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Formation of X-ray shift fringes and a new method for determination of the difference sign of interplanar distances

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A detailed investigation of the conditions for the formation of X-ray shift fringes is carried out, aiming to apply these patterns to investigations of crystal imperfections. Expressions for the amplitudes and X-ray intensity distribution are obtained for a two-crystal interferometer, in which the interplanar distance between two reflecting planes, d, has a relative change $\Delta d/d \simeq 10^{-8} - 10^{-5}$. It is theoretically proven and experimentally confirmed that the value of the period of interference bands essentially depends on the sign of Δd.

1. Introduction

It is known (Bezirganyan & Eyramjyan, 1970) that X-ray diffraction moiré patterns are very sensitive to structural distortions in the crystals from which the patterns have been obtained. Moiré patterns are one of the most precise methods for measuring very small shifts and rotations of different parts of the same crystal or different crystals with respect to one another (Hart, 1968).

It appears that this supersensitive method might be applied successfully to the investigation of small structural imperfections in quasi-ideal crystals. However, the application of X-ray diffraction moiré patterns for the investigation of structural distortions in crystals is considerably limited as a result of difficulties in unambiguous decoding of these patterns (unambiguous determination of the existence and location of the deformed area). On the one hand, these difficulties occur because X-ray moiré interference patterns result from small shifts and rotations existing between the reflecting planes of the interferometer block (Lang, 1968; Drmeyan, 1987). On the other hand, moiré patterns arise because of the phase shift of superimposed beams (Tanemura & Lang, 1973; Alajajyan *et al.*, 1979). The latter provides an opportunity for obtaining moiré patterns by means of two-block interference systems with a narrow air gap (Authier *et al.*, 1968).

Bezirganyan & Drmeyan (1979) showed that in the case of two-crystal interferometers, where the interplanar distances and directions of reflecting planes in the two blocks are similar, moiré patterns do not arise if the incident wave is plane. In the case of a spherical incident wave, moiré patterns are also obtained when the two crystals have the same interplanar distances and their reflecting planes are exactly parallel.

Bezirganyan & Drmeyan (1981) showed that in the case of deviations from the ideal construction of a multiblock interferometer, or the existence of a narrow air gap (non-diffraction zone) between the blocks of a two-block interferometer, phase shifts arise between the interfering waves (the beams are shifted with respect to one another), thus leading to the formation of interference bands. These patterns can be called shift patterns (shift fringes or lines).

The formation of shift bands in two-block interferometers is nearly unavoidable; even in ideal two-block interferometers, the superimposing beams are shifted with respect to one another, leading to the formation of shift fringes.

Ohler & Härtwig (1999) showed how the theory of moiré fringes on X-ray diffraction topographs of bi-crystals can be derived from the dynamical theory for reflection and transmission cases.

Yoshimura (1991, 1996) considered the gap width of a bicrystal in comparison with the *Pendellösung* length. X-ray moiré topography was discussed by Ohler *et al.* (1996, 1997) and Prieur *et al.* (1996); in particular, Ohler *et al.* (1996) described moiré fringes observed on X-ray diffraction topographs of silicon on insulator structures (produced by the implantation of oxygen) and determined the components of the relative strain tensor from these patterns.

The above-mentioned ideas justify the necessity of a detailed investigation of the mechanism of X-ray diffraction shift patterns in general, and the determination of the sign of the difference in interplanar distances in particular. These two issues are considered in the present paper.

2. Dependence of shift-fringe period on the sign of the difference between interplanar distances

As is very well known, if the reflecting planes of crystals are similarly oriented (exactly parallel) and the interplanar distances are different ($d_2 - d_1 = \Delta d$) then, regardless of the sign of Δd, *i.e.* regardless of on which block the primary beam is incident, we obtain the expression

$$\Delta = d_1 d_2/|d_2 - d_1| \simeq d_0^2/|\Delta d| \quad (1)$$

for the periods of dilatation moiré patterns, where d_0 is the mean value of d_1 and d_2. However, it has been shown both theoretically and experimentally that (1) is an approximation (Pinsker, 1982); it is inapplicable for real experimental conditions.

