Robust optic alignment in a tilt-free implementation of the Rowland circle spectrometer

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Abstract: High-resolution x-ray emission spectroscopy (XES) has recently been demonstrated in the laboratory setting, achieving nearly synchrotron-level count rates despite the use of only conventional x-ray tube sources. This holds high potential for greatly expanding the reach of x-ray spectroscopies beyond the specialist community of synchrotron users, but comes with its own unique technical challenges. Just as with most XES instruments at synchrotrons, the lab-based spectrometers use spherically bent crystal analyzers (SBCAs), however in the lab setting this comes with an added barrier to best implementation. The imperfect parallelness between the desired crystal plane and the wafer surface, usually called “wafer miscut”, introduces an ambiguous re-focusing error that typically requires a motorized two-axis tilt stage for fine alignment of the SBCA optic onto the ideal Rowland circle configuration. While the strong reference scattering signals at synchrotron light sources allow for straightforward alignment by scanning these angular degrees of freedom, the same is not true in laboratory instruments where weak XES signals and the low brilliance of the conventional x-ray source can make alignment onerous. Here a new, asymmetric Rowland geometry is demonstrated that eliminates all need for motorized fine-tilt adjustment and is shown to provide rapid, extremely reproducible re-insertion of any aligned SBCA, i.e., without the need for any subsequent reoptimization. These improvements strongly improve ease of use of laboratory based spectrometers, taking them an important step closer to turnkey operations.

Keywords: Spectroscopy; Instrumentation; XANES; XAS; Optics.

I. Introduction

One of the most important advances in hard x-ray spectroscopy has been the development and proliferation of spherically bent crystal analyzers (SBCAs). When employed in the Rowland circle geometry^{1,2}, these x-ray optics monochromatize and refocus point-source input via Bragg reflection. The intrinsically narrow bandpasses of the perfect crystals used in construction result in resolutions of ~ 1 -eV for the simplest applications to as little as a few-meV for diced analyzers in near-backscatter geometries³⁻⁵. Such qualities make SBCAs an integral component in synchrotron-based and, very recently, in laboratory-based instrumentation⁶⁻¹⁸.

SBCAs are manufactured by bonding or gluing flat Si or Ge wafers to a shaped surface, usually a concave glass lens¹⁹⁻²¹, so that the resulting crystal surface achieves the desired radius of curvature, i.e., equal to the Rowland circle diameter. However, due to limitations imposed during wafer slicing from large single-crystal boules and subsequent polishing, the underlying crystal planes typically have so-called miscut angles of $0.1 - 0.5^\circ$ with respect to the crystal surface. While this fabrication error generally does not significantly degrade the performance of the optic, as we will discuss in section V, it complicates the implementation of any Rowland circle spectrometer, previously leading to the use of a two-axis tilt for fine alignment of the SBCA as a compensation for wafer miscut even in the hypothetical absence of other positional or alignment errors. These fine alignment requirements increase instrument complexity and cost in each of design, construction, maintenance, and operations. In the present context, the lattermost issue, operations, is of primary concern.

X-ray absorption spectroscopies, whether x-ray absorption fine structure (XAFS), x-ray emission spectroscopy (XES, by which we mean x-ray fluorescence spectroscopy with energy resolution comparable to the core-hole lifetime), or more complex variants has been the almost sole purview of a modest community of specialists. This situation has occurred not because of the theoretical limitations requiring extended, high-level expert training for any reliable implementation, but instead because of the access limitations inherent to synchrotron radiation facilities and the consequent oversubscription of beamlines supporting these methods. Over the last several years there has been a growing effort to launching a rebirth of laboratory based XAFS and XES for routine materials characterization^{16-18,22-24} (and also for more advanced time-resolved applications²⁵⁻³¹). In the present manuscript, the development of a spectrometer that requires no optic realignment is an important step toward the level of turnkey operations needed for broader use of XES in communities that need the chemical information provided by XES but where training in x-ray optic design and implementation would be rare.

