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Further Tests of the Validity of X-Ray Crystal Methods of Determining *e*

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The authors have continued their efforts to absolve the x-ray method of determining the electronic charge of the criticism that Bragg reflection determines the lattice dimensions in only a very shallow surface region whereas the density determinations have been made on a much larger volume of crystal. In earlier work by a powder method we were able to show that the above-mentioned superficiality criticism could not explain the discrepancy between $e=4.803$ (the "x-ray value") and $e=4.770$ (computed by the oil-drop method with Harrington's value of air viscosity). The powder method, necessarily somewhat less precise than the macroscopic calcite method, yielded a mean of $e=4.799$, agreeing with the macroscopic calcite value well within the precision of the powder method. The present work gives a still more precise absolution of the macroscopic calcite results from the above-mentioned criticism. In the earlier work we abandoned efforts to measure lattice dimensions in the interior of a thin-cleaved slab of calcite by means of Laue reflection in a precision transmission spectrometer because photographs with the crystal adjacent to the slit, but on the same side as the photographic plate showed most of the Laue reflection coming from regions close to the front and back surfaces of the slab. Also strange unexplained fluctuations of intensity along the lengths of the spectral "lines" were observed with the crystal in this position. In a continuation of this work two thin crystal slabs have been studied. Each has been etched with HCl rather heavily all over one of its faces and over half the height of its opposite face. The upper half of the spectral lines selectively reflected to the photographic plate had thus traversed a crystal slab etched on both sides, while the lower half had traversed a slab etched on only one side. With this arrangement, and with the crystal on the side of the slit nearest the photographic plate, we are able to show that the enhanced Laue reflection from regions near the surface is completely suppressed by sufficient etching and that fainter reflection can then be definitely observed from the interior regions of the crystal. With the crystal on the opposite side of the slit from the plate we are able to compare very accurately on one and the same photographic plate the reflection angles for the enhanced surface reflection with the reflection angles for the interior planes. These angles turn out to be very precisely equal, thus furnishing clinching justification for the validity of the well-known precise determinations of *e* by Bragg reflection from the surface of macroscopic pieces of calcite. The density fluctuations along the length of the lines are absent for the interior reflection and we conclude that they are probably the result of local variations of surface disturbance set up in the process of cleavage.

I. INTRODUCTION

THE present paper describes the continuation of a program of investigation of the criticisms of the x-ray crystal method of determining the electronic charge *e*. In a previous paper[1] the authors have tried chiefly to answer the following criticism, namely, that while the density measurements on a macroscopic crystal are averages

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[1] DuMond and Bollman, Phys. Rev. **50**, 524 (1936). The schematic view, Fig. 5 of this paper, shows the precision transmission or "Laue reflection" type spectrometer. The slit and crystal are mounted on a turntable whose axis of rotation coincides accurately with the center line of the slit. The reflecting planes of the thin calcite crystal parallel to its narrow cleavage surface are used and the table and crystal are set by optical adjustments so that these planes stand accurately normal to the photographic plate holder surfaces. These latter are two in number (not shown in Fig. 5) and the distance between their accurately parallel plane surfaces has been measured by means of scratches on a bar in the same comparator used for measuring the line separations on the plates. Movable lead shields are provided to protect the plate from the straight-through transmitted beam on the side opposite to that on which the spectrum lines are being photographed. Lead x-ray shielding and light-tight housings are shown in Fig. 4. The Mo target of the x-ray tube has its surface nearly parallel to the plane of Fig. 5 so that the projected height of the focal spot (normal to the plane of the drawing) is very small. The focal spot is of large enough diameter in the direction parallel to the plane of the figure to furnish radiation of appropriate direction for the entire *K* spectrum. In Fig. 5 the crystal and slit are shown in the normal *A* position for making sharp spectrum lines and for determining Laue reflection angles with precision. Fig. 7 of the same reference shows schematically the position of slit and crystal after turning the turn table one-half revolution away from the normal position of Fig. 5. Only the first of the two photographic plate positions is shown. Before etching the exit or entry cleavage surfaces of the calcite, reflection from the regions near these surfaces predominated so strongly as to form intense lines on the plate at A_1 and B_1 or at A_2 and B_2 with the appropriate separations d_1 and d_2 easily computed from the thickness of the crystal and the geometry of the arrangement. (See Fig. 8.) After etching, these lines are extinguished and a uniform band of reflection by the interior planes of the crystal throughout the triangle *OAB* can be seen on the photographic plate filling the regions

