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Effect of Compton Scattering on the Borrmann Effect of X-Rays in Silicon Crystals

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Dedicated to Prof. G. Borrmann on his 65-th birthday

The absolute integrated intensities diffracted in anomalous transmission through thick, nearly perfect crystals of silicon were measured for AgKα and MoKα wavelengths and for the {220} reflection, at room and liquid nitrogen temperatures. There is good agreement between experimental values and those calculated by using the formulas of the dynamical theory of diffraction, provided the contribution of Compton scattering μ_C^* is included in the effective absorption coefficient μ^*. μ_C^* is a considerable fraction of μ^* (from 23 to 55 per cent) for the two wavelengths and temperatures used in the present work. The experimental values of μ_C^* agree well with those calculated by using the theory of the Compton contribution to the dynamical absorption coefficient of X-rays. A simple formula which is a good approximation of the rigorous expression of μ_C^* is also given. The Debye temperature Θ of Si was derived from the experimental dependence of the intensities on crystal thickness for the {220} reflection at room and liquid nitrogen temperatures by using CuKα radiation. It was found that $\Theta = 521 \pm 5$ and 543 ± 5 °K at 295 and 77 °K, respectively, in agreement with the results of other authors.

I. Introduction

In a previous study measurements of X-ray intensities diffracted in anomalous transmission (Borrmann effect) through nearly perfect crystals of Cu were found to be in good agreement with the intensity values calculated by means of the dynamical theory of diffraction [1]. The diffracted intensities in anomalous transmission depend greatly on the effective absorption coefficient μ^*, that is, on the difference between the average and the dynamical absorption coefficients. Therefore, as Prof. Borrmann suggested many years ago [2], it is important to evaluate the contributions of the various X-ray scattering processes to μ^*. In the case of Cu crystals only the photoelectric effect contributes significantly to absorption at the X-ray wavelengths used in diffraction. However, this fact is not generally valid: the relative importance of the Compton scattering contribution to the absorption of X-rays increases with a decrease in the atomic number or the wavelength of X-rays. Experiments on the anomalous transmission of MoKα radiation ($\lambda = 0.711$ Å) through nearly perfect crystals of silicon have shown that there is a discrepancy between experimental and calculated values of the diffracted intensities when only photoelectric absorption is included in the formulas of the dynamical diffraction theory. The measured intensities, diffracted at room temperature by the {220} planes of a thick Si crystal ($\mu_0 t \approx 50$), were about half the calculated ones [3]. Although the photoelectric effect accounted for more than 96% of the total average absorption coefficient, it was suggested that the origin of the discrepancy be in the neglect of the Compton scattering contribution to μ^*. In fact, the contribution of photoelectric absorption to the static part of the dynamical absorption coefficient is nearly independent from the scattering angle, the K-shell electrons being responsible for most of the absorption. The Compton contribution instead, should rapidly decrease with the scattering angle because the outside electrons of the atom are responsible for a great part of the Compton scattering.

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The purpose of the present work, the results of which were previously presented in abstract form [4], is a systematic investigation concerning the effect of Compton scattering on the {220} intensities of the AgKα and MoKα radiations diffracted in anomalous transmission through thick Si crystals. The Compton contribution to μ^* is derived from the experimental data of the integrated intensities and compared with the calculated values.

II. Experimental Procedure

The experimental method for the measurement of the integrated intensities was the same used in previous experiments. The MoKα and AgKα radiations were monochromatized by reflection on curved quartz and LiF crystals, respectively. The LOPEX Si crystals were cylindrical bars of various lengths (from about 2.2 to 7.3 cm) either phosphorus or boron doped (purchased from the Texas Instrument Inc.) with the cylindrical axis parallel to the $\langle 111 \rangle$ direction. They had a resistivity greater then 100 Ω cm and a dislocation density less than 1000 lines per cm^2. The perfection of the crystals was checked in a few cases as follows. Thin slices were cut from the bars and absolute integrated intensities of the {220} reflection in the symetrical Laue geometry, and of $\{h\,h\,h\}$-type reflections in the symmetrical Bragg geometry, were measured by using CuKα and MoKα radiations. In the Laue geometry, the lamellas behaved as thin crystals with MoKα radiation and as thick crystals (anomalous transmission) for CuKα radiation. There was always good agreement between the experimental data and the values calculated by using the formulas of the dynamical diffraction theory. X-ray topographs, taken with he Lang method, showed that the crystals were usually free of dislocations. However, pictures of the Borrmann beams with crystals of a thickness equal to about 2.5 cm, did not show an uniform darkening of the spots.

