
Reconstruction of spatial distribution of strains in crystals using the energy spectrum of X-ray Moiré patterns

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Abstract. We develop a new approach to the analysis of experimental Moiré patterns obtained with LLL-interferometer. Radial distributions of the energy spectra of Moiré patterns reveal different sensitivities to the sources of local mechanical strains in the low- and high-frequency ranges. This offers new possibilities for determining the magnitudes of total mechanical strains and reconstructing their spatial distributions in crystals.

Keywords: LLL-interferometers, Moiré patterns, strain fields, frequencies of radial distributions, Fourier energy spectra

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1. Introduction

Optical interferometric methods have given great impulse to novel techniques of multicrystalline interferometry of X-ray and neutron radiations [1–3]. The first studies [2, 4] have shown that the X-ray interferometry reveals extremely high sensitivity to any disturbance of crystalline structure [5, 6]. Currently, this method has many important practical applications in metrology, e.g. when determining the Avogadro constant [3, 7, 8] and standardizing X-ray wavelengths [3]. It also represents a powerful tool for the phase tomography studies of medical and biological samples [9–11]. Implementation of combined optical and X-ray interferometries in Refs. [12, 13] has discovered even such a fine effect as small influence of surface pressure on the interplanar distances in silicon crystals. A promising setup for a multicrystalline interferometer has been suggested in the work [14].

The effect of structural defects and external actions on the formation of Moiré images using a three-crystal LLL-interferometer has been studied in Refs. [15–19]. At the same time, it is worthwhile that the quality of interference images is influenced significantly by the structural defects and macroscopic mechanical strains present in the plates introduced in interferometer. Since the X-ray interferometry rests upon basic laws of optics [20], the studies of formation of X-ray interference patterns in the analyzer of a so-called LLL-interferometer, which creates a predetermined strain field, are important for the further development of methods aimed at reproducing the strain-field distributions and calculating the strain-tensor components created by defects [21].

Nowadays the dynamic theory of X-rays enables one to carry out any necessary numerical simulations of the Moiré patterns under conditions of predetermined strain fields [22–25]. If one analyzes some experimental Moiré patterns, the approach makes it potentially possible to obtain all the quantitative information about the sources of residual or active strains and their spatial distribution. The same refers to the nature of appropriate effects [24]. The models that involve

different distributed sets of concentrated forces, which have been used in the works [16, 19, 22–25], allow for investigating the influence of long-period or localized strain fields on the X-ray diffraction and the interference processes occurring in LLL-interferometers.

The Moiré images created by one-dimensional sets of local concentrated forces on the output surface of the analyzer of three-crystal LLL-interferometer have been investigated in the works [23, 24] for the both cases of orientations of those forces parallel or perpendicular to the diffraction vector. Different sets of force magnitudes and their spatial distributions selected in the above works have testified qualitative agreement of theoretical Moiré patterns with the experimental ones, which have been obtained from the residual strain fields created by the scratches during scribing the output surface of the analyzer [15].

New strain-induced Moiré bands occur near the places where the concentrated forces are localized, i.e. in a transition region where the strains change their signs. The shape of those bands reflects a general displacement field of the atomic planes created near the sources of forces [24]. On the other hand, increase in the period of the Moiré bands upon withdrawal of the concentrated forces reflects the behaviour of the total strain field.

The presence of a constant phase shift in one of the interfering beams in the interferometer analyzer induces a decrease in the period, the contrast and the size of the area where the strain-induced Moiré bands are formed [23]. The area of efficient interaction of the phase and strain Moiré components depends not only on the value of constant phase shift, but also on the magnitude and distribution of local force sources.

In the study [25] we have developed the approach that can reproduce dependence of the mean spatial frequency of the energy spectra of Moiré images on the magnitude of strain field generated as a total of local strain sources. This enables one to estimate the magnitude of the averaged residual strain, which has generated the Moiré pattern in the process of scribing the analyzer surface, using the average spatial frequency of the energy spectrum [15]. At the same time, one could not establish the relation between the parameters of energy spectra and the spatial configuration of strain distribution.

