EFFECT OF TEMPERATURE ON THE REGULAR REFLECTION OF X-RAYS FROM ALUMINUM FOIL

By E. H. Collins

Abstract

Effect of temperature on the reflection of x-rays from rolled Al foil.— The powder method of analysis was used, a coil of the foil being placed at the center of the spectrometer in an electric furnace provided with openings for the x-rays. Intensity measurements for the scattered rays, wave-length .710 A, were obtained with an ionization chamber, for temperatures 80°, 310° and 600°C. The scattering curve for 80°C shows peaks at 17.4°, 20°, 28.7° and 33.8° (for planes 111, 100, 110 and 311). These are displaced for the higher temperatures because of the expansion, and are also decreased in intensity, the ratios for I_{600} to I_{80} being respectively .86, .815, .56 and .71. These ratios are considerably less than those predicted by the Debye theory, .94 to .81.

Relative spacing of crystal planes in aluminum rod and foil.— The angles of the peaks are from 0.4° to 0.7° less for the foil than for the rod, showing that the planes in the foil are about 1.5 per cent farther apart as a result of the rolling. This difference is not decreased by heating to 600°C.

1. Introduction

A THEORETICAL study of the change in intensity of general scattered and regularly reflected x-rays with a change in temperature of the scattering material has been made by Debye[1] and by Darwin.[2] Experimentally this effect has been investigated by Bragg,[3] Jauncey[4] and Blackhurst.[5]

This paper is a report of an experimental study of the change of intensity of x-rays regularly reflected from a cylinder of aluminum foil at temperatures 80°, 310° and 600°C.

2. Apparatus and Method

The x-ray spectrometer used was the instrument described by Hewlett.[6] It employed the powder method of x-ray analysis, the relative intensities of the scattered x-rays being measured by an ionization

[1] Debye, Ann. der Physik **43,** 49-95 (1914)
[2] Darwin, Phil. Mag. **27,** 325 (1914)
[3] Bragg, Phil. Mag. **27,** 881 (1914)
[4] Jauncey, Phys. Rev. **20,** 421 (1922)
[5] Blackhurst, Proc. Roy. Soc. **102,** 341 (1922-23)
[6] Hewlett, Phys. Rev. **20,** 690 (1922)

chamber and electrometer. The scattering material was a cylinder, diameter 8 mm, made by a roll of aluminum foil, the amount used being the optimum for maximum reflection.

A furnace of special design, shown by the diagram Fig. 1, was constructed for heating the aluminum. The rotating aluminum cylinder *A* was enclosed in a pocket made from asbestos board with heating strips wound in spiral form at the top and bottom of the pocket. An open space all around the furnace was left for the entrance and exit of the x-rays. The dimensions of the furnace were such that the secondary scattering and tertiary radiation reaching the ionization chamber from the materials of the furnace were reduced to a minimum.

To determine the temperature of the aluminum a preliminary experiment was first performed to measure the difference in temperature between a point *T*, Fig. 1, just above and outside the cylinder and a

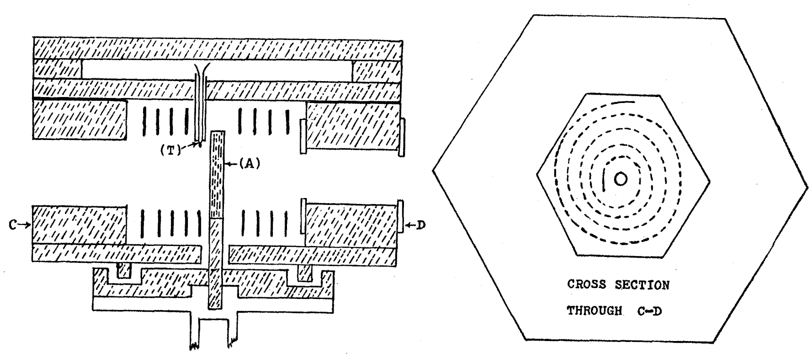

Fig. 1. Vertical and horizontal sections through furnace.

point within the cylinder itself. When observations were in progress it was only necessary to measure the temperature at that point *T* and subtract the difference observed in the preliminary experiment to obtain a very close approximation to the actual temperature of the aluminum. Hoskins chromel-alumel thermocouples and a sensitive millivoltmeter were used in the temperature measurements. The low temperatures were checked with a mercury thermometer.

The intensity of the scattered x-rays was taken as proportional to the rate of deflection of the electrometer. From this rate taken with the aluminum in place was subtracted the rate with the aluminum removed. The latter reading represented the drift of the electrometer plus any scattering of x-rays from the furnace and surrounding objects reaching the ionization chamber.

