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Stereometrical X-ray Diffraction Topography of Crystal Imperfections

The common X-ray diffraction projection topography of crystal imperfections is essentially deficient. First, in the process of projection topography the spatial pictures of crystal imperfections are projected on the plane (of an X-ray film or plate) and the three-dimensional picture is turned into two-dimensional image. Besides, the diffraction pictures of defects located at different crystal depths and giving image in the same diffracted beam (in the same direction) often overlap.

These inherent deficiencies of conventional X-ray diffraction projection topography essentially reduce the spatial and linear resolutions of the topograms.

Secondly, the displacement of scatterers (the motive atoms) in the reflecting planes is known not to lead to the rise of additional phase differences between the waves scattered in the direction of an observation point. Hence, the diffraction images are actually the pictures of such defects (or of their parts), which give rise to displacements in the direction of the normals to the reflecting planes. Thus, the common diffraction images of imperfections (defects) contained in the diffracted beams are not complete even before the projection,- they mainly represent the pictures of deformation fields formed in the direction of the normal to reflecting planes. In other words, the X-ray reflexes respond to structural defects only when deformational displacements of the atoms caused by these defects are perpendicular to the atomic planes, by means of reflections from which the given reflex is formed. Strictly speaking, only those defects do not affect the intensity of reflexes, the displacements of which are on the reflecting planes. In particular, the contrast (visibility) of the dislocation line (image) is strongly dependent on the orientation of the Burgers vector relative to the reflecting planes,- the image of dislocation has maximum contrast when the Burgers vector is perpendicular to reflecting planes. Further on, the contrast of segregation lines depends on the orientation of reflecting planes with respect to the crystal growth axis - they are visible with maximum contrast when the diffraction vector is parallel to the growth axis, and when the diffraction vector is normal to the growth axis, then the segregation lines are invisible. When in segregated regions of the crystal there are simultaneously also excretions, then the segregation bands are observed irrespective of the values of angle between the diffraction vectors and the crystal growth axis.

Thus, the topograms and interferograms obtained from one family of reflecting planes do not give a complete picture of imperfections of a crystal under investigation. Hence, to have more or less detailed description of the deformed state of crystals, it is necessary to obtain the defectogram from the same crystal if only with the help of two or three plane families having different orientations.

Hence, the acute need in completely stereometrical X-ray diffraction topography of single crystal imperfections is obvious.

Lang has proposed a stereometrical technique of projection topography [1] . This aim_in_view he proposed to use the reflecting planes hkl and $\bar{h}\bar{k}\bar{l}$, but as is mentioned above it is easy to see that the second deficiency is not overcome in this scheme. Indeed, the normals to hkl and $\bar{h}\bar{k}\bar{l}$ planes practically coincide and the diffracted rays bear the images of deformation field only in this direction. This technique is only partly adequate as regards the first of the mentioned deficiencies, because it gives limited information about the position of defects in the bulk of crystal under irradiation.

For more or less complete solution of the problem it is necessary to use symmetrically equivalent families of reflecting planes making comparatively large angles with each other and in the present work this scheme is discussed.

It is evident that unambiguous interpretation of defect images is essentially simplified if the radiograms are obtained from symmetrical reflections on families having similar interplane distances, i.e., belonging to the same system of planes,- the complex of symmetrically equivalent planes.

One should keep in mind that the interferometric methods for the investigation of crystal imperfections are more sensitive than the topographic ones. One-crystal (simple) topographic pictures, which allow direct observation of defects in crystals, are images of imperfections, while the interferometric pictures are interference patterns due to the deformation field of crystal lattice distortions. The resolution and sensitivity of interferometric methods much exceed those of the topographic ones and, besides, it is difficult to reveal uniform deformations in separate crystals by means of topographic methods. On the other hand, in interferometric investigations with the same interferometer it is rather difficult to use two or three families, belonging to the same complex of symmetrically equivalent, strongly reflecting planes.

In all the known X-ray interferometric methods [2,3] for the investigation of crystal imperfections, usually single-stage interferometers with one family of reflecting planes were utilized. As a rule, in such interferometers only one family of strongly reflecting planes, belonging to the same complex of symmetrically equivalent planes, is conveniently oriented for the reflection. Hence, the interferograms obtained with these interferometers do not give detailed picture of imperfections of the crystal under investigation.

In the present work we propose a novel X-ray interferometric method based on multiple interferometers, which allows to describe the deformation fields of crystals in more detail.

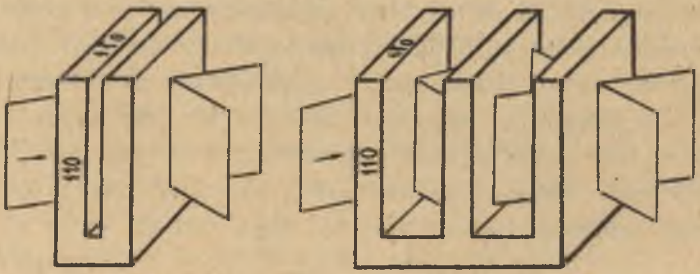
1.DOUBLE TWO-CRYSTAL AND THREE-CRYSTAL INTERFEROMETER

In all our experimental investigations we used silicon samples.