In the present study, we consider new approaches to the analysis of Moiré intensity distributions basing on the Fourier energy spectrum method [25, 26]. We demonstrate different sensitivities to the spatial distributions of local strain sources, which are revealed by the radial energy spectra of Moiré images in their low-frequency and high-frequency ranges. As a consequence, one can determine on this basis both the total magnitude of the strains and their spatial distribution in crystals.

2. Method for determining the distribution of force sources P_k

To calculate Moiré intensity distribution as a function of magnitude of the concentrated forces and their spatial arrangement, we have utilized the algorithm and the analytical relations given in Refs. [22–25]. A scheme of action of some local strain sources P_1, \dots, P_N on the output surface of the LLL-interferometer is shown in Fig. 1. A sum P_{NL} presented in relative units corresponds to the sum of forces P_d (in Newtons). For example, we have $P_d = 0.08 \text{ N}$ in the case of $P_{NL} = 100$, which characterizes a region of weak strains [15]. The relationship for the sum of forces P_{NL} and the P_d parameter is described as (see Ref. [22])

$$P_d = \sin \theta \frac{P_{NL}}{2\pi GL} |\vec{H}|, \quad (1)$$

where θ is the Bragg angle of X-ray reflection (e.g. for the (220) $\text{CuK}\alpha$ -radiation), G the shear modulus, \vec{H} the diffraction vector, and L the length of a line segment along which a series of forces P_1, \dots, P_N acts.

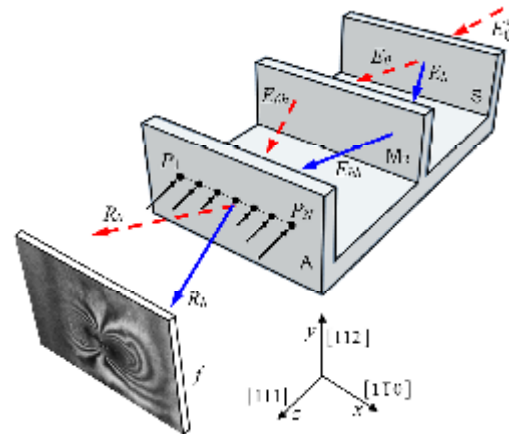


Fig. 1. Scheme of X-ray LLL-interferometer (see also Ref. [1]); S – splitter, M – mirror, A – analyzer, E_0^a – incident X-ray beam, E_0 and E_1 – refracted and diffracted beams after splitter S , E_{0b} and E_{1b} – diffracted beams after mirror M , and R_0 and R_1 – output beams from the interferometer, which form a Moiré pattern f due to the action of concentrated forces P_1, \dots, P_N .

In order to establish the relationship between the strain and the Moiré pattern parameters, we have performed the appropriate calculations for five different P_{ML} values (21, 52, 84, 105 and 147). This has been done for the following three characteristic shapes S_i of the distributions P_k : S_1 – a uniform distribution P_k^1 , S_2 – a parabolically curved distribution P_k^2 with a minimum force value corresponding to the centre, and S_3 – a curved distribution P_k^3 with a maximum in the centre [18]. This area of P_{ML} values reflects completely the initial and intermediate stages of formation of additional Moiré bands in Fig. 2.

