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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 development holds high potential for expanding the reach of x-ray spectroscopies beyond the specialist community of synchrotron users, but comes with its own unique technical challenges for instrument performance and also, just as importantly, for ease of use by non-experts in x-ray science. Here, we address spectrometer design and operations in the context of the imperfect parallelness between the desired crystal plane and the wafer surface in spherically bent crystal analyser (SBCAs), an effect usually called "wafer miscut". This 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. We instead demonstrate an asymmetric Rowland geometry that eliminates all need for motorized fine-tilt adjustment. We find rapid, extremely reproducible re-insertion of any aligned SBCA, i.e., without the need for any subsequent reoptimization. These improvements strongly benefit the ease of use of laboratory based spectrometers, taking them an important step closer to the level of turnkey operations needed for wide adoption outside of the existing specialist community.

1. 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 their construction result in resolutions of ∼1-eV for the simplest applications to as little as a few-meV for diced analyzers in near-backscatter geometries [3–5]. Such qualities make SBCAs an integral component in synchrotron-based and, more recently, in laboratory-based instrumentation [6–18].

SBCAs are manufactured by bonding or gluing flat Si or Ge wafers to a shaped surface, usually a concave glass lens [19–21], such 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 can have so-called miscut angles as large as 0.1–0.5° 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 5, it complicates the implementation of any Rowland circle spectrometer, requiring fine alignment capability that increases 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 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 intrinsic lifetimes), or more complex variants have 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 materi-

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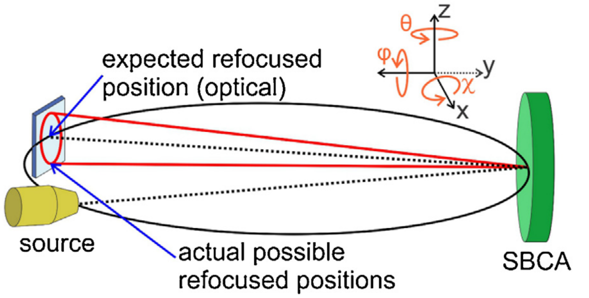

Fig. 1. The effect of crystal miscut on the refocused position of the monochromatized beam. Due to the symmetry–breaking of the miscut, the azimuthal, or “clock”, angle (ϕ) becomes a relevant parameter directly affecting the reflection geometry. The yaw (θ) and pitch (χ) of the SBCA are typically adjusted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

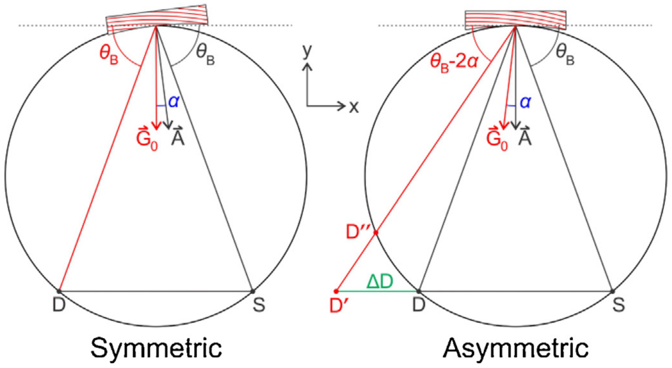

Fig. 2. Two methods of correcting for a wafer miscut of magnitudeα. Here the clock angle can be thought of as a rotation of $\vec{G}_0$about the SBCA symmetry axis $\vec{A}$. (Left) The symmetric Rowland case where θ and χ tilts are adjusted to steer the reflected beam (red line) back onto the expected 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 track on-circle (D'') to recover the focusing, asymmetric Rowland configuration of Suortti, et al [33].

als characterization [16–18,22–24] (and also for more advanced time-resolved applications [25–31]). 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 physical symmetry axis of the SBCA has dramatic consequences for spectrometer design and operations. In particular, we find 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 use an asymmetric configuration to adapt to the wafer-cut error rather than have the optic adapt to the most idealized symmetric Rowland configuration. The asymmetric Rowland circle configuration has been treated in great detail by Suortti, et al. [33].

This paper continues as follows. In Section 2, we introduce general considerations of wafer miscut and Rowland circle operations. In Section 3, 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 4, we instead consider 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 5 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 6 we summarize and conclude.

2. 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.

In synchrotron applications, there are two generic solutions to this problem. First, one may 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 approach is particularly prevalent in multi-analyzer systems, e.g., Fister, et al. [34], where other degrees of freedom, described below, are often not available.

Second, one may instead move to an asymmetric Rowland configuration, again see Fig. 2. For this case, the optic center is maintained tangent to the Rowland circle and $\vec{G}_0$ is oriented to reside in the plane of the Rowland circle. When the detector is placed at point D”, a perfect Rowland focusing configuration, subject to Johann error, is recovered. This approach, first described in fine detail by Suortti, et al. [33], sees use in synchrotron instrumentation where the source location is fixed and, importantly, this approach can be easily modified to operate dispersively.

From an instrumentation perspective in the context of laboratory-based instrumentation with low-weight x-ray sources [16–18], however, the full method of Suortti, et al. [33] using a fixed source and perfect detector tracking to the optic refocal point is unnecessarily complex, embodying extra motorized degrees of freedom. Operational advantages instead accrue from operating the spectrometer in a configuration where the source and detector