ber output while the true counting rate is available for dead-time corrections.

To test the performance of the system, we recorded the small-angle scattering from a standard specimen of polyethylene. Data were taken at an angle where the intensity was relatively angle-independent. The X-ray generator was a General Electric XRD-5, and the X-ray tube was a Siemens fine-focus high-intensity Mo tube operated at 50 kV and 18 mA. Data were taken once per day for a period of twelve days. The dashed curve of Fig. 4 illustrates the percentage change of the true scattered power (measured in counts/sec) on a given day from the true scattered power recorded on the first day, and is indicative of the long-term stability of the generator/collimation system without the ionization-chamber monitor. The solid curve illustrates the percentage change of the monitored scattered power (measured in counts/charge-integrator pulse), and is indicative of the stability of the improved system. The error bars are 1σ based on counting statistics. The peak-to-peak fluctuation without the monitor is 14.7% while the r.m.s. deviation is 5.3%. The use of the monitor reduces these values to 3-5% and 1.3% respectively. Counting statistics of the data were about 1%, thus indicating the inherent stability of our monitor system to be ≈0.8%. We believe these fluctuations may be a result of an escape peak and other statistical processes associated with such a low-absorption detector.

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References


The Measurement of Single-Crystal Lattice Parameters using a Double-Diffraction Technique

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A modified double-diffraction technique is described for the accurate measurement of lattice parameters of (100) slices of gallium arsenide. The method relies on the selection of appropriate reflexions for producing a pseudo-point of triple diffraction, and the determination of the wavelength necessary to produce such a triple point. Results are presented which show that lattice parameters can be determined rapidly and simply to better than 1 in 10^3.

1. Introduction

Isherwood & Wallace (1971) have described how the lattice parameters of certain cubic crystals can be determined very accurately by measuring the small angles produced between two doubly diffracted Renninger-type reflexions on the Bragg cone of a primary diffracting plane. The success of this technique relies on being able to predict a position of triple diffraction for the primary diffracting plane and the coupled doubly diffracting planes which is close to the Ewald sphere of reflexion for the monochromatic radiation used for the primary diffraction from all three planes. Although the doubly diffracted reflexions are very sharp, the contrast between the Renninger-type reflexions is greatly enhanced if the primary diffracting plane has very small or zero intensity, and the doubly-diffracting planes, together with the intermediate planes which ultimately give rise to the Renninger reflexions, have a reasonably high intensity. As shown by Isherwood & Wallace, the diamond-type structure is particularly amenable to this kind of investigation because the superlattice derived from the presence of the (00l) glide plane leads to forbidden reflexions which are particularly useful as the primary diffracting planes. This kind of structure is adopted by many semiconductors which are used as single crystals in electronic devices, and the possibility of using such a relatively simple technique for accurate lattice-parameter measurements as a characteristic descriptor of such materials is very attractive.

In applying the Isherwood and Wallace technique
to \{100\} slices of gallium arsenide, several difficulties have been encountered which prevented the adoption of the technique as described by them. High accuracy can only be obtained if the angle between the Renninger-type reflexions is very small; otherwise factors such as film orientation, film shrinkage and the distance between the X-ray source and film become important parameters which are difficult to measure accurately. Furthermore, if the line on the film due to the doubly diffracted beams of various wavelengths intersects the primary Bragg cone at a small angle, it is difficult to locate the exact position of the intense Renninger reflexion on the Bragg cone with the optical microscope used for measurement. It should be pointed out that this could have been overcome by the use of a recording microdensitometer if one had been readily available. Various difference and multiple-film techniques were used in an attempt to overcome all the foregoing problems, but cumulative errors rendered these unsuitable.

All these sources of error can be avoided by making measurements on the pseudo-triple point that is obtained by the intersection of the doubly diffracted beams at an unknown wavelength in the vicinity of the Bragg cones formed by the Ka1 and Ka2 beams. A simple interpolation formula can be used to obtain the value of the unknown wavelength with considerable accuracy, and the formula for the derivation of ao is correspondingly simple.