A more precise and detailed investigation shows that Δ depends on the sign of difference Δd, *i.e.* on the incidence direction of the primary beam.

We will consider this dependence first theoretically, in a spherical wave approximation, and then experimentally.

2.1. Theoretical considerations

Let us determine within the framework of Kato's spherical wave theory (Kato, 1968*a*,*b*; Polcarova, 1978*a*,*b*, 1980) the expressions for amplitudes and intensity distributions of X-ray waves diffracted in the direction of the first reflection in a two-crystal interferometer with a narrow air gap (Fig. 1), where the interplanar distance d between reflecting planes in one of the blocks has a fractional change $\Delta d/d \simeq 10^{-8} - 10^{-5}$.

To obtain moiré patterns with visible periods of fringes in X-ray topographs, the problem according to Bezirganyan & Drmeyan (1979) will be considered for the case of Borrmann transmission of X-rays in both crystals in the symmetric Laue case. The diffraction geometry in real space is shown in Fig. 2.

The intensity distribution of the interference field at the exit surface of the second block is given by the following expression (Tanemura & Lang, 1973):

$$\begin{aligned} I(\mathbf{r}) &= |\Phi_{0g}(\mathbf{r}) + \Phi_{gg}(\mathbf{r})|^2 \\ &= (1/16\pi)^2 E^2(|\beta|/kZ)\exp(-\mu_0 t \sec\Theta_{B1}) \\ &\quad \times \big\{|\xi_1|^2\exp(-2\beta_i u) + |\xi_2|^2\exp(-2\beta_i \nu) \\ &\quad + 2|\xi_1||\xi_2|\exp[-\beta_i(u+\nu)]\cos[\beta_r(u-\nu) \\ &\quad - k\Delta\Theta_B\alpha t_3 - 2t_2\alpha k\Delta\Theta_B + 2\pi\Delta\mathbf{g}\cdot\mathbf{r}]\big\}. \qquad (2) \end{aligned}$$

Here Φ_{0g} and Φ_{gg} are the waves diffracted in the first crystal in directions of transmission and reflection, respectively, and the waves diffracted in the second crystal in the direction of the first reflection,

$$|\xi_1| = \frac{(\eta_1 - \eta_2)u^{-1/2}(\beta_r\eta_1^2 - k\Delta\Theta_B\eta_2 u/2)}{(\eta_1\beta_r)^{1/2}(\beta_r\eta_1^5 - 3\alpha t_1 k\Delta\Theta_B\eta_2 u^3)^{1/2}},$$

$$|\xi_2| = \frac{(\eta_1 + \eta_3)\nu^{-1/2}(\beta_r\eta_1^2 - k\Delta\Theta_B\eta_3\nu/2)(\beta_r\eta_1 - k\Delta\Theta_B\nu)}{(\eta_1\beta_r)^{3/2}(\beta_r\eta_1^5 - 3\alpha t_1 k\Delta\Theta_B\eta_3\nu^3)^{1/2}},$$

$$u = (\eta_1^2 - \eta_2^2)^{1/2}, \quad \nu = (\eta_1^2 - \eta_3^2)^{1/2},$$

$$\eta_1 = \alpha t, \quad \eta_2 = x - \alpha t, \quad \eta_3 = x - \alpha(t + 2t_3), \qquad (3)$$

$$t = t_1 + t_2, \quad \alpha = \sin\Theta_{B1}, \quad \beta = k(\chi_{\bar{g}}\chi_g)^{1/2}\operatorname{cosec}2\Theta_{B1}, \qquad (4)$$

$$\mu_0 = k\chi_{0i}, \quad \Delta\mathbf{g} = \mathbf{g}_2 - \mathbf{g}_1, \quad \Delta\Theta_B = \Theta_{B2} - \Theta_{B1}, \quad |\mathbf{k}| = k,$$

