REFLECTION OF X-RAYS FROM ROCK SALT.

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SYNOPSIS.

Reflection of X-rays from Rock-salt Crystals.—By means of the double spectrometer arrangement previously used in studying the reflection from calcite, a narrow beam of nearly homogeneous rays from one crystal was reflected from a second crystal. The two natural cleavage surfaces used were obtained by splitting a good clear specimen. The *rocking curves* are irregular and more than 50 times as wide as corresponding curves for calcite, while the *per cent. reflection at parallelism* of the surfaces was much less than for calcite, being only 10 for 3° grazing angle and 5 for 7°. Polishing the reflecting surfaces increased the curve width somewhat but washing with water restored the surfaces to their original condition. By interposing a third slit between the crystals and moving it across the beam more detailed information was obtained as to the position and character of the imperfections in the crystal surface. Another selected pair of freshly split surfaces gave similar results, except that the per cent. reflection at parallelism was about twice as great.

Imperfections of Rock-salt Crystals.—Compared with calcite, rock-salt crystals are evidently very imperfect. The recent results of W. L. Bragg also prove this. The greater apparent reflectivity observed with rock-salt is due to the fact that its irregular structure reflects a much broader band out of a continuous x-ray spectrum.

IN a recent number of the PHYSICAL REVIEW (May, 1921) the authors presented some results of an investigation of the reflection of x-rays from calcite. This reflection was investigated by means of a double x-ray spectrometer so arranged that a nearly homogeneous beam of x-rays from the first crystal (*A*) was allowed to fall on a second crystal (*B*). Maximum reflection was obtained when the reflecting atomic planes of the two crystals were parallel. On rocking the second crystal about the position of parallelism interesting curves of energy distribution were obtained which were strikingly narrow, the width at half-maximum being as small as 16 seconds of arc in some cases.

A similar investigation has been made of the reflection from the (100) planes of rock-salt. The crystals were mounted on the double spectrometer as shown in Fig. 1. The reader is referred to the original paper for details of the spectrometer and the method of observation.

Two properties in particular of this reflection were investigated. (1) The degree of perfection or imperfection of the crystals as indicated by the width and regularity of the rocking curves. Imperfections in the surface of the crystals or slight local tilting of the reflecting planes have the effect of broadening the rocking curves very much. It was found in the case of calcite, that the more nearly perfect the crystal the narrower and more regular the rocking curves. (2) The ratio of the energy at maximum reflection from the second crystal to the energy of the beam from the first crystal before reflection from the second one. This we call *per cent. reflection*. It is not a true coefficient of reflection, but would probably be the true coefficient for perfect crystals.

Two sets of rock-salt crystals were investigated, which are designated in the tables as crystals No. 1 and crystals No. 2. These crystals were the best that could be obtained from a supply house. They were large, fairly clear and nearly perfect as viewed by the eye. These crystals were split along (100) cleavage planes and mounted on the spectrometer in such position that the two reflecting surfaces were the contiguous planes before splitting. In the case of crystals No. 1 observations were made with the surfaces (*a*) split only, (*b*) polished, (*c*) washed with water.

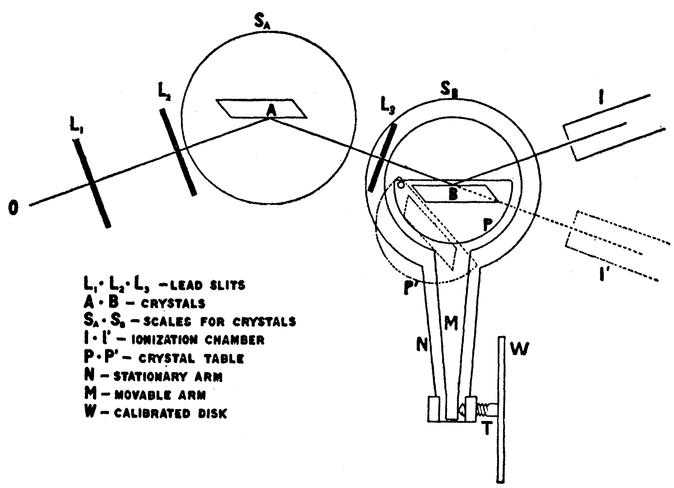

Fig. 1.

