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X-ray Diffraction Pattern of a Strained Rock-Salt Crystal

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Laue photographs were taken with a rock-salt crystal under compressing force applied to one of the natural cleavage surfaces and in the direction perpendicular to the incident X-ray beam. As the compressing load was increased step by step up to a certain value the spots on the photograph were lengthened into nearly concentric arcs which had not been observed in previous experiments. When the load reached a higher value broadening of the spots in the radial direction also occurred. These phenomena are interpreted as due to two stages of deformation of the crystal. The first stage is considered as a simple bending of the crystal about a fixed axis, which is found to be nearly perpendicular to the largest surface of the crystal. The second stage of deformation is complicated and has not been explained in detail; it may consist in bendings about axes parallel to the largest surface or inclined at small angles to it. The deformation was found to be of a plastic nature.

Introduction

It is known that the spots of a Laue photograph taken with a single crystal sufficiently strained appear to be lengthened; such a phenomenon has been called asterism.

The asterism of the rock-salt crystal was first studied by Joffé & Kirpitcheva (1922); they found that all the Laue spots, except those produced by the (110) plane, were elongated radially, and the elongated spots consisted of several separated spots. Joffé & Kirpitcheva explained that when a crystal is under strain there is slipping of the crystallographic planes, and this causes the bending of the planes about an axis in the direction of slipping. Later, Manteuffel (1931) took Laue photographs of a rock-salt crystal under different straining loads and found that, at a certain value of the load, the spots split into two, and, as the load was increased, into several component spots; on further increase of the load, the spots became diffuse and spread out in two directions. Manteuffel suggested that the separation of the Laue spots into several component spots indicates the existence of a crystal mosaic in the optically homogeneous crystal.

Yamaguchi (1929), in explaining the asterism of Laue photographs, treated the bending of a crystal as equivalent to a rotation, and pointed out that (1) if the axis of rotation is coincident with the incident X-ray beam, the loci of the Laue spots are concentric arcs around the central spot; (2) if the axis of rotation is perpendicular to the incident X-ray beam, the loci of the Laue spots resemble lemniscates; and (3) if the axis of rotation is normal to a crystallographic plane, the Laue spot of that plane is not disturbed.

The photographs published by Joffé & Kirpitcheva and by Manteuffel were, however, only portions of Laue photographs, and therefore did not seem to give the complete presentation of the crystal deformation. It is the purpose of the present investigation to make a further experimental study of the rock-salt crystal with the method similar to Manteuffel's and to give interpretations of the results on the basis of Yamaguchi's theoretical consideration.

Method for determining the axis of bending

Yamaguchi's treatment of the bending of a crystal as equivalent to a rotation does not involve much error if the incident X-ray beam is narrow and its path in the crystal is short. A slight modification and extension of his treatment will give a method for determining the axis of bending of a strained crystal. Calculations are simplified by using the gnomonic projection of a Laue spot instead of the spot itself. From Text-fig. 1, one can immediately write

$$\cos\beta = \cos\alpha\sin\theta + \sin\alpha\cos\theta\cos\eta, \quad (1)$$

$$\sin\theta = \cos\alpha\cos\beta + \sin\alpha\sin\beta\cos\psi, \quad (2)$$

$$R = L\cot\theta. \quad (3)$$

Now if the crystallographic plane MN rotates through a small angle $d\psi$ about the axis OA, η and θ are changed, while α and β remain the same. Differentiation of (1) and (2) and simplification of the results give respectively

$$\frac{d\eta}{d\psi} = \frac{\cos\alpha\cos\theta - \sin\alpha\cos\eta\sin\theta}{\sin\alpha\sin\eta\cos\theta}\frac{d\theta}{d\psi}, \quad (4)$$

and

$$\frac{d\theta}{d\psi} = -\frac{\sin\alpha\sin\beta\sin\psi}{\cos\theta}. \quad (5)$$

Substitution of (5) in (4) gives

$$\frac{d\eta}{d\psi} = \left[\frac{\sin\alpha\sin\theta\cot\eta}{\cos^2\theta} - \frac{\cos\alpha}{\cos\theta\sin\eta}\right]\sin\beta\sin\psi. \quad (6)$$