E is the amplitude, $\mathbf{k}$ is the wavevector of the plane-wave component in a vacuum, $\mathbf{g}_1$ and $\mathbf{g}_2$ are reciprocal-lattice vectors in the first and second crystals, respectively, vector $\mathbf{r}$ defines the location of the observation point at the outcome surface of the second crystal, X and Z are the components of this vector, χ_g is the Fourier factor of order g ($g = 0, g$) of the polarizability of the crystal for X-ray waves, $\chi_{\bar{g}}$ is the conjugate Fourier factor for polarizability, χ_{0i} is the imaginary part of χ_0, β_r and β_i are the real and imaginary parts of β, respectively, Θ_{B1} and Θ_{B2} are the exact Bragg angles in kinematic theory for the first and second crystals, respectively, t_1 and t_2 are the thicknesses of the first and second crystals, t_3 is the air gap thickness, and μ_0 is the normal linear absorption coefficient of X-ray waves.

In (2) for the intensity distribution, the first two terms within the braces {} are slowly varying functions of coordinate X; hence the oscillations of the intensity distribution are conditioned by the third term. From the condition

$$\beta_r(u - \nu) - k\Delta\Theta_B\alpha t_3 - 2\alpha t_2 k\Delta\Theta_B + 2\pi\Delta\mathbf{g}\cdot\mathbf{r} = 2\pi\ell, \qquad (5)$$

where ℓ is an integer, we can calculate the period of the interference bands.

Taking into account (3) and (4) and the condition $t_3 \ll t$, the first term on the left-hand side of (5) can be represented in the following approximate form:

$$\beta_r(u - \nu) = (2\pi x' t_3/t\Delta\tan\Theta_B)(1 - x'^2/t^2\tan^2\Theta_B)^{-1/2}, \qquad (6)$$

where x' is the coordinate of the observation point at the exit surface of the second crystal and $\Delta = \pi/\beta_r\sin\Theta_B$ is the extinction depth. The last term on the left-hand side of (5) is equal to

$$2\pi\Delta\mathbf{g}\cdot\mathbf{r} = 2\pi(\Delta d/d^2)x'. \qquad (7)$$

Substituting (6) and (7) into (5) and using the approximation $t \simeq 2t_2$, we obtain the following expression for the coordinate x'_k, where the intensity reaches its maximum value:

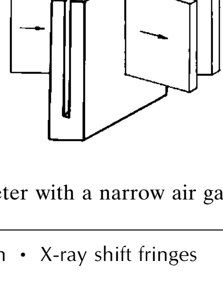

Figure 1
Two-crystal interferometer with a narrow air gap, showing the ray path.

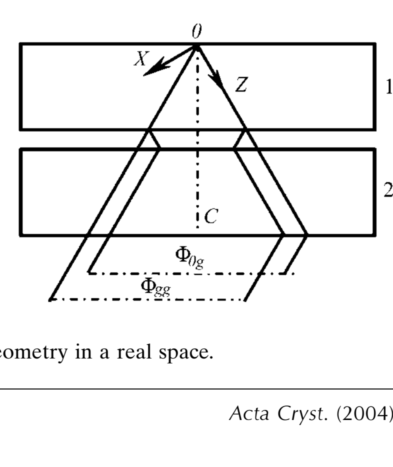

Figure 2
Diffraction geometry in a real space.

$$\frac{2\pi x'_k t_3}{t\Delta \tan \Theta_{\rm B}(1 - x'_k 2/t^2 \tan^2 \Theta_{\rm B})^{1/2}} + 2\pi \frac{\Delta d}{d^2} x'_k - 2\alpha t_2 k \Delta\Theta_{\rm B}(1 + t_3/t) = 2\pi\ell. \qquad (8)$$