Three striking differences are observed between the results for rock-salt and for calcite. (*a*) The rocking curves for rock-salt are many times wider (more than 50 times) than those for calcite. (*b*) The rocking curves were irregular in outline and not symmetrical. (*c*) The per cent. reflection was much less than for calcite.

A third slit L_3 (width .4 mm.) was introduced between the crystals for the purpose of limiting the beam of x-rays to a portion only of the crystal surfaces (see Fig. 1). The types of rocking curves obtained for crystals No. 1, with the slit L_3 so placed as to limit the beam to a region near the center of the crystals are shown in curves Nos. 1, 2 and 3. The widths at half-maxima and the per cent. reflection for the several grazing angles are given in the table.

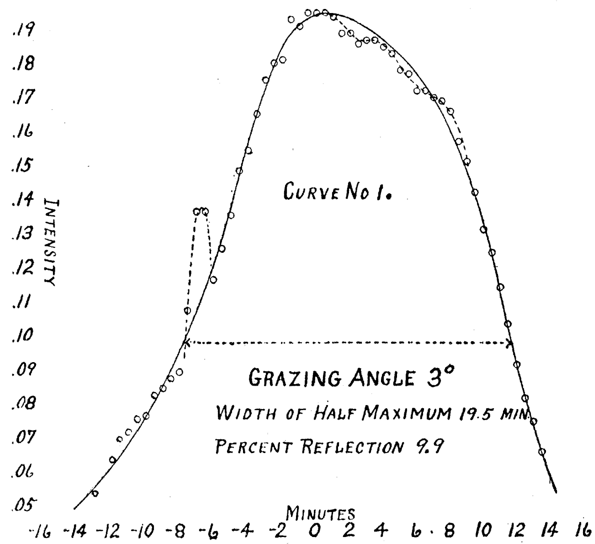

Fig. 2.

These curves all show an irregularity occurring on the same side for all three grazing angles. *However, when the slit was removed so that the beam of x-rays covered nearly the whole of the crystal surfaces the irregularities were ironed out.* The curves were regular in outline but were somewhat unsymmetrical.

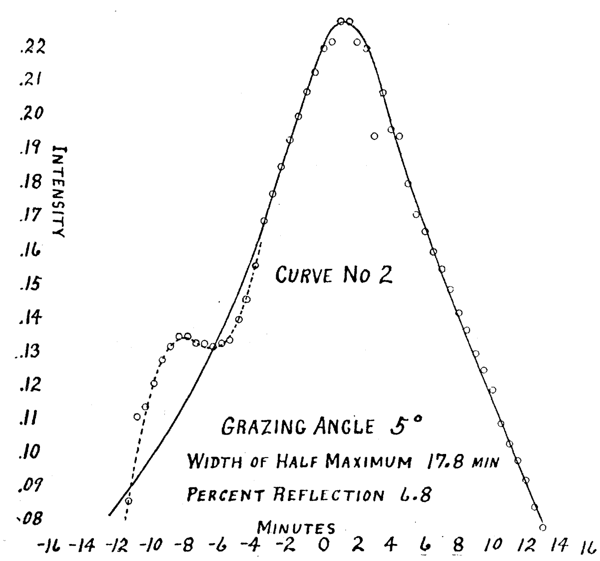

Fig. 3.

These results indicate that the crystals were quite imperfect. To check up these irregular effects, the slit L_3 was progressively moved to different positions across the beam of x-rays. The slit limited the reflection to a new portion of the crystal surfaces for each position. The successive positions of the slit were so adjusted that the regions on the crystals from which the rays were reflected were adjacent to one another. The results are given in the table for three positions *A*, *B* and *C*. The rocking curves obtained, without the irregular humps, are given in Fig. 5.

