Acta Cryst. (1997). A**53**, 410–414

X-ray Diffraction Structural Investigations of a Bicrystalline System by Section Topography under Conditions of Anomalous Borrmann Absorption

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(Received 19 January 1996; accepted 10 January 1997)

Abstract

The problem of spherical X-ray diffraction by a bicrystalline system with a thin surface layer is theoretically considered for a Laue set-up in the case $\mu t \gg 1$ under the condition of the existence of a crystal-lattice mismatch. The intensity distribution, contrast and period of the interference pattern are calculated. A method to determine the thickness and the lattice mismatch of the epitaxial or distorted surface layer of a bicrystal is suggested.

1. Introduction

The X-ray moiré effect is known to be produced from a bicrystalline system (BS) that has slightly different reciprocal-lattice vectors for the active reflections of the two parts (layers) of the system. This interference effect observed in the Laue set-up and appearing through superimposition of Bragg diffracted waves has a very high sensitivity to crystal lattice strain of the order of $\Delta d/d \simeq 10^{-8}$ to 10^{-5} (d is the interplanar spacing of the reflecting atomic planes). In experimental investigations, moiré interference fringes can be used for studying impurity (diffused atoms, implanted ions, precipitates) distribution and structural distortions caused by the impurities in thin surface layers of the crystals. Chikawa (1965) has observed by Lang's method moiré patterns obtained from two superposed CdS crystals, the thinner of which was grown epitaxially on the other and contained the higher concentration of the doped impurity. Lang & Miuscov (1965) recorded moiré patterns formed in a quartz single crystal containing a crack. The X-ray diffraction on a BS produced by bombarding an Si single crystal with a high-energy proton beam was investigated by Authier & Montenay-Garestier (1965) and by Simon & Authier (1968).

We have devoted a series of studies to X-ray diffraction of structural distortions (strains $< 10^{-5}$) in the surface layers of Si single crystals after mono-energetic Au-ion implantation. X-ray traverse-type topographs with traverse-type moiré fringes were recorded by Lang's method. In this study, two cases have been considered: an approximation of a 'thin' crystal, *i.e.* the case with negligible absorption, $\mu t \ll 1$ (Bezirganyan, Subotowicz, Sedrakyan & Paprocky, 1988; Sedrakyan, Haroutyunyan, Bezirganyan, Subotowicz & Trouni, 1991); and an approximation of an 'intermediate' thickness of crystal, *i.e.* the case with absorption $\mu t \simeq 1$ (Haroutyunyan, Sedrakyan & Subotowicz, 1996). In the theoretical model, the ion-implanted crystals have been considered as a bicrystal system (thin surface distorted layer with bulk perfect layer) with slightly different lattice constants.

For both above-mentioned approximations ($\mu t \ll 1$ and $\mu t \simeq 1$), the structural investigations of the objects were carried out using traverse topography both theoretically and experimentally, whereas for the method of section topography this problem has been considered by us only theoretically because of inefficiency of the relevant experiments (the corresponding justification for it is presented in §4).

However, as follows from the theoretical results obtained in the present paper, the method of section topography turns out to be effective in structural investigations of similar bicrystalline objects for an approximation of a 'thick' crystal corresponding to the case of heavy X-ray absorption, $\mu t \gg 1$ (see §2). Under this condition, the method of section topography could be applied for experimental determination of a lattice mismatch of the order of 10^{-7} to 10^{-5} as well as of distorted (or epitaxial) layer thicknesses of the order of 1 to 10 µm by means of section-type moiré fringes. From a practical standpoint, the consideration of this problem is important in particular for X-ray diffraction investigations of the bicrystalline systems epitaxial layer–thick substrate (see §3).