Let us introduce the following auxiliary notations in (8):

$$t_3/\Delta = a_1, \quad t \tan \Theta_B = a_2, \quad \Delta d/d^2 = a_3,$$
$$2\alpha t_2 \Delta\Theta_{\rm B}/\lambda(1 + t_3/t) = n, \quad \ell + n = m,$$

where λ is the wavelength. Then after some transformation we obtain the following equation of fourth order for x'_i:

$$a_3^2(x'_i)^4 - 2a_3 m(x'_i)^3 + (m^2 + a_1^2 - a_2^2 a_3^2)(x'_i)^2 + 2a_3 a_2^2 m x'_i - m^2 a_2^2 = 0. \qquad (9)$$

The solution of (9) gives the positions of the intensity distribution maxima, x'_i, where i is the number of each maximum. The period of interference bands can be found from the expression

$$\sigma_{i,i+1} = x'_{i+1} - x'_i. \qquad (10)$$

It is obvious that the period of the interference bands depends on the sign of Δd and the direction of $\Delta\mathbf{g}$, *i.e.* on the sign of the scalar product $2\pi\Delta\mathbf{g}\cdot\mathbf{r}$. From (9) and (10), the periods $\sigma_{i,i+1}$ ($i = 0, 1, 2, 3, 4$) for the first five interference bands, closest to the point C (Fig. 2), are calculated with an accuracy of 1 µm at values of $\Delta d/d = 2 \times 10^{-6}$ and -2×10^{-6} (*i.e.* $d_2 < d_1$ and $d_2 > d_1$) for a two-crystal interferometer with a $t_1 = t_2 = 0.5$ cm-thick silicon crystal and a gap width of $t_3 = 350$ µm for Mo $K\alpha$ radiation and reflection 220. The results are presented in Table 1.

In the case of $\Delta d < 0$ ($d_2 < d_1$) at $|\Delta d|/d = 2 \times 10^{-6}$, the period of the interference bands is significantly larger. Thus theoretical calculations show that the magnitude of interference-band period essentially depends on the sign of Δd.

2.2. Experimental

A two-crystal system prepared from a nearly perfect silicon single crystal grown by the floating-zone method, placed on a common base, was used for the experimental investigation of the dependence of shift fringes on the sign of the interplanar distance difference between the reflecting planes. The blocks were placed so close to one another that the waves diffracted in the first crystal were superimposed at the entrance surface