The integrated intensities of the two diffracted {220} (reflected and transmitted) beams were measured by rotating the counter rigidly fastened to the crystal. Two NaI scintillation counters were used with single-channel pulse-height analyzers. One of them monitored the X-ray radiation incident on the Si crystal: a small part of this radiation was scattered into the counter by a thin plexiglass or aluminium sheet inserted in the incident beam. Absolute values of the integrated intensities were obtained by measuring the intensity of the incident beam attenuated by calibrated Al filters. Contributions of half-wavelength radiation to the intensity measurements of the diffracted and incident beams were avoided by operating the X-ray tubes at suitable voltages; the purity of the diffracted radiation and of the attenuated incident beam was checked in various cases by a multichannel analyzer.

A precision better than $\pm 1\%$ and $\pm 4\%$ was achieved in the relative and absolute intensities, respectively. There were small differences, in some cases equal to a few percent, among the integrated intensities of the three {220} planes parallel to the $\langle 111 \rangle$ direction of the Si bar. The arithmetic average of six intensity measurements (one for each side of the three planes) was take for every crystal. The intensities were measured at room and liquid nitrogen temperatures. In the last case a double-wall cryostat was used with the crystal in a helium atmosphere in order to avoid appreciable temperature gradients across the length of the crystal.

The average absorption coefficients for MoKα and AgKα radiations were measured by using a few Si crystal slices. These lamellas, which reduced the X-ray intensity by a factor of 2 to 5, were inserted in the beam diffracted by a Si crystal. Many measurements were taken with different slices and on various regions of the same slice; the experimental results were then averaged out. The thicknesses of the slices were measured by using a sensitive capacitance bridge with an accuracy of $\pm 2\,\mu$. For each slice the thickness taken was equal to the average of many measurements done inside the area used in the experiment. The experimental values of the absorption coefficient are reported in Table 1 together with the calculated ones (see the following paragraph for the calculations). There is good agreement between the present experimental values and those reported by Hildebrandt and Wagenfeld [5].

III. Calculations of Average and Dynamical Absorption Coefficients

The expression for the contribution of Compton scattering to the dynamical absorption coefficient of X-rays was determined by Ohtsuki and co-workers [6]. Although reference is made to that paper for the theoretical treatment of this problem, we report here a brief formal derivation of the pertinent formulas used to calculate the Compton scattering contribution to the average and dynamical absorption coefficients.

The local absorption cross section $\sigma^{c\,\dagger}(\boldsymbol{r})$ for Compton scattering is given by the following expression:

† Both indices-c and C-stand for "Compton".

$$\sigma^{c}(\boldsymbol{r}) = 4\pi \sum_{i} \varrho_i(\boldsymbol{r}) - \sum_{ij} \int f_{ij} \frac{\mathrm{d}f_{ij}}{\mathrm{d}\boldsymbol{r}} \mathrm{d}\boldsymbol{k} \qquad (1)$$

where $\varrho_i(\boldsymbol{r})$ is the electric charge density due to the i-th electron of the atom and the sum is extended to all the atomic electrons;

$$f_{ij} = \int \psi_i^*(\boldsymbol{r})\, \psi_j(\boldsymbol{r}) \exp\{2\pi i\, \boldsymbol{k}\, \boldsymbol{r}\}\, \mathrm{d}\boldsymbol{r};$$

$\psi_i(\boldsymbol{r})$ and $\psi_j(\boldsymbol{r})$ are electron wave functions; $\boldsymbol{k}$ is the scattering vector and the integral in formula (1) is extended to the entire surface of the Ewald sphere.