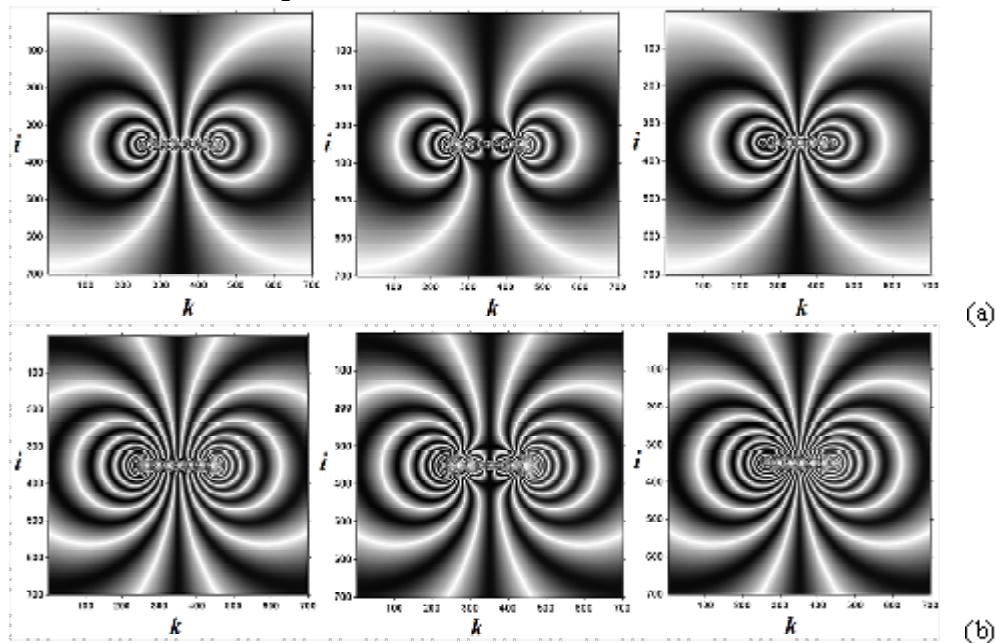


Fig. 2. Fragment of a set of X-ray Moiré patterns, which are presented as matrices $f(i, k)$ for the distributions of concentrated forces P_k^1 , P_k^2 and P_k^3 (with the shapes S_1 , S_2 and S_3 , respectively). P_{ML} is equal to 52 (panel a) and 105 (panel b).

To carry out Fourier transformation and calculate the energy spectra, the Moiré patterns $P_M(x, y)$ shown in Fig. 2 have been represented as digital matrixes $f = f(i, k)$, where the index i concerns the series $1, \dots, M_1$ and k the series $1, \dots, N_k$. Here M_1 is the height and N_k the width of the pattern in pixels.

The X-ray Moiré patterns f depend significantly on the magnitude and the type of local force distribution (see Fig. 2). We are to note that solving uniquely the inverse problem (i.e., reconstructing spatial distribution and magnitudes of the strains that generate a Moiré pattern, issuing from this pattern) is still hindered by essential difficulties. We offer an original method to overcome them and solve the problem.

For this aim, we perform a two-dimensional direct discrete fast Fourier transform of the matrix $f(i, k)$. The energy spectrum P_S (or the spectral power density) is then determined by the square module of F_C [26]:

$$P_S = |F_C|^2. \quad (2)$$

The radial distribution (or profile) $P_R(d)$ of the Moiré pattern is calculated using the energy spectrum P_S . Here d is the radial frequency number ($d = 1, \dots, N_R$) and $N_R = \min(M_1, N_k) / 2$.

In any Moiré pattern, a high-frequency noise is present in the most of cases [27]. It manifests itself as a background in the radial distributions $P_R(d)$, especially at high spatial frequencies ($v_r > 0.2 \text{ pel}^{-1}$). Therefore one should remove the noise component from the distributions P_R in order to calculate accurately the average spatial frequency \bar{v}_R . This spatial frequency is calculated from the radial distribution $P_R(d)$ of the energy spectrum [25] as follows:

$$\bar{v}_R = \frac{\sum_{d=2}^{N_R} P_R(d) \cdot v_r(d)}{\sum_{d=2}^{N_R} P_R(d)}. \quad (3)$$

Here the constant component $P_R(1)$ is not taken into account when calculating the average frequency. Note that, contrary to the P_R values for a given frequency, the average spatial frequency \bar{v}_R of the radial distribution P_R does not depend on the experimental conditions under which the image has been obtained.

