Express method of construction of accurate inverse pole figures

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Abstract. With regard to metallic materials with the FCC and BCC crystal lattice a new method for constructing the X-ray texture inverse pole figures (IPF) by using tilt curves of spinning sample, characterized by high accuracy and rapidity (express), was proposed. In contrast to the currently widespread method to construct IPF using orientation distribution function (ODF), synthesized in several partial direct pole figures, the proposed method is based on a simple geometrical interpretation of a measurement procedure, requires a minimal operating time of the X-ray diffractometer.

1. Introduction

The proposed X-ray diffractometer method of constructing the inverse pole figures (IPF) by using tilt curves of the sample has a high accuracy and rapidity. At the present time, following specialists in mathematical methods to describe the texture, it is considered that the most exact and universal method of obtaining IPF is regeneration of the orientation distribution function (ODF). For the construction of ODF it is necessary to have a set of the direct pole figures and a rather complicated computer software for mathematical treatment of data [1-2]. Indeed, this method allows to build IPF for any given section of the test sample without prior diffractometric measuring. However, in most practical cases it is advisable to have more available and express method, although not such universal.

At the simplicity of experimental procedure the proposed method of IPF construction provides a highly accuracy of calculation of weight express texture coefficients P(hkl) for an arbitrarily large number of points within the unit stereographic triangle (for a cubic lattice). This method distinguishes from the traditional ones of construction available IPF often used in practice of X-ray laboratories [3-4].

Proposed procedure for constructing the IPF is considered as applied to materials with a cubic lattice.

2. The basis of express method of constructing the inverse pole figures

Let the slope curves $I_{hkl}(\varphi)$ are registered during the continuous tilt of the test sample and its rapid rotation around the normal to the surface n, where I_{hkl} – the registered intensity of the X-ray reflection (hkl), φ - the tilt angle. Then values $I_{001}(\alpha)$, $I_{011}(\beta)$ and $I_{111}(\gamma)$ accurate within normalization coefficients are proportional to the probability of finding in the sample such grains whose crystallographic axes <001>, <011> and <111> are deviated from the normal to the surface sample n at the angles α, β and γ, respectively, with the accuracy up to normalization coefficients. Judging by

the angular sizes of the unit stereographic triangle any external direction n of a cubic lattice of the test material is spaced no more than at 54.74° from the crystallographic axis <001>; at 45° from <011> and 54.74° from <111>; i.e. $\alpha \le 54.74^o$, $\beta \le 45^o$ и $\gamma \le 54.74^o$. Therefore, tilting analyzed polycrystalline sample during recording diffractometric curve at an angle up to 55° for the registration of reflections (001) and (111) and at an angle up to 45° when registering the reflection (011,) we have obtained X-ray reflection at least from one of the families of crystallographic planes {001}, {011} and {111} for each grain (figure 1 *a, b, c*).

