

Science and Engineering Research Council (UK) for which we are grateful. Furthermore, we thank Professor Jack Dunitz for sending us his work in preprint form, and Dr Alan Hewat for many helpful suggestions during the writing of this paper, after giving considerable help with the experiment at the Institut Laue-Langevin, Grenoble.

References

- BACON, G. E., LISHER, E. J. & PAWLEY, G. S. (1979). *Acta Cryst.* B35, 1400–1403.
 BRIDGMAN, P. W. (1936). *Proc. Am. Acad. Arts Sci.* 72, 227–266.
 BROCK, C. P. & DUNITZ, J. D. (1982). *Acta Cryst.* B38, 2218–2228.
 CHAPLOT, S. L., LEHNER, N. & PAWLEY, G. S. (1982). *Acta Cryst.* B38, 483–487.
 CRUICKSHANK, D. W. J. (1957). *Acta Cryst.* 10, 504–508.
 LEHMANN, M. S. & PAWLEY, G. S. (1972). *Acta Chem. Scand.* 26, 1996–2004.
 MACKENZIE, G. A., PAWLEY, G. S. & DIETRICH, O. W. (1977). *J. Phys. C.* 10, 3723–3736.
 NATKANIEC, I., BOKHENKOV, E. L., DORNER, B., KALUS, J., MACKENZIE, G. A., PAWLEY, G. S., SCHMELZER, U. & SHEKA, E. F. (1980). *J. Phys. C.* 13, 4265–4283.
 PAWLEY, G. S. (1967). *Phys. Status Solidi*, 20, 347–360.
 PAWLEY, G. S. (1980). *J. Appl. Cryst.* 13, 630–633.
 PAWLEY, G. S. & YEATS, E. A. (1969). *Acta Cryst.* B25, 2009–2013.
 PONOMAREV, V. I., FILIPENKO, O. S. & ATOVMIAN, L. O. (1976). *Sov. Phys. Crystallogr.* 21, 215–216.
 RIETVELD, H. M. (1969). *J. Appl. Cryst.* 2, 65–71.
 RYZHENKOV, A. P. & KOZHIN, V. M. (1968). *Sov. Phys. Crystallogr.* 12, 943–945.
 SAKATA, M. & COOPER, M. J. (1979). *J. Appl. Cryst.* 12, 554–563.
 SCHMELZER, U., BOKHENKOV, E. L., DORNER, B., KALUS, J., MACKENZIE, G. A., NATKANIEC, I., PAWLEY, G. S. & SHEKA, E. F. (1981). *J. Phys. C.* 14, 1025–1041.
 WINDSOR, C. G. & SINCLAIR, R. N. (1976). *Acta Cryst.* A32, 395–409.

Acta Cryst. (1982). A38, 810–813

Observation of Effect of Temperature on X-ray Diffraction Intensities across the In K Absorption Edge of InSb

BY T. FUKAMACHI

Saitama Institute of Technology, Okabe, Saitama 369-02, Japan

T. KAWAMURA

Faculty of Education and Liberal Arts, Yamanashi University, Kofu, Yamanashi 400, Japan

K. HAYAKAWA

Central Research Laboratory, Hitachi Ltd, Kokubunji, Tokyo 185, Japan

AND Y. NAKANO AND F. KOH

Saitama Institute of Technology, Okabe, Saitama 369-02, Japan

(Received 5 April 1982; accepted 24 May 1982)

Abstract

Variations at room and liquid-nitrogen temperatures have been observed of the energy-dispersive integrated X-ray reflection intensity from an InSb single crystal across the In K absorption edge by the use of a multi-channel solid-state detector. The results show that the relative intensities from a nearly perfect crystal change as a function of the temperature above and below the absorption edge, but there is no such change in a nearly mosaic crystal. This variation observed in a

nearly perfect crystal is characteristic of an absorbing monatomic perfect crystal and can be explained in terms of the dynamical theory of diffraction including anomalous scattering.

Introduction

Across the absorption edge, the integrated X-ray reflection intensity $J(\omega)$ shows a characteristic variation as a function of X-ray energy $\hbar\omega$, depending on

the polarity and/or the perfection of the crystal. This variation was first pointed out theoretically by Cole & Stemple (1962) and later quantitatively studied by the use of energy-dispersive diffractometry with a solid-state detector (SSD), which made it feasible to study anomalous scattering very near the absorption edge (Fukamachi, Hosoya & Okunuki, 1976a,b; Fukamachi, Hosoya, Kawamura & Okunuki, 1977, 1979).

Recently, for an absorbing monatomic perfect crystal, the effect of temperature on $J(\omega)$ across the absorption edge has been pointed out theoretically by Kawamura & Fukamachi (1979). (This will be referred to as a temperature effect in short.) This temperature effect is peculiar to dynamical diffraction with absorption included and cannot be observed in a monatomic mosaic crystal.

