SHORT COMMUNICATIONS

Chart of accessible regions in reciprocal space when beam-to-crystal surface angles are limited.

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A chart has been constructed which shows accessible regions of reciprocal space when the angles between the crystal face and both the incident and diffracted beams are limited to \( \geq \delta \). The normal to the crystal face lies in the plane defined by the two beams. Both 'reflection' and 'transmission' geometries are considered. The chart is helpful in the design of various diffraction experiments with faceted crystals larger than the incident beam. As an example, it is shown how the chart can be used to help choose a sample geometry for the measurement of X-ray Debye–Waller factors in \( \beta \)-tin.

**Introduction**

In many diffraction experiments extended crystals that intercept the full incident beam are used. They are generally convenient to handle and maximize diffraction intensities. However, because of their relatively large size, problems arise in maintaining accurate diffraction geometries and in making absorption corrections. For these reasons it is often desirable to restrict the angles between the crystal face(s) and the beams to \( \geq \delta \). Frequently, other factors make such restrictions desirable or necessary. These include the limited lateral size of crystals, the manner in which they are mounted, limits on the area of the crystal to be irradiated and limits on the cross-sectional area of the diffracted beam. As a result of these restrictions, the regions of reciprocal space that can be studied are limited. Knowledge of the nature and extent of these limitations is important in the design of many diffraction experiments. For this reason a chart has been constructed which shows the accessible regions of reciprocal space when two commonly used diffraction geometries are employed and when various restrictions on beam-to-surface angles are imposed. The results obtained extend a discussion by Buerger (1960) on absorption in moving-film techniques.

**Construction**

Fig. 1 shows the regions of reciprocal space that can be studied by reflection and by transmission when a crystal larger than the incident beam is irradiated. 'Reflection' refers to the case in which the incident and diffracted beams enter and leave the crystal through the same face. 'Transmission' refers to the case in which the incident beam enters one face and the diffracted beam leaves through a second parallel face. As the crystal is rotated, all dashed lines in the figure rotate in the appropriate manner about the origin of the reciprocal lattice. The normal to the crystal face is assumed to lie in the plane defined by the incident and diffracted beams.

Consider the unshaded region above the large diameter in Fig. 1. This reflection region can be thought of as arising in the following way. Reflected beam, \( OP \), which is not necessarily a Bragg reflection, can occur when the crystal face makes any angle between 0° and 20° with the incident beam. For this range of orientations the points on arc \( AB \) in reciprocal space pass through point \( P \) on the sphere of reflection. For all points \( P \) on the upper half of the sphere arcs similar to \( AB \) can be constructed. Together, these arcs define a reflection region. The other reflection and transmission regions in this figure can be shown to arise in a similar fashion.

If the incident and reflected beams are constrained to make angles \( \geq \delta \) with the crystal face, beam \( OP \) can only occur when the crystal makes an angle between \( \delta \) and \( 20-\delta \) with the incident beam. This corresponds to sampling arc \( A'B' \) in reciprocal space. Arcs similar to \( A'B' \) can be constructed through different points \( P \) as before. Together they define a region of reciprocal space bounded by the limiting sphere and the contour \( \delta \) that can be studied by reflection when both the incident and reflected beams make angles \( \geq \delta \) with the crystal face.

Fig. 2 is a chart of reciprocal space which shows contours of constant \( \delta \) at 15° intervals. Each contour is the locus of points such that when they touch the sphere of reflection, either the incident or diffracted beam makes an angle of \( \delta \) with the crystal face while the other makes an angle \( \geq \delta \). When the singular point touches the sphere, both beams make angles of \( \delta \) with the crystal face. The origin of reciprocal space lies at the center of this figure, the dashed line is parallel to the crystal face and the face normal is vertical. See the Appendix for details of construction.

When a point in the unshaded reflection region lying between contour \( \delta \) and the limiting sphere touches the sphere of reflection, both the incident and reflected beams make...
angles $> \delta^\circ$ with the crystal face. When a point in the shaded transmission region lying inside contour $\delta$ touches the sphere of reflection, both incident and transmitted beams make angles $> \delta$ with the crystal faces.

**Application**

As an example of the use of this chart, consider the problem of determining X-ray Debye–Waller factors of $\beta$-tin using a large crystal. The atoms in $\beta$-tin lie at special positions of symmetry 42m in space group 141/amd. In such a crystal it is desirable to measure the integrated intensities of high-angle reflections close to the $c^*$ axis and the $a^*b^*$ plane. For instance, consider $h0l$ reflections. Fig. 3 shows the accessible portion of the $h0l$ net if the normal to the crystal face is assumed to lie at $45^\circ$ to $a^*$ and $c^*$, Mo $K\alpha$ radiation is used, $75^\circ > 2\theta > 160^\circ$ and $\delta > 30^\circ$. It is clear that only certain reciprocal lattice points are accessible and of these only a fraction lie in desirable regions of reciprocal space. These include the 16,0,0; 14,0,2; 15,0,3; 408 and 109 which all lie in the reflection region. By comparison, the reciprocal lattice points in the transmission regions are for the most part at lower $2\theta$'s and further away from the desirable regions of reciprocal space. It is clear that other surfaces whose normals lie at $45^\circ$ to the [001] might have been considered. Even more useful data could be obtained if the crystal were cut with two faces, one parallel to $(hk0)$ and the other parallel to $(00l)$.

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**APPENDIX**

The chart in Fig. 2 can be constructed by following the steps below. Directions or labels in italics are to aid in construction and are not part of the final chart.

1. Draw a circle, $LS$, of radius 2 to a convenient scale. *This represents the limiting sphere. Its center is 0.*
2. Draw a diameter, $D$. This is shown as a dotted line in Fig. 2.
3. To aid in construction draw $P$, a perpendicular to $D$ through 0. Draw $C$, a circle of radius 1 concentric with $LS$.
4. Call either intersection of $C$ with $D$ $n = 1$. Starting at this point divide the circumference of $C$ into $N$ equal segments. $N$ must be an even number. $N = 360^\circ / \Delta$ where $\Delta$ is the desired contour interval. In Fig. 2 $\Delta = 15^\circ$ and $N = 24$. The points on the circumference of $C$ at the boundary of each segment are consecutively numbered $n = 1, 2, 3, ..., N$.
5. Draw two circles of radius 1 about point $n = 1$ and $n = N/2 + 1$. These are the 0° contours. The areas within these circles represent the transmission regions and are shaded in Fig. 2.
6. With the exception of points 1, $N/2 + 1$ (and points $N/4 + 1$ and $3N/4 + 1$ if they exist) use each of the remaining points as the center of 2 arcs of radius 1. First swing and arc from $LS$ to $P$ by the shorter path and then from 0 to another point on $D$ by the shorter path. When these arcs are completed, contours similar to those in Fig. 2 will have been constructed. The first contour inside and outside the circles drawn in step 5 is labeled $\Delta^o$, the second 2$\Delta^o$, etc.

**Reference**