

Acta Cryst. (1950). 3, 396

Crystal orientation on the Weissenberg goniometer. By HORACE WINCHELL, *Department of Geology, Brush Mineralogical Laboratory, Yale University, New Haven, Conn., U.S.A.*

(Received 20 February 1950)

Setting a crystal zone axis accurately on a goniometer head is sometimes difficult because of the lack of crystal faces or cleavage surfaces on the specimen. There are several ways

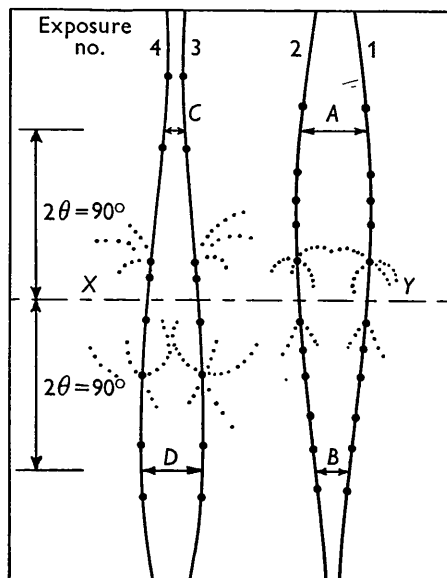


Fig. 1. Parts of four Laue patterns recorded on cylindrical film in a Weissenberg camera. The crystal was rotated 180° between exposures 1 and 2, and between 3 and 4, but only -90° between 2 and 3. The film was translated a short distance, Weissenberg fashion, between successive exposures. For radian-diameter camera, the angular corrections to the adjusting arcs of the goniometer head are $\frac{1}{2}(A-B)$ and $\frac{1}{2}(C-D)$, respectively. A , B , C and D are measured in millimeters, and corrections in degrees.

to adjust crystals by X-ray methods; each method is in general suited best for a particular apparatus. Bunn (1945, pp. 173-5) described the use of a standard oscillation camera to measure the inclination of the zero-layer line at

Acta Cryst. (1950). 3, 396

Silicon carbide of 594 layers. By GORO HONJO, SHIZUO MIYAKE and TAKANORI TOMITA, *Tokyo Institute of Technology, Oh-Okayama, Meguroku, Tokyo, Japan*

(Received 2 May 1950)

A number of modifications of the crystal structure of silicon carbide, SiC, have been reported, the difference between which, as is well known, arises from various sequences of the basic layers of three species A , B and C . The number of the layers stacked within one lattice period in the direction of the hexagonal c axis of these modifications are respectively 3, 4, 6, 15, 21, 33, 51, 87 (Jagodzinski, 1949) and about 230 (Zhudanov & Minervina, 1947). The crystal is also of special interest in respect to the stacking disorder which results in continuous distributions on the reciprocal-lattice rods parallel

the point where the primary beam strikes the film, so as to derive the corrections to be applied to each of the arcs of the goniometer adjusting head. In making analogous adjustments of a crystal for a Weissenberg instrument, the modification of Bunn's method here described has been found rapid and accurate.

Several Laue patterns can be recorded on different parts of a radian-diameter Weissenberg film with short exposures (e.g. 5 min. at 50 kV., 20 mA.) by simply moving the film carriage 2 or 3 cm. between successive exposures. Two such exposures are made with the plane of one of the goniometer adjusting arcs normal to the X-ray beam, the crystal-rotation axis being turned 180° between the two. The difference ($A-B$) in the separations A and B (Fig. 1) between the 'zero-layer' Laue zone lines at the positions corresponding to $2\theta = \pm 90^\circ$, is then proportional to the angular correction to be applied to the particular adjusting arc. Two additional exposures with the second adjusting arc normal to the X-ray beam provide similar data ($C-D$) for its correction. If four exposures are made as just described, the film may be measured with a pair of dividers immediately after development, without waiting for rinsing, fixing, washing and drying.

The exposure time can be reduced by one-half by eliminating the second exposure for each goniometer arc; but then the film must be processed completely and dried before the necessary construction line XY can be drawn accurately enough on it to determine the angular deviation (Bunn's method) of the 'zero-layer' Laue zone lines from perpendicularity thereto. If this construction is used, the primary beam must be allowed to strike the film for a second near each end of the film traverse. The few minutes saved in exposure time will then probably be more than offset by the additional time for processing the film and reading it.

Reference

BUNN, C. W., (1945). *Chemical Crystallography*. Oxford: Clarendon Press.

to the hexagonal c axis for which $h+2k=0 \pmod{3}$ (Jagodzinski, 1949; Honjo, 1949).

