CHINESE JOURNAL OF PHYSICS VOL. 23, NO. 2 SUMMER 1985

X-ray Polarization Ratio of Graphite Crystal Monochromator

Water LUR (盧火鐵) and R. CHEN (陳瑞虹)

Department of Physics, National Taiwan Normal University, Taipei, Taiwan, Republic of China

(Received 20 November 1984)

The x-ray polarization ratios of graphite monochromator are obtained by measuring its σ- and n-component of the x-ray after reflection from the monochromator. Both the σ and π polarizations are measured by x-ray anomalous transmission of perfect crystal. The results are 0.910(3) for Cu K_α and 0.952(10) for Mo K_α radiation. The diffraction data of Cu K_α and Mo K_α radiations show that the polarization status has a tendancy of moving away from an ideally perfect crystal value to mosaic crystal value as the wavelength of the radiation becomes shorter.

1. INTRODUCTION

Recently the improvement in the accuracy of x-ray intensity measurements has provided diffractionists with valuable data to study not only the precise molecular architecture but also the chemical and physical properties of materials via the deformation of electron distribution. One improvement to the precision of the diffraction data collection is the utilization of a crystal monochromator. But it introduces an uncertainty in the polarization factor and affects the interpretation of the result. It has been assumed for a long time that an ideally mosaic crystal is adequate to describe the crystal monochromator. In this case the polarization status of the unpolarized incident beam after monochromator can be written as

$$K = \cos^2 2\theta_M ,$$

where K is the ratio of the reflection power of the monochromator for the component of polarization in the diffraction plane to the for the perpendicular component. Here θ_M is the Bragg angle at the monochromator. Many techniques and methods have been used in the measurement of the polarization ratio since the first measurement done by Miyake, Togawa & Hosoya[1]. The results show that the factor $K(\theta_M)$ varies in a wide range, and the tacit assumption could be erroneous. The survey of polarization ratios through Internation Union of Crystallography (1978, 198 1) has also been proposed by Jennings.[2,3] The purpose is to establish the range of these values such that researchers can estimate it if the measurement is not done. In this paper we report our measurements on graphite monochromator using Cu K_α and Mo K_α radiation.

II. EXPERIMENT

The graphite monochromator which is commercially supplied by UCAR is installed on Rigaku 4-circle single crystal diffractometer in our laboratory. The measurement of polarization ratio was made by the perfect crystals suggested by Suortti & Paakkari.“ From the x-ray dynamical theory of perfect crystal, one might expect that the anomalous transmission, known as Borrmann effect ’ , could happen. That means if the perfect crystal is so thick ($\mu_0 t > 10$) that it transmits only a singly polarized radiation which fulfills the Bragg condition. In a usual case, (220) reflection of silicon perfect crystal is employed.

The x-ray beam coming from Cu tube has convergent and divergent angles 39 ’ and 53 ', respectively. The beam is collimated with a cross section of lx 1 mm at the center of the diffractometer. The x-ray is incident on a graphite monochromator M which was set for (002) reflection. The orientation of the perfect crystals was first determined from Laue diffraction patterns. The crystal was cut with some accuracy so that the direction [111] is perpendicular to the surface of crystal. The crystals were then prepared by griding with # 1500 SiC grit and etched 10 mm in CP-4 to polish the surface. The method of measurement is shown schematically in Fig. 1. The x-ray beam after monochromator contains two components: one is parallel to the diffraction plane (n-component), and the other is perpendicular to the plane (o-component). The perfect crystal is mounted on the goniometer head with a special holder. It is set in Laue diffraction condition to give the correct 2θ for the (220) reflection as shown in Fig. 1. The Borrmann effect of x-rays through the perfect crystal gives the forward transmitted beam and the diffracted beam. Both x-ray beams leaving the crystal are therefore highly polarized and have surprisingly high intensity. To match the diffraction geometry of Rigaku diffractometer, the integrated intensity of o-component was measured by setting counter at D_2 to monitor the diffracted beam. The other three circles were set at $\omega = \theta_{220}$, $\chi = 0°$, and $\phi = 0°$