In Fig. 1 is shown their numerical simulation of the integrated reflection power of $\bar{5}\bar{5}\bar{5}$ in the Bragg case near the Ga K absorption edge of both perfect and mosaic GaAs crystals. The temperature factor B , which is defined by the use of a Debye-Waller factor as $\exp(-M) = \exp[-B(\sin \theta/\lambda)^2]$, was assumed to be the same for both Ga and As atoms and varied from 0 to 4 \AA^2 . When B is zero, the integrated reflection powers

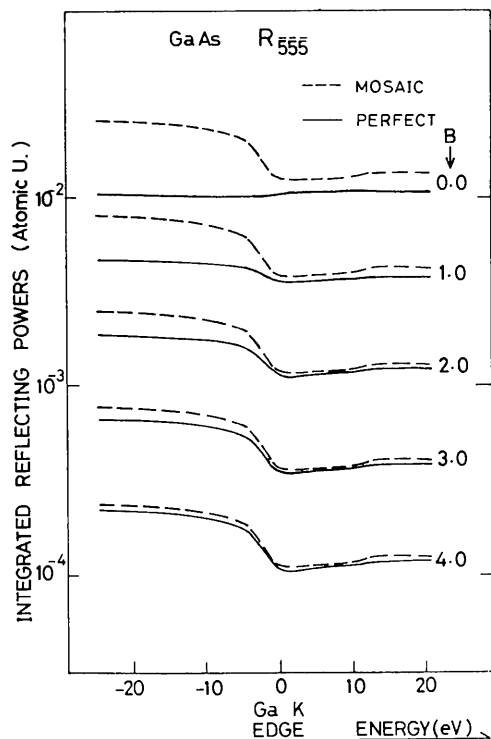


Fig. 1. The integrated $\bar{5}\bar{5}\bar{5}$ reflection intensities across the Ga K absorption edge from a perfect (solid curves) and an ideally mosaic (dashed curves) GaAs crystal calculated as a function of the temperature parameter B . The ordinate is on a logarithmic scale.

from a perfect crystal show relatively smaller intensities on the low-energy side of the absorption edge than on the high-energy side. When B is made larger, this intensity variation changes: the integrated reflection power on the low-energy side becomes relatively larger than that on the high-energy side. In a mosaic crystal, on the other hand, the integrated reflection power is always larger on the low-energy side than on the high-energy side, and their relative intensities do not change depending on the B value; B is assumed to be the same for Ga and As atoms (Morlon, Fukamachi & Hosoya, 1979).

Since the temperature dependence of the scattering factor is expressed by the Debye-Waller factor, the aforementioned temperature effect for an absorbing perfect crystal is expected to be conspicuous for high-order reflections. In the present experiment, this effect was measured on the 777 reflection from an InSb crystal near the In K absorption edge. The atomic numbers of In and Sb are very close and the temperature factors of the two atoms are expected to be almost the same. We measured the integrated reflection intensities at both room and liquid-nitrogen temperatures by the use of a multi-channel solid-state detector (MCSSD). The MCSSD made it possible to measure the integrated reflection intensities in a short time, which avoided any influence of variation of the specimen condition on $J(\omega)$ in the present experimental apparatus.

Experimental apparatus

In Fig. 2 is shown the experimental system. The X-ray tube with a copper target is operated at 30 kV and 30 mA. The specimen is put into a stylo-foam cup filled

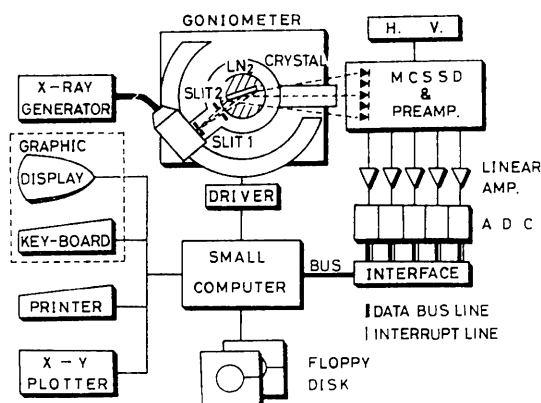


Fig. 2. Schematic diagram of the experimental system. Linear amplifiers: NAIG E511 (shaping time $2 \mu\text{s}$); ADC's: NAIG E551 (50 MHz); small computer (256 kbytes) and floppy disks (1.2 Mbytes \times 2); AICOM C6; graphic display and key-board: Tektronix 4006-1; printer: Texas Instrument OMNI 800; X-Y plotter: Watanabe WX 4671.

