

expected that the initial effect of pressure on sulfur will be a decrease in its energy gap to such an extent that thermal excitation of large numbers of electrons into the conduction band will occur at room temperature, making it a semiconductor. Then, at pressures above that necessary to produce this semiconducting state, a discontinuous increase in conductivity indicative of a phase change to a metallic state would be expected. Such an effect of pressure on sulfur has not been observed. However, Slykhouse and Drickamer⁴⁶ have shown by optical studies that the energy gap of sulfur decreases with increasing pressure as expected.

The calculations using the theoretical expressions for

Z_m and K_{el} , and the measured values of E_g point to the desirability of measurements of the thermal conductivity of tellurium as a function of pressure and temperature. These measurements would permit a direct comparison of the theory and experiment for K_{el} , testing the general validity of the theoretical model.

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Absolute Intensities of X-Rays Anomally Diffracted through Nearly Perfect Copper Crystals*

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Absolute integrated intensities of the x-ray beams "anomalously transmitted" through nearly perfect copper crystals have been measured for the (111) planes for values of μt ranging from 10 to 90. Results obtained for both the symmetric and asymmetric Laue geometries are compared with theory. These comparisons yield a value of 0.965 ± 0.003 for the ratio $I''(111)/I''(000)$ and indicate that the crystals investigated have a high degree of perfection. The measurements were made with Mo $K\alpha$ radiation using a double-crystal spectrometer arranged in the parallel condition and with the first crystal diffracting in the Bragg geometry. Measured profiles of the Bragg-Laue double-crystal rocking curves for $\mu t = 10$ and 90 are in semi-quantitative agreement with the theory.

INTRODUCTION

IT has been demonstrated recently¹ that large copper crystals having low dislocation densities ($\lesssim 10^4/\text{cm}^2$ as determined by etch-pit count) appear to be nearly perfect with respect to their x-ray diffraction properties. Such crystals exhibit anomalous transmission of x rays when set to diffract in the Laue geometry and give diffraction peaks when examined in the Bragg geometry which have half-widths in agreement with the prediction of Darwin's² theory for perfect crystals.

In the present investigation further examination has been made of the x-ray diffraction properties of nearly perfect copper crystals. In particular, absolute integrated intensities and peak profiles of the anomalously transmitted x-ray beams obtained by diffraction from the (111) planes have been measured and compared with theory.

THEORY

The theory of the diffraction of x rays through thick, absorbing, perfect crystals (Laue geometry) has been thoroughly investigated by Zachariasen,^{3,4} Von Laue,⁵ Hirsch,⁶ Kato,⁷ and others. The geometry under consideration is shown in Fig. 1 and the situation may be described as follows. When the direction of an incident parallel beam makes the appropriate angle θ_B for diffraction from the planes (hkl), two plane waves of x rays, one in the incident beam direction and one in the diffracted beam direction, are coupled inside the crystal. Their interaction produces a standing wave electromagnetic field with nodes at the planes of the atoms. Thus, there is a reduction in photoelectric absorption which results in an increase in the intensity of transmitted x rays. At the exit surface of the crystal, the wave field then splits into two uncoupled beams, the transmitted and the diffracted beams. This effect is very

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¹ M. C. Wittels, F. A. Sherrill, and F. W. Young, Jr., *Appl. Phys. Letters* **1**, 22 (1962); **2**, 127 (1963); *Phys. Letters* **5**, 183 (1963).

² C. G. Darwin, *Phil. Mag.* **27**, 325, 675 (1914).

³ W. H. Zachariasen, *Theory of X-Ray Diffraction in Crystals* (John Wiley & Sons, Inc., New York, 1945).

⁴ W. H. Zachariasen, *Proc. Natl. Acad. Sci.* **38**, 378 (1952).

⁵ M. V. Laue, *Acta Cryst.* **2**, 106 (1949).

⁶ P. B. Hirsch, *Acta Cryst.* **5**, 176 (1952).

⁷ Nario Kato, *J. Phys. Soc. Japan* **10**, 46 (1955).

striking; we have observed x rays diffracted through copper crystals which would ordinarily reduce the intensity of an incident Mo $K\alpha$ x-ray beam by a factor of e^{-90} .