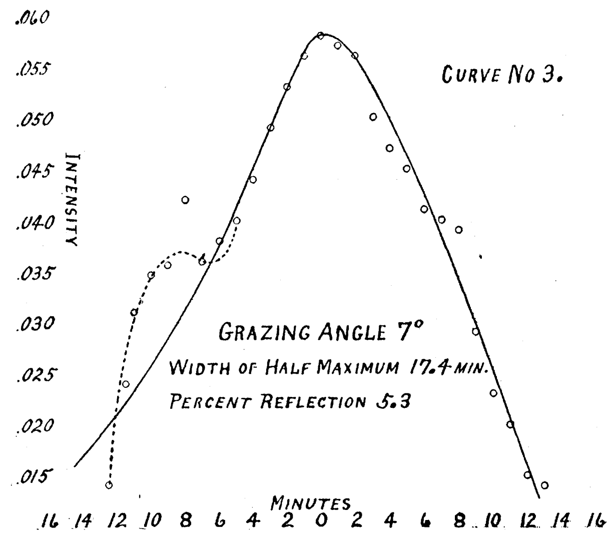

Fig. 4.

The curves *A*, *B* and *C* are given in the order of decreasing glancing angle. The angular position of the curves is displaced but not regularly, as the slit was progressively displaced across the beam. This means that the reflecting elements in the surface of the crystals did not lie in continuous planes, but that in certain regions the reflecting planes were tilted at a small angle to the planes near them. The greatest tilting for the two successive positions *A* and *B* was about 14 minutes of arc as indicated by the positions of the curves *A* and *B* in the figure. *Had the crystals been perfect these three curves would have been superimposed.*

The above results were obtained with the natural cleavage surfaces of the crystals. Polishing and etching of the surfaces had been observed to produce a great effect on the reflection and width of rocking curves in the case of calcite. The effect of polishing and washing the rock-salt crystal surfaces was investigated and the results are shown at the bottom of the table for crystals No. 1. Observations were made at only one glancing angle. It will be observed that polishing increased the width of rocking curve to some extent, but that when the surfaces were washed with water to remove the polish they were restored to their original condition. No measurements were made of the per cent. reflection in this instance, as it was not desirable to disturb the setting of the instrument between the processes of polishing and washing.

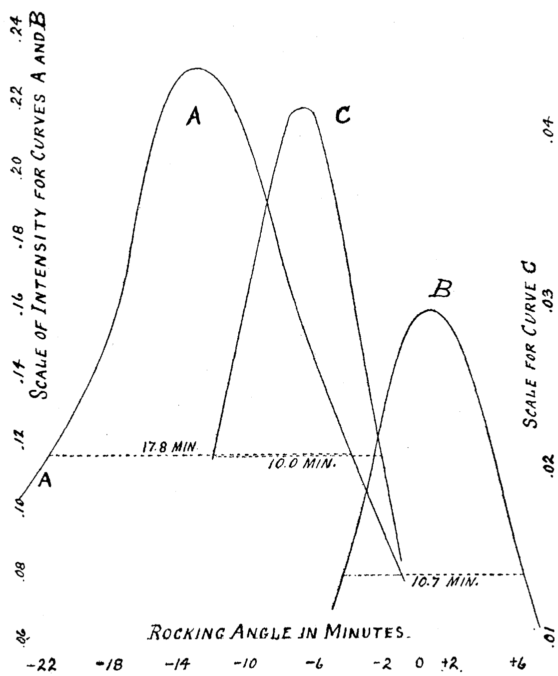

Fig. 5.

To further test this matter of irregular reflection, a new specimen of rock-salt was obtained. This was a large clear piece, in fact, nearly perfect as viewed by the eye. It was found, however, after splitting that the two cleavage surfaces showed small ripples and undulations when examined by reflected light. The crystal was split a number of times until fairly satisfactory surfaces were obtained.

Observations were made with this pair of crystals, (*a*) with freshly split surfaces, (*b*) with the surfaces both polished and washed. The results for three different glancing angles are given in the table for crystals No. 2.