2. Intensity distribution in X-ray section topographic images of a BS

The problem is considered under the following assumptions:

(*a*) the reciprocal-lattice vectors **h** and **h**′ of the thin surface layer and the thick layer of the BS differ from one another only by their modulus so that both layers are simultaneously in positions of Bragg reflection for

ISSN 0108-7673 © 1997

X-rays [$|\Delta d/d| \leq 10^{-5}$, $\Delta d/d = (d' - d)/d$, $d = 1/|\mathbf{h}|$, $d' = 1/|\mathbf{h}'|$, where d and d' are the interplanar spacings of the reflecting atomic planes in thin and thick layers, respectively];

(*b*) both layers have a parallel-sided shape;

(*c*) the incident X-ray wave is spherical and monochromatic, the point source S is located at the BS entrance surface [approximation of so-called incident δ-type wave (Kato, 1968)];

(*d*) X-ray diffraction occurs in compliance with the symmetrical Laue reflection scheme;

(*e*) the polarizability of both layers for X-rays is the same;

(*f*) the conditions

$$t_1 < \Lambda, \quad t_2 \gg \Lambda, \tag{1}$$

$$\mu t = \mu(t_1 + t_2) \gg 1 \tag{2}$$

are fulfiled [t is the thickness of the bicrystal, t_1 and t_2 are the thicknesses of the thin and the thick layers, respectively; Λ is the extinction length of the active reflection, μ is the normal linear absorption coefficient of X-rays, which coincides for the two layers in accordance with point (*e*)].

The origin of the rectangular coordinate system xyz, point 0, coincides with the point source S of X-rays, the direction of the z coordinate coincides with the direction of incidence of the X-rays on the thin layer at the exact Bragg angle in kinematical theory and the y axis is perpendicular to the plane of scattering.

Our main aim is the calculation of the section-type topograph intensity distribution in the reflected beam, *i.e.* in the direction h' (see Fig. 1).

Using Kato's spherical-wave theory (Kato, 1968), in our previous work (Sedrakyan, Haroutyunyan, Bezirganyan, Subotowicz & Trouni, 1991), we have obtained for the present problem for an asymmetrical Laue set-up the corresponding spherical-wave solutions

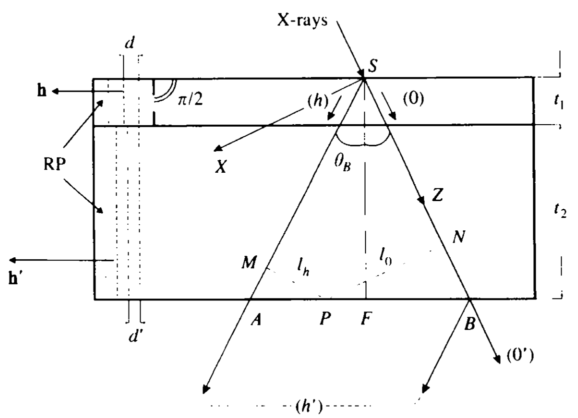

Fig. 1. The diffraction geometry. S is the X-ray point source, RP are the reflecting atomic planes, which are perpendicular to the entrance surface, P is the observation point, $PN = l_0$, $PM = l_h$, $AF = FB$.

$\Phi_{0h'}(\mathbf{r})$ and $\Phi_{hh'}(\mathbf{r})$ on the exit surface of the BS for the asymptotic case $\mu(t_1 + t_2) \ll 1$, *i.e.* for a nonabsorbing bicrystal. These solutions have the following physical sense: the wave field $\Phi_{0h'}(\mathbf{r})$ is the superposition of the plane waves diffracted in thin and thick layers in the directions of transmission (0) and Bragg reflection (h'), respectively, whereas the wave field $\Phi_{hh'}(\mathbf{r})$ is the superposition of the plane waves diffracted in these layers in the directions of reflection (h) and (h'), respectively; $\mathbf{r}$ is the position vector of the observation point. It should be noted that according to the statement of the present problem the following directions of X-ray wave propagation in BS are close to each other: 0 and 0', h and h' (0' is the transmission direction for a wave in a thick layer). The wave fields $\Phi_{0h'}(\mathbf{r})$ and $\Phi_{hh'}(\mathbf{r})$ are given in analytical form by Sedrakyan, Haroutyunyan, Bezirganyan, Subotowicz & Trouni (1991) in expressions (54) and (55), respectively.