The Compton cross section $\sigma^{c}(\boldsymbol{r})$ is a periodic function of the position in the crystal and can be expanded in a Fourier series:

$$\sigma^{c}(\boldsymbol{r}) = \sum_{H} \sigma_H^{c} \exp\{2\pi i\, \boldsymbol{k}_H\, \boldsymbol{r}\}$$

where $\boldsymbol{k}_H$ is the scattering vector corresponding to the reflection $H \equiv h, k, l$. The expression of σ_H^{c} is:

$$\begin{aligned} \mu_0^{c} &= \sum_{n} \int \varrho_n(\boldsymbol{r}) \exp\{2\pi i\, \boldsymbol{k}_H\, \boldsymbol{r}\}\, \mathrm{d}\boldsymbol{r} \\ &\quad - \sum_{m,n} \int\int f_{mn} \frac{\mathrm{d}f_{mn}}{\mathrm{d}\boldsymbol{r}} \exp\{2\pi i\, \boldsymbol{k}_H\, \boldsymbol{r}\}\, \mathrm{d}\boldsymbol{k}\, \mathrm{d}\boldsymbol{r} \qquad (2) \\ &= f_H - \sum_{m,n} \int f_{mn}(\boldsymbol{k})\, f_{mn}(\boldsymbol{k}+\boldsymbol{k}_H)\, \mathrm{d}\boldsymbol{k} \end{aligned}$$

where f_H is the value of the atomic scattering factor for the reflection H.

The expressions for the Compton contributions to the average and dynamical absorption coefficients are then:

$$\mu_0^{c} = 2\pi N r_0^2 \lambda^2 \int_0^{1/\lambda} P(K)\, (1+\lambda_c \lambda K^2/2)^{-2} \times (Z - F(0))\, K\, \mathrm{d}K, \qquad (3)$$

$$\mu_H^{c} = N r_0^2 \lambda^2 e^{-M_H} \int_0^{1/\lambda}\int_0^{2\pi} P(K,H)\, (1+\lambda_c \lambda K^2/2)^{-2} \times (f_H - F(H))\, K\, \mathrm{d}K\, \mathrm{d}\Phi$$

where: N = number of atoms per cmc; r_0 = classical radius of the electron; λ = wavelength of the X-ray radiation; λ_c = Compton wavelength; e^{-M_H} = the Debye-Waller factor for the reflection H; $P(K)$ and $P(K,H)$ are polarization factors; $(1+\lambda_c \lambda K^2/2)^{-2}$ is the Breit-Dirac recoil factor; Z and f_H are the atomic number and the atomic scattering factor for the reflection H, respectively; $F(0) = \Sigma |f_{ij}|^2$;

$$F(H) = \sum_{ij} f_{ij}(\boldsymbol{k}) \cdot f_{ij}(\boldsymbol{k}+\boldsymbol{k}_H).$$

The numerical values of μ_0^{c} and μ_{220}^{c} were calculated for Si in the cases of AgKα and MoKα characteristic radiations by using a computer. The values of the f_{ij} functions needed for the calculations were taken from the work of Freeman [7] and these functions were extrapolated by smooth curves whenever necessary.

Cross sections for the photoelectric absorption of AgKα and MoKα radiations were computed by using atomic hydrogen-like wavefunctions for both the bound and the continuum electronic states. For these calculations we used the inner screening constants of Slater for the effective atomic number $Z-s$ and the expressions of the electric dipole, quadrupole and dipole-octupol terms as reported by Wagenfeld [8]. The calculated values of the total photoelectric cross section (the sum of the contributions of the $K+L+M$ shells) agree to better than 1% with those tabulated by Hildebrandt, Stephenson and Wagenfeld [9]. The electrons of the K, L and M shells contribute in percent 0.97, 0.03 and 5×10^{-5} respectively, to the total cross section. Since the contribution of the M shell electrons is negligible even with respect to those of the Compton and thermal scatterings, it can be omitted with no influence on the considerations presented in this work.

The computed contributions to the average linear absorption coefficient by the photoelectric, Compton and thermal scatterings, are reported in Table 1. The contributions of the thermal scattering were evaluated by using formulas reported in a previous paper [10]. The total calculated absorption coefficients are in good agreement with the experimental values.

Table 1. Comparison between calculated and experimental values of the linear absorption coefficients in cm^{-1} of Si, for AgKα and MoKα radiations. The total calculated average absorption coefficient ($\mu_{0,\mathrm{TOTAL}}$) is the sum of the contributions of photoelectric (μ_{PH}), thermal (μ_{T}) and Compton (μ_{C}) scatterings.