In the course of X-ray study on the single crystals of this substance we recently found a sample which is distinguished by the fact that it contains partly a new modification having an extraordinarily long period, the other part of it being composed of the structure with 6-layer period (SiC(6)). Fig. 1 shows an oscillation photograph taken by oscillating the crystal through 5° around the c axis so as to record specifically the $(10\bar{1}l)$ rod for which $h+2k=1$. It looks at a first glance as if the pattern

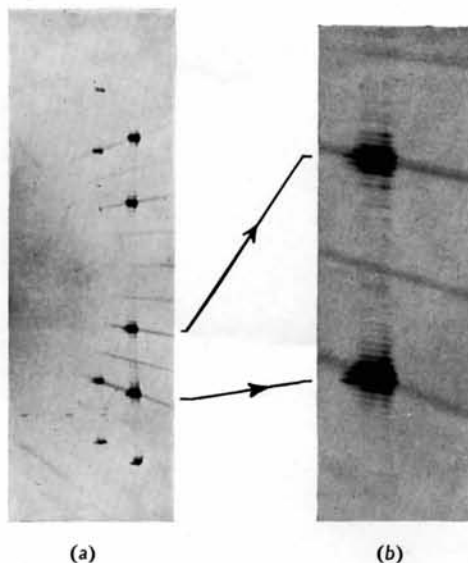


Fig. 1. Oscillation photograph about the c axis of the new modification of SiC. (a) $R=8$ cm., Cu target; (b) showing the detail of (a).

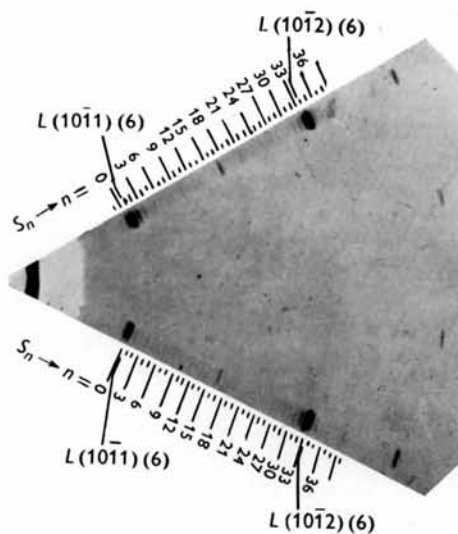


Fig. 2. Laue photograph taken on cylindrical film ($R=8$ cm.) with Cu target. X-ray beam nearly parallel to c axis, $[1120]$ parallel to cylindrical axis.

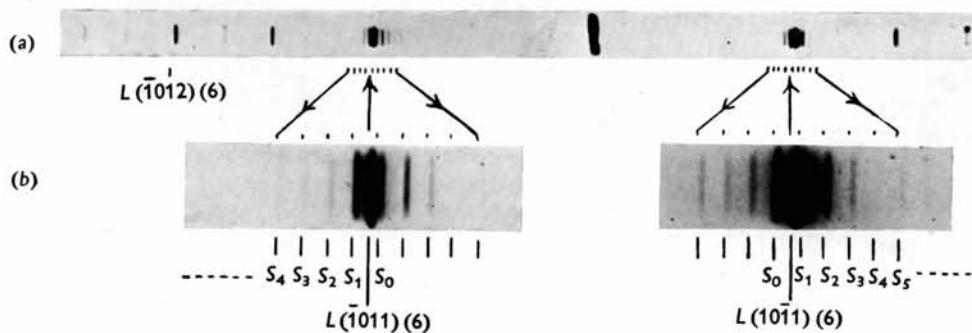


Fig. 3. (a) Laue photograph taken on cylindrical film ($R=8$ cm.) with Cu target, X-ray beam parallel to c axis, $[1100]$ parallel to cylindrical axis; (b) showing the detail of (a).

consists of the spots due to the crystal of 6-layer structure and the continuous streak connecting these spots. It is found, however, that the streak is actually composed of a number of spots, due to the new modification, separated from each other by an equal short distance, about 0.3 mm. on the film taken in a camera of radius 8 cm. with Cu $K\alpha$ radiation, and we can enumerate 33 spots within the distance which corresponds to the c period of SiC(6). This observation shows that the reciprocal-lattice points of the new modification are distributed on the $(10\bar{1}l)$ rod with an interval $\frac{1}{3}$ of that for SiC(6), indicating a lattice period along the c axis corresponding to at least $6 \times 33 = 198$ layers.

The fine structure of the reciprocal-lattice rod, as found above for the $(10\bar{1}l)$ rod, can also be confirmed by observations on Laue photographs, on which the Laue lines due to SiC(6) as well as those due to the new modification appear simultaneously. Figs. 2 and 3 (a) show such Laue patterns taken on cylindrical films ($R = 8$ cm.) with the incident X-ray beams nearly parallel to the c axis and perpendicular to the $[11\bar{2}0]$ and $[1\bar{1}00]$ axes respectively, the latter axes being set parallel to the axis of the cylindrical films. Besides the intense Laue lines belonging to SiC(6) (denoted by $L(10\bar{1}1)(6)$, $L(10\bar{1}2)(6)$, etc.), there are regular line groups with moderate intensities which correspond to the new modification (denoted by s_n , $n = 0, 1, 2, \dots$). We found just 33 lines of this kind between two Laue lines of SiC(6), say $L(10\bar{1}1)(6)$ and $L(10\bar{1}2)(6)$. The appearance of these s_n lines is explained on assuming for these reciprocal-lattice rods the same structure as deduced from the oscillation photograph, with due regard