of a large volume, say 10 cc of crystal, the x-rays are diffracted from a thin surface layer (Bragg reflection) which is limited by "extinction" to about 4×10^{-4} cm thickness, a very small fraction of the total volume in an entirely specialized location.

In the earlier work an effort was made to measure by means of Laue reflection the calcite lattice dimensions for the *interior* of a thin crystal slab. A precision transmission spectrometer, whose arrangement is schematically evident in Fig. 5 of the above-mentioned previous paper,[1] was used. Normally the slit is situated on the same side of the crystal as the photographic plate, so as to form sharp spectral lines. We shall designate this as the A position. We found, however, upon turning the circular table halfway round so that the crystal stood between the slit and the photographic plate, (hereafter called B position) as indicated in Fig. 7 of the previous paper, that the Laue reflection from those parts of the internal planes closely adjacent to the front and back surfaces of the crystal slab (at O, A or B) strongly predominated over the reflection from the interior of the slab. See Fig. 8 of the previous paper. The results of the A arrangement could, therefore, not be regarded as representative of the crystal interior. Also in the B position there were observed certain mysterious fluctuations of intensity along the lengths of the spectral lines reflected by the regions of the planes near the front and back surfaces of the crystal.

These difficulties led us to the adoption of the powder method for determining e with the result, $e=4.799\times 10^{-10}$ e.s.u. slightly lower than the value 4.803 obtained by ordinary methods with macroscopic calcite. We did not attach any especial significance to this slight difference of four units in the third decimal place which is within the limits of accuracy of the powder method. Rather the result was interpreted as a corroboration in favor of the value 4.803 obtained by Bragg reflection on macroscopic calcite and against the much lower value 4.770. The powder method is necessarily somewhat less precise (as judged by internal consistency of the measurements) than the macroscopic calcite method and it is therefore desirable to increase, if possible, the precision with which the macroscopic calcite results may be absolved of the criticism regarding their superficiality.[2] We believe the work here reported accomplishes this.

II. EFFECT UPON LAUE REFLECTION OF ETCHING EXIT AND ENTRY SURFACES

K. V. Manning[3] has described specifications for etching a cleaved or polished calcite surface with HCl in order to reduce the "rocking curve" widths of the crystal as observed with the two-crystal spectrometer. We have etched one side of our thin slab of cleaved calcite and have found that the enhanced Laue reflection from the regions near the surface can be completely suppressed if the etching be continued considerably longer than Manning specifies for his purpose. We found some 20 minutes with 0.7 normal HCl were necessary. With the spectrometer set in the B position and a prolonged exposure,

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d_1 and d_2. (See Fig. 7.) In this position the spectrometer is useless for precise measurements on the lines as these are too broad. Its use is solely to show which regions of the crystal are active or predominant in reflection. The exposure in Fig. 8 was made with the plate in the more remote plate holder (see Fig. 1) so that the α-doublet spectral separation would be sufficient to avoid confusion with the doublet character of the lines caused by enhanced reflection near the crystal surfaces. Because of the large plate distance the fluctuations of intensity along the lengths of the lines are spread so far apart that they are few in number and do not show clearly in this reproduction.