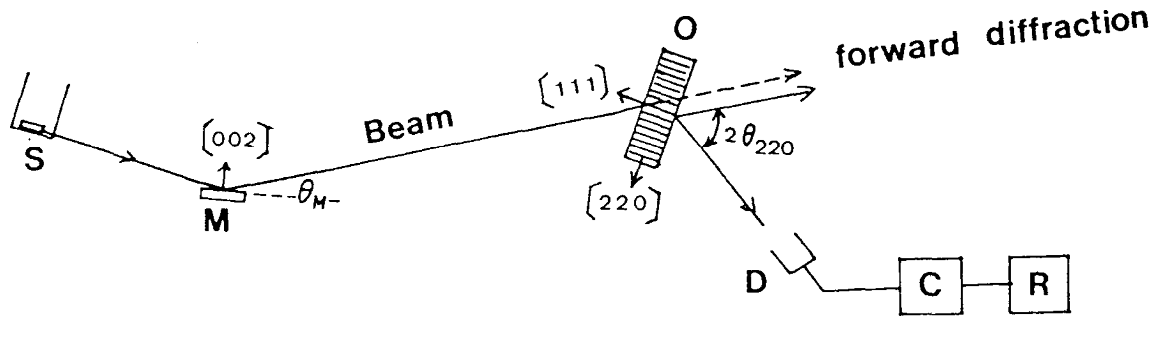

FIG. 1. Schematic view of the experimental arrangement. The unpolarized x-ray after reflection from the graphite monochromator M is polarized. The polarization status is analysed with a Borrmann crystal which is set at 0 for (220) Laue diffraction condition. D: scintillation counter; C: single-channel pulse analyzer; R: Recorder.

respectively. The schematic representation of this arrangement is shown in Fig. 2. To measure the n-component, the counter is then moved up to D_1 by $2\theta_{220}$ degrees as measured from the z-axis counterclockwise on the x-z plane, with $\omega = 0°, \chi = 90°$, and $\phi = \theta_{220}$. The ϕ scan was used for all the measurements. Two silicon perfect crystals with different thickness in which their normal absorption coefficients, μ_O t, are 70 and 30 respectively were used to measure the polarization ratio of graphite for Cu K_α radiation, while for Mo K_α radiation a germanium perfect crystal with $\mu_O t = 61$ was used. In addition, the x-ray tube was operated at a voltage such that second harmonics would be minimized, while the integrated counts of the diffracted beam was maintained high enough to get a reliable measurement. The x-ray beam reflected from the graphite crystal mono-

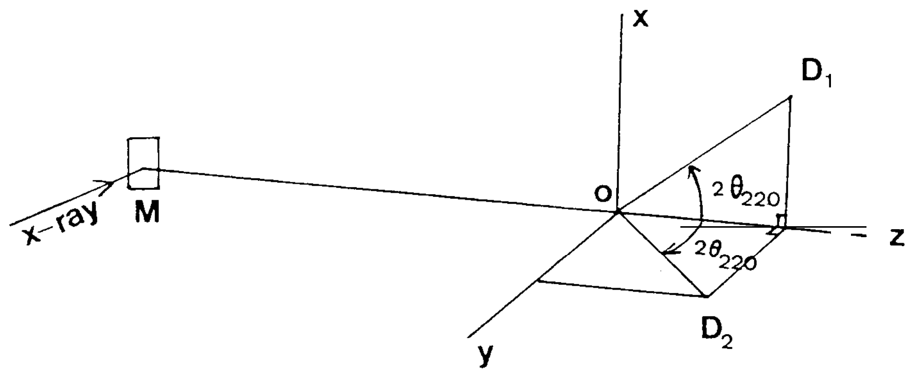

FIG. 2. Schematic representation of the geometric coordinate and counter positions D_1 and D_2 in measuring the polarized diffracted beams caused by a Borrmann crystal at 0. Here M is the graphite monochromator.

chromator was examined in order to make sure that the half-wavelength contribution was negligible.