with liquid nitrogen when measured at liquid-nitrogen temperature. The X-ray detector is a five-channel MCSSD; these SSD's are 0.4 mm in width, separated by 0.8 mm, and the electrodes between them are 0.4 mm wide. The detail of the MCSSD and its data processing system has been described elsewhere (Nakano, Fukamachi, Kotani, Hirata, Hosoya & Iitaka, 1982) and a block diagram is shown in Fig. 2. An example of diffraction spectra measured by this system is shown in Fig. 3. The goniometer is set so that the 777 reflection is at the In *K* absorption edge. In the present experimental system, the incident X-ray intensity distribution is not homogeneous at each electrode. The results in Fig. 3 are scaled to show the same intensities for the 444 reflection, which is well away from the absorption edge. These were measured at the room temperature on (a) a nearly perfect and (b) a nearly mosaic crystal. It is noted that the difference between two $J(\omega)$ values above and below the absorption edge is more conspicuous in (b) than in (a).

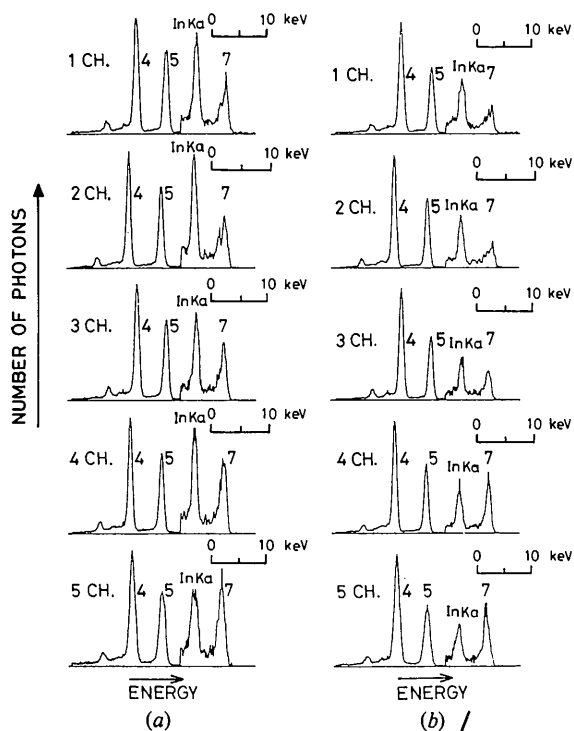


Fig. 3. Diffraction spectra of (a) a nearly perfect and (b) a nearly mosaic InSb crystal at each channel of a MCSSD measured at room temperature. The numbers 4, 5 and 7 indicate 444, 555 and 777 reflections, respectively. The vertical scale on the high-energy side (In *K*α and 777 reflection) is ten times that on the low-energy side. The energy of the 777 reflection is across the In *K* absorption edge; the energy of the third channel is just set on the absorption edge. The energy scale is different at each channel and is denoted at upper right.

Results

In the diffraction spectra of Fig. 3 several components of fluorescent X-rays are included, especially the In *K*β component for a 777 reflection peak. The fluorescent X-rays were subtracted using fluorescent X-ray spectra measured by setting the goniometer away from the Bragg condition. We measured these spectra from a nearly perfect crystal at both room and liquid-nitrogen temperatures. Then, after polishing this crystal surface by emery paper, we measured the spectra from this nearly mosaic crystal at both room and liquid-nitrogen temperatures. We measured both crystals twice: each measurement time was 1000 s at room temperature and 700 s at liquid-nitrogen temperature. The fluorescence spectra were measured in the same manner.

The results are shown in Fig. 4, after normalization at the second channel. For a nearly mosaic crystal, the intensity variation across the absorption edge is marked and the difference at room and liquid-nitrogen temperatures is not observed in the limitation of the experimental error. For a nearly perfect crystal, on the other hand, the intensity variation is larger at room temperature than at liquid-nitrogen temperature, although the difference across the absorption edge is smaller than that of a nearly mosaic crystal.

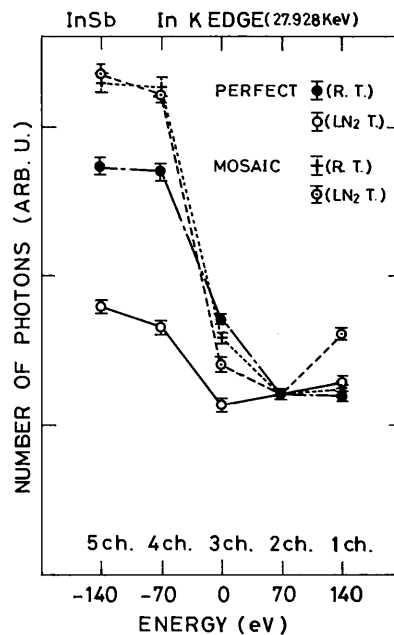


Fig. 4. Normalized 777 integrated reflection intensities from an InSb crystal just across the In *K* absorption edge measured by the MCSSD. The data are normalized at the second channel. The dotted and dashed lines are obtained from a nearly mosaic crystal at room and liquid-nitrogen temperatures, respectively. The dot-dashed and solid lines are from a nearly perfect crystal, taken at room and liquid-nitrogen temperatures, respectively.