[2] In a paper entitled "The Most Probable 1938 Values of the Electron and Related Constants," R. A. Millikan, Ann. d. Physik **32**, 42 (1938), the question as to a slight residual "mosaic effect" is again raised as a possible justification for a value of $e=4.796$ very appreciably lower than 4.803, the mean of the best x-ray results obtained with macroscopic calcite. Millikan in this paper has frankly attempted to harmonize the best experimental values of e, e/m, and h/e so that when these are substituted into the Bohr formula for the Rydberg constant a value in accord with the spectroscopic determinations of that constant shall be obtained. In our opinion this required doing some violence both to the most reliable values of h/e as measured by means of the short wave-length limit of the continuous x-ray spectrum and to the value of e obtained by the x-ray crystal method. The results reported in the present paper, we believe, remove any justification for revising downward the x-ray crystal value, $e=4.803$, obtained with macroscopic calcite. For the purpose of determining *the most probable values of the constants, e, m, and h in 1938* such an effort to harmonize these discrepancies is perhaps the best compromise that can be made. We take the stand, however, that the discrepancy between the measured values of these constants and the requirements of the Bohr-Rydberg formula is something interesting in itself which may lead to important revisions of theory. Whether such revisions must be made in the form of the Bohr-Rydberg formula itself or in the theory governing some of the experimental determinations we, of course, do not pretend to know. Experiments are now under way which we hope may answer such a question.

[3] K. V. Manning, Rev. Sci. Inst. **5**, 316 (1934).

reflection from the interior of the crystal could now be brought out on the photograph as a band of the appropriate breadth bounded on one side only with the sharp intense line from the region of the crystal near the remaining unetched face. This remaining surface line still exhibited the marked fluctuations of intensity along its length which characterize all such surface lines in all the samples we have observed.

Next the crystal slab was etched in the same way on the upper half of its opposite face (the face nearest the slit). The boundary line between the etched and unetched regions of this face was horizontal and cut across the slit at very near the midpoint of its height. By carefully adjusting the

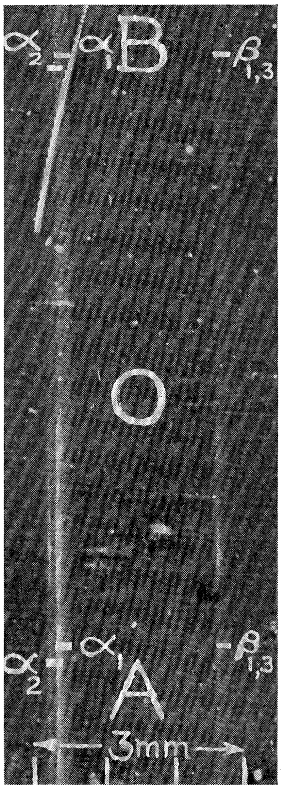

FIG. 1. Showing an enlargement of the reflected Mo K spectrum from an etched crystal slab (spectrometer in B position). In this case the exit surface of the crystal slab has been rather strongly etched with HCl and the entry surface has been similarly etched over its upper half. That segment of the spectrum lines marked AO came from the part of the crystal with one unetched face. The segment OB came from the region with both faces etched. The etching has caused the intensified surface reflection to disappear completely. The fainter reflection from the interior of the crystal can now be seen as a uniform band and in the region AO it is bounded on one side only by the enhanced reflection from the single remaining unetched part of the entry face. In the region OB each line can be seen spread out as a uniform band of reflection from the crystal interior (region, OAB, Fig. 7 previous paper) with no enhancement at the edges. The apparent width of the enhanced region is mainly caused by the finite width of the spectrometer slit and should not be understood as indicating any great depth for the crystal region from which the enhanced reflection comes. This exposure was taken with the plate in the nearer of the two plate holders and there is, therefore, some slight overlapping of the bands from $K\alpha_1$ and $K\alpha_2$. The band from $K\beta_{1,3}$ is also clearly visible in the original. Also because of the closer plate distance several of the fluctuations of intensity along the length of all the enhanced surface lines are visible in the original in the region AO. These fluctuations are completely absent for the reflection from the interior of the crystal (segment OB). The reader is asked to disregard the accidental scratch in the region OB of the alpha-lines. The scale indicates the magnification.