To be specific, here we demonstrate that careful consideration of the orientation of the miscut-error with respect to the symmetry axis of the SBCA has dramatic consequences for spectrometer design and

operations. In particular, we demonstrate that the two-axis tilt can be removed from a Rowland circle SBCA spectrometer without loss of performance and, in fact, with considerable simplification in instrument design and ease of use.³² The key technical point rests in choosing to have the Rowland circle configuration adapt to the wafer-cut error rather than have the optic adapt to the most idealized symmetric Rowland configuration.

This paper continues as follows. In section II, we introduce general considerations of wafer miscut and Rowland circle operations. In section III, we demonstrate the expected sensitivity of SBCA alignment to both the magnitude of the miscut and also to its orientation with respect to the Rowland circle, all when using a conventional spectrometer that includes the two-axis tilt, *i.e.*, in the usual "symmetric Rowland" configuration. In section IV, we instead introduce the "asymmetric Rowland" configuration and provide the major results of this paper. We find that alignment of the miscut into the Rowland plane allows full compensation with a detector offset, enabling complete removal of the two-axis tilt behind the SBCA. In this configuration, we find easy initial SBCA alignment which is then permanent, in that an aligned SBCA can be removed and then later reinstalled with no loss of performance and with strongly, immediately reproducible energy scales. In section V we provide additional results in support of these assertions, including especially ray-tracing results for the focal errors induced by our approach. Finally, in section VI we summarize and conclude.

II. Compensating for wafer miscut in SBCA alignment

The effect of wafer miscut is illustrated in Fig. 1. A miscut crystal has a tilt in its reciprocal lattice vector, $\vec{G}$, (*i.e.* the vector perpendicular to desired crystal plane) with respect to the surface normal, $\vec{n}$, at every point on the wafer and hence also on the curved optic. This introduces a serious hurdle to SBCA alignment. In standard practice, an SBCA is prealigned using optical reflection of a diverging laser. In this specular reflection the refocusing geometry is determined by the surface normal, $\vec{n}$. X-ray scatter, however, occurs in the crystal bulk and its reflection is wholly dependent on $\vec{G}$. The result of miscut is therefore an ambiguous deviation in the x-ray reflection away from the optical light used in alignment (*i.e.* the expected geometry in the absence of wafer miscut).

The ambiguity, as encompassed by a circle of possible refocus positions shown in Fig. 1, is due to the new degree of freedom introduced into the system by the symmetry-breaking of the wafer miscut: the azimuthal, or "clock" angle (φ) of the SBCA is now relevant and couples directly to the orientation of $\vec{G}$ and thus to the re-focus position. The miscut is not known a priori, and consequently even the most carefully engineered instruments require a means for compensation.

The canonical solution has been to use a two-axis (θ, χ) tilt on each SBCA to steer the monochromatized beam back to the optically-determined, symmetric detector position. This places the reciprocal lattice vector at the center of the SBCA, $\vec{G}_0$, at the high-symmetry bisector of source and detector. This is shown schematically as the “symmetric Rowland” configuration in Fig. 2.

This alignment process is often technically straightforward at synchrotron light-sources where a strong elastic scatter line can always be obtained at the desired alignment energy. However, two-axis tilt alignment of the SBCA is often more arduous for laboratory-based spectrometers which employ low-brilliance, x-ray tube sources. In particular, alignment is especially challenging for XES configurations investigating weak emission lines, or for nominally strong emission lines from very dilute species.