To take into account X-ray absorption, we assume χ_m ($m = 0, h, \bar{h}$) to be complex, *i.e.*

$$\chi_m = \chi_{mr} + i\chi_{mi}, \tag{3}$$

where χ_m is the mth-order Fourier coefficient of the polarizability of crystal for X-rays, $\chi_{\bar{h}}$ is the conjugate Fourier coefficient, the indices r and i mean the real and imaginary parts of the relevant quantities, respectively. Using the asymptotic representations

$$\begin{aligned} J_0(u) &\simeq (2/\pi u)^{1/2} \cos(u - \pi/4), \\ J_1(u) &\simeq (2/\pi u)^{1/2} \cos(u - 3\pi/4) \end{aligned} \tag{4}$$

with argument values

$$u = \beta(l_0 l_h)^{1/2} \gg 1 \tag{5}$$

for the zeroth- and first-order Bessel functions involved in equations (54) and (55) of Sedrakyan, Haroutyunyan, Bezirganyan, Subotowicz & Trouni (1991) and substituting (3) into (54) and (55), one obtains, for the section-type topograph intensity distribution of the interference pattern formed in the h' direction for the symmetrical Laue reflection case,

$$\begin{aligned} I_{h'}(\mathbf{r}) &= |\Phi_{0h'}(\mathbf{r}) + \Phi_{hh'}(\mathbf{r})|^2 \\ &= (1/16\pi^2)E^2\beta_r(Kz)^{-1}(l_0 l_h)^{-1/2} \\ &\quad \times \exp(-\mu t_2/\cos\theta_B)\{\cos^2(u_r - \pi/4) \\ &\quad + \sinh^2 u_i + a^2 l_0^{-1} l_h[\cos^2(u_r - 3\pi/4) \\ &\quad + \sinh^2 u_i] - 0.5a(l_h/l_0)^{1/2}\exp(-2u_i)\sin\varphi\}, \end{aligned} \tag{6}$$

where

$$\varphi = 2\pi(\mathbf{h}' - \mathbf{h}) \cdot \mathbf{r} + 0.5K\chi_{0r}t_1/\cos\theta_B + 2\sin\theta_B \Delta s t, \tag{7}$$

$$\beta = KC(\chi_h \chi_{\bar{h}})^{1/2}/\sin 2\theta_B, \quad \beta_r = KC|\chi_{hr}|/\sin 2\theta_B, \tag{8}$$

$$u_r = (l_0 l_h)^{1/2}\beta_r,$$
$$u_i = (l_0 l_h)^{1/2}\beta_i = -(l_0 l_h)^{1/2} K C |\chi_{hi}| / \sin 2\theta_B, \quad (9)$$

$$\mu = K\chi_{0i}, \quad \Delta s = K(\theta_B - \theta'_B), \quad a = \beta_r t_1 \sin\theta_B, \quad (10)$$

sinh is hyperbolic sine, $E, K = 2\pi/\lambda$ and Δs are the amplitude, the vacuum wave number and the incidence parameter of the incident X-ray wave, λ is the radiation wavelength, θ_B and θ'_B are the exact Bragg angles in the thin and thick layers, respectively, C is the polarization factor of X-rays, $l_0 = PN$ and $l_h = PM$ are the distances from an arbitrary observation point P of the region AB at the exit surface to the ASB Borrmann triangle sides SB and SA, respectively (see Fig. 1), $\mathbf{r} = \mathbf{OP} \equiv \mathbf{SP}$ and z is the position vector and z coordinate of the observation point at the exit surface of the BS. Expressions (6) to (10) are derived for a centrosymmetric crystal. Let us restrict ourselves to the consideration of the intensity distribution in the central part (in the neighbourhood of the point F, see Fig. 1; $SF \perp AB$) of the interference pattern, since in the case of a highly absorbing crystal [see (2)] the margin effect (in the vicinities of points A and B) disappears and the X-ray intensity at the margins is much less than in the central part. For observation points from the central part of the interference pattern, the following approximation can be made:

$$l_0 \simeq l_h \simeq (t_1 + t_2)\sin\theta_B. \quad (11)$$

Therefore, taking into account (11), the small imaginary part $|\chi_{hi}| \ll |\chi_{hr}|$ and $|\chi_{0i}| \ll |\chi_{hr}|$, from (2), (5) and (8) the following estimation can be obtained:

$$|u| \simeq \beta_r(t_1 + t_2)\sin\theta_B \gg \mu(t_1 + t_2) \gg 1. \quad (12)$$

Hence, for the asymptotic representation (6) of the solution of the problem under consideration, we need only fulfilment of condition (2).