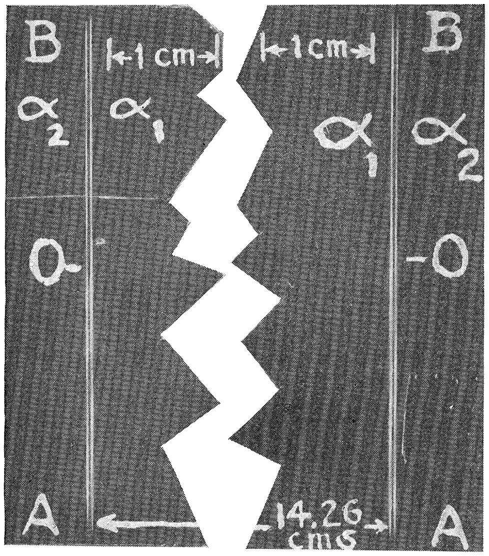

FIG. 2. Enlarged reproduction of spectral lines (Mo $K\alpha_1$, α_2) taken with the spectrometer in the A position. (Normal arrangement to give sharp lines for accurate angle determinations, the crystal being on the opposite side of the slit from the photographic plate.) Here the crystal slab has been etched completely over its entry face and over the upper half of its exit face (the face nearest the slit). Thus the somewhat fainter upper half of the spectrum line (segment here indicated as OB) has been formed by Laue reflection throughout the entire interior of the crystal slab, while the stronger lower half (segment here indicated as OA) has been formed chiefly by Laue reflection from the regions near the unetched surface. On the original the fluctuations of intensity along the length of the line can be seen in the segment OA. Attention is called to the complete absence of any jog or offset at the junction of the line segments OA and OB. Careful measurements, on lines such as these, of the separation between left- and right-hand reflected images for the top and bottom segments (OB and OA) showed the reflection angles for the interior of the crystal to be accurately identical with the reflection angles for the regions near the surface.

height of the focal spot of the x-ray tube it was easy to arrange so that the upper half of the spectral lines selectively reflected to the photographic plate had thus traversed a crystal slab etched on both sides while the lower half has traversed a slab etched on only one side. Fig. 1 is an enlarged reproduction of an exposure with the spectrograph in the *B* position and with the crystal etched fully on one side and halfway down on the other side. The band reflected from the interior of the crystal is clearly visible and its lower half is bounded on one side by the enhanced reflection from the unetched part of one face of the crystal.

Now with the above-described crystal and with the spectrometer in the *A* position (slit between crystal and photographic plate) a number of exposures were taken for the purpose of comparing precisely the diffraction angle for the reflection near the surface with the diffraction angle from the interior planes. Both α_1- and α_2-lines of the molybdenum *K* spectrum were measured. The upper part of the spectral lines formed by Laue reflection in a crystal with both entry and exit surfaces etched appears of uniform density over its length but considerably less intense than the lower part of the spectral lines formed by Laue reflection chiefly from the part of the crystal with one unetched surface. This latter part of each line exhibits the fluctuations of intensity along its length and clearly comes mainly from Laue reflection near the unetched surface which strongly predominates over the reflection from the interior. The sharp boundary between the part of the line where surface reflection predominates and the part formed by interior reflections is clearly visible, the difference in reflected intensity being marked. Fig. 2 is an enlarged reproduction of one such spectrum line.

TABLE I. *Line separations in cm on plate for* Mo$K\alpha_1$ *and* $K\alpha_2$.

PLATE NO.	$\Delta\alpha_1$ FOR INTERIOR	$\Delta\alpha_1$ FOR SURFACE	$\Delta\alpha_2$ FOR INTERIOR	$\Delta\alpha_2$ FOR SURFACE	
1	4.4155	4.4162	4.4431	4.4430	Nearer plate holder
2	4.4124	4.4112	4.4392	4.4378	
3	4.4141	4.4135	4.4406	4.4403	
Average	4.4140	4.4136	4.4410	4.4404	
4	14.2612	14.2610	14.3480	14.3483	Farther plate holder
5	14.2568	14.2579	14.3435	14.3437	
6	14.2565	14.2560	14.3444	14.3432	
Average	14.2582	14.2583	14.3453	14.3436	

FIG. 3. Illustrating the fluctuations of intensity along the lengths of the lines. Enlarged reproduction of an entire *K* spectrum formed by Laue reflection from an unetched crystal with the spectrometer in the *B* position and with the plate on the nearer of the two plate holders. The broadening and doubling of the lines combined with the low dispersion because of the proximity of plate to slit has caused the alpha-doublet to overlap confusingly. This proximity of plate to slit permits a large number of the intensity fluctuations to appear in good contrast on the plate.