In this work we overcome this limitation and demonstrate an alternate correction scheme with two main benefits. First, in the new approach a two-axis tilt is not necessary. Second, after an initial alignment of the SBCA optic with a strong emission line or direct bremsstrahlung illumination, no further re-orientation is needed: the optic can be pulled from the shelf, inserted into the instrument and tune-up is immediate. To achieve this, the tilts are held fixed at $\theta = \chi \cong 0^\circ$ as a consequence of two changes in operation: (1) the optic clock angle, i.e., the rotation of the optic about the cylindrical axis of its support, usually a glass lens, is chosen such that $\vec{G}_0$ is rotated into the Rowland plane; and (2) the detector position is offset to the actual refocused path. This is shown in Fig. 2 as the “asymmetric Rowland” configuration. The remainder of this paper looks at how the miscut can be determined for instruments in the symmetric or asymmetric configuration, and, more importantly, demonstrates substantial simplification and improved ease of operation for the latter design. We furthermore demonstrate how the asymmetric Rowland configuration requires an SBCA to be aligned only a single time, allowing for “plug and play” operation upon subsequent use.

III. Symmetric Rowland configuration

It has previously been shown that SBCA miscut can be accurately determined through careful optical alignment and subsequent collimated-beam x-ray diffraction measurements from the analyzer center³³. Here we demonstrate an alternative method that is useful for illustrating the consequences of the conical locus of possible refocus positions discussed above (Fig. 1) and the usual adaptation to wafer miscut, which is to orient the optic so to best conform to the requirements of a symmetric Rowland circle implementation.

This method uses only the equipment already standard for symmetric Rowland circle spectrometers, namely a two-axis tilt. The first step of this procedure is the same as for any alignment: the optic is mounted and its yaw (θ) and pitch (χ) are adjusted to maximize detected signal. The clock angle (φ),

meaning the rotation about the cylindrical axis of the optic's physical support, is then rotated by a known amount and the above step is repeated. When this procedure is carefully carried out through a full 360° revolution the optimized θ and χ tilts trace out a circle, corresponding to the rotation of $\vec{G}$ at the SBCA center, $\vec{G}_0$, about the optic's physical symmetry axis, $\vec{A}$. While the spatial map of $\vec{G}$ as a function of position along the spherically curved surface is indeed more complex, one must remember the key point of Rowland circle operation is that, absent the well-known inherent geometric errors of Johann-typed SBCAs, the angle of incidence of the x-rays from a point source is the same at every point on the SBCA surface. Note that the angular radius of the circle traced in θ and χ is equal to twice the miscut angle, α .

To test this premise we performed the above procedure on two Si 111 and one Si 551 SBCAs (all from XRS Tech), each having 100-mm wafer diameter and 1-m radius of curvature. Data for this study was collected using a laboratory-based spectrometer recently developed at the University of Washington¹⁶⁻¹⁸. Reference x-ray fluorescence signals are generated by emission from Cu or Ni foils excited by the output from a conventional tube source (Moxtek) with an Au anode operated at 35 kV and 200 μ A. The Cu and Ni emission lines were used to provide strong, narrow signals for accurate calibration and for fine tests of changes in spectrometer alignment, resolution and energy scale upon removing and re-installing the corresponding SBCA. The detector is a solid-state Si drift detector (Amptek XR-123SDD) with a 5-mm diameter active area. The region of interest was set appropriately to reduce background signal.

The results are shown in Fig. 3. Data were taken in incremental steps $\Delta\varphi = 15^\circ$ with fiducial markings insuring accuracy to within $\pm 1^\circ$. For this study the rotation was applied manually, though this could easily be replaced with a motorized stage if desired. For the Si 111 optics, the Cu $K\alpha_2$ line was used for alignment (80.1° Bragg angle at 444 reflection) while for Si 551, the Ni $K\beta_{1,3}$ emission (80.5° Bragg angle) was used. The data shows excellent agreement to the expected circular shape with extracted miscut values of $0.45 \pm 0.01^\circ$, $0.13 \pm 0.01^\circ$, and $0.081 \pm 0.002^\circ$ accordingly.

