Acta Cryst. (1952). **5**, 723

Two Aids for the Orientation of Crystals in a Precession Camera

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(*Received* 12 *May* 1952)

Crystals may be oriented in a precession camera by the aid of (*a*) an attached microscope, (*b*) Laue photographs. These supplement the methods described by Buerger.

Buerger (1944) describes the operation of a precession camera and gives two methods for orienting crystals. Two additional methods are described here.

The first method involves the viewing of the crystal in a direction normal to the X-ray beam. This may be accomplished by building a microscope on the other end of the beam which carries the goniometer head. Many crystals show cleavages or faces, the traces of which are parallel to the crystal axes. By setting these traces along a cross-wire in the microscope (the cross-wire being parallel to the X-ray beam) it is possible to align the crystal axis parallel to the X-ray beam. It is extremely difficult to do this with the commonly-used auto-collimator unless the crystal happens to have good reflecting faces. A suitable procedure for roughly aligning crystals is (*a*) adjust the desired axis normal to the axis of the goniometer head by the use of an optical goniometer or the auto-collimator; (*b*) then adjust the axis parallel to the X-ray beam by means of the microscope. By suitable design, the microscope need not interfere with the precession motion of the camera.

Occasionally, no morphological evidence of the position of the axes can be found. The alignment of an axis parallel to the X-ray beam must then be found by trial. If the Buerger small-angle precession method is used, it is not possible to interpret the resulting photograph if the error in the alignment is large. A more suitable method is to take a Laue photograph with the film normal to the X-ray beam. On the resulting photograph, the reflections from a zone normal to the desired crystal axis will lie on an ellipse which touches the direct X-ray beam. The method of formation of this ellipse is shown in Fig. 1. The incoming X-ray beam on the stereographic projection is denoted by X, the position of the axis by A, and the zone by $DEFG$. The X-ray reflections from planes in the zone will have directions given by the small circle $X'BC$. This small circle will project onto the flat photographic film as an ellipse $X''B'PC'$. The distance PX'' equals $r \tan 2\theta$, where r is the crystal-to-film distance and θ is the angle between the axis and the X-ray beam. The alignment of the axis is easily carried out by moving the axis along the diameter of the ellipse by an angular amount given by the above equation. The recognition of the ellipse is usually easy because the corresponding zone has low indices. The planes which would reflect at angles close to the reflections from the zone will therefore have high indices and low intensities. The zone may also be a symmetry zone which also augments its intensity with respect to the neighbouring reflections. By use of this method, crystals have been aligned when their axes were originally 20° from the X-ray beam. The theoretical limit is 30° with the usual size film holder. The angle of precession for orientation photographs is limited by the increasing overlap of the higher levels with the zero level according to the formula given by Buerger (1944, p. 26) and can often not exceed 10°.

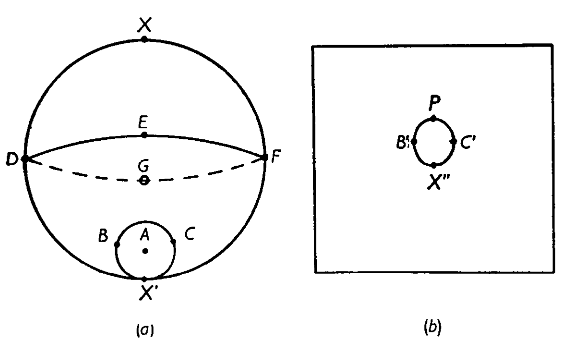

Fig. 1. Construction of the Laue photograph. (*a*) Stereographic projection. (*b*) Photographic film.

The procedure for aligning crystals is (*a*) take a small-angle precession photograph; if uninterpretable, (*b*) take a Laue photograph and correct the crystal orientation, and (*c*) take a small-angle precession photograph to obtain the final correction (this cannot be obtained from a Laue photograph as the ellipse becomes too small). If the error in the alignment of the axis is greater than 20°–30°, additional photographs are necessary with the crystal moved 30°–40° between the photographs.

Reference

Buerger, M. J. (1944). *The Photography of the Reciprocal Lattice*, ASXRED Monograph, No. 1.