Precise measurements of the diffraction angles could be made by measuring the separation of the images formed by Laue reflection both to left and to right. The angles for surface and interior reflection were very accurately compared by use of the measurements between the top halves of the lines and the bottom halves on one and the same plate. The absolute angles themselves were also measured precisely by successively photographing spectra on plates mounted in two plate holders (visible in Fig. 4 of the previous paper)[1] one of which was much nearer the slit than the other and whose separation was accurately measured (by means of a trammel bar) on the same comparator as was used for measuring the plates. Table I gives the comparison of line separations for surface and interior reflections, while Table II gives the absolute angles we have

TABLE II. *Laue reflection angles for interior and surface reflection.*

$\theta\alpha_1$ interior	$\theta\alpha_1$ surface	$\theta\alpha_2$ interior	$\theta\alpha_2$ surface
6° 42′ 21″	6° 42′ 22″	6° 44′ 47″	6° 44′ 45″
Comparison with Larsson's results in Bragg reflection			
$\theta\alpha_1 = 6°\ 42'\ 35''$		$\theta\alpha_2 = 6°\ 45'\ 2''$	

TABLE III. *Measured Laue reflection angles for second crystal sample before etching.*

$\theta_{\alpha 1}$	$\theta_{\alpha 2}$
6° 42′ 35″	6° 44′ 59″

measured compared with those observed by Larsson,[4] whose results were given highly satisfactory confirmation by Compton,[5] Tu,[6] and Bearden.[7]

The first thin slab we studied, while showing satisfactory agreement between surface and interior reflection angles, gave the absolute values for the angles of Laue reflection about six-hundredths of one percent smaller than the results observed by Larsson in Bragg reflection. This discrepancy is only slightly decreased by the correction for index of refraction and as it is a little too large to be interpreted as an error of measurement, we concluded that our first sample of calcite was perhaps slightly imperfect. We therefore selected another thin slab of calcite which, before etching, as shown in Table III, gave for the absolute Laue angles excellent agreement with the accepted Bragg values, and we repeated on this sample all the studies we have just described for our first sample with the completely satisfactory results as regards agreement between the line separations for the surface and for the interior reflections as shown in Table IV. Table V shows the absolute angles for surface and interior Laue reflections for the second calcite. Comparison with the results of Larsson (Table II) shows excellent agreement with the Bragg values.

The very slight differences between the Laue reflection angles for surface and interior reflection are entirely within the limits of observational error and quite without significance, for both crystal samples.

Regarding the absolute values of the reflection angles, the correction for refractive index of 3.25 seconds of arc must be subtracted from Larsson's value but is entirely negligible in our case because in our Laue reflection the entry and exit beams are nearly normal to the surface of the calcite. The deviation from Larsson's corrected results is inside the limit of our observational error for the second crystal sample.

The important conclusion from these observations is that when Laue reflection is used with a calcite crystal etched on both faces in the *A* arrangement the observed diffraction angle *representative of the entire interior of the crystal* is just the same as the diffraction angle for reflection occurring near the surface so that the validity for the purpose of determining *e* of the well-known precision measurements made in Bragg reflection is now, we believe, firmly established to about the same order of precision as could be claimed for the observational precision of those measurements themselves.