In addition to the magnitude of the miscuts, their orientations are obtained by careful note of the clock angle associated with each set of (θ, χ) values. The convention for clock angle is as follows: 0° corresponds to $\vec{G}_0$ in the Rowland-plane and tilted towards the source-side of the circle; 90° corresponds to $\vec{G}_0$ tilted out-of-plane upwards, etc. (see Fig. 3).

In Fig. 4 we test the consequence of the SBCAs clock angle φ on the measured non-resonant $K\beta$ emission spectrum of Ni metal. In each case the tilts have been adjusted as described above. All spectra presented in this study were collected at 15 seconds of integration per point in 0.1 eV steps. Here the resolution is dominated by the K-shell corehole lifetime (1.44 eV) and we expect the difference between orthogonal orientations to be indistinguishable. Indeed the peak-normalized spectra are identical to within statistical

noise. Furthermore, the count rates agree favorably with peak rates of 4270/s and 3990/s for in-plane ($\varphi = 0^\circ$) and out-of-plane ($\varphi = 90^\circ$) orientations respectively. These results indicate that for experiments that are intrinsically limited by corehole broadening to ~ 1 -eV or coarser resolution the SBCA performance after conventional alignment with a two-axis tilt is independent of clock angle.

IV. Asymmetric Rowland configuration

Armed with the above knowledge, we now investigate how the careful selection of the orientation of $\vec{G}_0$ can simplify overall instrument alignment and operations. As a first scenario, in the symmetric Rowland geometry the miscut can be chosen to be rotated either into or out of the Rowland plane. Doing so removes the need for either, but not both of, the χ or θ adjustment, effectively reducing the required motorized degrees of freedom to a 1-D tilt, with obvious saving in both instrument cost and alignment time.

A second, more aggressive, approach is to eliminate two-axis fine tilt adjustments entirely. This can be realized by rotating $\vec{G}_0$ into the Rowland plane and offsetting the detector position to position D' as shown in the asymmetric Rowland configuration in Fig. 2. The magnitude of the offset, ΔD , is determined geometrically to be

$$\Delta D = R \sin^2(\theta_B) [\cot(\theta_B - 2\alpha) - \cot(\theta_B)] \quad (1)$$

where R is the SBCA radius of curvature, θ_B is the expected Bragg angle absent any miscut, and α is the crystal miscut. In principle the detector offset is a function of θ_B which is the exact parameter altered during an energy scan. However, for $\alpha/\theta_B \ll 1$, which is true for typically modest miscut and operation close to backscatter, eq. (1) reduces to

$$\Delta D \cong 2R\alpha + \mathcal{O}(\alpha/\theta_B)^2 \quad (2)$$

which is independent of θ_B . This constant offset approximation holds quite well over a wide range of Bragg angles. For example, for a 1-m curvature SBCA having 0.5° miscut the optimal detector offset as determined by eq. (1) is 17.5 mm at perfect backscatter and 17.6 mm at $\theta_B = 70^\circ$. Thus any reasonably sized detector can be used in an energy scan with constant detector offset. While this approach will alter the energy calibration with respect to the perfectly symmetric case, this issue is addressed pragmatically by, e.g., measurement of reference standards, just as would be done with the usual experimental configuration.

This method, of course, hinges on orienting $\vec{G}_0$ into the Rowland plane which requires prior knowledge of the SBCA miscut. While the analyzer could first be characterized in a symmetric Rowland instrument

with a two-axis tilt and then transferred to the fixed, asymmetric design, this negates much of the intended benefits. We have therefore identified a related procedure for optimizing the orientation of the SBCA miscut entirely within an asymmetric Rowland geometry.

This is accomplished by measuring the count rate of a reference signal as a function of φ . Any out-of-plane tilt in $\vec{G}_0$ deflects the reflected beam out of the Rowland plane shown in Fig. 2 and cannot be compensated by the simple 1-D detector offset. For a finite sized detector, or a large detector constrained by appropriately sized entrance slits, the count rate will therefore be maximized when $\vec{G}_0$ is oriented in the Rowland plane. Once the maximum signal is found, a simple fiducial mark on the cylindrical exterior of the SBCA's lens-support allows for easily reproduced alignment, as we show below.