Our analysis of the Moiré patterns calculated for the cases of different sums of forces P_{MZ} enables finding a correlation between the \bar{v}_R and P_{MZ} parameters. This makes it possible to reconstruct the sums of the concentrated forces P_{MZ}^Y on the basis of the frequency \bar{v}_R , using a piecewise linear fitting [25]. However, no correlation has been found between the frequency \bar{v}_R and the nature of the force distribution P_R . As a result, there is a need in examining the correlation between the distributions P_R and P_R .

We develop a theoretical approach to calculate the distribution of forces P_R , issuing from the parameters of Moiré patterns. For this aim, we have analyzed a set of reference images shown in Fig. 2. The relationship between the nature of P_R and the frequency of radial distribution P_R can be ascertained after analyzing the shape of P_R in different ranges of the v_r frequency. We have performed the analysis for the dependence $P_R(v_r)$ not only for the distributions P_R containing high-frequency oscillations, but also for the distributions P_{R^*} , which are obtained after transforming P_R to the logarithmic scale and fitting it with a polynomial of 20th degree (see Fig. 3).

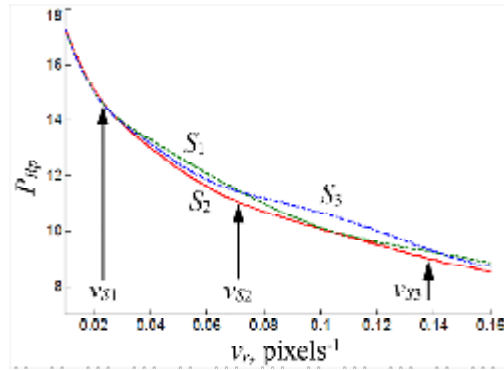


Fig. 3. Fragments of fitted radial distributions $P_R(v)$ of the energy spectra P_S of Moiré patterns on a semi-log scale. Frequencies v_{S1} , v_{S2} and v_{S3} are the boundaries of different ranges, $P_{NL} = 105$, and S_1 , S_2 and S_3 are the shapes of force distributions.

It has been demonstrated that the distributions $P_R(v)$ exhibit different sensitivities to the nature of force distribution in the two alternative frequency ranges (see Fig. 3). Here the range #1 is limited by the spatial frequencies v_{S1} and v_{S2} , while the range #2 is limited by the frequencies v_{S2} and v_{S3} . The frequencies v_{S1} , v_{S2} and v_{S3} , which are the abscissas of intersection points of the distributions P_R with the shapes S_1 and S_3 , have been determined for each sum of forces P_{NL} separately (see Fig. 3).

Fig. 4 shows dependences of the range boundaries v_{S1} , v_{S2} and v_{S3} on the sum of forces P_{NL} , which have been obtained for a whole set of reference Moiré patterns. Using a piecewise linear fitting such dependences can be used to calculate the frequencies v_{S1} , v_{S2} and v_{S3} for random P_{NL} values. Then the relative areas S_{R1} and S_{R2} can be estimated for the radial distribution $P_R(v)$ inside the calculated frequency ranges (v_{S1} , v_{S2}) and (v_{S2} , v_{S3}):

$$S_{R1} = \frac{\sum_{v_r=v_{S1}}^{v_{S2}} P_R(v_r)}{\sum_{v_r=v_{S1}}^{v_{S3}} P_R(v_r)}, \quad (4)$$

$$S_{R2} = \frac{\sum_{v_r=v_{S2}}^{v_{S3}} P_R(v_r)}{\sum_{v_r=v_{S1}}^{v_{S3}} P_R(v_r)}. \quad (5)$$