INTEGRATED INTENSITIES

The integrated intensities of the transmitted (R_T) and diffracted (R_H) beams are given by Kato⁷ for a perfectly parallel plane polarized x-ray beam as (with $h \gtrsim 6$):

$$R_T = \frac{K(e^2/mc^2)N\lambda^2}{\pi(|b|)^{1/2}\sin 2\theta_B} |F_H'| \left(\frac{\pi}{8h}\right)^{1/2} \times e^{-(\mu t - h)} \left\{ 2D_0 - 1 + \frac{2D_1 - 1/8}{h} + \dots \right\}, \quad (1)$$

$$R_H = \frac{K(e^2/mc^2)N\lambda^2}{\pi(|b|)^{1/2}\sin 2\theta_B} |F_H'| \left(\frac{\pi}{8h}\right)^{1/2} \times e^{-(\mu t - h)} \left\{ 1 + \frac{1}{8h} + \frac{9}{2(8h)^2} + \dots \right\}, \quad (2)$$

where N is the number of unit cells/cm³, λ is the x-ray wavelength, θ_B is the diffraction angle, e^2/mc^2 is the classical electron radius, F_H' [H stands for (hkl)] is that part of the structure factor determined by the real part of the atomic scattering factors, and μ is the linear absorption coefficient. K is equal to 1 or $\cos 2\theta_B$ for polarizations of the incident beam normal (n) or parallel (p) to the plane of incidence. The other quantities are defined as follows:

$$t = \frac{t_0}{2} \left(\frac{1}{\gamma_0} + \frac{1}{\gamma_H} \right),$$

$$h = \mu t_0 K \epsilon (1 + g^2/k^2)^{1/2} / (|\gamma_0 \gamma_H|)^{1/2} \cong \mu t K \epsilon,$$

$$k = F_H''/F_H',$$

$$g = \frac{(1-b)\mu}{4K(e^2/mc^2)\lambda N |F_H'| (|b|)^{1/2}},$$

$$b = \gamma_0/\gamma_H,$$

and

$$\epsilon = F_H''/F_0'' \quad (\epsilon = f_H''/f_0'' \text{ for Cu}).$$

Here γ_0 and γ_H are the direction cosines of the incident (or transmitted) and diffracted beams with respect to the surface normal, respectively, f_H'' and f_0'' are the imaginary parts of the atomic scattering factors [$0 = (000)$], F_H'' and F_0'' are the corresponding structure factors, and t_0 is the distance between the (parallel) surfaces of the crystal. D_0, D_1, \dots are complicated functions of b for $b \neq 1$ (asymmetric Laue geometry). For $b = 1$ (symmetric Laue geometry) $D_0 = 1$ and $D_1 = 9/8$. To a very good approximation $h = \mu t K \epsilon$, for the values of b considered in the present work.

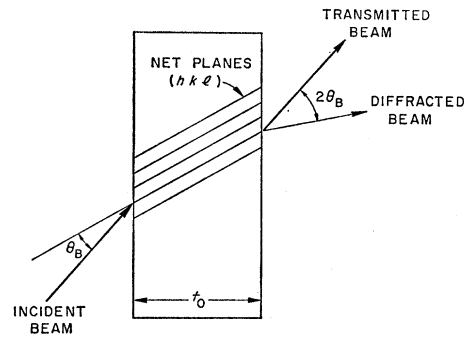


FIG. 1. Laue geometry for anomalous transmission.

In the present work a sample under investigation was made the second crystal in a double-crystal spectrometer arranged in the parallel condition. The first crystal was a nearly perfect copper crystal set for diffraction from the (111) planes in the Bragg geometry. Thus, the beam incident on the second crystal was only partially polarized and not strictly parallel. If R_B^n and R_B^p denote the integrated intensities for the beams diffracted in the Bragg geometry from the first crystal (n and p correspond to the two states of polarization $K = 1$ and $\cos 2\theta_B$, respectively), and if R_T^n and R_T^p denote the corresponding transmitted intensities in the Laue geometry, then it can be shown⁸ that for the experimental arrangement used, the observed intensity of the transmitted beam is given by

$$R_T = \frac{E_T \omega}{I_0} \frac{R_B^n R_T^n + R_B^p R_T^p}{R_B^n + R_B^p}. \quad (4)$$