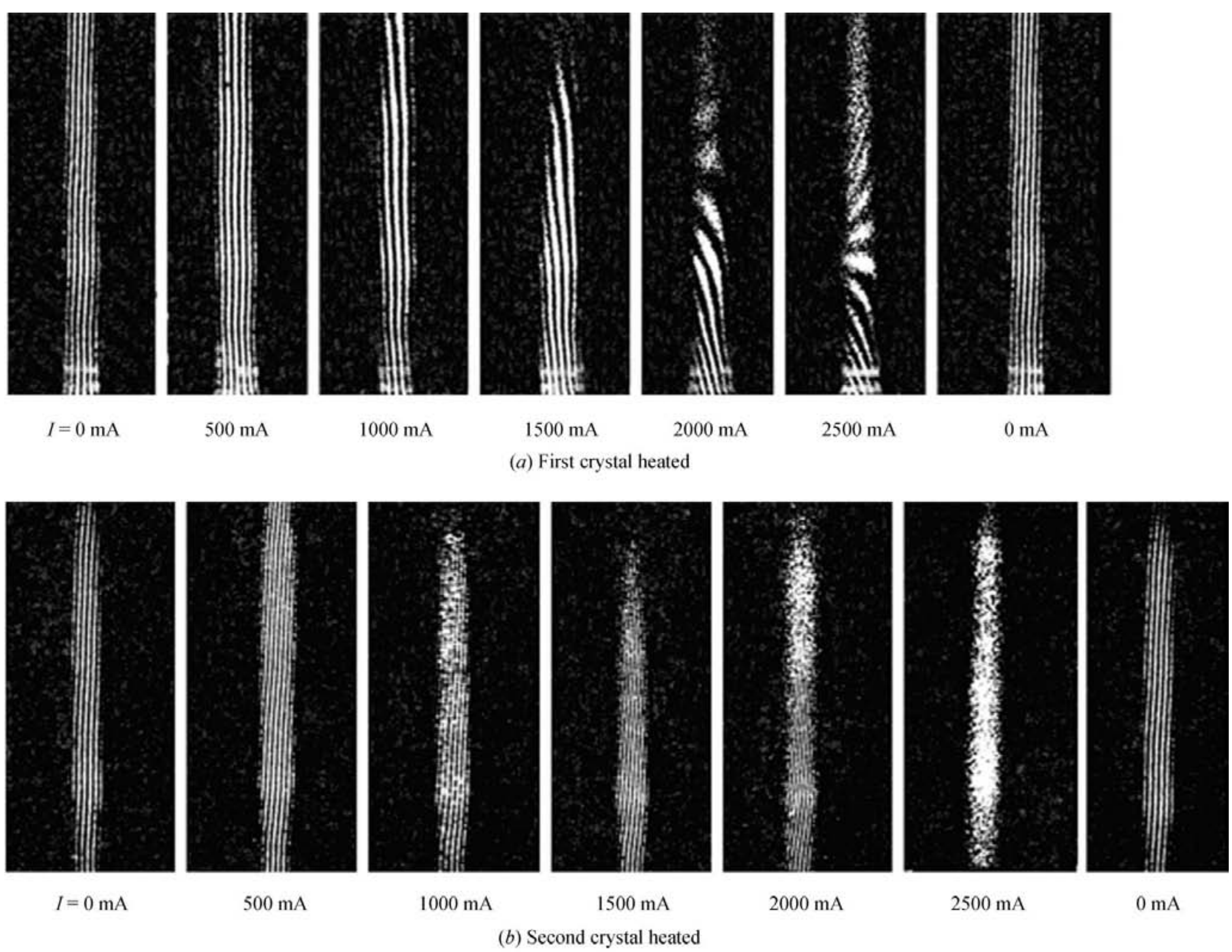

Figure 3
Photographs of section topographs for various cases.

Table 1
The dependence of interference-band period on $\Delta d/d$.

$\Delta d/d$	$\sigma_{0,1}$	$\sigma_{1,2}$	$\sigma_{2,3}$	$\sigma_{3,4}$	$\sigma_{4,5}$
2×10^{-6}	122	122	120	118	115
-2×10^{-6}	373	323	246	184	140

of the second crystal. The directions of the reflecting planes in both crystals were strictly parallel. The sample was prepared from a dislocation-free silicon single crystal, cut by a thin diamond saw out of a parallelepiped silicon bar (Fig. 1). To obtain the difference of interplanar distances between the reflecting planes, one of the crystals was heated, trying to avoid a temperature gradient.

Experiments investigating the dependence of shift-fringe period on the sign of the interplanar distance difference Δd were carried out in three different ways:

(i) with both crystals at room temperature,

(ii) with the first crystal heated and the second at room temperature,

(iii) with the second crystal heated and the first at room temperature.

Experiments were carried out under conditions corresponding to those used for the theoretical calculations. The photographs of section topographs are presented in Fig. 3. The first and last topographs shown in Figs. 3(*a*) and 3(*b*) were obtained at room temperature. Other topographs were obtained during a gradual increase of temperature in separate crystals. The current strength in the heater is given below the topographs (a current increase leads to an increase of crystal temperature). From the obtained photographs we can see that

(i) Heating the first crystal results in an increase of shift-fringe period, while heating the second crystal decreases this period.

(ii) After heating (zero current in the heater), the periods of the shift fringes regain their initial values [see the first and last topographs in Figs. 3(*a*) and 3(*b*)].

(iii) At high temperatures (high current in the heater), the shift fringes are initially distorted (impact of the temperature gradient), then they disappear.

Thus, our experiments (in agreement with theoretical calculations) reveal that the period of the shift fringes depends on the sign of the difference in the interplanar distances. Similar results were obtained for the case in which the incidence direction of the initial beam was changed while heating the same crystal.

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