In obtaining the data for the scattering curves the following method was used: The intensity of the scattered x-rays from the aluminum cylinder was determined at 80°C over the angular region desired, then the aluminum was heated to the higher temperature (310°C or 600°C) and the observations of intensities again taken over the same angular region; finally the procedure was repeated for aluminum at 80°C. The average of the two intensities at 80°C was plotted on the same set of axes as the intensity for the scattered x-rays at the higher temperature. This procedure was necessary because in the interval of time (about four hours) necessary to take the data for one of these curves the density of methyl bromide gas in the ionization chamber changes slightly. The temperature 80°C was used to save time because a relatively much longer time was required for the aluminum cylinder to cool from 80°C to room temperature due to the presence of the furnace than for the aluminum to cool from 310°C or 600°C to 80°C.

3. Experimental Results

The experimental results are shown graphically by curves I, II, III, IV, Fig. 2 and curve V, Fig. 3 which represent the intensity of the scattered radiation plotted against angle from the incident beam. The arbitrary unit of intensity is not the same in the curves I, II, III, IV because of changes in the density of the methyl bromide gas.

4. Comparison with the Debye Theory

For the purposes of comparison of the experimental results with Debye's theory, Debye's formulas are put in the following form:

$$\log\left(\frac{I_1}{I_2}\right) = 418.\left[\left(1-\cos\phi_1\right)\frac{\varphi\ (x_1)}{\Theta\ x_1} - \left(1-\cos\phi_2\right)\frac{\varphi\ (x_2)}{\Theta\ x_2}\right]$$

for no zero point energy; and a similar formula with $\varphi(x_1)/x_1$ replaced by $(\varphi(x_1)/x_1+1/4)$, and $\varphi(x_2)/x_2$ replaced by $(\varphi(x_2)/x_2+1/4)$ for zero point energy.

Where I_1 = intensity of reflected x-ray at a high temperature;

φ_1 = angle of observation at a high temperature;

x_1 = ratio of the characteristic temperature Θ of the substance to the absolute temperature T.

$\varphi(x_1)$ is a function of Θ/T which Debye evaluates. The letters with the subscripts 2 have a similar meaning for a lower temperature.

A comparison of the observed and calculated ratios of the intensity of regularly reflected radiation at high temperatures to the intensity of the regularly reflected radiation at low temperatures is shown by Table I. The "observed" intensity of the regular reflection is proportional to the length of the ordinate at the crest of the peak minus the length of a portion due to the general scattered radiation. The value of the portion subtracted is found by plotting the intensities at each side of the peaks as shown, then interpolating graphically for the value of the intensities