A similar expression holds for R_H . Here E_T is the total energy that enters the detector as the second crystal is rotated through the diffracting position at a constant angular velocity ω , and I_0 is the intensity of the beam incident on the second crystal. It is easily shown, using the theoretical expressions for R_B^n and R_B^p as given by Hirsch and Ramachandran,⁸ that for the (111) copper reflection, $R_B^p \approx (\cos 2\theta_B) R_B^n$ so that Eq. (4) simplifies to

$$\frac{E_T \omega}{I_0} \frac{R_T^n + (\cos 2\theta_B) R_T^p}{1 + \cos 2\theta_B}, \quad (5)$$

and similarly

$$\frac{E_H \omega}{I_0} \frac{R_H^n + (\cos 2\theta_B) R_H^p}{1 + \cos 2\theta_B}. \quad (6)$$

We compare later the observed intensities with theory by using Eqs. (5) and (6). Since a theoretical value of ϵ for copper is not yet available, these comparisons are used to yield an experimental value for ϵ .

⁸ P. B. Hirsch and G. N. Ramachandran, Acta Cryst. 3, 187 (1950).

PEAK PROFILES

Peak profiles are plots of intensity versus the angular position of the second crystal. Theoretically (for our particular experimental arrangement), this profile is given by the convolution of the Bragg geometry peak profile and the Laue geometry peak profile. If we let $R_B^n(\alpha)$ and $R_B^p(\alpha)$ denote the intensity as a function of the deviation α from θ_B for the Bragg geometry and for the two polarization components n and p , respectively, and let $R_H^n(\beta)$ and $R_H^p(\beta)$ [or $R_T^n(\beta)$ and $R_T^p(\beta)$] denote similar function for the Laue geometry, then, following the same analysis that leads to Eq. (4), the double-crystal peak profiles in the present work theoretically are given by

$$R_H(\beta) = \frac{\int_{-\infty}^{\infty} R_B^n(\alpha)R_H^n(\alpha-\beta)d\alpha + \int_{-\infty}^{\infty} R_B^p(\alpha)R_H^p(\alpha-\beta)d\alpha}{\int_{-\infty}^{\infty} [R_B^n(\alpha) + R_B^p(\alpha)]d\alpha} \quad (7)$$

A similar expression holds for $R_T(\beta)$. The integrations indicated in Eq. (7) were performed on a CDC 1604 computer using the single-crystal peak profile functions given by Hirsch⁶ and by Hirsch and Ramachandran.⁸ These theoretical results are given later and compared with the observed profiles.

EXPERIMENT

The first crystal of the double-crystal spectrometer used in this investigation was a nearly perfect copper crystal with (111) planes essentially parallel (within 0.5°) to the surface. The dislocation density of this crystal was $\sim 5 \times 10^3/\text{cm}^2$ as determined by etch-pit count.

The samples investigated were very carefully prepared in order to maintain control of the variable parameters in the experiments. A $1 \times 1 \times 2$ -cm parallelepiped, with crystal directions shown in Fig. 2, was cut from a nearly perfect copper crystal,⁹ by an acid saw technique.¹⁰ This crystal was annealed at 1075°C for two weeks, after which it was electropolished and etched. The dislocation density was determined to be $< 500/\text{cm}^2$ by etch-pit count. The crystal was then hardened by irradiation with fast neutrons so that damage due to handling would be minimized (see below). After irradiation the crystal was again electropolished and etched, and no change in dislocation density was observed. The crystal was sliced into $1 \times 1 \times Z$ -cm (z between 0.025 and 0.205) samples, some having (110) faces and others having (111) faces as

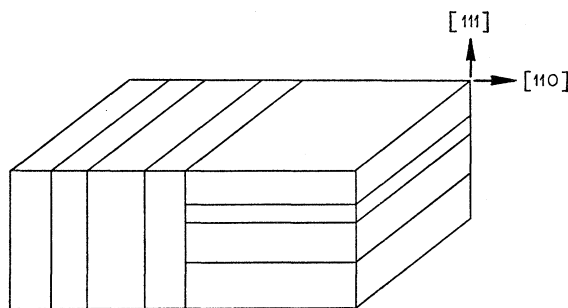


FIG. 2. Relative orientation of samples having (110) and (111) faces as grown.

indicated in Fig. 2. Those samples having (111) faces were again electropolished and etched, and the dislocation densities were still $< 500/\text{cm}^2$. Three samples having (110) faces and two having (111) faces were examined. For the (110) samples the (111) planes provided a symmetric Laue geometry, i.e., $b=1$. For the (111) samples the (111) planes provided an asymmetric geometry, because these planes make a 70° angle with the sample face. The value of b for these samples could be made 0.885 or 1.13 depending on whether the crystal was oriented as shown in Fig. 1 or rotated 180° about the normal to the entrance surface.

