

Table I

<i>R</i>	1/ <i>R</i> <sup>2</sup>	<i>I</i> (counts in 5 min.) for rekha parallel to		
		[100]	[010]	[110]
4.5	0.0494	98	133	139
3.0	0.111	149	236	235
2.0	0.25	257	434	460
1.5	0.444	401	719	720
1.25	0.64	590	—	—
Mean slope of the straight line		783	1493	1587
Background count		60	60	60

It is estimated that the accuracy of the elastic ratios is 5%. The compressibility of germanium is obtained by extrapolating the Bridgman (1949) values to zero pressure:

$$\beta = 12.79 \times 10^{-13} \text{ cm.}^2 \text{ dyne}^{-1}.$$

Since  $\beta = 3/(c_{11} + 2c_{12})$  we may combine this value with the ratios given above to obtain the values

$$c_{11} = 13.3, \quad c_{12} = 5.1, \quad c_{44} = 7.0 \times 10^{11} \text{ dyne cm.}^{-2}.$$

Bond *et al.* (1950) and McSkimin (1953), using an ultrasonic method, have also determined the elastic constants of germanium and the values obtained were as follows:

	<i>c</i> <sub>11</sub>	<i>c</i> <sub>12</sub>	<i>c</i> <sub>44</sub>
Bond, 1st crystal	12.92	4.79	6.70
2nd crystal	12.98	4.88	6.73
McSkimin	12.89	4.83	6.71

The elastic ratios obtained from these values agree very well with those obtained in the present investigation; however, the absolute values are about 4% higher. The values obtained in the present investigation, using a frequency of approximately 10<sup>11</sup>, do not differ significantly from those obtained by the previous workers at ultrasonic frequencies.

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## Precession Goniometry to Identify Neighboring Twins

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The two limiting circles which appear on a properly chosen orientation picture, taken with the precession camera, are used to identify the twin law in the case of neighboring twins.

Whenever the crystal lattice or one of its multiple lattices possesses pseudo-symmetry, the crystal may twin (twinning by pseudo-merohedry or by reticular pseudo-merohedry). If the pseudo-symmetry of the 'twin lattice' is pronounced and sufficiently high, several twin laws may lead to nearly identical orientations of the twinned individual. The resulting twins have been called 'neighboring twins' (*macles voisines*, Friedel, 1926). Because the relative orientation of one of the twinned crystals with respect to the other is known to morphologists only within the limits of error of optical goniometry, the identification of neigh-

boring twins may be a difficult problem, as is well illustrated by cryolite, staurolite, harmotome, morvenite, etc. During a study of staurolite twinning (Hurst, Donnay & Donnay, 1955), an X-ray procedure for such an identification was developed. It makes use of the precession camera (Buerger, 1944).

If reflection in (*hkl*) is a possible twin law, the twin is adjusted on the precession instrument so that the X-ray beam lies in (*hkl*). The zero level to be photographed then contains [*hkl*]\*. If (*hkl*) is the twin plane, [*hkl*]\*<sub>I</sub> and [*hkl*]\*<sub>II</sub> coincide so that [*hkl*]\* shows as a single row of reflections. (The subscripts I and II

Table 1. *Angles between nearly parallel rows in the two individuals of a 60° staurolite cross*Cell dimensions used in calculations:  $a=7.86_{\text{a}}$ ,  $b=16.61_{\text{b}}$ ,  $c=5.67_{\text{c}}$ ,  $\text{Å} \pm 0.3\%$ .