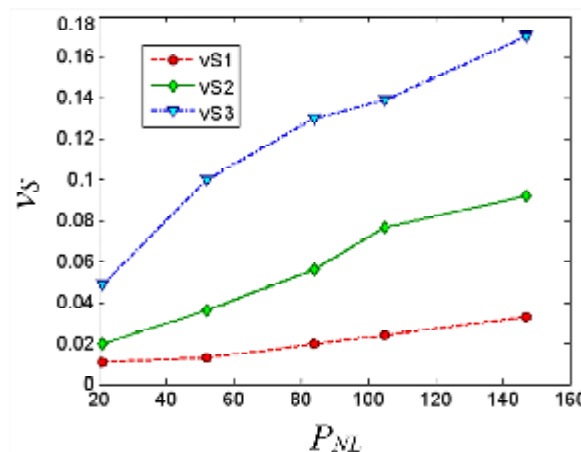


Fig. 4. Dependences $v_{S1}(P_{NL})$, $v_{S2}(P_{NL})$ and $v_{S3}(P_{NL})$ for the boundaries of frequency ranges, as calculated for the Moiré patterns shown in Fig. 2.

The S_{E1} and S_{E2} values have been averaged for all the Moiré patterns with the same sum value P_M . The result is given by the average areas S_{A1} and S_{A2} . The approximate mean areas S_{A1}^a and S_{A2}^a have been calculated by piecewise linear fitting of the dependences $S_{A1}(P_{M1})$ and $S_{A2}(P_{M2})$, basing on the calculated P_{M1} and P_{M2} (for P_R) are then determined by the equations

$$S_{M1} = \frac{S_{F1}}{S_{A1}^a}, \quad S_{M2} = \frac{S_{F2}}{S_{A2}^a}. \quad (6)$$

A transformation of the areas from S_{E1} and S_{E2} to S_{M1} and S_{M2} suggested above yields in the fact that the values S_{M1} and S_{M2} are no longer dependent on the force sum P_{M1} but depend mainly on the shape of the force distribution P_R .

The values S_{M1} and S_{M2} have been averaged for the reference Moiré patterns corresponding to the same force distribution (S_1 , S_2 or S_3), thus resulting in the corresponding mean normalized values S_{F1} and S_{F2} for each of the force distributions. The force distributions S_1 , S_2 and S_3 correspond to the theoretical amplitudes of sine function A_R which are equal respectively to 0, -0.7 and 0.7. However, the dependences obtained thus far are still ambiguous and do not allow one to calculate A_P following from S_{F1} or S_{F2} (see Table 1).

Table 1. S_{F1} , S_{F2} and S_{F3} values depending on P_R as derived for the set of images in Fig. 3.

Shape of forces distribution	A_P	S_{F1}	S_{F2}	S_{F3}
S_2	-0.7	1.0224	0.8622	0.9612
S_1	0	1.1110	0.8208	1.0000
S_3	0.7	1.0237	1.2904	1.1256

This is why we have calculated the parameter S_{F3} from Table 1, which is determined as a linear combination of S_{F1} and S_{F2} using the proportion of gold cross section. In this manner we obtain a unique dependence of A_P on the parameters of energy spectrum of the Moiré pattern:

$$S_{F3} = S_{F1} \cdot 0.618 + S_{F2} \cdot 0.382. \quad (7)$$

Eq. (7) implies the following conditions: (i) if $S_{F3} = 1$, then there is a uniform shape S_1 of force distribution ($A_P = 0$). (ii) if $S_{F3} < 1$, we have the shape S_2 with a minimum at the centre ($A_P < 0$) and, finally, (iii) the inequality $S_{F3} > 1$ means the shape S_3 with a maximum at the centre ($A_P > 0$).

The final empirical dependence of the reconstructed amplitude A_P of sine function, which describes the force distribution P_R (see Fig. 5), on the parameter S_{F3} of the energy spectrum is as follows:

$$A_P = 2 \left(\frac{1}{1 + \exp(-(S_{F3} - 1) \cdot C_1)} - \frac{1}{2} \right). \quad (8)$$

It is described according to the $S_{F3}(A_P)$ values (see Table 1). In our case we have $C_1 = 43$ (at $S_{F3} \leq 1$) or $C_1 = 14$ (at $S_{F3} > 1$). The dependence $A_P(S_{F3})$ provides the A_P changes occurring in the acceptable range (from -1 to 1) for arbitrary S_{F3} values. The dependences $A_P(S_{F3})$ are different for the force distributions with the shapes S_2 and S_3 , which is taken into account by different C_1 coefficients.

