Redetermination of the Temperature Dependence of X Rays Anomalously Transmitted through Germanium

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We have redetermined the temperature dependence of the anomalously transmitted x rays through single crystals of germanium of effective thicknesses, $\mu_0 t = 20$ and 30. We have established, as suggested by Okkerse, that the earlier measurements were subject to error due to a thermal gradient perpendicular to the diffracting planes. By suitable rearrangement of the heating geometry this type of gradient was substantially removed. The current results are in agreement with those of Okkerse and indicate that the data can be explained by allowing the imaginary part of the scattering factor to depend on temperature as $\exp(-M)$ where M is based on a Debye temperature of 291°K. It is felt that there is insufficient evidence at present to conclude in general that the temperature dependence in dynamical diffraction is $\exp(-M)$.

In a previous publication[1] we reported measurements on the temperature dependence of the anomalous transmission of x rays in germanium single crystals. We obtained a linear dependence of the logarithm of the integrated intensity with temperature ranging over an order of magnitude in intensity. [Reference to this work will be abbreviated as (I).] The data in (I) were fitted to theory assuming that $\epsilon(=F_H''/F_0'')$, the normalized angular dependence of the imaginary part of the scattering factor, could be expressed as $\epsilon = \epsilon_0 \exp[-(\alpha M)]$ where ϵ_0 is the static value, M is the exponent in the usual Debye-Waller factor e^{-M} and α is an adjustable constant. It was found that the data for the (220) and (400) reflections were consistent with a single $\alpha = 1.32$.

In a symposium associated with the 50th anniversary celebration of the discovery of x-ray diffraction, Okkerse[2] presented experimental data indicating a similar logarithmic dependence but with an α value of 1.0 based upon a Debye temperature of 291°K.

To resolve the experimental discrepancy, we repeated our past measurements with several variations. In the first series of experiments the crystals were mounted in the same manner as before (crystal in the shape of the letter T, held by the vertical arm) as in (I), Fig. 4, but with the crystal extended in the furnace so that both the forward diffracted (anomalously transmitted) and diffracted beams could be measured. (Previously only the diffracted beam was measured.) The experiment was repeated with the x-ray beam striking different portions of the surface of the crystal. The results of these measurements were: (1) the slopes of log intensity versus temperature of the diffracted beams were constant for different points on the crystal surface to within 5%, but did not agree in value with the previous measurements for a crystal of the same thickness, (2) the curves of the forward diffracted beam were not linear but curved toward lower intensities at higher temperatures such that at sufficiently high temperatures, the forward beam had less intensity than the diffracted beam. The amount of curvature depended upon where the beam struck the crystal surface.

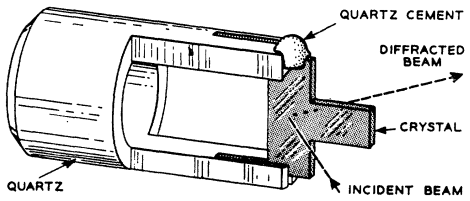

FIG. 1. Details of crystal mount. The crystal is attached on only one arm of the yoke.

These results strongly indicated the presence of thermal gradients even though the slopes of the diffracted beam curves (which were measured in our previous paper) did not vary with beam position on the crystal.

We subsequently repeated our previously reported measurements on the (400) reflection on the same crystal and experimental setup as in (I). The results reproduced those in Fig. 4, Ref. 1, i.e., they showed the curvature when the beam was near the supported part of the crystal, and a linear plot of the same slope as before, when the beam was at the center of the tee-shaped crystal. However, when the beam was moved still further from the support, the plot remained linear, but had a progressively smaller slope the further the beam was from the quartz-supporting rod. This definitely proved that the results in (I) were in error because of a thermal gradient due to heat flow down the supporting rod. This substantiates Okkerse's[2] hypothesis concerning the results in (I) that a linear dependence of the log of intensity versus temperature is not the correct criterion for a strain gradient-free crystal. In this arrangement heat flow down the quartz-support rod produced a gradient perpendicular to the diffracting planes which strongly affected the integrated intensities.

To avoid this difficulty we now mounted the crystal as in Fig. 1. Any gradient caused by heat flow down the supporting rod would now be parallel to the diffracting planes and should have a minimal effect on the integrated intensity. As before, the germanium (220) and

[1] B. W. Batterman, Phys. Rev. **126**, 1461 (1962).
[2] B. Okkerse, Philips Res. Rept. **17**, 464 (1962).