To test the efficacy of this proposed method, we modified the University of Washington instrument described in section III (see Fig. 5). Specifically we removed the two-axis tilt module and in its place installed a simple, rigid right-angle bracket, aligned and shimmed a single time with assistance of a laser and flat mirror to ensure correct placement with respect to the Rowland circle. The flat back of the SBCA lens-support is simply clamped flush to the face of this bracket as shown in the inset in Fig. 5. The alignment procedure was carried out using the 0.45° -miscut Si 111 analyzer and Cu foil reference sample from section III. At each φ the detector offset was scanned to find the maximum Cu $K\alpha_2$ signal as indicated in Fig. 6. The shift in the shape when φ is rotated by 180° indicates a small error in our geometry with the detector sitting slightly below the Rowland plane. This, however, does not affect the maximum count rate, compared to the symmetric Rowland geometry, nor does it significantly degrade resolution as will be discussed in section V.

In addition to the reduction of mechanical components, this fixed bracket design allows for remounting of SBCAs with only minor irreproducibility in their position. Realignment is therefore not needed. The proof of this claim is demonstrated in Fig. 7. For each sample the SBCA miscut was rotated in-plane as discussed above. An initial detector scan (< 5 min) was performed to find the optimal offset and a spectrum was recorded. The sample and optic were then entirely removed. After performing other measurements with the spectrometer we then reinstalled the same sample/SBCA combinations and repeated the spectral scans using the same detector offsets and SBCA clock angles found previously (i.e. without any re-optimization scans). The agreement is excellent with only minor changes in the peak count rate (13600/s vs. 13400/s for Cu and 3200/s vs. 3920/s for Ni). The small discrepancy in intensity is most likely due to degradation of the He space over time. We stress that no relative shift has been applied between the initial and revisited spectra thus demonstrating a reproducible energy scale to ~ 50 meV without recalibration.

This new asymmetric Rowland configuration and its implementation form the major results of this paper. Our design successfully removes all motorized tilt degrees of freedom, substantially easing instrument design and construction without degrading spectrometer performance.³² Furthermore, the simplified alignment and robust reproducibility upon reinstalling SBCAs offers obvious benefits in user operations and towards the study of dilute samples and/or weak emission lines where direct alignment is impractical due to lack of a strong signal. While the results have only addressed x-ray emission spectroscopy, they more broadly apply to any operation of a Rowland circle spectrometer using SBCAs, including when used as a monochromator for laboratory-based x-ray absorption fine structure (XAFS)^{16,17}.

V. The effect of wafer miscut on SBCA performance

Having established the importance of careful control of the orientation of the wafer miscut, before concluding we examine how the relative orientation of $\vec{G}$ affects overall instrument performance. We begin by addressing energy resolution. The factors limiting resolution of spherical analyzers fall into four main categories³⁴: (1) intrinsic contributions from the crystal Darwin width and strains induced during the bending process; (2) the finite angular size of the source (or sample) as seen by the SBCA; (3) Johann errors due to only the center of the SBCA being on the Rowland circle; and (4) axially aberrations caused by the out-of-plane spherical bending being correct only at perfect backscatter. These factors are taken to be independent and therefore combine in quadrature to give the final instrument resolution. The latter three categories are purely geometric aspects and all affect energy resolution as $\cot(\theta_B)$. For this reason instruments requiring highest possible resolution are always designed to work at extreme backscatter geometries.

Of the factors listed above, Johann and axial errors are clearly the most sensitive to crystal miscut. Both are due to variation across the face of the analyzer in the angle between incident radiation and $\vec{G}$, i.e. the complementary Bragg angle. These geometric aberrations have long been understood and are well documented^{35,36}. Crystal miscut directly influences the direction of $\vec{G}$ and therefore requires a direct assessment in the context of Johann error.