Twin law $[u_i v_i w_i]$	$j=1$	$j=2$	$j=3$	$j=4$	
	$\varrho_j = \langle 102 \rangle_{\text{I}} : \langle 102 \rangle_{\text{II}}$	$\varrho_j = \langle 320 \rangle_{\text{I}} : \langle 320 \rangle_{\text{II}}$	$\varrho_j = \langle 013 \rangle_{\text{I}} : \langle 013 \rangle_{\text{II}}$	$\varrho_j = \langle 313 \rangle_{\text{I}} : \langle 313 \rangle_{\text{II}}$	
$i=1; [102]_{120^\circ}$	$[102]_{\text{I}} : [102]_{\text{II}}$ 0°	$[\bar{3}20]_{\text{I}} : [320]_{\text{II}}$ 33' (20'-44')	$[013]_{\text{I}} : [0\bar{1}3]_{\text{II}}$ 28' (17'-38')	$[3\bar{1}3]_{\text{I}} : [313]_{\text{II}}$ 20' (12'-26')	
$i=2; [320]_{120^\circ}$	$[\bar{1}0\bar{2}]_{\text{I}} : [\bar{1}02]_{\text{II}}$ 1° 2' (51'-1° 13')	$[320]_{\text{I}} : [320]_{\text{II}}$ 0°	$[0\bar{1}3]_{\text{I}} : [013]_{\text{II}}$ 54' (44'-1° 7')	$[\bar{3}13]_{\text{I}} : [\bar{3}\bar{1}3]_{\text{II}}$ 38' (31'-45')	$[\bar{3}13]_{\text{I}} : [\bar{3}\bar{1}3]_{\text{II}}$ 1° 11' (1° 0'-1° 34')
$i=3; [013]_{90^\circ}$	$[102]_{\text{I}} : [\bar{1}02]_{\text{II}}$ 21' (12'-30')	$[\bar{3}20]_{\text{I}} : [\bar{3}\bar{2}0]_{\text{II}}$ 22' (14'-39')	$[013]_{\text{I}} : [013]_{\text{II}}$ 0°	$[3\bar{1}3]_{\text{I}} : [\bar{3}\bar{1}3]_{\text{II}}$ 26' (16'-37')	
$i=4; [313]_{180^\circ}$	$[10\bar{2}]_{\text{I}} : [\bar{1}02]_{\text{II}}$ 1° 55' (1° 22'-2° 29')	$[\bar{3}20]_{\text{I}} : [\bar{3}\bar{2}0]_{\text{II}}$ 27' (7'-1°)	$[0\bar{1}3]_{\text{I}} : [0\bar{1}3]_{\text{II}}$ 2° 3.5' (2° 3'-2° 4')	$[313]_{\text{I}} : [313]_{\text{II}}$ 0°	$[\bar{3}\bar{1}3]_{\text{I}} : [\bar{3}\bar{1}3]_{\text{II}}$ 55' (14'-1° 36')

The  $\varrho$  angles of column 1 do not overlap so that crystal plates were cut normal to  $[102]_{\text{I}}$ .

refer to the two individuals of the twin.) Because of the presence of a center of symmetry, added by X-rays if not present in the structure,  $[hkl]^*$  is also a twofold twin axis.† It follows that  $[hkl]^*$  is a symmetry line of the photograph as far as positions, although not necessarily intensities, of reflections are concerned. Thus a twin plane can readily be recognized; only twin axes will be considered henceforth.

Let the  $n$  possible twin axes be designated  $[u_i v_i w_i]$ ,  $i = 1, 2, \dots, n$ . The 60° cross of staurolite with possible twin axes  $[102]_{120^\circ}$ ,  $[320]_{120^\circ}$ ,  $[013]_{90^\circ}$ , and  $[313]_{180^\circ}$  may be taken as an example. If  $[u_1 v_1 w_1]$  is the twin axis, the reciprocal-lattice planes  $(u_1 v_1 w_1)_{\text{I}}^*$  and  $(u_1 v_1 w_1)_{\text{II}}^*$  coincide. If one of the other rows  $[u_i v_i w_i]$ ,  $i \neq 1$ , is the twin axis, the plane  $(u_1 v_1 w_1)_{\text{I}}^*$  nearly coincides with another plane of the form  $\{u_1 v_1 w_1\}_{\text{II}}^*$ , say  $(\bar{u}_1 v_1 w_1)_{\text{II}}^*$ . The angle between these two planes is equal to the angle  $\varrho_1 = [u_1 v_1 w_1]_{\text{I}} : [\bar{u}_1 v_1 w_1]_{\text{II}}$  between the corresponding direct-lattice rows. The fact that this angle can be measured with high accuracy on a precession photograph  $(u_1 v_1 w_1)_{\text{I}}^*$  is the basis of the method. If  $[u_1 v_1 w_1]_{\text{I}}$  is made the vertical axis of the sphere of projection, the angular co-ordinates of  $[\bar{u}_1 v_1 w_1]_{\text{II}}$  can be designated by  $\varphi_1, \varrho_1$ , where  $\varphi_1$  is referred to some convenient zero meridian.

