UNUSUAL REFLECTING POWER OF A PAIR OF CALCITE CRYSTALS

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Abstract

Reflection characteristics of the calcite (Iceland spar) crystal are reported. Two split calcite crystals were mounted on double x-ray spectrometer in parallel position. Coefficient of reflection, percent reflection and width of rocking curve were measured at both first and second order reflections and for three wave-lengths: λ0.209A, λ0.7077A and λ1.537A. The percent reflection was as great as 48.5 at first order for λ1.537. The corresponding coefficient of reflection was small. The rocking curves were surprisingly narrow, being as small as 1.25″ at second order for λ0.7078A. This width is less than is to be expected from the Darwin equation using previously determined values of the structure factor. Crystals of these properties are especially valuable for use on the double crystal spectrometer, when high resolving power is desired.

THE resolving power of the double x-ray spectrometer is largely dependent on the perfection of the crystals. In the search for the best crystals a pair was found whose properties were so near those of a "perfect" crystal that it seemed desirable to bring it to the attention of those who are interested in theories of crystal reflection.

The specimen of calcite (Iceland spar) was obtained from the Ward Natural Science Establishment of Rochester, New York. A portion about $2\times2\times10$ cm was split from the large crystal. This was then carefully split into two equal crystals 1 cm thick and 10 cm long. They were mounted so that the two contiguous faces of the last splitting formed the two reflecting surfaces. These crystals were mounted on the spectrometer in the parallel position in the manner previously described.[1] When the crystals are placed in this position the width of rocking curves is independent of the properties of the radiation. It depends only on the crystals.

The instrument and the technique have been improved so that it is possible to read rocking angles to a part of a second of arc. An important matter in the technique of two-crystal spectrometry is that the axis of rotation of the second crystal B should be parallel to that of the first crystal A. Lack of parallelism increases the width of the rocking curves.

Measurements were made at first and second orders on both crystals. The wave-lengths used were W $K\alpha_1$(0.209A), Mo $K\alpha_1$(0.7077A) and Cu $K\alpha_1$(1.537A). The rocking curves obtained are shown in the figures. They are plotted on the same scale with first order curves in Figure 1, and second order curves in Figure 2. The results are also given in Table I. The coefficient

[1] Bergen Davis and W. M. Stempel, Phys. Rev. **17**, 608 (1921).

of reflection is the value of $E\omega/I$. The unit of angle is one degree of arc. The intensity I of the beam coming from crystal A is obtained by throwing crystal B back out of direct path of beam and allowing it to enter the ionization

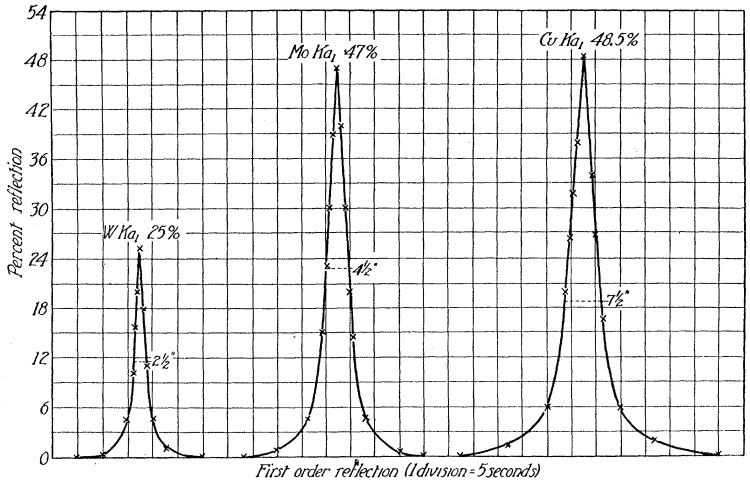

Fig. 1. Rocking curves, first order.

chamber. The term $E\omega$ which is the integrated intensity after reflection from crystal B is often obtained by the method of sweeps. In the present instance it was obtained directly from the rocking curves by a method previously

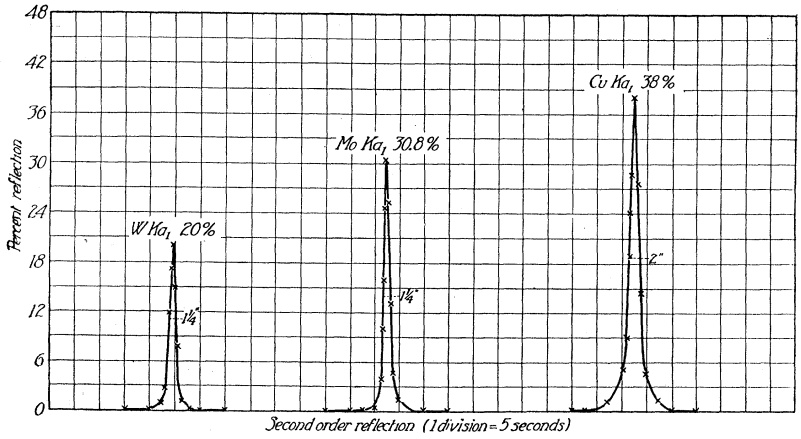

Fig. 2. Rocking curves, second order.