Differentiation of (3) and substitution of (5) in the result give

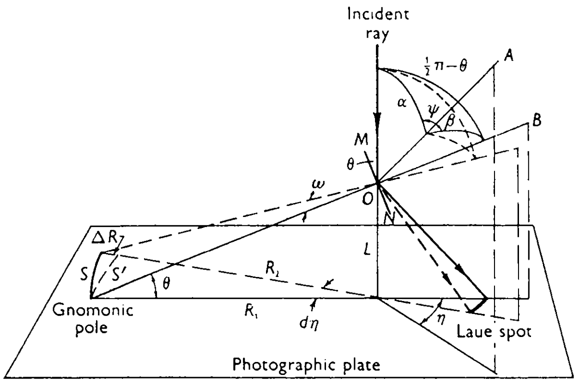
$$\frac{dR}{d\psi} = L\frac{\sin\alpha\sin\beta\sin\psi}{\cos\theta\sin^2\theta}. \quad (7)$$

Dividing (6) by (7), one gets

$$\frac{d\eta}{dR}=\frac{1}{L}\left[\frac{\sin^3\theta\cot\eta}{\cos\theta}-\frac{\sin^2\theta\cot\alpha}{\sin\eta}\right]. \quad (8)$$

Equations (6) and (7) give the change of position of the gnomonic projection of a Laue spot when the crystallographic plane rotates through a small angle $d\psi$ about an axis OA which makes an angle α with the incident X-ray beam as shown in Text-fig. 1. To calculate the direction of the axis of rotation, one solves (8) for α, thus

$$\cot\alpha=\tan\theta\cos\eta-\frac{L\sin\eta}{\sin^2\theta}\frac{d\eta}{dR}. \quad (9)$$



Text-fig. 1. Relations of various quantities in the treatment of the bending of a crystallographic plane as a rotation about a fixed axis. MN is the trace of the reflecting plane in the reference plane BOL.

Now, when a Laue spot is elongated owing to the bending of the crystallographic plane, which can be treated approximately as rotation as suggested by Yamaguchi, its gnomonic projection is also elongated into a strip. From the symmetrical property of the appearance of the elongated spots, one knows the plane in which the axis of bending lies; thereby the angle η in (9) for each spot is known. θ is determined from the Miller indices of the spot; thus, $\tan\theta=L/R$ and $\sin^2\theta=L^2/(L^2+R^2)$. Again, $d\eta/dR$ can be put as $S'/(R\Delta R)$, where S' and ΔR are the lengths indicated in Text-fig. 1. With these considerations, (9) can be put as

$$\cot\alpha=\frac{L}{R}\cos\eta-\left(\frac{L}{R}+\frac{R}{L}\right)\frac{S'}{\Delta R}\sin\eta, \quad (10)$$

from which α can be calculated.

Experimental

A crystal-compressing apparatus similar to that used by Manteuffel was constructed. The rock-salt crystal used in this experiment had the dimensions

$$0{\cdot}671\times0{\cdot}507\times0{\cdot}173\text{ cm.}$$

The compressing load was applied to one of the natural cleavage surfaces, the surface 0·507 × 0·173 cm., and was in the direction perpendicular to the incident X-ray beam, which fell on the largest surface, the surface 0·671 × 0·507 cm., as nearly perpendicularly as the crystal could be set.

Laue photographs were taken at 5 cm. behind the crystal for different compressing loads increasing step by step up to 2·44 kg.mm.$^{-2}$ The value of the compressing load expressed in kg.mm.$^{-2}$ was taken as the total weight applied on the crystal divided by the area of that portion of the crystal surface which was in contact with the compressing rod of the apparatus.

Results and interpretation

Out of the twelve photographs taken, two are shown in Plate 5, figs. 1 and 2.

General nature of the crystal deformation. When a compressing load of 0·51 kg.mm.$^{-2}$ was applied, no elongation of the spots appeared. When the load was increased to 1·15 kg.mm.$^{-2}$, the spots were lengthened (Plate 5, fig. 1) into nearly circular arcs, except those at one corner of the photograph, which were somewhat shortened. This type of elongation had not been observed previously. Further increase of the load, step by step, gave at first further lengthening of the spots, but later the lengthening was very slight. Splitting of the spots into components appeared at 1·46 kg.mm.$^{-2}$ Radial broadening occurred at 2·13 kg.mm.$^{-2}$ With further increase of the load, the spots were further broadened into triangular shape (Plate 5, fig. 2).