Several experimental problems associated with the extreme softness of copper crystals having low dislocation densities should be mentioned. The data could not have been obtained at all without the irradiation hardening of the samples. Most of the data reported here were obtained from crystals which had received a dose of 10^{17} fast neutrons/ cm^2 (shielded from thermals with Cd). Earlier work by one of us¹¹ indicated that such a dose produces sufficient hardening. Since the irradiation introduces a variety of defects in the crystals, the possible influence of these defects on the intensity measurements was investigated and is discussed below. Even for the crystals which were irradiated, it was impossible to use any mechanical method, such as lapping, to make the entrance and exit surfaces of a sample accurately flat and parallel and accurately oriented with respect to a given crystallographic direction. Thus it was very difficult to provide samples with well-known and uniform thicknesses t_0 and with well-known values of b . An optical comparator was used to measure, to the nearest 0.0001 in., the thickness of each sample at several places. These measurements were then appropriately averaged to yield a value for t_0 . An autocollimator optics system and back reflection Laue photographs were used to determine that the orientation of the crystal surface with respect to the diffracting planes was in error by no more than $\frac{1}{2}^\circ$.

Mo $K\alpha$ radiation and a NaI-thallium-doped scintillation detector were used for all the measurements. To eliminate half-wavelength contributions, the x-ray

⁹ F. W. Young, Jr., and J. R. Savage, J. Appl. Phys. **35**, 1917 (1964).

¹⁰ F. W. Young, Jr., and T. R. Wilson, Rev. Sci. Instr. **32**, 559 (1961).

¹¹ F. W. Young, Jr., J. Appl. Phys. **33**, 3553 (1962).

tube was generally operated below 35 kV, but to obtain usable intensities from the thickest sample investigated (0.205 cm) a higher tube voltage was required. Subsequent data obtained from this sample with the use of electronic pulse-height discrimination indicated that half-wavelength contributions to the earlier data were negligible.

Multiple foil techniques were used to check the linearity of the detection circuits and to determine the dead time corrections required at various high counting rates. The peak intensity of each reflection was always kept well within the linear range of the detection circuits.

The intensity I_0 of the incident beam was measured after reduction by carefully calibrated absorbers to a counting rate for which dead time corrections, if any, were generally less than 3%. For an integrated intensity measurement, the crystal was rotated with uniform angular velocity from background on one side of the peak into the background on the other side, and the total counts were accumulated on a scaler. Background counts were taken at a fixed point far off the peak and then subtracted from the integrated counts to yield values for E_T and E_H [as in Eqs. (5) and (6)] for each crystal.

RESULTS

A value of ϵ was obtained from each measured value of R_H by an iterative procedure programmed for a CDC 1604 computer. This procedure was necessary because it was not possible to obtain an analytical solution of Eq. (5) or (6) for ϵ . The experimental results are shown in Table I along with theoretical and experimental values for the ratio R_H/R_T . This ratio is nearly independent of ϵ [see Eqs. (1), (2), (5) and (6)] but depends on the degree of asymmetry in the Laue geometry, on sample thickness, and possibly on the concentration and type of defects present in the crystal. The structure factor used for the (111) reflections was that measured by Batterman *et al.*¹² The values of ϵ obtained for

different samples are in good agreement. The error limit indicated is one standard deviation. In view of the large experimental range of μt , this sample to sample agreement indicates that the theory accurately predicts the dependence of R_H on μt for several values of b for crystals which, as discussed below, clearly are not ideally perfect. The small sample to sample differences are largely due to uncertainties in t_0 and b as mentioned above.