	Calculated				Experimental
λ (Å)	μ_{PH}	μ_{T}	μ_{C}	$\mu_{0,\mathrm{TOTAL}}$	μ_0
0.561 (AgKα)	6.89	0.143	0.337	7.37	7.46 ± 0.10
0.711 (MoKα)	14.11	0.180	0.316	14.61	14.68 ± 0.12

The contributions of the photoelectric and thermal scatterings to the dynamical absorption coefficient for the perpendicularly polarized component of the X-ray radiation (the only one of interest for the present work) were computed by using the expressions given in pervious papers [1, 8, 10]. For the photoelectric part we used the electric dipole, dipole-octupol and quadrupole terms of the K and L electrons. The ratios ε_0 between the photoelectric dynamical and average absorption coefficients were 0.9975 and 0.9968 for the {220} reflection of the AgKα and

MoKα radiations, respectively. The contribution of Compton scattering to the dynamical absorption coefficient for the perpendicularly polarized component was calculated by using the second of the formulas (3).

We have reported in Table 2 the contributions of photoelectric (μ_{PH}^*), thermal (μ_T^*) and Compton (μ_C^*) scattering processes to the effective absorption coefficient μ^*, for the {220} reflection and the characteristic radiations AgKα and MoKα, at liquid

Table 2. Calculated contributions of photoelectric (μ_{PH}^*), thermal (μ_T^*) and Compton (μ_C^*) scatterings to the effective absorption coefficient μ^* for anomalous transmission in Si crystals, in cm^{-1}, for AgKα and MoKα radiations at liquid nitrogen and room temperatures. μ^* is the sum of the three contributions.

λ (Å)	T (°K)	μ_{PH}^*	μ_T^*	μ_C^*	μ^*
0.561 (AgKα)	77	0.116	0.006	0.158	0.280
	295	0.240	0.015	0.161	0.416
0.711 (MoKα)	77	0.247	0.012	0.157	0.416
	295	0.497	0.022	0.160	0.679

nitrogen and room temperatures. The Debye-Waller factor was included in the contributions of photoelectric and Compton scatterings to the dynamical absorption coefficient. We want to emphasize that the Compton contribution is 40% and 23% of the total for the AgKα and MoKα radiations, respectively, at room temperature. By comparison, this contribution is less than 5% of the total (only 2% in the case of MoKα radiation) in the case of the average absorption coefficient. At liquid nitrogen temperature the importance of the Compton contribution is even higher: 55% and 30% of the total effective absorption coefficient for the AgKα and MoKα radiations, respectively. As already mentioned in the introduction, the physical explanation of these facts is the following. Electrons of the K shell primarily contribute to photoelectric absorption. Then the atom acts as a point scatterer and the part of the dynamical absorption coefficient which is due to the photoelectric effect, depends on the scattering vector mainly through the Debye-Waller factor. For Compton scattering, instead, the outside electrons of the atom play an important role in the scattering process. Therefore, contribution to the dynamical absorption coefficient depends strongly on the scattering angle and is not much affected by the Debye-Waller factor. The consequence is that Compton scattering contributes greatly to the effective absorption coefficient even though it is only a small part of the average absorption coefficient. An approximate formula for μ_C^* can be obtained from formulas (3) by noting that $F(H)$ is a slowly varying function of the scattering vector. If we assume that $F(H) \approx F(0)$, it follows from a series development of the expressions under the integral signs:

$$\mu_C^* \approx \frac{8\pi N r_0^2}{3} \frac{\lambda^2}{(\lambda+\lambda_c)^2} (Z - f_H e^{-M_H}).$$

This approximate formula gives values which do not differ by more than 4% from those evaluated by means of formulas (3), for the cases of interest to the present work.