It is advisable, although not imperative, to prepare the following angle table before beginning the experimental procedure. From measured cell dimensions the angles  $\varrho_{ij}$  are computed for each angle  $\varrho_j$ ,  $j = 1, 2, \dots, n$ , and each twin law  $[u_i v_i w_i]$ . Angle  $\varrho_{ij}$  is zero, of course, when  $i = j$ . The permitted angular range due to the uncertainty in cell dimensions is calculated. Table 1 shows the angle table for the 60° cross of staurolite. Inspection of the table shows which  $\varrho_j$  angle gives no overlap or the smallest range of overlap for the different twin axes. If  $\varrho_1$ , say, is the most favorable angle, the plane  $(u_1 v_1 w_1)_{\text{I}}^*$  is the one to be examined.

† It is necessary to specify the order of the twin axis or the angle of the twin rotation, as it is known (Friedel, 1904) that rotations through angles other than 180° may be twin operations. Not all twin axes are 'axes of hemitropy'! (See following paragraph:  $[102]_{120^\circ}$  is a threefold twin axis, etc.)

In the case of the staurolite 60° cross (Table 1) the plate should be cut perpendicularly to one of the  $\langle 102 \rangle_{\text{I}}$  rows, namely the one that is near a  $\langle 102 \rangle_{\text{II}}$  row. A measured  $\varrho$  angle of about 2° would indicate twin law  $[313]_{180^\circ}$ . The  $\varphi$  angle is then determined graphically on a stereographic net and measured on the photograph (Fig. 1) as a check.

If only microscopic specimens are available, one of them is mounted with  $[u_1 v_1 w_1]$  along the X-ray beam. From a sufficiently large specimen, a plane-parallel plate is ground roughly normal to  $[u_1 v_1 w_1]_{\text{I}}$  by the standard mineralogical technique of preparing oriented sections. The plane of grinding may be chosen by visual inspection; it is not critical, nor is the plate thickness, whose optimum value  $1/\mu_i$  is the reciprocal of the linear absorption coefficient. The plate is mounted normal to the beam, the twin boundary is centered in the pinhole, and a 10-minute orientation picture is taken (unfiltered radiation, no layer-line screen, precession angle  $\mu = 10^\circ$  or  $15^\circ$ ). The advantage of the plate over a specimen bathed in the beam (Donnay & Donnay, 1954) lies in the fact that it enhances the intensity of the white-radiation streaks.

If  $[u_1 v_1 w_1]$  is the twin axis, only one limiting circle (given by the intense cut-offs of the white-radiation streaks) appears, even if the adjustment is not perfect. This finding may be confirmed by taking a zero-level picture (filtered radiation, layer-line screen,  $\mu=30^\circ$ ), which should be indistinguishable from that of a single crystal. We found this to be so perfectly the case that using the plate we even doubted the presence of two individuals in the beam. Such doubts can be forestalled by letting first one individual diffract, and then without changing the film moving the plate parallel with itself to irradiate the other individual.

If  $[u_1 v_1 w_1]$  is not the twin axis, two distinct limiting circles are visible (Fig. 1). One of them should be accurately centered on the film, by making the usual arc adjustments, before the final picture is taken. To have the two limiting circles stand out well on that photograph, one selects a  $\mu$  angle such that some radial streaks will be cut off a few millimeters beyond strong reflections. (The radius of the limiting circle is  $2F \sin \mu$ ,