to the effect of higher harmonics. The Laue photographs, however, reveal more clearly that none of the reciprocal points of the new modification coincides with those of SiC(6) which have hexagonal symmetry. As is obvious from Fig. 3 (b), the reciprocal points of the latter structure are situated at a position one-third the way between the nearest two reciprocal-lattice points of the new modification. This one-third deviation is not a result of considerations arising from some trivial origins, since, as we have confirmed, it takes place for six rods $(10\bar{1}l)$, $(1\bar{1}0l)$, $(0\bar{1}1l)$, $(\bar{1}01l)$, $(\bar{1}10l)$ and $(01\bar{1}l)$ in common, but by changing the sense alternately, so that the distribution of the reciprocal points, as a whole, manifests the rhombohedral symmetry of the structure. The ultimate c period of the new modification, therefore, does not correspond to the above-mentioned $6 \times 33 = 198$ layers, but the triple of it, that is to say, $3 \times 6 \times 33 = 594$ layers. The factor 3, together with the one-third deviation of s_n -line group relative to SiC(6) Laue lines, is understood by the extinction rule of reflexions for the rhombohedral lattice.

The lattice period along the hexagonal c axis of the new modification is $2.51 \times 594 \approx 1500$ Å. The range of the ordering force for layers in this structure, therefore, should be at least $2.51 \times 594 \div 3 \approx 500$ Å., which cannot be understood in terms of the conventional type of force between layers.

References

- HONJO, G. (1949). *J. Phys. Soc. Japan*, **4**, 352.
 JAGODZINSKI, H. (1949). *Acta Cryst.* **2**, 298.
 ZHUDANOV, G. S. & MINERVINA, Z. V. (1949). *J. Exp. Theor. Phys.* **17**, 3.

Acta Cryst. (1950). **3**, 397

Largest likely values for the reliability index. By A. J. C. WILSON, *Viriamu Jones Laboratory, University College, Cardiff, Wales*

(Received 11 May 1950)

The reliability index

$$R \equiv (\sum |F_{\text{obs.}}| - |F_{\text{alc.}}|) / (\sum |F_{\text{obs.}}|)$$

is widely used as a test of the quality of a structure determination. Values of R for trial structures are not often published, but it would seem that a structure giving R in the range 0.3–0.5 is accepted as showing promise of refinement. Dunitz (1949) gives 0.37 for his trial structure for 1, 2, 3, 4-tetraphenylcyclobutane. Completed structure determinations usually have R less than 0.25, though higher values have been reported; Fenimore (1948) gives $R = 0.31$ – 0.36 and Geller & Hoard (1950) give 0.32. It seems of some interest to find the probable value of R for an entirely wrong structure, that is, one with the same symmetry as the correct structure, but with an unrelated arrangement of the same atoms.

Let F_1, F_2 be the values of $|F_{hkl}|$ for the two structures, X be $|F_1 - F_2|$, and M be the number of reflexions on which R is to be based. Then

$$R = M \langle X \rangle / M \langle |F| \rangle \\ = \langle X \rangle / \langle |F| \rangle, \quad (1)$$

where the angle brackets indicate average values. Since F_1 and F_2 are due to structures with the same symmetry and atoms, the probability, $P(F) dF$, that F lies between F and $F + dF$ is the same for both F_1 and F_2 (Wilson, 1949,

1950). A particular value of X can arise in two ways: (i) a value of F_1 may be paired with the value $F_2 = F_1 + X$, and (ii) a value of $F_1 > X$ may be paired with the value $F_2 = F_1 - X$. The probability, $Q(X) dX$, that X lies between X and $X + dX$ is therefore given by

$$Q(X) = \int_0^\infty P(F) P(F+X) dF + \int_X^\infty P(F) P(F-X) dF \\ = 2 \int_0^\infty P(F) P(F+X) dF. \quad (2)$$

Since $Q(X)$ is a probability-distribution function, a partial check of (2) is obtained by showing that the integral of $Q(X)$ from 0 to ∞ is unity, whatever the form of $P(F)$. For conciseness of expression in this and other manipulations below it is convenient first to introduce the functions $N(F)$ and $G(F)$ defined by the equations

$$\left. \begin{aligned} N(F) &\equiv \int_0^F P(F) dF, \\ dN(F) &= P(F) dF, \\ N(\infty) &= 1, \end{aligned} \right\} \quad (3)$$

$$\left. \begin{aligned} G(F) &\equiv \int_0^F FP(F) dF, \\ dG(F) &= FP(F) dF, \\ G(\infty) &= \langle |F|^2 \rangle. \end{aligned} \right\} \quad (4)$$