As is seen from formula

$$\cos\alpha = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{(h_1^2 + k_1^2 + l_1^2)^{1/2}(h_2^2 + k_2^2 + l_2^2)^{1/2}} \qquad (1)$$

the angle α between the symmetrically equivalent planes (110) and $(1\bar{1}0)$ in cubic crystallographic system is 90°.



a

b

Fig.1 Double interferometers: a- two-crystal, b- three-crystal.

In Figs.1a and 1b we show two-crystal and three-crystal double interferometers with silicon crystals. The two-crystal double interferometer consists of two parallelepipedal blocks with parallel air spacing in between. The thicknesses of the blocks (equal to 4.5 mm) are such that the Borman anomalous absorption of Mo K_α radiation took place in the silicon crystal. The width of air spacing is l ≈300 mm.

As is seen from Figs.1a and 1b, the families of strongly reflecting planes (110) and $(1\bar{1}0)$ are perpendicular to each other and the dimensions of interferometers were chosen so as the conditions of reflections 220 and 440 were satisfied. When one of these families is set in the reflecting position, then to bring the other family in the same position it is necessary to rotate the interferometer by 90° around the axis normal to the surface of interferometer entrance. Thus, the conditions are produced for obtaining completely similar interference patterns from double two- and three-crystal interferometers. Of course, it is acievable only when the interferometer blocks are almost ideal crystals. But if the interferometer blocks (or the crystal) contain defects having different orientations relative to the reflecting planes of different families of the double interferometer, then the interference patterns of these interferometers will be different. This fact enables us to judge about the spatial orientation of defects and the distribution of deformations caused by these defects.

a b

Fig.2 Topograms from one crystal: a- reflection 220; b- reflection $2\bar{2}0$.

In Figs.2a and 2b; 3a and 3b; 4a and 4b we show the topograms and interferograms obtained from one crystal, double two-crystal and double three-crystal interferometers with

220 and $2\bar{2}0$ reflections.

As is seen from Figs.2a and 2b, the reflection 220 does not display any defects (Fig.2a), while the reflection $2\bar{2}0$ displays a segregation (in Fig.2b we find the segregation lines).

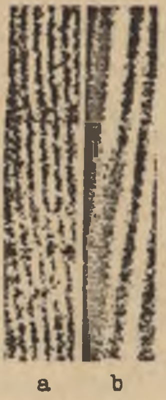


Fig.3 Interferograms from double two-crystal interferometer: a-reflection 220; b-reflection $2\bar{2}0$

These figures show that the reflection 220 in double two-crystal interferometer displays only the displacement lines (Fig.3a), and the reflection $2\bar{2}0$ - both the displacement lines and the segregation lines. In Fig.3b the picture is a result of interference superposition of displacement and segregation lines.

In the first crystal of the double three-crystal interferometer we find a dislocation and, hence, in Fig.4a (reflection 220) we have an interferometrical Moire pattern of the deformation field of dislocation and in Fig.4b (reflection $2\bar{2}0$) we see a picture obtained as a result of coherent superposition of segregation lines and the dislocation Moire.

Thus, by means of the same double interferometer it is possible to identify the segregation lines, the displacement bands as well as the Moire patterns of different imperfections.

One can easily see from these pictures, that as the segregation lines are obtained at the reflection from (110) planes and not from $(1\bar{1}0)$ planes, hence, first, the diffraction vector of reflection $2\bar{2}0$ is parallel to the crystal growth

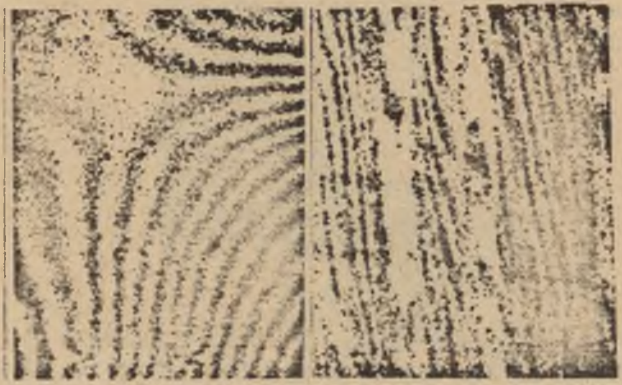


Fig.4 Topograms from double three-crystal interferometer: a- reflection 220; b- reflection $2\bar{2}0$.

axis and the diffraction vector of reflection 220 is normal to it, and, second, the excretions in interferometer crystals are absent.