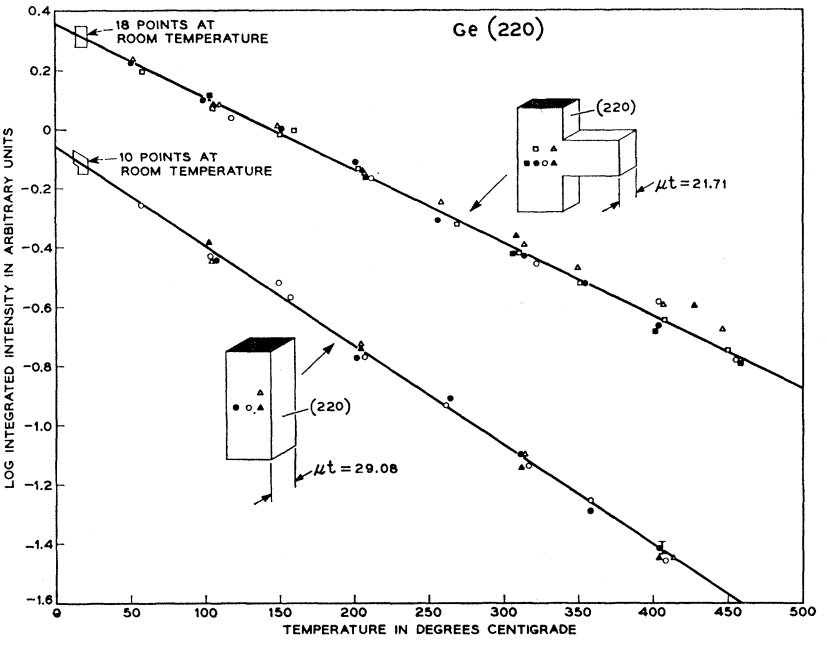

FIG. 2. (220) temperature dependence for two crystals of $\mu_0 t = 21.71$ and 29.08. The insets show the crystal shape and beam position. The shading indicates the mounting face of the crystal.

(400) anomalously diffracted beams were investigated using Cu K_α radiation. Two new specimens oriented for (220) and one new specimen for the (400) were prepared as described in (I). The (400) used in (I) was still available and made the fourth sample. The crystal thicknesses were such that for each reflection we investigated the region of $\mu_0 t \approx 20$ and ≈ 30.

The quantitative measurements were performed in the same manner as in (I), the only variation being the new manner in which the crystals were held (Fig. 1). The data for the two reflections are presented graphically in Figs. 2 and 3. The insets show the positions of the intersection of the primary beam with the crystal surface. This beam had a rectangular cross section of 0.5×0.5 mm^2. The intensity values for the various positions are included in the same plot. The intensities for the different points were taken generally in a random sequence. After measurements at several temperatures for any one crystal position, a room temperature check was made.

The internal consistency of the data for the many beam positions for each crystal gives strong evidence that thermal gradients are not affecting the measurements.

The slope of the curves is related to α of the assumed Debye-Waller factor, $e^{-\alpha M}$, by Eq. (9) in (I) which is repeated below for convenience:

$$\text{slope} = -(1-\alpha/2)k - \mu_0 t \epsilon_0 \alpha k e^{-\alpha M},$$

where $k = M/T$ and M is based on an x-ray Debye temperature for germanium of 291°K.[3] Table I gives the parameters and resulting values for α. The static values, ϵ_0, are very close to unity. For the computation we took Wagenfeld's[4] theoretical values, $\epsilon_0(220) = 0.993$ and $\epsilon_0(400) = 0.985$.

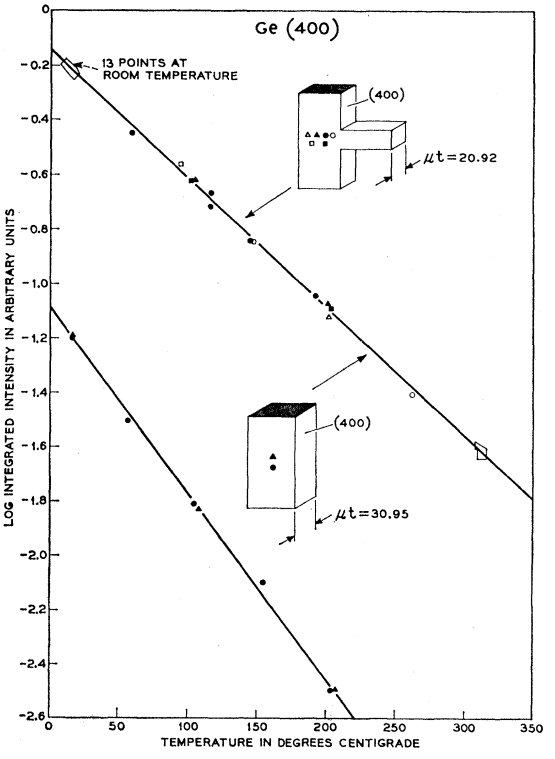

FIG. 3. (400) temperature dependence for two crystals of $\mu_0 t = 20.92$ and 30.95. Details as in Fig. 2.