From (2), (9) and (11), it follows that

$$\sinh^2 u_i \gg 1 \geq \cos^2(u_r - \omega), \quad \omega = \pi/4, 3\pi/4. \quad (13)$$

Using (11) and neglecting due to (13) the cosinusoidal terms in (6), one finally finds

$$I_{h'}(\mathbf{r}) \simeq (1/16\pi^2)E^2\beta_r(Kz)^{-1}[(t_1 + t_2)\sin\theta_B]^{-1} \times \exp(-\mu t_2/\cos\theta_B)[(1 + a^2)\sinh^2 u_i - 0.5a\exp(-2u_i)\sin\varphi]. \quad (14)$$

Because condition (2) corresponds to the case of anomalous Borrmann transmission of X-rays, only the weakly absorbing, *i.e.* σ-polarized, wave can be taken into account. Therefore, in quantities given by (8) and (9) and involved in (14), the value $C = 1$ can be substituted. The intensity oscillations in (14) result from the second term in square brackets. The spacing of the interference pattern (section-type moiré fringes) can be easily obtained from (7) and is

$$g = d^2/|\Delta d|, \quad (15)$$

where $\Delta d = d' - d$. These fringes are parallel to the y axis provided that the statement of the present problem does not change along this axis. To observe the interference fringes, the following condition should be fulfilled:

$$AB/g = 2(t_1 + t_2)\tan\theta_B/g \geq 2, \quad (16)$$

where AB is the base of Borrmann's triangle (see Fig. 1). Condition (16) has the following physical sense: experimental measurements will be effective provided that topographic recording of more than two interference fringes is possible. From (15) and (16) and the condition $t_2 \gg t_1$ [see (1)], it follows that

$$d/t_2\tan\theta_B \equiv (\Delta d/d)_{\min} \lesssim \Delta d/d, \quad (17)$$

which defines the lower limit $(\Delta d/d)_{\min}$ of the strain (or lattice mismatch) $\Delta d/d$, which could be experimentally determined by the approach under consideration. It is obvious from (17) that the greater t_2 (the thickness of the bulk layer) the smaller the lower limit of the measurable strain.

From (14) with (2), (9), (11) and (13) taken into account, the interference pattern contrast in its central part can be found as

$$V = \frac{I_{\max} - I_{\min}}{I_{\max} + I_{\min}} = \frac{a\exp(-2u_i)}{2(1 + a^2)\sinh^2 u_i} \simeq \frac{2a}{1 + a^2}. \quad (18)$$

From (18) and (10), it can be seen that in considering the approximation the contrast does not depend on the thickness t_2 of the thick layer.

Expressions (8), (10) and (18) lead to the following dependence of the thin-layer thickness on the interference fringe contrast:

$$t_1 = (\Lambda/\pi)V^{-1}[1 - (1 - V^2)^{1/2}], \quad (19)$$

where the extinction length $\Lambda = \lambda\cos\theta_B/|\chi_{hr}|$. In the asymptotic case, $V \ll 1$, (19) becomes

$$t_1 = (\Lambda/2\pi)V. \quad (20)$$

3. On the possibility of experimental determination of the epitaxial or distorted-layer thickness and lattice mismatch in a BS

The results obtained in the previous section could be applied to X-ray diffraction experimental investigations of the structure (lattice mismatch and deformations) and determination of the thickness of thin layers of the bicrystal objects epitaxial layer–thick substrate and thin surface distorted layer–thick perfect layer.

From the interference-pattern (moiré fringes) contrast V measurements and substitution of its value into (19), the thickness t_1 of an epitaxial layer (or distorted layer) in a BS can be determined. For instance, a system consisting of a thick Si substrate and a relatively thin epitaxially grown Si layer containing in particular homogeneously distributed impurities (Milvidsky, 1986) can be considered as such a BS. According to Vegard's law, the homogeneously distributed impurities cause in a thin layer the homogeneous lattice strain $\Delta d/d$ (Chikawa, 1965), therefore our theoretical model and hence (19) can be applied to objects with such a homoepitaxial layer for measurements of its thickness [the fulfilment of conditions (1), (2) and (16) is suggested]. From (8), (10) and (18), the plot of contrast of the interference pattern as a function of the epitaxial layer thickness t_1 for the Si bicrystal, Mo $K\alpha$ radiation, 220 reflection is represented in Fig. 2. The curve in Fig. 2 indicates that the contrast is high ($V \gtrsim 0.5$) for thicknesses $t_1 \gtrsim 3\,\mu m$, therefore the accuracy of the measurements of the epitaxial layer thickness will be relatively high for this region.