2. TRIPLE TWO-CRYSTAL AND THREE-CRYSTAL INTERFEROMETER

For more detailed description of crystal defect structure it is necessary to increase the degree of interferometer multiplicity,- to increase the number of reflecting plane families belonging to the same complex of symmetrically equivalent planes.

As is seen from formula (1) and Fig.5, the angle α made by $(1\bar{1}0)$ and $(0\bar{1}1)$ as well as by $(0\bar{1}1)$ and $(\bar{1}01)$ symmetrically equivalent planes is 60°.

In Figs.6a and 6b the photographs of triple two-crystal and three-crystal interferometers with $(1\bar{1}0)$, $(\bar{1}01)$ and $(0\bar{1}1)$ families are shown.

To achieve the correct setting, the X-rays are directed at the Bragg angle to one of three families of reflecting planes of the triple interferometer, the diffracted radiation is detected, then the interference pattern from the second and third families is obtained by turning the interferometer by 60° and the radiation is detected again. To form an opinion about the crystal imperfection, the obtained pictures are compared.

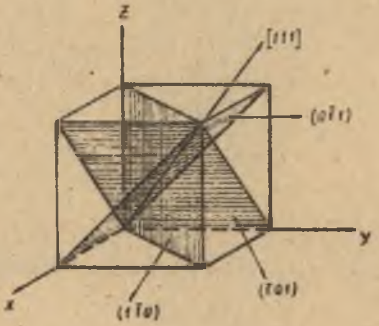


Fig.5 Orientation of symmetrically equivalent planes $(1\bar{1}0)$, $(0\bar{1}1)$ and $(\bar{1}01)$.

In Figs.7a - 7c the sectional interferograms obtained from the triple two-crystal interferometer by the reflections $2\bar{2}0$, $\bar{2}02$ and $0\bar{2}2$ (Mo K_α radiation) are shown.

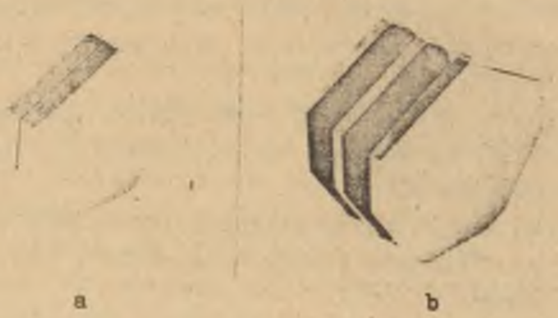


Fig.6 Photographs of triple interferometers: a- two-crystal, b- three-crystal

Fig.7 Sectional interferograms from triple two-crystal interferometer obtained by reflections: a- $2\bar{2}0$, b- $0\bar{2}2$, and c- $\bar{2}02$.

In Figs.8a - 8c one can see topographic interferograms obtained from the triple three-crystal interferometer by the reflections $\bar{2}02$, $2\bar{2}0$ and $0\bar{2}2$.

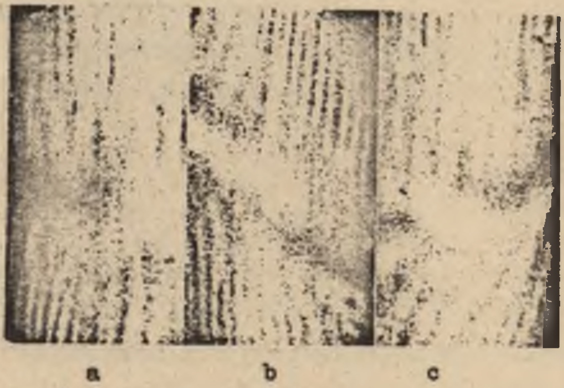


Fig.8 Topograms from triple three-crystal interferometer obtained by reflections: a- $2\bar{2}0$, b- $0\bar{2}2$ and c- $\bar{2}02$.

A careful examination of these pictures allows one to come to the following conclusions:

- The crystals of the triple two-crystal interferometer do not contain any dislocations,- in radiograms of Figs.7a-7c we have obtained nondeformed (almost ideal) displacement lines. In these sectional interferograms the segregation lines are also absent, as in the cases under consideration the diffraction vectors are not normal to the interferometer crystal growth axis.

- The interferometric topograms of Figs.8a - 8c show that the distribution of deformations due to the dislocation line located in the first crystal of the triple three-crystal interferometer are different,- they depend on the orientation of the families of symmetrically equivalent planes relative to the dislocation line.

This is an experimental evidence of the fact, that the interferometric patterns obtained from the same crystal with the help of symmetrically equivalent planes will be similar only when the interferometer crystals are ideal.

To determine the form and place of dislocation we availed ourselves of the simple topographical method - the topograms were taken after each crystal of the triple three-crystal interferometer and made sure that there was only one dislocation line and it was located in the first crystal. The images of dislocations obtained from different families of the triple interferometer differ only by lengths and orientations.

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