The precise value of ϵ for an ideally perfect copper crystal is not yet known. However, Hildebrandt and Wagenfeld¹³ have shown both experimentally and theoretically for germanium that $\epsilon = \epsilon_0 e^{-M}$, where ϵ_0 is a few tenths of one percent less than unity for low order reflections and e^{-M} is the Debye-Waller factor. Thus ϵ for the copper (111) reflections may also be $\leq e^{-M}$. The value for ϵ of 0.960 ± 0.003 obtained from these crystals is indeed a little less than the value for e^{-M} of 0.969.¹⁴ However, this result was unexpected in view of the relatively large concentration of defects and impurities known to exist in these crystals which should reduce ϵ below its value for a perfect crystal. In addition to a dislocation density of $\sim 500/\text{cm}^2$ (or equivalently 10^8 cm of dislocation length/cm³), there exist $\sim 10^{15}/\text{cm}^3$ vacancy and interstitial loops ~ 50 Å in diameter which result from the radiation dose of 10^{17} fast neutrons/cm² (Ref. 15), 10^{17} – 10^{18} impurities/cm³, and $\sim 10^5/\text{cm}^3$ vacancy loops ~ 100 μ in diameter which were formed during the preparation of these crystals. It has been demonstrated that a dislocation density in silicon¹⁶ and germanium¹⁷ of the magnitude encountered here has essentially no effect on the peak widths and integrated intensities obtained in the Bragg geometry, and similar results have been obtained on these copper crystals.¹ However, Okkerse¹⁸ reports that a dislocation density as low as $50/\text{cm}^2$ affects the intensity of x rays anomalously diffracted through nearly perfect Ge crystals. Thus one might expect the dislocations in the copper crystals studied here to have some effect on the value of ϵ . In addition, it is surprising that the radiation damage, impurities, etc., do not have a larger effect. The effect is indeed small since ϵ is already very close to e^{-M} .

It is clear that impurities do not affect the intensities of the anomalous beams in copper to the extent reported for germanium.^{19,20} If corrections were applied to our values of ϵ , according to the ϵ versus impurity concen-

TABLE I. Results for copper crystals irradiated with 10^{17} neutron/cm². Here $e^{-M} = 0.969$.

Sample	μt^a	b	R_H (10^{-7} rad)	ϵ	R_H/R_T Exptl.	R_H/R_T Theory
X116-N4-1	16.8	1.00	12.6	0.955	0.879	0.868
X116-N4-2 ^b	46.6	1.00	1.97	0.960	0.974	0.956
X116-N4-2 ^c	37.6	1.00	3.14	0.959	0.955	0.943
X116-N4-3	91.5	1.00	0.254	0.962	0.988	0.977
X116-N4-5	29.0	0.885	6.25	0.963	1.039	1.055
X116-N4-5	29.0	1.13	6.16	0.965	0.800	0.812
X116-N4-6	10.4	0.885	25.4	0.955	0.914	0.898
X116-N4-6	10.4	1.13	22.8	0.957	0.666	0.677
				(Average)	0.960 ± 0.003	

^a $\mu = 439.4$ cm⁻¹.

^b Before anneal.

^c After 1000°C anneal.

¹² Boris W. Batterman, David R. Chipman, and John J. Demarco, *Phys. Rev.* **122**, 68 (1961).

¹³ G. Hildebrandt and H. Wagenfeld, *Acta Cryst.* **16**, part 13, A160, paper 19.11 (1963).

¹⁴ Based on a Debye temperature of 320°K and a room temperature of 300°K.

¹⁵ M. J. Makin, A. D. Whaphamand, and F. J. Minter, *Phil. Mag.* **7**, 285 (1962).

¹⁶ J. R. Patel, R. S. Wagner, and S. Moss, *Acta Met.* **10**, 759 (1962).

¹⁷ Boris W. Batterman, *J. Appl. Phys.* **30**, 508 (1959).

¹⁸ B. Okkerse, *Philips Tech. Rev.* **21**, 340 (1959).

¹⁹ Susumu Maruyama and Goro Honjo, *J. Phys. Soc. Japan* **17**, 1803 (1962).

²⁰ O. N. Efimov and A. M. Elistratow, *Fiz. Tverd. Tela* **5**, 1869 (1963) [English transl.: *Soviet Phys.—Solid State* **5**, 1364 (1964)].

tration data reported for Ge, ϵ_0 for a perfect copper crystal would be made >1 , a result which is physically unreasonable.

Investigations have been initiated to determine the influence of the other defects present in these crystals on ϵ . Because of the relatively low density of the larger vacancy loops, which occur during crystal preparation, one might not expect to detect their affect on ϵ in the presence of the radiation damage. However, transmission topographs which were made of the crystals indicate that the loop images, which are regions on the topograph of reduced x-ray intensity, are of sufficient size and number to cause a significant decrease in the total transmitted x-ray intensity. It would appear that their removal would produce a significant increase in the integrated intensity and hence in ϵ . To examine this point, sample X116-N4-2 was annealed at 1000°C for two hours and then reirradiated with 10^{17} fast neutrons/cm². Topographs of the annealed crystal indicated that these large vacancy loops had indeed been removed. However, intensity measurements made on the annealed crystal yielded the same value of ϵ as before. (The integrated intensity changed because of the unavoidable thickness changes which occurred in the anneal-irradiate-polish cycle.) Thus the influence of this type of defect on the intensities of the anomalously transmitted x-ray beams in copper is not yet understood. The apparent contradiction between the evidence obtained from the topographs and the intensity measurements is a problem for further research.