The curve width is somewhat less than that first obtained with crystals No. 1, but is approximately the same as that obtained for No. 1, after the slit L_3 had been once removed and then replaced. It will be noticed that polishing and washing did not affect the width of rocking curves materially.

The per cent. reflection for this pair of crystals was considerably greater than for the first pair (see column four of the tables).

The decrease in reflection for increase of wave-length was more marked for this pair than for the first pair of crystals.

Table I.

Rock-salt.

Crystals No. 1 (A_1 and B_1).

Condition of Crystal.	Grazing Angle.	Width at Half-Max.	Per Cent. Reflect.
With Slit L_3 between Crystals.			
Split only		18.7 min.	10.1
	3°	19.9 min.	9.9
		19.3 min.	10
		16	8.9
	5°	17.8	6.8
		16.9	7.8
	7°	17.4 min.	5.3
Without Slit L_3 between Crystals.			
Split only	5°	12.3 min.	9.5
	7°	11.7	7.6
	9°	12.5	5.7
Slit L_3 Moved across X-ray Beam.			
Split only—*A*	5°	17.8 min.	
B	5°	10	
C	5°	10.7	
With Slit L_3 between Crystals.			
Split	5°	10.4 min.	
Polished	5°	13.7	
Washed	5°	10.8	

TABLE II.

Crystals No. 2 (A_2 and B_2).

Condition of Crystal.	Grazing Angle.	Width at Half-Max.	Per Cent. Reflect.
Slit L_3 between Crystals.			
Split	3°	10.6	24.8
	5°	10.4	15.3
	7°	8.7	13
Surfaces polished and washed	3°	10.9	22.1
	5°	11.5	14.7
	5°	11.1	15.5
	7°	10.9	11.5

These crystals showed so much irregularity in structure, when examined by this method, that but little effect was produced by changing the condition of the reflecting surfaces. In this respect there is a striking contrast between rock-salt and calcite.

The double spectrometer has been applied to crystal reflection by A. H. Compton,[1] and more recently by W. L. Bragg[2] to the investigation of the reflection from imperfect crystals (rock-salt). In this last paper, as an illustration of effect of regular and irregular structure on reflection, Professor Bragg gives two rocking curves. The second crystal in these cases was a thin slab and the curves were taken by transmission. The curve which is given in illustration of regularity of structure is very broad, however. Its breadth is about 30 minutes of arc at half-maximum. This indicates that (*a*) the beam coming from the first crystal to the second was very non-homogeneous, or (*b*) that the second crystal was very irregular in structure, or (*c*) that both of these were true.

Our rocking curves for rock-salt specimens which are manifestly imperfect are more narrow than this one given by Professor Bragg. The experiments previously described for the case of calcite gave rocking curves that are 100 times as narrow as this curve. Calcite approximates to a perfect crystal but is not, of course, completely perfect. This great width of curve given by Professor Bragg would indicate that the crystals were quite imperfect.

Curves Nos. 1, 2 and 3 were all taken with slit L_3 in place. *When*

[1] Proc. Am. Phys. Society, PHYS. REV., July, 1917.

[2] Phil. Mag., March and July, 1921.

slit L_3 was removed so that the rays came from a large portion of the crystal surface, these irregularities were washed out. The curves were regular and smooth, although the crystal was quite imperfect.

It has been noted by a number of experimenters that greater energy is apparently obtained from rock-salt at a first reflection than from calcite. Our results, however, indicate that rock-salt is not so good a reflector as calcite. The calcite *gives a more complete reflection and a much more homogeneous beam of x-rays than rock-salt.* The greater energy observed from rock-salt is due to the fact that at any one position of the crystal the portion ($\Delta\lambda$) selected from the continuous x-ray spectrum is much wider than in the case of calcite.

The results obtained indicate that:

Rock-salt crystals are more apt to be irregular and defective than calcite.

Rock-salt is not so good a reflector of x-rays as calcite.

Rock-salt, owing to irregularities, gives a much less homogeneous beam of x-rays by reflection than calcite.

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Columbia University,
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