From the facts just mentioned, it is clear that the crystal deforms non-uniformly with the increasing compressing force; a certain type of deformation takes place suddenly when the load reaches a certain value, and further increase of the load does not give much further yield of this type. The crystal in the present experiment evidently underwent two stages of deformation which set in at different loads.

To give a quantitative representation of the crystal deformation the angle subtended by the gnomonic projection strip at the crystal was calculated for three different crystallographic planes, and plotted against the compressing load (Text-fig. 2). It can be easily shown that the angle is given by

$$\cos\omega=\frac{2L^2+R_1^2+R_2^2-S^2}{2(L^2+R_1^2)^{\frac{1}{2}}(L^2+R_2^2)^{\frac{1}{2}}}, \quad (11)$$

and that it represents approximately the amount of bending of the crystallographic plane within the width of the X-ray beam.

In making the gnomonic projection for this purpose, and for the determination of the axis of bending to be presented later, two sharp points were marked on each Laue spot at a short distance from the ends of the spot equal to half the natural length of the spot, which was found from a photograph taken with the same crystal before the compressing load was applied. Thus, the distance between the two marked points is the actual elongation of the spot. Then the gnomonic projections of these two points were made. The strip between these projected points is marked S in Text-fig. 1.

In order to test whether the deformation is plastic or elastic, the crystal was allowed to stand under the same

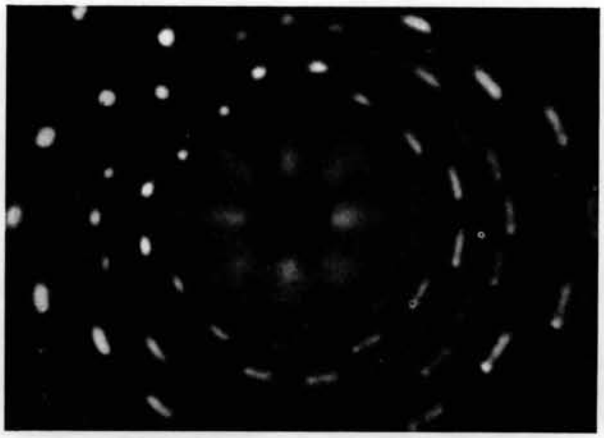


Fig. 1. Laue photograph of the rock-salt crystal under a compressing load of 1·15 kg.mm.$^{-2}$

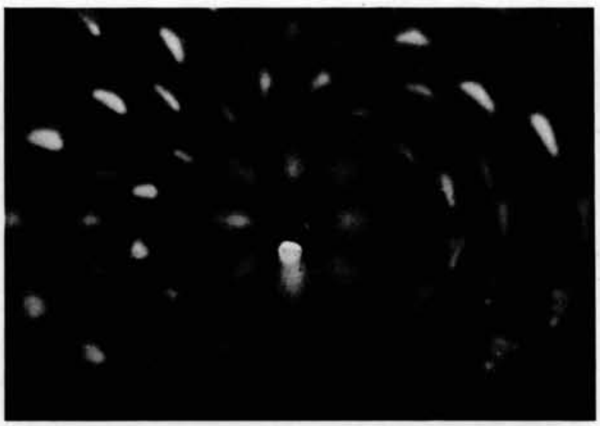


Fig. 2. Laue photograph of the rock-salt crystal under a compressing load of 2·44 kg.mm.$^{-2}$

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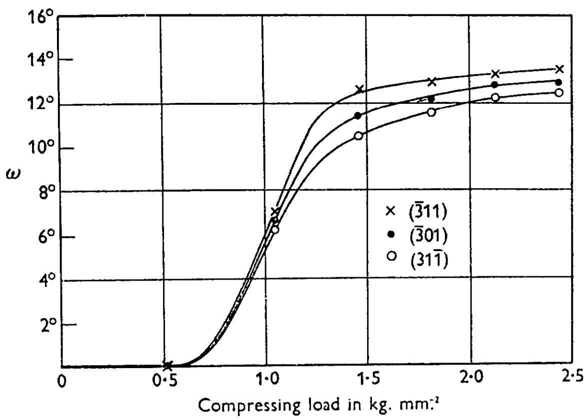
load for 9 hr. after the photograph in Plate 5, fig. 1 was taken; after this lapse of time, another photograph was taken. Then the load was removed; immediately one more photograph was taken, and after 5 hr. without load on the crystal another was taken. These four photographs did not show the slightest difference. It is therefore quite certain that the deformation of the crystal in this experiment under compression was of a plastic nature.