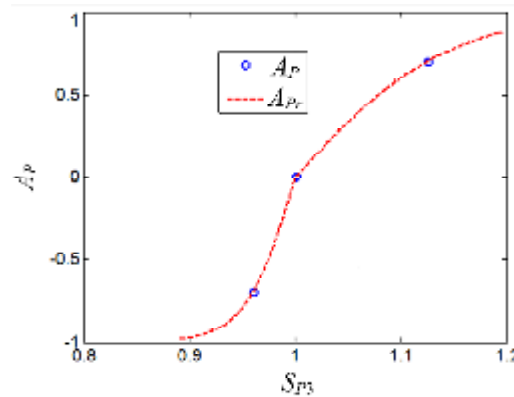


Fig. 5. Dependence $A_p(S_{PJ})$: S_{PJ} is the average normalized area of the radial distributions P_R for the images shown in Fig. 2, the parameters A_p are given by Table 1, and A_{p_r} is the amplitude value reconstructed using Eq. (8), which describes the force distribution P_r .

3. Comparison of calculated and experimental Moiré patterns

Our approach suggests a solution to the inverse problem of finding the sum P_{NL} of forces and the force distribution P_n (with $n = 1, \dots, N$), following from the analysis of Moiré patterns. The appropriate algorithm is as follows. First, the total force P_{NL}^r is determined from the mean frequencies ν_R of the radial distribution P_R [18] (see Fig. 6). The mean square error of P_{NL}^r calculations is negligible for the set of reference images: we obtain the absolute error $R_{pL} = 1.4$ and the relative error $\varepsilon_{pL} = 1.7\%$.

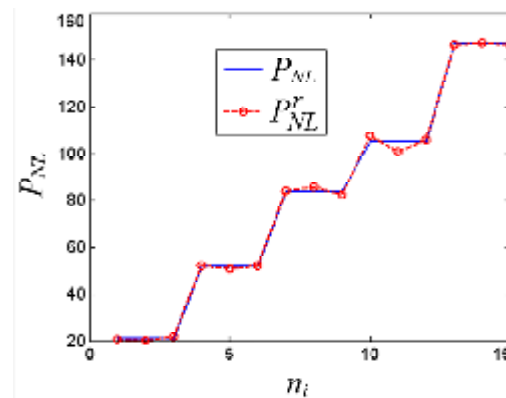


Fig. 6. Comparison of experimentally reconstructed (P_{NL}^r) and theoretical (P_{NL}) forces: the P_{NL}^r values found from the ν_R values coincide in practice with the P_{NL} values that correspond to the images n_i (see Fig. 2). The cases $P_{NL} = 21, 52, 84, 105$ and 147 are considered.

The force distribution P_n (with $n = 1, \dots, N$) can be determined issuing from the analysis of P_R shape. The force values P_n (of which sum equals to P_{NL}) are described by the sine function with the amplitude A_{p_r} and the period $T_p = 2N - 1$, where the amplitude A_{p_r} can be calculated basing on the P_R values and Eq. (8).

As a result, Fig. 7 shows the force distributions P_{n_r} reconstructed using the analysis of Moiré patterns depicted in Fig. 2. The root mean square error, i.e. the discrepancy between the P_{n_r} and P_n values remains acceptable for all of the reference images. Namely, we have the absolute error $R_p = 1.5$ and the relative error $\varepsilon_p = 1.8\%$. Different panels of Fig. 7 illustrate the following specific cases:

(a) the shape S_3 , $F_{MZ}^* = 105.3$, and $A_{F^*} = 0.66$; (b) the shape S_1 , $F_{MZ}^* = 107$, and $A_{F^*} = 0.09$; (c) the shape S_2 , $F_{MZ}^* = 102.4$, and $A_{F^*} = -0.63$; (d) a new shape similar to S_2 , $F_{MZ}^* = 100.7$, and $A_{F^*} = -0.99$. Here n is the number of concentrated forces. In particular, the relative P_{nr} error is $\varepsilon_P = 1.6\%$ for the reference images (see panels a, b and c in Fig. 7) and it becomes only slightly higher ($\varepsilon_P = 3.1\%$) for the test image of Fig. 7d.