Here we implement a method for calculating the combined Johann and miscut errors using ray tracing code in *Mathematica*. As the performance of SBCAs working in the symmetric Rowland configuration has been well-established experimentally, for brevity we present only results for the asymmetric geometry. This code calculates $\vec{G}$ at all points on the SBCA by first applying the miscut rotation to the lattice planes of a flat crystal and then bending its surface onto the partial analyzer “sphere”. The incident Bragg angle is then mapped across the analyzer surface for a given source location. This gives the added functionality of being able to fold in the effects of finite source size so as to account for all the geometric

factors limiting resolution. We do not consider the finite penetration of the x rays into the crystal bulk. While this is a primary energy-broadening mechanism far from backscatter, it is a minor effect for the angle ranges presented in this study. We furthermore consider the misorientation of $\vec{G}$ with respect to $\vec{n}$ to be coherent at every point, in that we ignore solid mechanics effects such as have been discussed in detail by ³⁷.

We emphasize that this ray tracing considers only geometric effects assuming perfect Bragg surfaces. In practice the elastic bending of flat wafers introduces crystal strains. These defects, which vary from optic to optic, are non-trivial and can modify the rocking curve and refocused shape significantly. Nonetheless, such strain-induced effects are independent of $\vec{G}$ orientation and the following discussion gives valuable insight into the effects of crystal miscut.

Calculations were run at several Bragg angles and miscut magnitudes for both a point source and 1-mm diameter source size. When introducing miscut, the central source location is adjusted along the Rowland circle such that the Bragg angle at the SBCA center is constant, allowing for a functionally correct assessment of the consequences of miscut. To ensure reasonable computation times we employ a stochastic approach where an x-ray generated from a random point in the source area strikes a random point on the SBCA. This process is repeated many thousands of times.

The effective angular spread of the incident rays on the SBCA is shown in Fig. 8, where we have assumed the standard 100-mm diameter wafer bent to 1-m radius of curvature. Calculations were also run for the pristine case (i.e. zero miscut) in order to provide a fair distinction between contributing errors. We have defined the figure of merit, $\Delta\theta$, to be the root mean square deviation from the desired θ_B of the actual incident Bragg angles across the SBCA surface.

As expected, the effect of crystal miscut varies with φ and becomes more severe when θ_B is further from backscatter. For modest miscuts and Bragg angles close to backscattering, the magnitude of this modulation is at most a factor of ~ 0.25 compared to the other geometric effects and occurs when $\vec{G}_0$ is oriented out-of-plane. Most importantly, our calculations show that the contribution from crystal miscut can be negated by proper choice of φ . Interestingly, we find that for $\varphi = 180^\circ$ ($\vec{G}_0$ in-plane slanted towards the detector) the angular spread is actually improved upon by the addition of miscut. This is somewhat unexpected but is due to our allowance for movement in source position. At $\varphi = 180^\circ$ the source location required to achieve an equivalent Bragg angle is closer to the nominal backscatter location which in part lessens the geometric aberrations discussed above.

One of the major benefits of SBCAs is their ability to refocus the analyzed radiation onto a small spot, allowing for the use of smaller-area detectors and also providing outstanding geometric rejection of stray

scatter, hence it is important to determine the influence of wafer miscut on the SBCA exit focus. We again use the ray-tracing code discussed above. With $\vec{G}$ being known across the analyzer surface the reflected ray is determined and traced back to a specified detector plane. Again the penetration of x rays into the crystal before reflection is not accounted for. These penetration depths, however, are small on the scale of the total travel distance and thus do not significantly affect the refocused position.