[3] B. W. Batterman and D. R. Chipman, Phys. Rev. **127**, 690 (1962).

[4] H. Wagenfeld, J. Appl. Phys. **30**, 2907 (1962).

TABLE I. Experimental slopes from Figs. 2 and 3 and corresponding values of α.

Reflection	μt	Slope	α
220	21.71	-2.48×10^{-3}	0.99_9
220	29.08	$-3.39_5\times10^{-3}$	1.02_7
400	20.92	$-4.65_6\times10^{-3}$	1.02_7
400	30.95	-6.85×10^{-3}	1.01_8

It can be seen from the table that a single value of $\alpha=1.01\pm0.015$ will fit the measurements for the four crystals. This is in contradiction to the results in (I) which were subject to error due to thermal gradients in the specimens perpendicular to the diffracting planes. These results now substantially agree with Okkerse's and indicate that Debye-Waller factor for germanium diffracting in the symmetric Laue case is very close to e^{-M}. This, coupled with the same experimental temperature dependence for symmetric Bragg reflection,[5] suggests that in general the Debye-Waller factor for perfect crystals is e^{-M}. This should not, however, be taken as proof of its general validity without further experiments and the development of a sound theoretical treatment of thermal vibrations in an absorbing perfect crystal.

[5] B. W. Batterman, Phys. Rev. **127**, 686 (1962).

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Kinetic Secondary Electron Ejection from Tungsten by Cesium Ions*

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Initial measurements of the number of electrons ejected per incident ion, γ_i, have been made for medium-energy (1–21 keV) cesium ions at normal incidence on a clean polycrystalline tungsten surface. The residual gas pressure was $\lesssim5\times10^{-9}$ Torr. The tungsten surface was cleaned by prolonged bakeouts and by flashing before each measurement. A dc ion-beam pulse method of measurement was used to prevent cesium coverage of the tungsten surface. In the energy range of 3–21 keV γ_i is a linear function of the ion kinetic energy with $\partial\gamma_i/\partial E\simeq0.04$ electron per ion per keV. At 3 and 21 keV γ_i is 0.02 and 0.74, respectively. If the data are extrapolated to $\gamma_i=0$, a threshold energy of 2.5 keV is obtained. Below this energy the measured values of γ_i were equal to zero within the accuracy of the measurement. The change of γ_i with cesium adsorption on the tungsten was also recorded.

USE of cesium as propellant in ion propulsion has increased the interest in the interaction of medium-energy cesium ions with solids. One of the interactions in which there has been a basic as well as a practical interest is that of the kinetic ejection of electrons from metals due to the bombardment of cesium ions. In the energy range of interest, 1–20 keV, there is little published data. Waters,[1] measured secondary electron yields for low-energy (150–1500 eV) cesium and lithium ions on tungsten. Arifov *et al.*,[2] and Petrov[3] have published data for alkali ions up to 10 keV impinging on various metals. In the energy range of 1–4 keV Brunnee[4] has measured γ_i for the alkali ions on molybdenum.

No close agreement exists among published values. This paper presents the measured yields of secondary electron emission from clean polycrystalline tungsten by normally incident cesium ions of energies of 1–21 keV. Since the ionization energy of cesium, 3.87 eV, is less than the work function of clean tungsten, about 4.5 eV, the electron yields presented are due to the kinetic ejection process only and not to the Auger or potential-ejection[5] process. The apparatus used is shown in Fig. 1.

The vacuum chamber is a stainless-steel cylinder, 3 ft in length with a diameter of 1 ft. It is pumped by a liquid-nitrogen trapped, 6-in. silicon oil diffusion pump. The chamber can be baked to 300°C with either an oven or strap heaters. Copper gaskets are used for all flanges. Within the tank a liquid-nitrogen liner is used to freeze out all condensibles. It can be baked out by passing hot gas through it. Pressure is measured with a Bayard-Alpert type ionization gauge. The system is capable of pressures lower than 1×10^{-9} Torr if proper care and technique are used.

The cesium ions are produced by a surface-ionization ion source using a porous tungsten ionizer operating at 1000–1200°C.[6] This source can generate up to 5 mA of cesium ion current with a maximum current density at the target of approximately 1–2 mA/cm^2. The fraction of neutral cesium atoms for the currents used in this

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[2] U. A. Arifov and R. R. Rakhimov, Transactions of the Ninth All Union Conference on Cathode Electronics, Moscow, 1959 (unpublished), p. 666.
[3] N. N. Petrov, Fiz. Tverd. Tela **2**, 949 (1960) [English transl.: Soviet Phys.—Solid State **2**, 865 (1960)].
[4] C. Brunnee, Z. Physik **147**, 161 (1957).
[5] H. Hagstrum, Phys. Rev. **96**, 336 (1954).
[6] G. Kuskevics and B. Thompson, AIAA J. **2**, 284 (1964).