IV. Experimental Results and Discussion

The integrated intensities of the two X-ray beams, diffracted in anomalous transmission, are to a good approximation proportional to $t^{-\frac{1}{2}} \cdot \exp\{-\mu^* t\}$ for $\mu_0 t \geqq 20$, $t = t_0/\cos\vartheta$ being the effective thickness of the crystal in the direction of the incident beam. Since the Debye-Waller factor appears in the photoelectric and Compton parts of the dynamical absorption coefficient, it was opportune to know the value of the Debye temperature Θ of Si with precision. This temperature was determined again by following the procedure used by Kyutt and Efimov [10]. From the formula

$$\ln I + \tfrac{1}{2} \ln t = -\mu^* t + \text{const}$$

where I is the integrated intensity in anomalous transmission, it follows that the plot of the expression in the first member of the formula vs. t is a straight line, whose slope is equal to the effective absorption coefficient. The Debye-Waller factor and then the Debye temperature can be derived from the experimental determination of μ^*. Measurements were done by using CuKα radiation and the {220} reflection of various Si crystals cut from the same bar (thickness of the crystals were in the range of $\mu_0 t$ from about 20 to 60), at both room and liquid nitrogen temperatures. Te set of measurements at liquid nitrogen temperature is illustrated in the semilogarithmic plot of Figure 1.

The experimental values of μ^* were corrected for the contribution of the thermal scattering [10], the contribution of Compton scattering being negligible with CuKα radiation. By taking a calculated average absorption coefficient $\mu_0 = 141.3$ cm^{-1} for CuKα radiation, we found that Θ is equal to 521 ± 5 °K

at room temperature and 543 ± 5 °K at liquid nitrogen temperature, the error bar on the value of Θ being the probable error relative to the fitting of the experimental points by the least square method. Agreement with the results by Kyutt and Efimov [11] is good. In fact, if we take $\mu_0 = 146\ \mathrm{cm}^{-1}$ as the above mentioned authors did, our results give values of Θ equal to 533 and 559 °K at room and liquid nitrogen temperature respectively, compared with 535 and 549 °K at room temperature and 100 °K respectively, by Kyutt and Efimov.

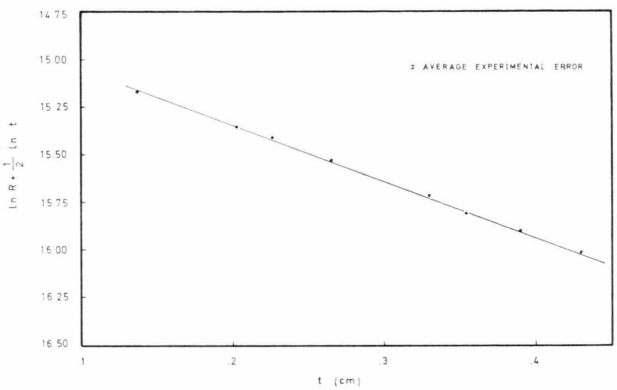

Fig. 1. Logarithm of the relative values of the integrated intensities R vs. the effective thickness of the crystal t for the {220} reflection of Si crystals, with CuKα radiation, at liquid nitrogen temperature. The Debye temperature Θ of the solid can be derived from the effective absorption coefficient μ^* which is equal to the slope of the line illustrated in the figure. By taking an average absorption coefficient $\mu_0 = 141\ \mathrm{cm}^{-1}$, $\Theta = 543 \pm 5$ °K at liquid nitrogen temperature.

Two different methods were used to obtain the contribution of Compton scattering to the effective absorption coefficient from the experimental data of the integrated intensities. In one case, integrated intensities were calculated by taking into account only the contributions of photoelectric and thermal scatterings to the effective absorption coefficient. These calculated values were then compared with the experimental absolute intensities. In fact, from the approximate formula for the integrated intensity, it follows that the contribution μ_C^* of the Compton scattering to the effective absorption coefficient is given by

$$\mu_C^* = \ln(R_{cal}/R_{exp})/t\,.$$

A few values of μ_C^* derived from the absolute experimental intensities with this method are illustrated in Table 3. The agreement with the calculated value of μ_C^* is quite satisfactory.

Table 3. Calculated and experimental values of absolute integrated intensities and of the Compton contributions μ_C^* to the effective absorption coefficient for Si crystals of various effective thicknesses ($\mu_0 t$), for AgKα and MoKα radiations at room and liquid nitrogen temperatures. The calculated values of the absolute intensities were computed by taking into account only photoelectric and thermal scattering contributions to the effective absorption coefficient μ^*. The experimental values of μ_C^* were obtained from the comparison of calculated and experimental values of the absolute intensities, as explained in the text.