III. PROBABLE CAUSE OF THE INTENSITY FLUCTUATIONS ALONG THE LENGTH OF THE SPECTRAL LINES

We think the fluctuations of intensity along the length of the spectral lines in the case of Laue reflection near the exit or entry surface of a calcite crystal may be explained as follows. We suppose that in spite of the remarkable uniformity of calcite both as regards density and lattice perfection as revealed by x-rays there exists in the body of the crystal a multitude of regions where the cleavage planes do not resist rupture quite as strongly as elsewhere. An incipient crack is too strong a term to describe such a weak region. We have no reason to think that these regions exhibit any marked regularity

TABLE IV. *Line separations in cm on plate for* Mo $K\alpha_1$ *and* $K\alpha_2$ *(second crystal sample).*

PLATE No.	$\Delta\alpha_1$ FOR INTERIOR	$\Delta\alpha_1$ FOR SURFACE	$\Delta\alpha_2$ FOR INTERIOR	$\Delta\alpha_2$ FOR SURFACE	
7	4.4149	4.4138	4.4416	4.4407	Nearer plate holder
8	14.2646	14.2630	14.3518	14.3504	Farther plate holder

TABLE V. *Measured Laue angles for surface and interior reflections, second crystal sample.*

$\theta\alpha_1$ interior	$\theta\alpha_1$ surface	$\theta\alpha_2$ interior	$\theta\alpha_2$ surface
6° 42′ 35″	6° 42′ 33″	6° 45′ 1″	6° 44′ 59″

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[4] Larsson, Phil. Mag. (7) **3**, 1136 (1927).

[5] A. H. Compton, Rev. Sci. Inst. **2**, 365 (1931).

[6] Y. C. Tu, Phys. Rev. **40**, 662 (1932).

[7] J. A. Bearden, Phys. Rev. **38**, 1389 (1931).

of distribution but feel tempted to regard them as accidents in the detailed history of the crystal growth. These regions though numerous enough to insure fairly ready cleavability of a macroscopic piece of calcite in almost any portion of its structure are, we suppose, separated by strong homogeneous domains which constitute the overwhelmingly preponderant volume of the entire crystal. This volumetric preponderance of the homogeneous strong domains is necessary to explain the remarkably reproducible density and the x-ray structural perfection of this crystal. It also agrees with the annoying difficulty sometimes encountered in inducing cleavage in calcite at some exactly specified point. A further fact supporting this picture is that a large piece of calcite, if hit or dropped, will almost always cleave along cleavage planes whereas when finely powdered calcite is examined with a microscope one sees many curved irregular and conchoidal fractures. It seems reasonable to suppose that when calcite is cleaved, the tearing action progresses by a sort of propagation of stress from one weak region to another lying as nearly as possible in the same plane. The lattice structure is greatly disturbed by the tearing action in the strong regions and little disturbed in the weak regions. Hence the surface after cleavage has patches of greater and less disturbance. Indeed something of this sort seems frequently visible under close scrutiny with a low power microscope. The more disturbed regions will reflect more x-ray energy than the less disturbed regions.[8] The fluctuations of intensity along the length of the spectral lines we have observed are, we think, a direct result of this. It is interesting to note that the above phenomenon is less pronounced in the case of our second crystal sample whose reflection angle showed it to be the more perfect of the two samples.

Figure 3 is an enlargement of a K spectrum of lines exhibiting these fluctuations of intensity taken with the first crystal sample.

ACKNOWLEDGMENTS

It is a pleasure to express our gratitude for the financial assistance from the Watters Memorial Research Fund established by Dr. Leon L. Watters of New York City which has supported this research work, establishing the validity of the x-ray crystal method of determining the electronic charge.

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[8] That mechanical disturbance of the crystal lattice produces stronger reflected spectral lines is well known. We believe it can be easily understood in the present case of Laue reflection in calcite as follows: Two-crystal spectrometer studies show calcite to be such a perfect crystal that the angular domain over which selective reflection occurs in a good undisturbed sample is very small (only a few seconds of arc). As a result, the solid angle into which a source of x-rays (say any atom radiating in the target) must radiate a given wave-length in order to have it selectively reflected by the crystal is very restricted. Now if the calcite lattice structure is mechanically disturbed, new, slightly differing orientations will be set up in different regions of the crystal so that a larger solid angle of radiation from each atom in the source can find regions appropriately oriented for selective reflection and the total energy reflected will be greater but will cover a wider angular domain. In a spectrometer of high resolving power (such as the two-crystal spectrometer) this results in an angular broadening of the lines rather than in an intensification of them. But in a spectrometer such as the one here described where the breadth of the spectral lines is determined chiefly by the breadth of the slit this latter masks to a large extent the angular broadening set up by mechanical disturbance and the overlapping of the broadened diffracted images transmitted through different regions of the slit width builds up a more intense spectral line.