III. RESULTS AND DISCUSSION

First the polarization ratio of the x-ray beam from the Cu target was measured with a b-filter, instead of using a crystal mono chromator. The measured ratios are 1.006(6) and 0.999(5) for the 4.6 mm and the 2.0 mm thick silicon crystals respectively. It indicated that the Cu K_α x-ray before the monochromator was unpolarized. The polarization ratios of the x-ray beam after the monochromator using Cu K_α and Mo K_α radiation were then measured. The results are tabulated in Table 1 and compared with ideally perfect and mosaic cases. It shows that the graphite monochromator has a polarization ratio for Cu K_α radiation close to that of an ideally perfect crystal, which is equal to $|\cos 2\theta_M|$. In contrast to the Cu K_α case, the value we obtained for Mo K_α radiation is close to that of an ideally mosaic crystal. Our results for Cu K_α and Mo K_α radiations are comparable to those obtained by Vincent and Flack[6]. In their experiment, the polarization ratio for Cu K_α radiation falls in between the expected values of mosaic and perfect

	Silicon crystal thickness	Polarization ratios	perfect crystal case $\cos 2\theta_M$	ideally mosaic crystal case $\cos^2 2\theta_M$
Cu K_α 25 KV	4.6 mm	0.907(7)		
			0.8948	0.8005
Cu K_α 20 KV	2.0 mm	0.911 (3)		
	Germanium crystal thickness			
Mo K_α 32.5 KV	1.9 mm	0.952(10)	0.9776	0.9558

TABLE 1. Summary of the experimental results compared with theories.

graphite monochromators. But their values for Mo K_α and Ag K_α radiations are much smaller than those of the ideally mosaic case. It also appears that the assumption that the monochromators have a degree of perfection being somewhere between ideally mosaic and ideally perfect,’ is not applicable to our case. If the value of polarization ratio is represented through the power parameter n given by $\cos^n 2\theta_M$, the averaged n-value obtained in our Cu K_α case is 0.84 which agrees with the measurement of LePage, Gabe, and Calvert[7]. It also agrees with the theoretical value 0.8 to 1.2 as calculated by Jennings[3] for graphite for Cu K_α radiation. The n-value obtained in our Mo K_α measurement is 2.2. This value is close to that of the ideally mosaic crystal. An extremely small value of n, 0.10, is also found by Bardhan and Cohen[8] for Cu K_α radiation. An extremely large value of n is obtained from the above mentioned measurements by Vincent and Flack[6]. Their values are 4.29 for Mo K_α and 15.4 for Ag K_α radiation. From our results with Cu K_α and Mo K_α radiations and the results of Vincent and Flack[6] with Cu K,, Mo K_α, and Ag K_α radiations, it is shown that the polarization status has a tendancy of moving away from the perfect crystal value to the mosaic crystal value as the wavelength of the radiation becomes shorter. The method used in this work is characterized as direct because it gives the beam polarization ratio directly as the quotient of the two measured polarized intensities. Since a perfect Borrmann crystal can serve as a polarizer and monochromator at the same time, we consider this method more reliable than the methods suggested by the other workers.’ ,[9] The wide spread of the measured n-values encourages diffractionists to measure the polarization ratio of their own apparatus, especially when the polarization correction is needed. It also indicates that the theoretical explanation of polarization ratio is not conclusive. There is much to be done in order to resolve the discrepancies between theoretical predictions and measured values. While this work was in progress in our laboratory, Jennings has recently published the survey of the polarization ratios of many crystal monochromators.[10]

ACKNOWLEDGEMENTS

We would like to thank Dr. P. Trucano for his kindly supplying silicon perfect single crystals and valuable suggestion in measurement. This work was supported through a grant from the National Science Council of the Republic of China under contract No. NSC73-0208-M003-05.

REFERENCES

S. Miyake, S. Togawa, and S. Hosoya, Acta Cryst. 17, 1083 (1964)
2. International Union of Crystallography, Acta Cryst. A34, 159 (1978)
3. L. D. Jennings, Acta Cryst. **A37,** 584 (1981)
4. P. Suortti, and T. Paakkari, J. Appl. Cryst. 1, 121 (1968)
5. B. W. Battermann, and H. Cole, Rev. Mod. Phys. 36,681 (1964)
6. M. G. Vincent, and H. D. Flack, Acta Cryst. **A36,** 614 (1980)
7. Y. Le Page, E. J. Gabe, and L. D. Calvert, J. App. Cryst. **12,** 25 (1979)
8. P. Bardhan and J. B. Cohen, Acta Cryst. **A32,** 597 (1976)
9. H. Hope, Acta Cryst. **A27,** 392 (1971)
10. L. D. Jennings, Acta Cryst. A40, 12 (1984)