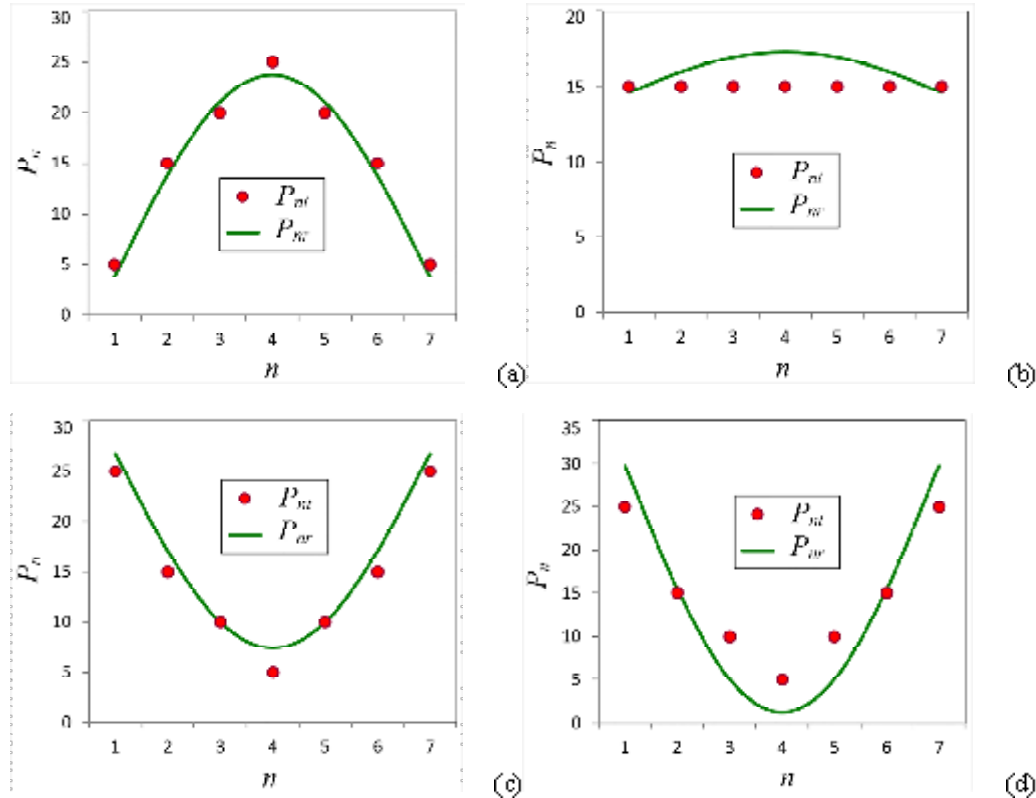


Fig. 7. Theoretical (P_n^t) and reconstructed (P_n^r) force distributions as functional dependences. The distributions are obtained basing on both the reference Moiré patterns (see Fig. 2b) and the test Moiré pattern for the cases of $P_{MZ}^* = 105$ and various shapes (S_1 , S_2 and S_3).

It is important that a satisfactory agreement between the reconstructed (P_n^r) and theoretical (P_n^t) forces has been obtained not only for the reference images and the distribution shapes S_1 , S_2 and S_3 , but also for the test image with a new distribution shape (see Fig. 7d). The root mean square deviation between the P_n^r and P_n^t values for the test image ($P_{MZ}^* = 21, \dots, 147$) is quite acceptable: the absolute error amounts to $R_P = 2.3$ and the relative error to $\varepsilon_P = 2.7\%$. This confirms the fact that our method is versatile when reconstructing the force distributions with various shapes.