Discussion

Calculated curves are shown in Fig. 5 for 777 integrated reflection intensities $J(\omega)$ from InSb for both a perfect and a mosaic crystal near the In K absorption edge. The temperature factors are assumed to be the same for In and Sb atoms. In this calculation, anomalous scattering factors were calculated for K , L and M electrons using the relationship of Parratt & Hempstead (1954) with the oscillator strengths due to Cromer (1965). The contributions from other outer electrons were neglected. Each integrated reflection intensity is normalized at a value 200 eV above the absorption edge. The difference between the integrated reflection intensities above and below the absorption edge in a perfect crystal becomes gradually large with the temperature factor B increased from 0 to 3 \AA^2 in steps of 1 \AA^2 . The integrated reflection intensity from a mosaic crystal shows a steeper change across the absorption edge but is independent of the temperature factor B .

The measured results in Fig. 4 are qualitatively in good agreement with the theoretical calculation in Fig.

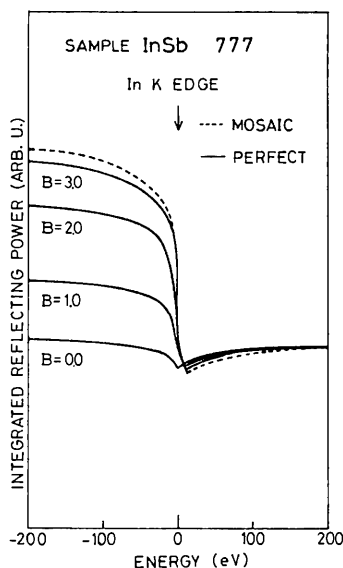


Fig. 5. Calculated integrated reflection intensities from InSb crystals across the In K absorption edge. The curves are normalized at 200 eV above the edge. The temperature factor B is changed from 0.0 to 3.0 \AA^2 for a perfect crystal (solid lines). The dashed line is for a mosaic crystal.

5, but quantitatively the agreement is not especially good about the intensity variation across the absorption edge. For example, in a nearly mosaic crystal the intensity ratio below and above the edge is approximately 3:1 in experiment but 2:1 in calculation. The origin of this disagreement cannot be accounted for in the present experiment and calculation.

It is noted, however, that two effects are experimentally confirmed. One is that the variation of $J(\omega)$ across the absorption edge is independent of the specimen temperature in a nearly mosaic monatomic crystal. Therefore, from the result of Morlon *et al.* (1979), a common temperature factor B can be used for In and Sb atoms. The other is that, in a nearly perfect crystal, the variation of $J(\omega)$ across the edge depends on the temperature. This effect is peculiar to an absorbing monatomic perfect crystal, and this has been theoretically pointed out by Kawamura & Fukamachi (1979).

The authors would like to thank Mr M. Yoshizawa for technical assistance. This work was partly supported by Grants-in-Aid for Scientific Research 346030 (TF) and 474127 (TK).

References

- COLE, H. & STEMPLE, N. R. (1962). *J. Appl. Phys.* **33**, 2227–2233.
- CROMER, D. T. (1965). *Acta Cryst.* **18**, 17–23.
- FUKAMACHI, T., HOSOYA, S., KAWAMURA, T. & OKUNUKI, M. (1977). *Acta Cryst.* **A33**, 54–58.
- FUKAMACHI, T., HOSOYA, S., KAWAMURA, T. & OKUNUKI, M. (1979). *Acta Cryst.* **A35**, 828–831.
- FUKAMACHI, T., HOSOYA, S. & OKUNUKI, M. (1976a). *Acta Cryst.* **A32**, 104–109.
- FUKAMACHI, T., HOSOYA, S. & OKUNUKI, M. (1976b). *Acta Cryst.* **A32**, 245–249.
- KAWAMURA, T. & FUKAMACHI, T. (1979). *Acta Cryst.* **A35**, 831–835.
- MORLON, B., FUKAMACHI, T. & HOSOYA, S. (1979). *Acta Cryst.* **A35**, 714–717.
- NAKANO, Y., FUKAMACHI, T., KOTANI, H., HIRATA, H., HOSOYA, S. & IITAKA, Y. (1982). *J. Appl. Cryst.* **15**, 89–93.
- PARRATT, L. G. & HEMPSTEAD, C. F. (1954). *Phys. Rev.* **19**, 1593–1600.