In Fig. 9 we present ray tracing results for several source-size diameters. A 100-mm diameter SBCA with 1-m radius of curvature and 0.5° miscut, oriented at 180° clock-angle, working at 80° Bragg angle was assumed. Results are shown for two different detector constraints. In Fig. 9a the detector center is constrained to lie along the x-axis of the source (position D' in Fig. 2). We also present the case in Fig. 9b where the detector face lies tangent to the Rowland circle (position D'') to demonstrate that focusing is recovered.

For comparison we overlay the shape of the full refocused beam for a pristine analyzer (red line). From these plots we see that in-plane focusing is strongly influenced by miscut orientation. The same is not true of the out-of-plane displacement where the increase from the pristine case, in which the spread is due to approximated spherical bending, is minimal. The out-of-plane spread, however, is dominant in magnitude compared to the in-plane direction even when working at the slightly off-circle position. The same considerations for detector size are thus clearly present regardless of miscut.

As a final test of spectrometer performance in the asymmetric Rowland configuration we present in Fig. 10 a comparison of a Ni $K\beta$ emission spectrum taken in the asymmetric configuration without a two-axis tilt to a Ni $K\beta$ emission spectrum instead recorded in the symmetric Rowland geometry which incorporates the two-axis tilt. After correcting for the energy shift caused by detector offset, the two setups produce nearly identical resolutions and count rates (4270/s for the symmetric configuration and 3920/s for the asymmetric). This result, combined with the analysis above, validates the asymmetric design by showing that the motorized tilt degrees of freedom can be removed without detriment to data quality for nonresonant XES using SBCAs relatively near to backscatter.

VI. Conclusion

In summary, we propose and demonstrate that the ubiquitous two-axis tilt assembly used to align SBCAs can be eliminated by careful orientation of wafer miscut in an asymmetric Rowland geometry. This approach delivers equivalent performance to the typical symmetric Rowland configuration while providing clear benefits to instrument design, maintenance, and especially operations. We find that this design simplifies SBCA initial alignment while also providing extremely reproducible alignment upon reinstallation. We hope that the streamlined, one-time alignment procedure of the asymmetric Rowland

configuration lower the technical barrier to further implementations of lab-based advanced x-ray spectroscopy using SBCAs.

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Figure 1 The effect of crystal miscut on the refocused position of the monochromatized beam. Due to the symmetry-breaking of the miscut the azimuthal, or “clock”, angle (φ) becomes a relevant parameter directly affecting the reflection geometry. The yaw (θ) and pitch (χ) of the SBCA are typically adjusted to steer the refocused beam back onto the expected focal location on the Rowland circle.

Figure 2 Two methods of correcting for a wafer miscut of magnitude α . Here the clock angle can be thought of as a rotation of $\vec{G}_0$ about the SBCA symmetry axis $\vec{A}$. (Left) The symmetric Rowland case where θ and χ tilts are adjusted to steer the reflected beam (red line) back onto the expected detector position (D). Note that such an adjustment means the SBCA is no longer tangent to the Rowland circle. (Right) The asymmetric Rowland configuration where the SBCA is held fixed and the detector is moved to find the monochromatized beam. The simplest scheme offsets the detector by ΔD along the x-axis of the source (S) to an off-circle location (D'). Alternatively, the detector could be moved back on-circle (D'').

Figure 3 The θ and χ tilts of the refocused x-ray beam relative to the expected (optical) position. Tilts were determined by maximizing the $\text{Cu K}\alpha_2$ ($\text{Ni K}\beta_{1,3}$) signal for a Si 111 (Si 551) SBCA operating in the symmetric Rowland configuration. Data points were taken every 15° in φ . A clock angle, $\varphi = 0^\circ$ corresponds to $\vec{G}_0$ oriented in the Rowland-plane tilted towards the source-side of the circle, 90° corresponds to $\vec{G}_0$ tilted out-of-plane upwards, etc. The black dotted lines show a circular fit to each dataset, revealing miscuts of $0.45 \pm 0.01^\circ$, $0.13 \pm 0.01^\circ$, and $0.081 \pm 0.002^\circ$ accordingly.