Finally, we have estimated a sum of residual forces F_{MZ}^* and a possible force distribution P_n^r (see Fig. 3b), using our method and analyzing the experimental Moiré pattern obtained in Ref. [15] (see Fig. 8a). The obtained sum of forces, $F_{MZ}^* = 43.7$, is close to the data derived with the independent method [25] ($F_{MZ}^* = 41.7$), while the relative calculation error for the sum of forces is equal to $\varepsilon_{F^*} = 4.6\%$. This testifies correctness and great potentials of our approach to the quantitative analysis of Moiré patterns.

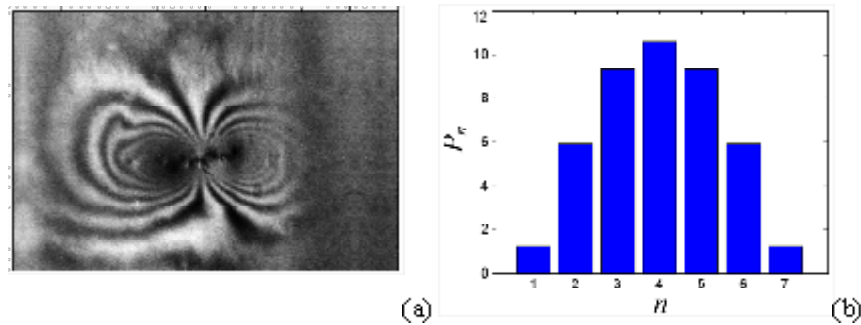


Fig. 8. (a) Experimental Moiré pattern (800×541 pel) caused by residual strains resulted from an indenter on the output surface of LLL-interferometer analyzer [15]; (b) reconstructed force distribution (profile) that causes these strains and results in the Moiré pattern shown in Fig. 8a. The parameters thus obtained are $F_{\Sigma} = 43.7$, $P_d = 0.035$ N and $A_{Pr} = 0.75$.

4. Conclusions

Summarizing the main results of the present work, we state the following. The X-ray Moiré intensity distributions depend essentially on both the magnitude and the nature of spatial distribution of local forces acting on the surface of LLL-interferometer analyzer. In particular, we have revealed that the $P_{\Sigma}(v_R)$ dependence shows different sensitivities to the nature of force distribution in the low-frequency and high-frequency ranges. The approach to analysis of the Moiré patterns suggested by us allows determining the total magnitude of active forces and, moreover, reconstructing their spatial distribution.

The total force P_{Σ} that causes a particular energy spectrum f can be determined from the mean spatial radial frequency v_R by fitting the dependences $P_{\Sigma}(v_R)$. The relative error for calculating the P_{Σ} parameter is acceptable for both the reference ($\varepsilon_{PL} = 1.7\%$) and experimental ($\varepsilon_{PL} = 4.6\%$) Moiré patterns.

The spatial distribution of the acting forces P_n can be determined from the analysis of the shape of P_{Σ} using the energy spectrum f and taking into account the total force P_{Σ} . The shape of the spatial distribution P_n is functionally described by sinusoids, whose amplitudes can be calculated following from the normalized areas of distributions P_{Σ} for the two alternative frequency ranges. The relationship between the force distribution P_n and the parameter P_{Σ} has been found after analyzing a set of reference Moiré patterns.

The method for reconstructing the spatial distributions of concentrated forces P_n suggested in this work can be used to find random forces and corresponding mechanical strains in crystals, which are spatially localized along a linear segment and of which values can be fitted by the sine function.

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Анотація. Розвинуто новий підхід до аналізу експериментальних муарових картин, одержаних за допомогою LLL-інтерферометра. Радіальні розподіли енергетичних спектрів муарових зображень виявляють різну чутливість до джерел локальних механічних напружень у низькочастотному та високочастотному діапазонах. Це пропонує нові можливості для визначення загальної величини механічних деформацій та реконструкції їхнього просторового розподілу в кристалах.