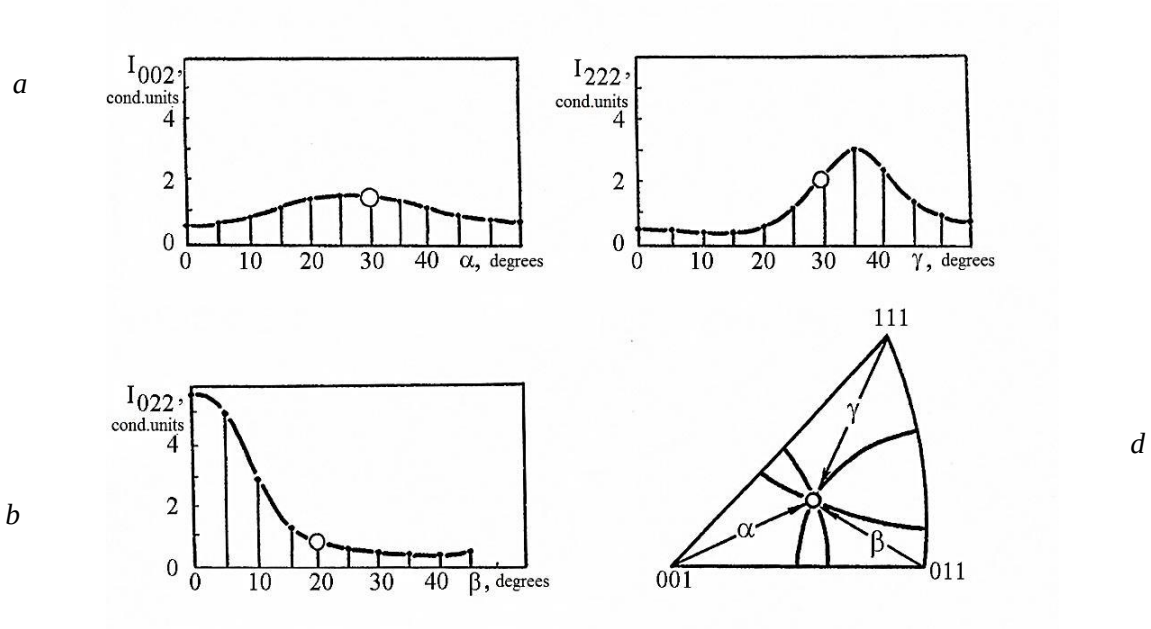

Figure 1. Construction of IPF on sample tilt curves: *a, b, c* – tilt curves $I_{001}(\alpha)$, $I_{011}(\beta)$, $I_{111}(\gamma)$; *d* - a partition of stereographic triangle by circles centered at the poles [[001]], [[011]] и [[111]].

If the axes <001> and <011> of the grains are spaced respectively through angles α and β from the normal n to the sample surface, it may be considered, that these events are as a combination of mutually independent, although fully they are not such. Because at the fixed angle α the angle β cannot take on any value in the range of 0-45°, but only lie within a smaller range, the length and boundary of which depend on the angle α.

Orientation direction of n within the unit stereographic triangle is uniquely defined by two angles: either (α, β), or (α, γ), or (β, γ). At the same time, for the convenience of practical computer build OPF this orientation would be best to set the Cartesian coordinates (x, y) in the plane of stereographic projection.

P(x, y) is denoted the probability that there is in the test polycrystal grain in which the normal n to the sample surface in the stereographic projection plane has coordinates (x, y). Then:

$$\begin{aligned} P(x,y) &= k_1 I_{001}(\alpha) \times k_2 I_{011}(\beta) \\ P(x,y) &= k_1 I_{001}(\alpha) \times k_3 I_{111}(\gamma) \\ P(x,y) &= k_2 I_{011}(\beta) \times k_3 I_{111}(\gamma) , \end{aligned} \qquad (1)$$

where k_1, k_2, k_3 - coefficient of proportionality. Each of the equations (1) with some error defines the same probability of P (x, y). Multiplying together all equations (1), we obtain:

$$P(x,y) = K [\Pi(x,y)]^{2/3}, \qquad (2)$$

where

$$\Pi(x,y) = I_{001}(\alpha) \times I_{011}(\beta) \times I_{111}(\gamma) . \qquad (3)$$

Thus calculated value of P(x, y) coincides with the probability that in the studied polycrystalline there is a grain whose axes <001>, <011> and <111> have angles α, β and γ with the direction n, respectively (see. figure 1-d).

If the value of P (x, y) is calculated for n points of an elementary stereographic triangle, the normalization coefficient K is found from the following relation:

$$K = \{ \sum^{N} [\Pi(x,y)]^{2/3} \}^{-1} . \quad (4)$$

When normalizing the sum $\{ \sum^{N} P(x, y) \}$ is not on 1, but on the number of calculation points N, we thereby partially change the meaning of values P (x, y). In this case, P (x, y) is in n times more than the probability of finding the grain with the appropriate orientation, but it already coincides with the meaning of P(hkl), which appears on the IPF constructed by using any of the conventional methods.