To investigate the influence of radiation damage on ϵ , we have examined crystals which were irradiated with 10^{16} fast neutrons/cm². The crystals were prepared as described earlier except that after they were sliced from a parallelepiped, they were annealed at 1000°C for 1 h, to remove the damage produced by the first dose of 10^{17} neutrons/cm². They were then irradiated with 10^{16} neutrons/cm² (the annealing also removed the large vacancy loops, discussed above, that were present).

TABLE II. Results for copper crystals irradiated with 10^{16} neutrons/cm².

Sample	μt ^a	b	R_H (10^{-7} rad)	ϵ	R_H/R_T Exptl.	R_H/R_T Theory
U-3	10.3	0.885	27.7	0.963	0.895	0.898
	10.3	1.13	24.6	0.964	0.670	0.678
U-4	26.6	0.885	8.08	0.966	1.015	1.048
	26.6	1.13	7.34	0.967	0.802	0.807
				(Average) 0.965 ± 0.002		

^a $\mu = 439.4 \text{ cm}^{-1}$.

These crystals were softer than those which received the higher dose. However, with careful handling, it was possible to obtain data from two samples having (111) faces. The results are shown in Table II. There is some evidence that the apparent ϵ increased owing to the decrease in irradiation dose. A change in ϵ was not unexpected; rather, it is surprising that such a small change resulted from a decrease in the concentration of radiation produced defects of probably a factor of 10. We have also obtained intensity data, which will be reported in another communication, from one copper crystal that has received various doses of irradiation. These data are consistent with those reported here and also indicate that ϵ for an unirradiated crystal is probably larger than 0.960 but not significantly larger than 0.965. Thus, although the increase in ϵ for the samples irradiated with 10^{16} fast neutrons is barely outside experimental error, it is felt to be real and we conclude that our best experimental value for ϵ for the (111) reflection of copper is 0.965 ± 0.003 .

The experimental values of the ratio R_H/R_T agree quite well with theory. It is of interest to note that this ratio can be significantly larger or smaller than unity depending on the value of b and μt . The small differences between theory and experiment seen in Tables I and II are within experimental error. Such agreement may also indicate that with respect to the integrated intensity measurements, these copper crystals are very perfect.

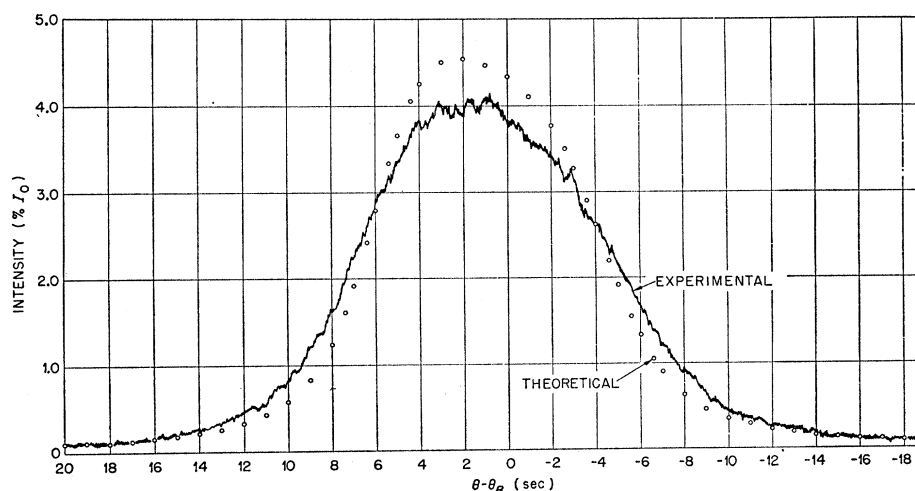


FIG. 3. Bragg-Laue double-crystal peak profile for copper crystal X116-N4-6, $\mu t = 10.4$.