described.[2] The percent reflection is the ratio of the intensity from crystal A to that from crystal B, when B is in the position of maximum reflection. In the next column is given the observed width at half-maximum of the rock-

[2] Bergen Davis and H. M. Terrill, Phil. Mag., Vol. XLV, March, 1923.

ing curves in seconds of arc. If the width depends on some property of the crystals it will be present equally in both crystals. Each crystal will contribute to the width. This contribution, however, is not directly additive. A reasonable method of correction has been given by Ehrenberg and Mark[3] and by M. Schwarzschild.[4] The contribution of crystal A to the width observed from crystal B is the factor $2^{1/2}$. The corrected width given in the last column is obtained by dividing the observed width by $2^{1/2}$. This column should represent the actual width of rocking curve due to crystal B when a strictly parallel beam is reflected from it.

TABLE I.

λ	Order	Coef. R	Percent R	Width (seconds)	Corrected width
0.209A	1	0.00026	25	2.5	1.78
	2	0.0001	20	1.25	0.9
0.7077	1	0.00035	47	4.5	3.2
	2	0.0001	31	1.25	0.9
1.537	1	0.0011	48.5	7.5	5.4
	2	0.00022	38	2	1.67

The structure factor:—An expression for the width of rocking curve for reflection from a perfect crystal has been derived by C. G. Darwin.[5] It has the form (for unpolarized rays)

$$\Delta\Theta = \frac{4}{3\pi} nF \frac{e^2}{mc^2} \lambda^2 \cot\theta,$$

where n is the number of molecules per cc, θ is the angle of reflection, and F is the term known as the structure factor. It represents the effect of distribution of electrons on the reflection. If the electrons belonging to a molecular layer all lie in one plane $F=Z$, where Z is the number of electrons in a molecule. As used here $Z=50$ for a calcite crystal.

The values of the structure factors for the constituents of calcite have been experimentally determined by the powdered crystal method. We are indebted to Dr. R. W. G. Wyckoff and Miss Alice H. Armstrong, of the Rockefeller Institute for Medical Research, for advanced unpublished values of F for oxygen and carbon. The values of F for calcium are taken from Havighurst.[6] The structure factor is a constant function of $\sin\theta/\lambda$ and has the same value for all wave-lengths but depends on the order of reflection. These values of the structure factor are designated by F_1 in the third column of Table II. The next column of the table gives the widths calculated by the

[3] Ehrenberg and Mark, Zeits. f. Physik **42,** 807 (1927).
[4] M. Schwarzschild, Phys. Rev. **32,** 1928.
[5] A. H. Compton, X-rays and Electrons, p. 141.
[6] R. J. Havighurst, Phys. Rev. **28,** 869 (1926).

Darwin equation for $F=Z$. The last column gives the widths for $F=F_1$. This column ($F=F_1$) is to be compared to the last column of Table I. It will be noticed that the observed widths are less than the calculated values for a perfect crystal except in the case of W $K\alpha_1$(0.209A).

TABLE II.

λ	Order	F_1	Calculated width $F=z$	Calculated width $F=F_1$
0.209A	1	25.1	2.55″	1.28″
	2	11	1.27	0.28
0.7077	1	25.1	8.6	4.3
	2	11	4.2	0.92
1.537	1	25.1	17.2	8.6
	2	11	12.3	2.7

The crystals, however, can not be regarded as perfect. The percent reflection does not rise to a greater value than 48.5 percent in any case, while theory predicts 100 percent reflection for a perfect crystal. The observed widths for W $K\alpha_1$ are greater than is to be expected from theory. The experimental difficulties were much greater at this small wave-length. The glancing angle is quite small. It was difficult to exclude the scattered radiation from the ionization chamber.

The resolution that is to be expected with the double x-ray spectrometer is about the same order of magnitude as the width of rocking curves with parallel crystals. Fine structure in spectral lines should be measureable to this degree of refinement provided the natural breadth is not greater than this. Also the natural breadth of a spectral line can be measured to this small angle provided it contains no fine structure. This may be illustrated by the case of second order reflection for wave-lengths near the Mo $K\alpha_1$, where the width is one second of arc. The resolution is given by $2\Delta\lambda = 2d\cos\theta_2\, d\theta_2$. If $d\theta_2 = 1''$ arc, the measureable fine structure will be 0.015X-units. Also the breadth of a spectral line may be measured to 0.015 X-units, which is much less than the classical breadth of 0.12 X-units.

We wish to express our appreciation of the trouble taken by Miss Alice H. Armstrong in preparing for us the structure factor data used in this article.