduce the amount of glitches, the following procedures should be taken into account.

(i) During the crystal-cutting procedure of the Si(111) DCMs, the orientations of the planes that are perpendicular to the primary diffraction planes of the two crystals should be chosen at $\varphi_{1,2} = (2n + 1)\pi/6$ ($n = 0, \pm 1, \pm 2 \ldots$) corresponding to the $11\bar{2}$ direction, *i.e.* the $01\bar{1}$ direction.

(ii) During the setting-up procedure of the Si(111) DCMs, first of all an adjustment of the relative azimuthal angle should be taken into consideration in order to keep the following relationship rigorously satisfied: $|\varphi_1 - \varphi_2| = n\pi/3$ ($n = 0, \pm 1, \pm 2 \ldots$), *i.e.* the two sets of glitch patterns caused by the two crystals of the DCMs merge into one glitch pattern. In addition, an adjustment of the Si(111) DCMs as a unit should be made to ensure that the azimuthal angles rigorously satisfy $\varphi_{1,2} = (2n + 1)\pi/6$ ($n = 0, \pm 1, \pm 2 \ldots$), *i.e.* the two crystals of the DCMs are situated at the optimal azimuthal orientation where the least amount of glitches are achieved in the hard X-ray region.

4. Related literature

The following references are mentioned in the supporting information: Hümmer & Weckert (1995), Weckert & Hümmer (1990, 1997).

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References

Arthur, J. (1989). *Rev. Sci. Instrum.* **60**, 2062–2063.
Bunker, G. (2010). *Introduction to XAFS: A Practical Guide to X-ray Absorption Fine Structure Spectroscopy.* Cambridge University Press.
Chang, S. L. (2004). *X-ray Multiple-Wave Diffraction: Theory and Applications.* Berlin: Springer-Verlag.
Hümmer, K. & Weckert, E. (1995). *Acta Cryst.* A**51**, 431–438.
Huang, X. R., Siddons, D. P., Macrander, A. T., Peng, R. W. & Wu, X. S. (2012). *Phys. Rev. Lett.* **108**, 224801.
Konigsberger, D. C. & Prins, R. (1987). *X-ray Absorption: Principles, Applications, Techniques of EXAFS, SEXAFS and XANES.* New York: John Wiley and Sons.
Van Der Laan, G. & Thole, B. T. (1988). *Nucl. Instrum. Methods Phys. Res. A*, **263**, 515–521.
Weckert, E. & Hümmer, K. (1990). *Acta Cryst.* A**46**, 387–393.
Weckert, E. & Hümmer, K. (1997). *Acta Cryst.* A**53**, 108–143.
Yang, C. Z., Hao, J. M. & Pei, G. W. (2000). *Rigaku J.* **17**, 46–57.