

Modern methods of experimental construction of texture complete direct pole figures by using X-ray data

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Abstract. Currently used methods for constructing texture complete direct pole figure (CDPF) based on the results of X-ray diffractometric measurements were considered with respect to the products of Zr-based alloys and, in particular, used in a nuclear reactor cladding tubes, for which the accuracy of determination of integral texture parameters is of the especial importance. The main attention was devoted to technical issues which are solved by means of computer processing of large arrays of obtained experimental data. Among considered questions there are amendments of the defocusing, techniques for constructing of complete direct pole figures and determination of integral textural parameters. The methods of reconstruction of complete direct pole figures by partial direct pole figures recorded up to tilt angles of sample $\psi=70-80^\circ$: the method of extrapolation of data to an uninvestigated region of the stereographic projection, and the method of "sewing" of partial pole figures measured for three mutually perpendicular plane sections of the product. The limits of applicability of these methods, depending on the shape of the test product and the degree of inhomogeneity of the layer-by-layer texture, were revealed. On the basis of a large number of experimental data, the accuracy of the integral parameters used for calculation of the physical and mechanical properties of metals with a hexagonal crystal structure was found to be equal to 0.02, when taking into account the texture heterogeneity of regular products from Zr-based alloys.

1. Introduction

The emergence of new diffractometers of latest generation at X-ray laboratories, which are busy studying of the texture of metallic materials, significantly changed the character of the routine work directly performed by researchers, shifting its centre of gravity toward the trivial manipulation with the enclosed software when calculating the orientation distribution function (ODF) or building 3D pole figures. Automatization of processes of texture diffractometer measuring and computer processing of the measurement results significantly accelerate these procedures and immeasurably reduce their labour input, allowing to operate by the vast arrays of experimental data that had previously been almost impossible. However, while a number of important methodological aspects of the X-ray study of the product texture is out of view of researchers, which consciously or unconsciously rely on the competence of developers of the device and the used software. If you need only information about the general character of texture and about the most remarkable features of its development, the situation is acceptable. But in cases where the texture parameters define the technology and performance of the product or you need a more precise analysis of texture to identify certain processes of structure development in the material, it is necessary to critically estimate the



sources of possible errors of the received information and to improve the accuracy of the calculated texture parameters.

Namely such situation is observed at the study of the texture of products from zirconium alloys for nuclear energy and, in particular, the cladding tubes, for which the so-called integral texture Kearns parameters [1] characterize their suitability as an extremely critical elements of reactor design. Meanwhile, during 30 years publications, where the most important for the physical and applied metallurgy questions would be addressed with due methodical thoroughness, do not appear in the scientific literature. Although the fundamental principles of the X-ray method of construction of the direct pole figures (DPFs) for the past decade have not been changed, the current practice of texture analysis is strongly needed in the description of methods of X-ray study of textures, upgraded in accordance with the process automatization and computerization of the treatment process of results. In this work, the gap is filled with reference to the study of the texture of products from zirconium-based alloys. The following methodological developments, which are necessary for the construction and treatment of complete direct pole figures (CDPF) with an accuracy corresponding to the increased possibility of experimental techniques, are described:

- 1) methods of defocus effect calculation at X-ray measurement of incomplete DPFs (IDPFs);
- 2) techniques of automatized experimental construction of complete DPF (CDPFs) for products of different types and, in particular, for the cladding tubes;
- 3) the calculation of integral texture Kearns parameters by using CDPF (0001) for α -Zr.

2. Principles of the construction method of direct pole figures

Crystallographic texture analysis of polycrystalline materials is generally carried out by X-ray diffractometer measurement of direct pole figures $\{hkl\}$ by tilting the sample (Figure 1) for the corresponding reflections (hkl) [2-6].

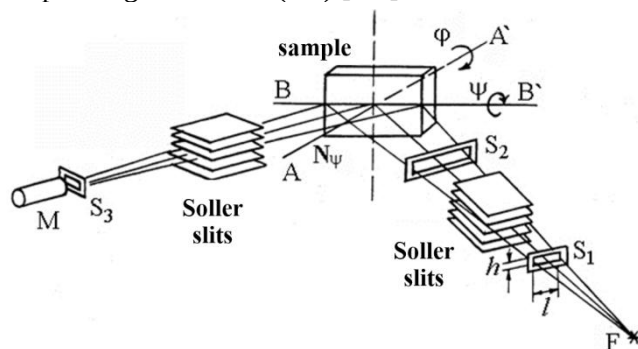


Figure 1. The scheme of DPF measurement by tilt method on reflection of diffracted beam: F – projection of X-ray tube focus, M – detector, S_i – slits; AA' and BB' – rotation axes of sample.