In principle, distribution P(x, y) can be calculated by using any of two equations (1). However, in the interest of reducing the possible systematic errors associated with the use of the proposed method of construction of IPF, at the calculations it is advisable to use all three equations (1).

3. The procedure for obtaining experimental IPF

The procedure for obtaining experimental data required to the construction of IPF comprises the following steps:

- diffractometric measurement of tilt curves of sample during spinning around the normal to the its surface at the registration of X-ray reflections from the crystallographic planes {001}, {011} и {111};
- measurement of similar curves by the detector fixed in position of background registration;
- evaluation of the amendments to defocusing when tilting the sample.

When using a computer calculation of IPF by the proposed method, the initial data are ordinates of diffractometric curves of sample tilt $I_{001}(\alpha)$, $I_{011}(\beta)$ и $I_{111}(\gamma)$ measured at regular angular interval $\Delta\varphi$, and the corresponding background values.

The program of the IPF calculation includes:

- determination of the coordinates (x_i, y_i) of all points of ensemble G_o, which are the points of mutual intersection within the unit stereographic triangle of circles with centers at the poles [[001]], [[011]] and [[111]] and with angular radii, multiple $\Delta\alpha$, $\Delta\beta$ and $\Delta\gamma$, respectively (see figure 2-a);

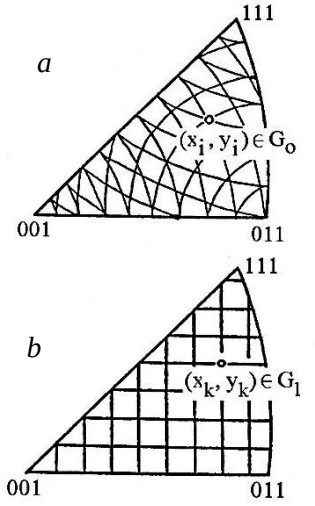

Figure 2. To the calculation of IPF: *a* - G_o set of data points (xi, yi) within the unit stereographic triangle, which are located at the nodes of intersection of the circles with centers at the poles [[001]], [[011]] and [[111]], and with the angular radii, multiple $\Delta\alpha$, $\Delta\beta$ and $\Delta\gamma$, respectively; *b* - G_1 set of calculation points (x_k, y_k), which are located at the nodes of a rectangular grid.

- determination of coordinates (x_k, y_k) of a given set of points G_1, for which will be calculated values *P*(*hkl*) and which are arranged in sites (nodes) of a rectangular grid covering the entire area of the elementary stereographic triangle (see figure 2-b);
- the calculation of the values of $\Pi(x_i, y_i)$ for all the points of ensemble G_o;
- the calculation of the values of Π (x_k, y_k) for the specified points of the ensemble G_1 by interpolation using as basic units of values *P* (x_i, y_i) in four (or three) points of set G_o, closest to the given point of the set G_1;

- determination of the coefficients λ_k, which characterize the relative area of a spherical triangle parts corresponding to the different points of ensemble G_1 and differing from each other due to the inhomogeneous distortion of space at a stereographic projection;

- normalization of sum $[\sum^{N} \lambda_k P(x_k, y_k)]$ on the number N, equal to the number of calculation points in the set G_1;

- organization of the array of calculated values P (hkl) in the form of IPF and print its image.

The measurement of three tilt curves by means of texture set GP-2 provided with an additional engine requires no more than 0.5 hours.

Figure 3 shows IPFs for the surface layer of the same sample rolled low-alloy chromium constructed by means of the proposed method (a) and method described in [2], based on the procedure for the restore of IPF using ODF (b). (The calculation of the last IPF made by S.F. Kurtasov by the program developed himself.) These IPFs are almost identical in character and only slightly differ by values of pole density at mutual corresponding points.