2. Experimental

With copper radiation it can be shown, using the Kossel plane construction and the approximate value for the lattice parameter as suggested by Isherwood & Wallace, that double diffraction will occur in gallium arsenide from both the (511) and (51T) planes when the crystal is oriented for primary reflexion from (060) using a slightly divergent incident beam with the [001] axis of the crystal vertical. The doubly diffracted beams emerging from the intermediate planes (060)-(511) = (551) and (060)-(51T) = (551) lie on the Bragg cones of (060) and give rise to intense spots on the film, which is placed about 1 m from the crystal in order to obtain a reasonably large image. Both the primary reflexion from (511) and the secondary from (551) have strong intensities compared with the very weak intensity from (060), so that, using an exposure time which gives reasonably intense Ka1 and Ka2 arcs on the film, a fine but well developed line is produced which intersects the (060) arcs at A and B, as shown in Fig. 1. The extent and density of the line between A and B depends on the strength of the continuous X-radiation emitted by the target in the vicinity of the Ka1 and Ka2 lines. The symmetrical nature of the (511) and (51T) planes ensures that a line corresponding to doubly diffracted radiation from (51T) also appears on the film, intersecting the Bragg cones at C and D. These fine lines thus correspond to rays of differing wavelengths between Ka1 and Ka2 which are doubly diffracted along Bragg cones of (060) corresponding to those wavelengths.

The point of intersection E corresponds to a point of double diffraction for both the (511) and (51T) planes using a wavelength which is intermediate between that of Cu Ka1 and Cu Ka2. If a suitably strong line of this wavelength were emitted by the target, the Bragg cone of (060) for this wavelength would pass through E, which would then be a point of triple diffraction for (060), (511) and (51T). It is the production of this point of intersection E and the determination of the wavelength \(\lambda\) which gives rise to this pseudo-triple diffraction which forms the essential part of the present technique for obtaining an accurate value of ao.
3. The wavelength measurement

An interpolation technique is used to determine the value of \( \lambda \). Experimentally, a good quality travelling microscope is used to scan a line such as \( LM \) on the film, illustrated in Fig. 1, and readings taken of the positions of \( P, E \) and \( Q \). These can be measured much more accurately than the positions of \( A, B, C \) or \( D \), which are required in the method as described by Isherwood and Wallace. The assumption is made that \( \frac{PE}{PQ} = \frac{(\lambda - \lambda_1)}{(\lambda_2 - \lambda_1)} \), where \( \lambda_2 \) and \( \lambda_1 \) are the wavelengths of the \( K\alpha_2 \) and \( K\alpha_1 \) lines respectively of the X-ray target. Since \( \lambda_2 \) and \( \lambda_1 \) are accurately known, the value of \( \lambda \) can be determined from the relation \( \lambda = \frac{PE}{PQ(\lambda_2 - \lambda_1)} + \lambda_1 \). As only the ratio \( \frac{PE}{PQ} \) is required by measurement, the position of the film is unimportant except for resolution and knowledge of film shrinkage is not required.

The justification for using this simple interpolation technique is given by considering the geometry in real space for the reflected rays contributing to the images at \( P, E \) and \( Q \). Since the horizontal plane is a plane of symmetry for the planes \((hkl)\) and \((hkl)\), the triple point lies in this plane for the horizontal beam that is utilized, so that only the incident and reflected rays contained in the horizontal plane need be considered in determining the relative positions of \( P, E \) and \( Q \). In Fig. 2 these rays are drawn for wavelengths \( \lambda_1 \) and \( \lambda_2 \) and \( \lambda \), incident at the corresponding Bragg angles \( \theta_1 \), \( \theta_2 \) and \( \theta \) respectively for reflexion from the \((0k0)\) planes of the crystal. For convenience the film is placed at \( F \) in Fig. 2 in such a way that \( TP \) is perpendicular to \( PQ \).

From geometry it follows that \( \frac{PE}{PQ} = \frac{\sin (\theta - \theta_1)}{\sin (\theta_2 - \theta_1)} = x \), say. In our calculations, we assume that \( \frac{PE}{PQ} = \frac{\lambda - \lambda_1}{\lambda_2 - \lambda_1} = \frac{\sin \theta - \sin \theta_1}{\sin \theta_2 - \sin \theta_1} \).