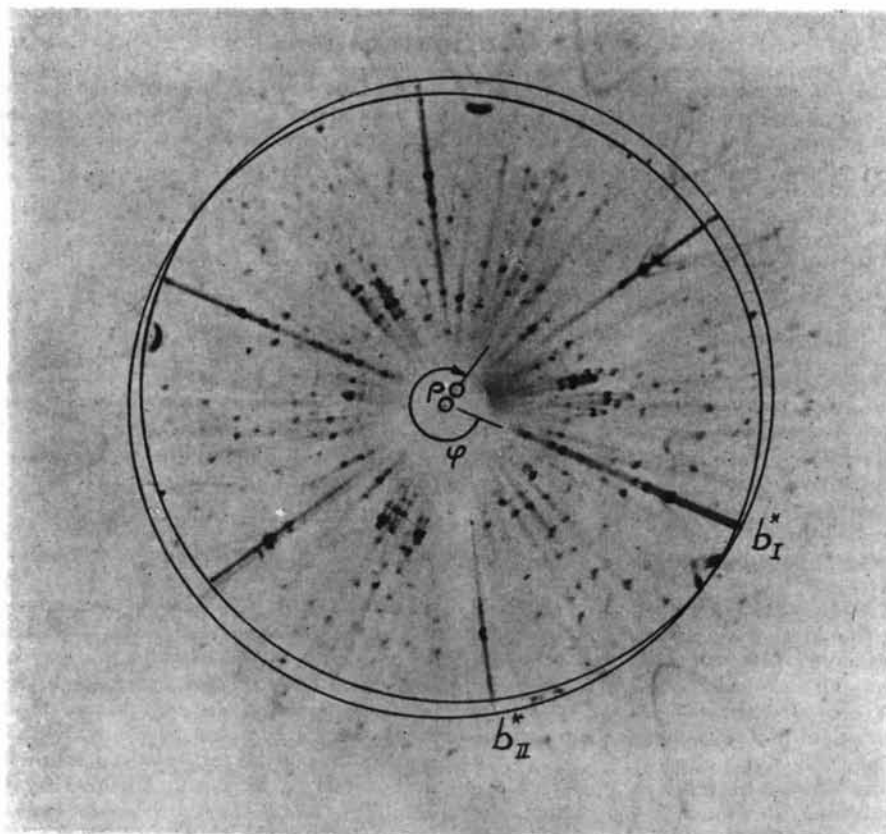


Fig. 1. Orientation film ( $\mu = 20^\circ$ , Mo K,  $\frac{1}{2}$  hr.) of staurolite plate cut normal to  $[10\bar{2}]_I$ . Limiting circles of net planes  $(10\bar{2})_I^*$  and  $(\bar{1}02)_{II}^*$  are drawn in. Angles  $\rho$  and  $\varphi$  indicate the twin law  $[313]_{180^\circ}$ .

where  $F$  is the crystal-to-film distance.) In the case of the plate, the exposure time is increased to half an hour when unfiltered molybdenum radiation (45 kV., 15 mA.) is used. For the small specimens it may have to be increased to several hours. To locate a limiting circle, draw it to scale on transparent paper, mark its center with a pin point, move it over the film until one family of radial streak ends on the circumference of the circle, and then prick the center through. If film shrinkage is not uniform, the streaks will terminate on an ellipse instead of a circle. By letting opposite ends of each diametral streak terminate at equal distances from the superposed circle, the center of the ellipse can be located precisely. The operation is performed for both families of streaks. The distance between the two centers thus found gives the angle  $\rho_1 = [u_1v_1w_1]_I : [u_1v_1w_1]_{II}$  to about  $\pm 3'$ . We checked this accuracy on three photographs of the same twin, taken with the same setting and  $\mu$  angles of  $15^\circ$ ,  $20^\circ$  and  $25^\circ$ . The azimuth  $\varphi$  of the line that connects the two centers on the film is also measured from the appropriate zero meridian. If the centers lie close together, this azimuth is poorly defined. The azimuth of the chord common to the two circles, which is normal to the desired direction, is determined instead. Comparison of the measured angles with the predicted ones leads to the twin law.

The result can be checked by reorienting the specimen so that the twin axis lies along the X-ray beam and by taking a zero level as described above. The plate will no longer be mounted normal to the beam, and the exposure time will have to be increased. Even in the worst possible case, however, where the beam lies in the plane of the plate, usable films can be obtained in less than an hour.

In this method the precession camera is used as a goniometer. In contradistinction to the optical goniometer, which gives angles between face normals or reciprocal-lattice rows, the 'precession goniometer' measures angles between direct-lattice rows.

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