The splitting of the Laue spots into several components can be explained as due to the fact that when the crystal has been bent or otherwise distorted to such an extent that it can no longer stand the strain, fracture takes place, and it breaks into small crystals with slightly different orientations, but mechanically still connected.



Text-fig. 2. Variation of ω, the angle subtended by the gnomonic projection strip at the crystal, with the compressing load.

Determination of the axis of bending. On the basis of Yamaguchi's theory, one can see from the spreading of the spots that the first stage of deformation must be a bending of the reflecting planes about an axis nearly parallel to the incident X-ray beam. Again, the symmetrical appearance of the photograph about one of its diagonals indicates that the axis must lie in the (110) plane of the crystal. Thus, after the gnomonic projection of a photograph is carefully made, all the values in (10), except α, the angle between the axis of bending and the incident X-ray beam, are known for each Laue spot; α then can be calculated. Table 1 is the result obtained from the photograph in Plate 5, fig. 1.

The values of α in Table 1, which are calculated from widely distributed spots corresponding to differently oriented planes, agree very closely. This shows that the whole crystal has undergone a simple bending about a fixed axis, which, as found by calculation, is inclined at a small angle to the incident X-ray beam. This conclusion may be expected from the appearance of the photograph on the basis of Yamaguchi's theory. As the incident X-ray beam is almost perpendicular to the (001) plane it can safely be said that the axis of bending for the first stage of deformation observed in this experiment is nearly perpendicular to the (001) plane of the crystal. One can then imagine that all the crystallographic planes parallel to this axis bend into cylindrical surfaces; all other planes, except those perpendicular to this axis, must also be curved, and the smaller the inclination to this axis, the greater is the curvature.

Attempts can then be made to explain the shortening of the Laue spots at the upper left corner of the photograph shown in Plate 5, fig. 1. Let it be imagined that the crystal is bent about the axis just mentioned in such a way that the (1$\bar{1}$0) plane, say, forms a cylindrical surface with its concave side toward the upper left-hand corner of the photograph. As all the planes responsible for the shortened spots are inclined at small angles to the (1$\bar{1}$0) plane, they must be curved in a similar way as that plane. Then the incident X-ray beam, which has a finite width, must have fallen on the concave side of these curved surfaces. Thus, after the Bragg reflexion, the beam is somewhat focused. By a similar reasoning, the spots towards the lower right-hand corner are lengthened by reflexion on the convex side of the curved surfaces. In other directions on the photograph the shortening or lengthening of the spots is intermediate. This shows that the crystal is bent symmetrically with respect to the (110) plane, which is a plane passing through the diagonal line just mentioned; it is then understandable that the crystallographic planes responsible for spots not on or near the diagonal line are in general less bent.

The second stage of deformation, judged from the appearance of the photograph as shown in Plate 5, fig. 2, is not a simple bending. As the crystal has already undergone a bending and breaking up in the first stage of deformation, it is not likely that it can have a simple bending again about another axis. The radial broadening of the spots, however, seems to indicate that this stage of deformation consists partly of bendings about one or more axes parallel to or inclined at small angles to the photographic plate, and partly of other forms of distortion.

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Table 1. *Angle between the axis of bending and the incident X-ray beam*

Crystal plane	$\bar{3}01$	$\bar{4}01$	$32\bar{1}$	331	231	321	041	$41\bar{1}$	$22\bar{1}$
	− 1° 29′	− 1° 8′	− 1° 36′	− 1° 30′	− 1° 59′	− 1° 58′	− 1° 11′	− 2° 20′	− 2° 9′
Average					− 1° 42′				