$\mu_0 t$	λ in Å	T in °K	$R_{H(T)} \times 10^8$ Calc.	Exp.	μ_C^* in cm^{-1} Calc.	Exp.
55	0.561 (AgKα)	295	2.81	0.78	0.161	0.164
46			5.05	3.01		0.161
79	0.711 (MoKα)	295	1.12	0.47	0.160	0.157
112			0.285	0.082		0.159
46	0.711 (MoKα)	77	12.47	7.65	0.157	0.152

The second method is based on the dependence of the diffracted intensities on the thickness of the crystal. As explained above, the plot of $\ln I + \frac{1}{2} \ln t$ vs. t is a straight line, whose slope is equal to the effective absorption coefficient. Data referring to the relative intensities diffracted by 10 crystals of $\mu_0 t$ in the range from 30 to 112, with MoKα radiation at room temperature, are reported in Figure 2. After subtraction of the calculated contribution of

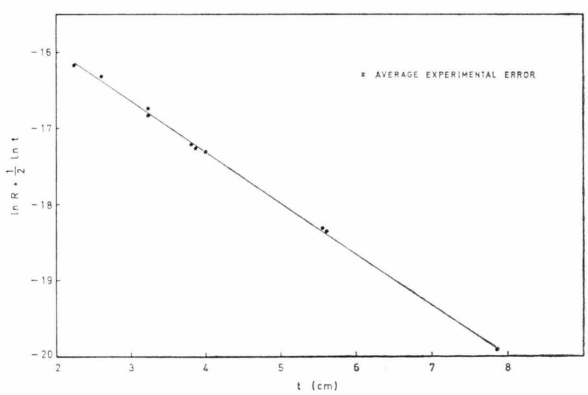

Fig. 2. Logarithm of the relative values of the integrated intensities R vs. the effective thickness of the crystal t for the {220} reflection of Si crystals, with MoKα radiation, at room temperature. The slope of the line illustrated in the figure is equal to the effective absorption coefficient μ^*. The contribution of Compton scattering μ_C^* is obtained by subtracting from μ^* the calculated contributions of photoelectric and thermal scatterings.

photoelectric and thermal scatterings we obtained $\mu_C^* = 0.15 \pm 0.01\ cm^{-1}$ from the slope of the line, in good agreement with the calculated value ($\mu_C = 0.160\ cm^{-1}$).

V. Summary

Absolute values of the {220} integrated intensities in anomalous transmission were measured for nearly perfect Si crystals varying in thickness from about 2.2 to 7.75 cm. MoKα and AgKα radiations were used at both room and liquid nitrogen temperatures. The contribution of Compton scattering μ_C^* to the effective absorption coefficient μ^* for anomalous transmission was derived from the experimental values of the intensities by using the two following methods:

a) μ_C^* times the effective thickness of the crystal is equal to the logarithm of the ratio between calculated and experimental intensities when the calculated intensities are computed by including only the contributions of photoelectric and thermal scatterings in μ^*;

b) if the logarithm of the intensities is plotted vs. the effective thickness of the crystal, the slope of the plot is proportional to μ^*. μ_C^* is obtained by subtracting from μ^* the calculated contributions of thermal and photoelectric scatterings.

Values of μ_C^* were computed by means of the formulas for the contributions of Compton scattering to the average and dynamical absorption coefficients. The agreement between the calculated and experimental values of μ_C^* is quite good. We also give a simple formula which approximates well the correct but complex expressions of μ_C^*.

In order to have a precise value for the Debye-Waller factor used for the calculation of the integrated intensities in anomalous transmission, we determined again the Debye temperature Θ by measuring intensities of CuKα radiation diffracted in anomalous transmission through Si crystals of various thicknesses. The values of Θ, derived from the plot of the logarithm of the diffracted intensities toward crystal thickness, were $521 \pm 5\ ^\circ K$ and $543 \pm 5\ ^\circ K$ at room and liquid nitrogen temperatures, respectively. These are in good agreement with the results reported by other authors.

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