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Crystal alignment on the Weissenberg goniometer.

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In two recent communications Winchell (1950) and Jerslev (1951) have indicated modifications of the method of Bunn (1945) for aligning accurately a crystallographic axis with the rotation axis of an X-ray camera. The rapidity and accuracy with which the angular corrections can be made are the criteria for the most suitable method. Shorter X-ray exposures can be obtained using the continuous spectrum (no monochromator) and simultaneously oscillating the crystal through some 15°. This, as can readily be seen from the sphere of reflection, allows the possibility for more planes to diffract and permits more planes to pass through the Bragg angle of the stronger characteristic X-ray lines. Shorter X-ray exposures can be utilized if one measures the curved zero layer line close to the incident beam ($2\theta \le 90°$) to take advantage of the larger atomic scattering factor in this region.

Two oscillation patterns using continuous radiation can be made on a Weissenberg camera with exposure times ranging from one to five minutes at 35 kV. and 20 mA. One of the two exposures is made with the crystal oscillating 15° about the point at which ohe goniometer arc is normal to the incident X-radiation. The second exposure, taken with the film stage moved two or more centimetres, is made with the crystal oscillating about a point 180° different from the first. As in Winchell's note, the two zero layer lines appear curved (Fig. 1).

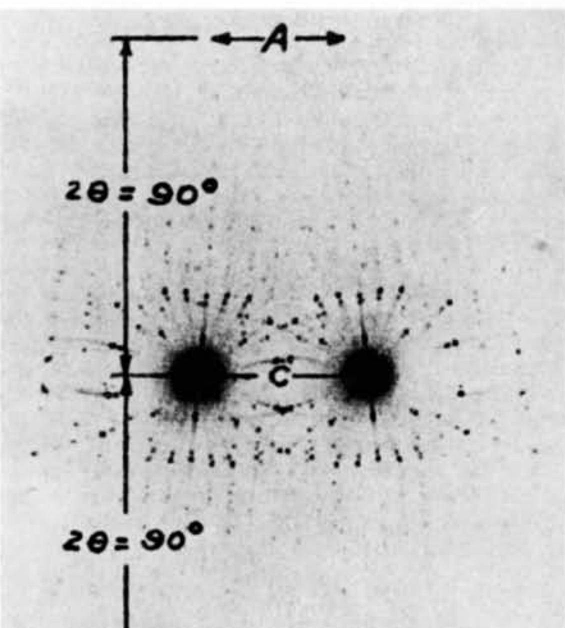

Fig. 1. Two 15° oscillation patterns of an unaligned single crystal taken on a Weissenberg camera utilizing continuous X-radiation.

Hendershot (1937) has calculated the distance these spots lie off the layer line in terms of the diffraction angle 2θ and the angle of inclination of the crystallographic axis to the rotation axis. The inclination angle may be produced by two rotations through angles i_1 and i_2; i_1 is the angle of rotation about the X-ray beam as an axis and in a plane perpendicular to the X-ray beam, and i_2 is the angle of rotation about the intersection of the X-ray beam with the axis of rotation in the plane determined by the incident beam and the axis of rotation. For small angles of inclination Hendershot showed that

$$a = Ri_1 \sin 2\theta,\ b = Ri_2 (1-\cos 2\theta)\,, \qquad (1)$$

where a and b are the displacements of the diffraction spot from the true equator by the angles of inclination i_1 and i_2 respectively. R is the radius of the camera, and θ is the Bragg angle. On a radian-diameter camera

$$a\ (\text{mm.}) = \tfrac{1}{2}i_1° \sin 2\theta,\ b\ (\text{mm.}) = \tfrac{1}{2}i_2° (1-\cos 2\theta)\,.$$

The total displacement of a diffraction spot is then the sum of the two rotations on one side of the incident direction and the difference on the other side. Translating this into separations A and B (Fig. 1), one easily obtains

$$i_2(1-\cos 2\theta)=\tfrac{1}{2}(A-B) \text{ and } i_1 \sin 2\theta=\tfrac{1}{2}(A+B)-C\,, \qquad (2)$$

where C is the distance the film was translated between the two exposures. For the Bragg angle $\theta = 45°$

$$i_2 = \tfrac{1}{2}(A-B) \quad \text{and} \quad i_1 = \tfrac{1}{2}(A+B)-C\,.$$

For shorter exposures smaller Bragg angles could be used with the appropriate substitution in equations (2).

It is apparent that only one of the two curves in the figure need be made. However, the necessity of drawing a line on the film through the locality of the undeviated zero layer line would require, in addition to the central spot, that the primary beam be allowed to strike one end of the film. The accuracy of this latter method would be less and the time consumed in making the measurements would be appreciably greater than that required for the two exposures as originally outlined.

References

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