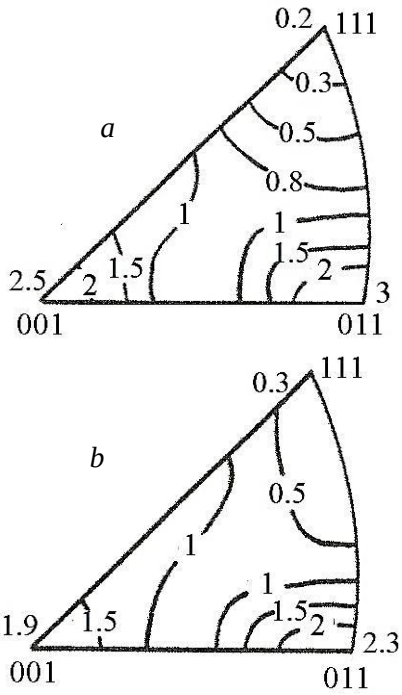

Figure 3. IPFs for the surface layer of the same sample from rolled low-alloy chromium constructed using the proposed method (*a*) and as described in [2], based on the procedure for the recovery of IPF using ODF (*b*).

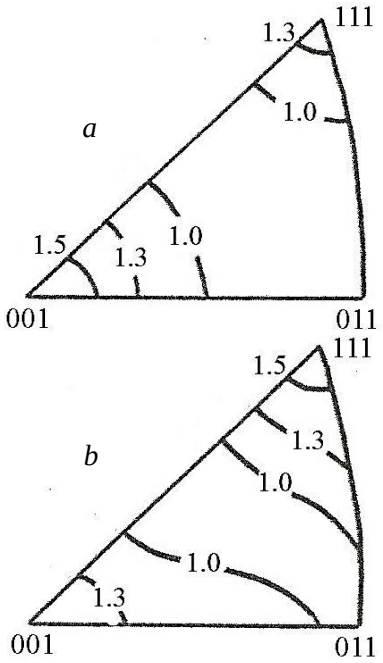

Figure 4. IPF for compound Nb_3Sn in the cross section of plane wire (conductor), calculated by the proposed method (*a*) and by the more precise method [5] using 14 reflections.

4. Examples of constructing IPFs

The effectiveness of the proposed method is illustrated by the results of the study of the texture of the superconducting compound Nb_3Sn, which has a cubic crystal lattice of type A15. A large number of reflections are observed in the diffraction spectrum of this compound, which allows to obtain relatively accurate information about the texture of the sample, even when using the original method of Harris [5], based on measuring the intensity of the recorded lines. Figure 4 shows IPFs of a sample of compounds Nb_3Sn, calculated by the proposed (a) and improving methods [5] when using 14

reflections (b). A comparison of obtained IPFs, as in the previous example, confirms that the proposed rapid method is quite accurately conveys features of the studied texture.

It should be noted that the proposed method of construction of IPF is associated with bringing some bias (systematical errors) in calculations, the origin of which is illustrated in figure 5. In the case of increasing the tilt angle of sample above 45° at registering the reflection from the {001} and above 35° for the reflections from the {011} and {111} circle with angular radius φ centered at the corresponding pole oversteps the limits of the stereographic triangles which are mutually symmetric relatively to this pole.

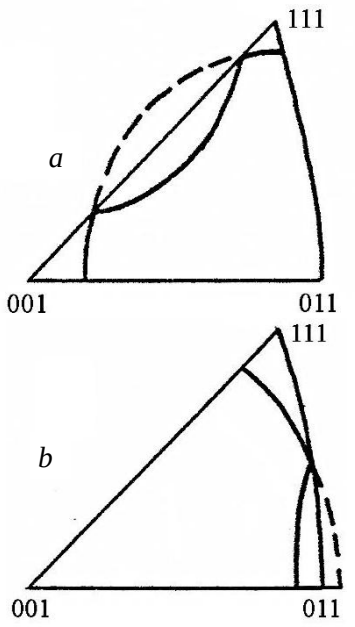

Figure 5. To analysis of the reliability of the proposed method of IPF construction: reflection of outer portion of a circle centered at the poles [[011]] (*a*) and [[001]] (*b*) shown by dotted line inside the stereographic triangle (see explanations in the text).