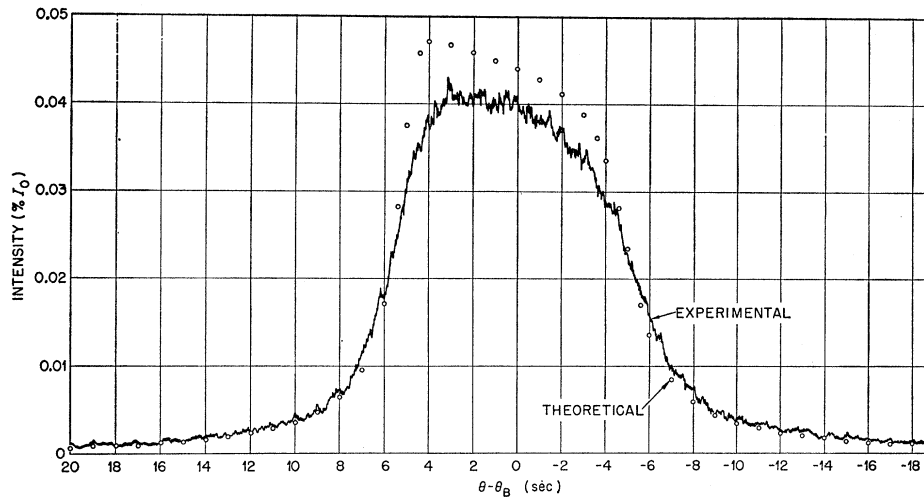


FIG. 4. Bragg-Laue double-crystal peak profile for copper crystal X116-N4-3, $\mu t = 91.5$.

It has been shown by Okkerse and Penning²¹ that this ratio is quite sensitive (in Ge) to elastic strain resulting from temperature gradients and bending. However, Patel and Batterman²² have shown that in Si this ratio is insensitive to the concentration of clusters of oxygen impurities, while the absolute intensities decreased markedly with increasing concentration. Therefore, the importance of the agreement between theoretical and experimental values for R_H/R_T as an indication of crystal perfection is not well understood.

The results of the theoretical calculations of the Bragg-Laue double crystal geometry are compared in Figs. 3 and 4 with experimental curves for the two crystals which represent the highest and lowest value of μt in Table I. The intensity scales are in units of the percentage of I_0 and both experimental and theoretical curves are on an absolute scale. No matching of half-widths or peak heights was attempted, but since the origins of the experimental angles were necessarily arbitrary, these curves were positioned horizontally to agree best with the theoretical curves. The theoretical curves were calculated using the value of ϵ determined experimentally. Thus, the areas of the theoretical and experimental curves should agree. The shapes of the experimental curves are in semiquantitative agreement with theory. The theoretical (111) Bragg geometry single-crystal curve is quite asymmetric and has a half-width of 10.5 sec and the theoretical (111) Laue geometry single-crystal curves are symmetric for the values

of b considered here and have half-widths of 3.3 sec for $\mu t = 10$ and 1.2 sec for $\mu t = 91.5$. Both curves show the strong influence of the asymmetric Bragg peak, but the asymmetry is more pronounced for $\mu t = 91.5$ because of the relatively narrow Laue peak. The half-widths of the experimental curves are $\sim 10\%$ larger and their heights are $\sim 10\%$ smaller than the theoretical curves. The differences could be due to the influence of dislocations in the first crystal on the peak shape of the beam diffracted from that crystal. Some double-crystal profiles obtained with both crystals in the Laue geometry indicate that the differences from theory seen in Figs. 3 and 4 could not be due to the second crystal. Further experiments on peak shapes are anticipated.

SUMMARY

The copper crystals examined in this investigation appear very perfect with respect to their x-ray diffraction properties even though they contain a relatively large concentration of structural defects and impurities. The dependence of the intensity of x rays anomalously diffracted through these crystals on the concentrations and types of imperfections is not yet understood. The data indicate that there is probably a small increase in ϵ with a decrease of radiation dose from 10^{17} fast neutrons/cm² to 10^{16} fast neutrons/cm², while the elimination of $\sim 10^5$ large vacancy loops by annealing had little or no effect on ϵ . Very good agreement between theory and experiment was found with regard both to the dependence of R_H and of R_H/R_T on μt for the symmetric and asymmetric Laue geometries and to the shape of the convoluted Bragg-Laue diffraction peaks.

²¹ B. Okkerse and P. Penning, Philips Res. Rept. 18, 82 (1963).

²² J. R. Patel and B. W. Batterman, J. Appl. Phys. 34, 2716 (1963).