In an analogous way, the bicrystal system thin distorted layer–thick perfect layer created through ion implantation, atomic diffusion or other influences can be investigated by the present approach. As in the case of X-ray traverse topography of ion-implanted Si crystals with damaged depth $t_1 \simeq 0.1\,\mu m$ (Haroutyunyan, Sedrakyan & Subotowicz, 1996), a linear dependence of the interference pattern contrast *versus* the distorted layer thickness for relatively small values of t_1 (of the order of $0.1\,\mu m$) takes place in the present case [see (20) and Fig. 2].

From (15), a lattice mismatch (distortion) $\Delta d/d$ can be determined from the fringe period g measurement. For the situation related to Fig. 2 (provided that $t_2 \gtrsim 1000\,\mu m$), from (15) and (16) and our initial assumption $|\Delta d|/d \leq 10^{-5}$, one obtains for lattice mismatch $|\Delta d|/d$ values of the order of 10^{-7} to 10^{-5}, which could be revealed by the suggested approach.

4. Discussion

(*a*) Using the possible approximations, for the asymptotic case $\mu(t_1 + t_2) \gg 1$ provided that $t_1 \ll t_2$, we have obtained the intensity distribution of the X-ray section-type moiré pattern [see (14)] in analytical form, which is simple mathematically and is suitable for theoretical analysis.

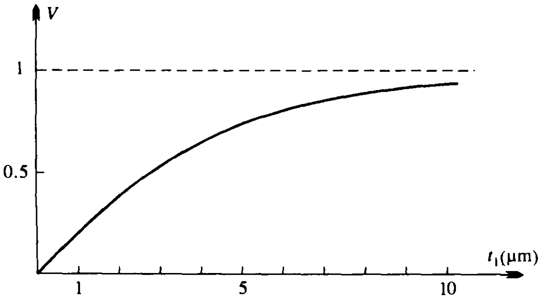

Fig. 2. The X-ray section-type topographic contrast V from a silicon BS (Mo $K\alpha$ radiation, 220 reflection) *versus* the thin-layer thickness t_1 (theoretical curve).

(*b*) As follows from Fig. 2, in the case under consideration, the interference pattern contrast is close to 1 within the range $t_1 \gtrsim 10\,\mu m$. Therefore, the corresponding measurements of epitaxial (distorted) layer thickness for the above-mentioned range could be carried out by means of (19) with a higher accuracy.

(*c*) It is well known that even a small difference $\Delta a/a < 10^{-5}$ (a is the lattice constant) between the lattice constants of epilayer and substrate, *i.e.* misfit boundary, causes the elastic strains in them. However, if the thickness of the epitaxial layer is much smaller than the thickness of the substrate (this condition is fulfiled in the case under consideration), the strain in the substrate is negligibly small in comparison with the strain in the epilayer. Therefore, for epitaxial bicrystals, the theoretical model under consideration for $t_2 \gg t_1$ will be more realistic in comparison to case $t_1 \simeq t_2$.

(*d*) For the given value of μ (*i.e.* for the given material and the X-ray wavelength) in the case under consideration ($\mu t \gg 1$), we have an opportunity to determine experimentally the deformation (mismatch of lattice) $\Delta d/d$ within its wide range of variation (see §3), whereas for $\mu t \ll 1$ and $\mu t \simeq 1$ (when the thickness of the BS is relatively small), these measurements are impossible because of margin effects and violation of the condition (16).

(*e*) In experimental investigations at relatively great values of t_2, a sharp decrease of the X-ray intensity [see (14)] because of heavy absorption could be compensated by increasing the incident-wave amplitude E with monochromatized synchrotron radiation instead of X-radiation from a conventional tube.

(*f*) In the present paper, we have considered the case $\chi_1 = \chi_2$ (the present theory is also applicable to the case $|\chi_1 - \chi_2| \ll |\chi_1|$). However, these results can be easily generalized to the case $|\chi_1 - \chi_2| \simeq |\chi_1|$ (χ_1 and χ_2 are the polarizabilities of the X-rays of the thin and thick layers, respectively).

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