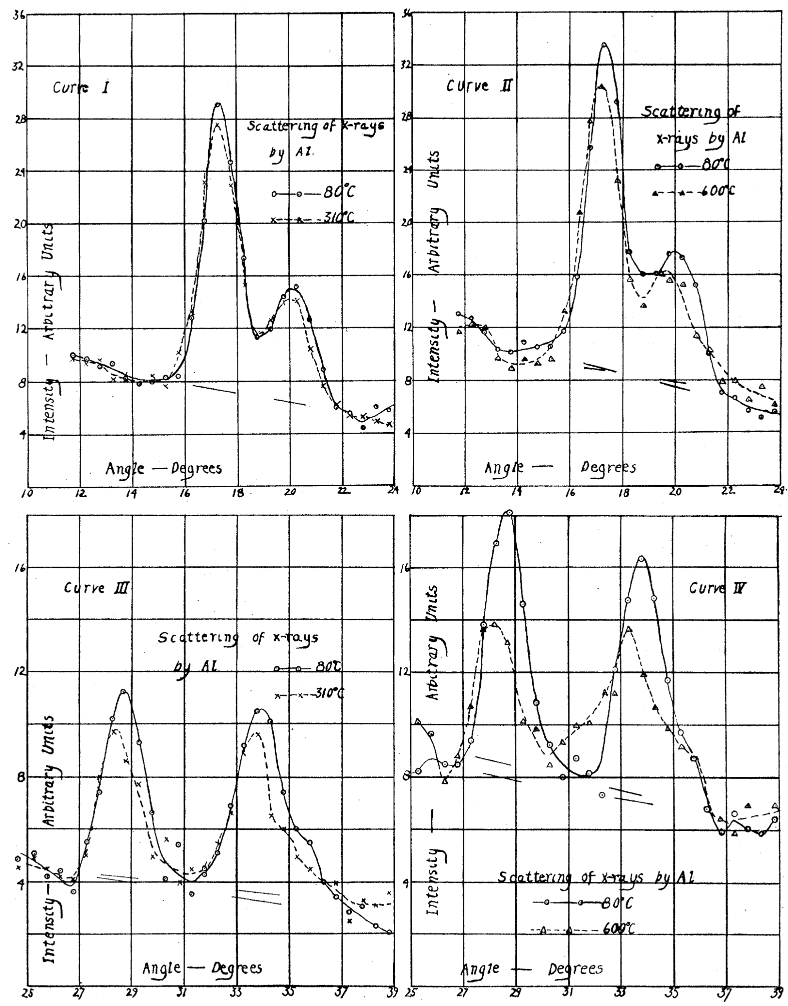

Fig. 2. The temperature effect on the regular reflection of of x-rays from aluminum foil.

of the general scattered radiation just under the peaks (see the straight lines directly under the peaks).

In Table I the subscripts indicate the temperature, I the intensity and φ the angle of regular reflection. This table and the scattering curves of Fig. 2 show that there is not satisfactory quantitative agreement with

Debye's theory. The decrease in intensity ratio with increase in temperature is markedly larger than that predicted by Debye's theory. Again the decrease in intensity ratio with increase in angle is larger than that predicted by Debye's theory, and the increase in ratio with angle is contradicted. Debye's theory is therefore inadequate.

TABLE I

Comparison of experimental and theoretical results

φ_{80}	Crystal planes	I_{310}/I_{80} obs.	I_{310}/I_{80} calc.*	I_{310}/I_{80} calc.†	I_{600}/I_{80} obs.	I_{600}/I_{80} calc.*	I_{600}/I_{80} calc.†
17.4	111	.92	.973	.976	.86	.936	.942
20.	100	.88	.966	.967	.815	.926	.928
28.7	110	.77	.935	.936	.56	.857	.857
33.8	311	.84	.913	.914	.709	.809	.809

* Assuming zero point energy.
† Assuming no zero point energy.

5. DISCUSSION OF DETAILS

It is difficult to draw a definite conclusion in regard to the general scattered radiation, because its intensity is too small in comparison with other sources such as the regular reflection of other wave-lengths which get through the zirconium oxide filter.

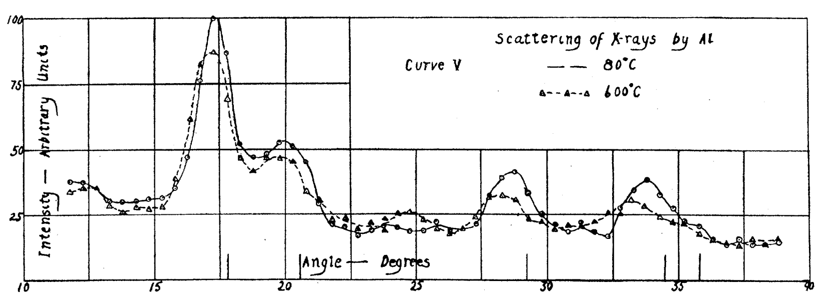

Fig. 3. The relative height and positions of the interference maxima at 80° and 600°C.

Due to the expansion of the crystal, and the resulting change in grating space, the maxima at high temperatures are shifted toward the small angle side. The observed shifts of the maxima are in good agreement with the calculated values.

Curve V, Fig. 3 is a composite of curves II and IV, Fig. 2. This shows the relative heights of the interference maxima.

A discrepancy exists between the angles of the regularly reflected x-ray beams obtained by A. W. Hull[7] and the angles obtained in this experiment. The angles obtained by Hull are indicated by the short lines ruled on the angle axis of curve V. This means that the crystal structure of aluminum was changed, the distance between the planes being increased, by rolling the aluminum into foil. The scattering from an aluminum rod gave results which were in agreement with Hull's values; this shows that the difference between the values here obtained and those of Hull is not due to experimental error. This effect has been noticed by J. Czochralski.[8]

In conclusion the writer wishes to express his appreciation to the Physics Department of the State University of Iowa for valuable assistance, especially to Professor G. W. Stewart under whose direction the work was carried on, and also to acknowledge indebtedness to Dr. C. W. Hewlett, now of the General Electric Company, whose x-ray spectrometer was used.

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April 12, 1924.

[7] Hull, Phys. Rev. **10**, 661 (1917)
[8] Czochralski, Science Abstracts, No. 2035, 1923.