This leads to an error in \( \lambda \) of \( \Delta \lambda \), so that more correctly

\[
\frac{PE}{PQ} = \frac{(\lambda + \Delta \lambda) - \lambda_1}{\lambda_2 - \lambda_1} = x, \\
\frac{\Delta \lambda}{\lambda} = \frac{x(\sin \theta_2 - \sin \theta_1) + \sin \theta_1}{\sin \theta} - 1. \quad (1)
\]

When the triple point coincides with \( P \) or \( Q \) (i.e. \( \theta = \theta_1 \) or \( \theta_2 \) respectively) then \( x = 0 \) or \( 1 \) respectively, and
\[ \Delta \lambda / \lambda = 0 \text{ as expected. We would expect a maximum error for } \lambda \text{ in the region of } \theta = (\theta_1 + \theta_2)/2, \text{ and computing a value of } \Delta \lambda / \lambda \text{ from equation (1) using the appropriate parameters gives a value of about } 10^{-6}, \text{ which is almost an order of magnitude less than the error } \Delta \lambda / \lambda \text{ due to measurement.} \]

4. Calculation of the lattice parameters

Using reciprocal-lattice geometry, the equation of the Kossel plane for a diffracting plane \((hkl)\) in a cubic lattice is given by:

\[ hx^* + ky^* + lz^* = h^2 + k^2 + l^2. \]

If the two planes \((hkl)\) and \((hk\bar{l})\), which are related by mirror-plane symmetry, give rise to triple diffraction with \((0k'0)\) which is normal to that mirror plane, then, as pointed out by Isherwood & Wallace, the three Kossel planes corresponding to \((hk\bar{l})\), \((hkl)\) and \((0k'0)\) will intersect at a point \(x^*_1, y^*_1, z^*_1\) defined by:

\[ hx^*_1 + ky^*_1 + lz^*_1 = h^2 + k^2 + l^2 \]
\[ hx^*_1 + ky^*_1 - lz^*_1 = h^2 + k^2 + l^2. \]

It follows that the point of intersection is given by:

\[ x^*_1 = \frac{h^2 + k^2 + l^2 - k'}{h}, \quad y^*_1 = k', \quad z^*_1 = 0. \]

Furthermore, from the Ewald construction for the 0k0 reflexion, it follows that since the pseudo-triple point \((x^*_1, y^*_1, z^*_1)\) must lie on the sphere of reflexion of diameter \(2a_0^2/\lambda^2\):

\[ x^*_1^2 + y^*_1^2 + z^*_1^2 = 4a_0^2/\lambda^2. \]

Thus, providing the wavelength corresponding to this triple point is known, \(a_0\) is given by:

\[ \frac{(h^2 + k^2 + l^2 - k')^2}{h^2} + k^2 = \frac{4a_0^2}{\lambda^2} \]
\[ \therefore a_0 = \frac{\lambda}{2h} \left[ (h^2 + k^2 + l^2 - k')^2 + h^2k'^2 \right]^{1/2}. \]

For the planes used in this determination \(h = 5, k = 1, l = 1, k' = 6\) whence

\[ a_0 = \frac{\sqrt{1341}}{10} \lambda. \]

Thus, three simple measurements on the film and the use of equation (2) yield an accurate value of the lattice parameter.

5. Results

In order to illustrate the capability of the technique for measuring the lattice parameters of cubic crystals, we have obtained films of the 'triple' point formed by the \((511), (51\bar{1})\) and \((060)\) planes of GaAs before and after neutron irradiation. The shift in the 'triple' point formed by the intersection of the doubly diffracted beams after irradiation is clearly shown in Fig. 3.

The values of the lattice parameters are as follows:

- Fig. 3(a): unirradiated \(5.65323 \pm 0.00005 \) Å
- Fig. 3(b): intermediate dose \(5.65462 \pm 0.00006\) Å
- Fig. 3(c): high dose \(5.65779 \pm 0.00005\) Å.

N.B. The photographs were all taken at \(20^\circ\)C and the wavelengths used were taken from Bearden (1967) viz.

\( \text{Cu } K\alpha_1 = 1.540562, \text{ Cu } K\alpha_2 = 1.544390 \) Å.

6. Conclusions

The modification of the technique used by Isherwood & Wallace (1971) for the measurement of the lattice parameters of cubic crystals has the advantage of extremely simple measurement and calculation. As in the Isherwood & Wallace method, some care in selecting appropriate reflexions for producing the pseudo-triple point is required and this is best done by plotting Kossel-plane intersections in the manner suggested by them.

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References