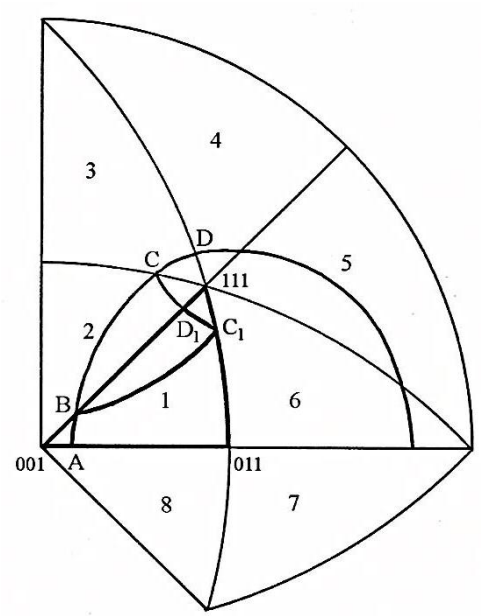

Figure 6. To the question about the formation of the X-ray reflections, recorded at fast rotation of the sample around the normal to its surface. The case of recording the X-ray line (011) (see explanations in the text).

For example, in the case shown in figure 6, the circle with angular radius 40° centered at pole [[011]] oversteps the limits of the stereographic triangles 1, 6, 7 and 8. Therefore, the intensity of X-ray reflection recorded at a rapid rotation of sample about the normal to its surface is determined by averaging the pole density along the polyline ABC_1D_1 constructed within the triangle 1 and consisting from individual segments which correspond to the different sections of the neighboring stereographic triangles. In particular, the segment BC1 corresponds to the section BC of triangle 2 and the segment C_1D_1 - the section CD of triangle 3. Then, the averaged intensity I_{011} (40°), attributed to the points of the arc AB under calculating, actually also depends on the pole density distribution in other areas of the triangle. If the pole density of actual IPF near the poles [[111]] is higher than near the poles [[001]], pole density values attributed to the points near the poles [[001]] under the calculation, are overstated.

However, the error introduced mentioned way in the calculation of IPF is largely reduced due to the simultaneous use of other reflections, at work with which obtained tilt curves characterize the same area IPF without such errors. In this example, these are the reflections from the {001}. The recommended use of three reflections at the sufficiency of IPF construction by any two of them just

provides a significant reduction of the pole density in the areas of IPF after the normalization, where recorded average intensity of one of the reflections are too high.

The sharper the investigated texture is, the less the significant errors are due to the procedure of the intensity averaging during measuring of tilt curves of sample. At the same time, when texture is weak, errors of building IPF are insignificant due to the fact that the recorded intensity varies relatively little, depending on the length of the line along which averaging occurs. The analysis shows that the errors of averaging can significantly affect the IPF as a measuring of some specially "designed" texture, almost never occur in real materials.

Routines conversion of polar coordinates to Cartesian and interpolation to calculate the intensity at given points of stereographic projection, which are used in the construction of IPF described by the proposed method are the main components of the program developed by the automated construction of IPF.

Acknowledgements

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References

[1] Bunge H J 1969 *Matematische Metoden der Texturanalyse* (Berlin: Akademie-Verlag) p 330 (in Russian)
[2] Kurtasov S F 1978 *Zavodskaya laboratoriya* **44** (11) 1359-61(in Russian)
[3] Borodkina M and Spector E N 1981 *X-ray analysis of texture of metals and alloys* (Moscow: Metallurgiya) p 272 (in Russian)
[4] Borodkina M M and Kurtasov S F 1979 *Zavodskaya laboratoriya* **45** (9) 830 (in Russian)
[5] Harris G.B. 1952 *Phil. Mag*. **43** 113