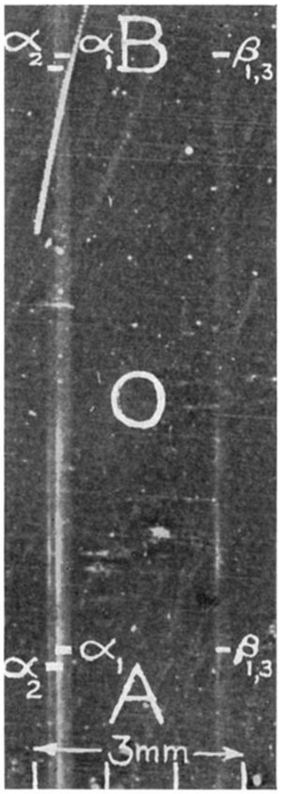
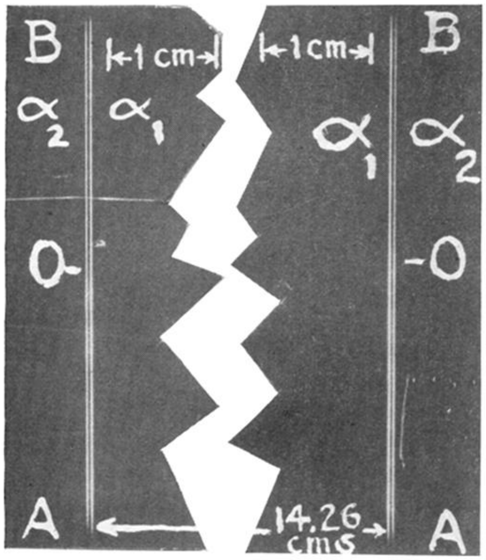


FIG. 1. Showing an enlargement of the reflected Mo K spectrum from an etched crystal slab (spectrometer in B position). In this case the exit surface of the crystal slab has been rather strongly etched with HCl and the entry surface has been similarly etched over its upper half. That segment of the spectrum lines marked AO came from the part of the crystal with one unetched face. The segment OB came from the region with both faces etched. The etching has caused the intensified surface reflection to disappear completely. The fainter reflection from the interior of the crystal can now be seen as a uniform band and in the region AO it is bounded on one side only by the enhanced reflection from the single remaining unetched part of the entry face. In the region OB each line can be seen spread out as a uniform band of reflection from the crystal interior (region, OAB, Fig. 7 previous paper) with no enhancement at the edges. The apparent width of the enhanced region is mainly caused by the finite width of the spectrometer slit and should not be understood as indicating any great depth for the crystal region from which the enhanced reflection comes. This exposure was taken with the plate in the nearer of the two plate holders and there is, therefore, some slight overlapping of the bands from $K\alpha_1$ and $K\alpha_2$. The band from $K\beta_{1,3}$ is also clearly visible in the original. Also because of the closer plate distance several of the fluctuations of intensity along the length of all the enhanced surface lines are visible in the original in the region AO. These fluctuations are completely absent for the reflection from the interior of the crystal (segment OB). The reader is asked to disregard the accidental scratch in the region OB of the alpha-lines. The scale indicates the magnification.



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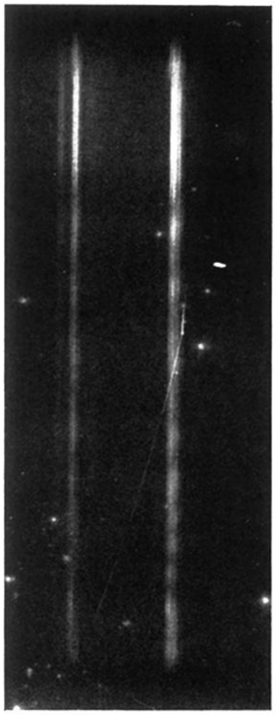

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