Figure 4 Peak normalized Ni K β spectra for an in-plane ($\varphi = 0^\circ$) and out-of-plane ($\varphi = 90^\circ$) orientation of $\vec{G}_0$ in the symmetric Rowland configuration. For clarity of presentation the first spectra has been offset as indicated. Unnormalized count rates were 4270/s and 3990/s at peak for 0° and 90° clock angle respectively. The two datasets agree so well as to be nearly indistinguishable – the green curve shows the residual between the two normalized spectra. Both spectra were collected at 15 seconds of integration per point in 0.1 eV steps.

Figure 5 A perspective rendering of the University of Washington instrument modified to work in the asymmetric Rowland configuration. Inset: The new SBCA module where the two-axis tilt has been replaced with a rigid, right-angle bracket. The flat back of the SBCA lens is clamped flush to the bracket surface.

Figure 6 Maximum count rate as a function of clock angle for the asymmetric Rowland geometry. Cu $K\alpha_2$ emission was used as the reference signal measured by a 0.45° -miscut Si 111 (444 reflection) SBCA. Count rates are given as absolute peak counts/second. The horizontal dashed line indicates the counting rate achieved when employing the same analyzer in the symmetric Rowland configuration. The shift in the shape when the clock angle is rotated by 180° indicates a small error in the spectrometer design with the detector sitting slightly below the Rowland plane.

Figure 7 Peak normalized Cu K α and Ni K β spectra recorded using the previously characterized Si 111 and Si 551 SBCAs in an asymmetric Rowland geometry. All spectra were collected at 15 seconds of integration per point in 0.1 eV steps. Spectra have been vertically offset for clarity. For each analyzer the clock angle was set at 180° to orient $\vec{G}_0$ into the Rowland plane. An initial detector scan was performed to find the optimal detector offset and a spectrum was recorded. The same sample/SBCA combinations were later revisited and spectral scans were rerecorded using the same clock angles and detector offsets found previously (i.e. without a re-optimization scan). The agreement is excellent with only small changes in the peak count rate (13600/s vs. 13400/s for Cu and 3200/s vs. 3920/s for Ni). No relative shift has been applied to the energy scales, which reproduce to within ~ 50 meV without recalibration.

Figure 8 The angular spread due to combined geometric errors for both a point (top) and 1-mm diameter (bottom) source size as a function of clock angle for several miscut magnitudes. The analyzer is taken to be a 100-mm diameter SBCA with 1-m radius of curvature. A clock angle of 0° corresponds to $\vec{G}_0$ oriented in the Rowland-plane tilted towards the source-side of the circle, 90° corresponds to $\vec{G}_0$ tilted out-of-plane upwards, etc. A clock angle of $\varphi = 180^\circ$ is shown to minimize $\Delta\theta$ at all θ_B and miscut magnitudes α .

Figure 9 The relative refocused positions on the detector face for randomly generated rays. A 100-mm diameter SBCA with 1-m radius of curvature and 0.5° miscut, oriented at $\varphi = 180^\circ$, working at 80° Bragg angle was assumed. Results are shown for several different source-size diameters at two different detector constraints: a) the center of the detector lies on the line determined by the x-axis of the source/sample (D' in Fig. 2); b) the detector face is tangent to the Rowland circle (D'' in Fig. 2). In either case the out-of-plane spread is dominate and comparable to the zero-miscut results (shown in red).

Figure 10 A comparison of peak normalized Ni K β spectra collected with both symmetric and asymmetric correction Rowland geometries. For clarity of presentation the second spectra has been offset as indicated. The two datasets show near perfect agreement. The green curve shows the residual between the normalized spectra. Absolute peak count rates are 4270/s for the symmetric configuration and 3920/s for the asymmetric. Both spectra were collected at 15 seconds of integration per point in 0.1 eV steps.

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