DPF $\{hkl\}$ is the pole density distribution of normals (axes) of the selected type $\langle hkl \rangle$ in the stereographic projection. Pole density is directly proportional to the intensity of the X-ray beam reflected from the respective planes $\{hkl\}$. Therefore, to measure the PPF the intensity of diffracted X-ray radiation from the planes $\{hkl\}$ is recorded for all possible orientations of the sample relative to the direction of the primary and reflected beams. Planes of the selected type $\{hkl\}$ reflect at different orientations of the sample if their normals lie in plane containing the primary and reflected beams and divide the angle between them in half. The value of the recorded intensity is proportional to the volume of grain involved in the formation of the diffracted beam.

The procedure of X-ray diffractometer measurement of texture are describe in detail in [2-4] and consist in recording the intensity of X-ray scattering at a certain Bragg angle 2θ and successive positions of the sample, which are determined by the angles ψ and ϕ , where ψ - the tilt angle of the sample varying between $0 - 80^\circ$, and ϕ - the rotation angle of the sample around the normal to the surface, varying from $0 - 360^\circ$ (Figure 1). Since these measurements are taken at different angles of the sample, they correspond to the different geometry of the X-ray diffraction - unchanged preserved only angle 2θ , but changing the angle between the surface normal and the incident beam between the

normal to the surface and the diffracted beam, changing the area of the irradiated surface of the sample and X-ray penetration depth into the material. The partial violation of the terms of focusing (i.e. the "defocus") caused by sample tilt leads to a broadening of the X-ray line profile and a drop in recorded intensity becoming significant when $\psi > 50^\circ$; moreover, defocus can lead to the angular displacement of the X-ray line. To reduce the effect of defocus it is necessary to limit the vertical divergence of the primary beam and the height of its projection in the sample plane [2]. The system of collimating slit was shown in Figure 1. Soller slits with an angle of divergence $1,5^\circ$, installed on the primary beam, help to reduce its vertical divergence, and Soller slit with an angle of divergence $2,5^\circ$, set on the detector, prevent it from getting scattered radiation. Even at optimal geometry of measuring it is necessary to consider the maintaining defocus effect by adjusting the obtained data on the basis of a periodic measuring of calibration samples.

Thus, the input data for the construction of PPF are [2]:

1) I_{ij} - the measured intensity values at successive points orientation space (ψ_i, φ_j) , where ψ_i varies from 0 to a maximum value $\psi_{\max}$, φ_j - from 0 to 360° ; for polycrystalline materials the uniform step for both corners ($\Delta\psi = \Delta\varphi = 2.5$ or 5°) is commonly used; for sharp texture or single crystals a detailed measuring near the texture maximum is used;

2) $I_{ibg} = I_\psi$ - background intensity values of the sample (at $2\theta_{bg}$) for the respective tilt angles ψ_i ;

3) K_i - the magnitude of correction coefficient of defocus also varying with the tilt angle ψ_i . Methods of their measurement or calculation are given below.

Then the calculation of the pole density at each point of the stereographic projection carried by the following formula: $P_{ij} = [I_{ij} - I_{ibg}] \cdot K_i / \bar{I}$, where average value of intensity for studied region of IDPF is

$$\bar{I} = \frac{\int_0^{2\pi} \int_0^{\psi_{\max}} [I(\psi, \varphi) - I_\psi] \cdot \sin \psi \cdot d\psi \cdot d\varphi}{\int_0^{2\pi} \int_0^{\psi_{\max}} \sin \psi \cdot d\psi \cdot d\varphi},$$

and $\int_0^{2\pi} \int_0^{\psi_{\max}} \sin \psi \cdot d\psi \cdot d\varphi = s$ - area of studied orientation space of DPF, which equal 2π in case construction of complete DPF.

3. Accounting of defocus of X-ray beam in the construction IDPF by tilting

Construction of DPF is impossible without accurate knowledge of the correction coefficients K_i . The following methods are used for their determination: 1) "sewing" of tilt curves for special composite samples; 2) measuring tilt curves for textureless standard; 3) analytical calculation for sample of small size (the sample size less than area of primary X-ray beam projection on the sample).

The practical difficulty of obtaining powder textureless standards with a density close to the theoretical does not allow to rely on defocus coefficients obtained by the second mentioned methods. In addition, when preparing the textureless powder the crystallographic texture of compression or stacking may be formed in them, what also results in an error in the determination of K_i . Therefore in this paper we consider only the first of these method as more original, since the other two is not difficult for researchers on texture analysis.

As cold-rolled Zr-based alloys are characterized by texture $(0001) \pm 30-40^\circ \text{ND-TD} < 10\bar{1}0 >$, in which the basal normals distributed mainly in the section ND-TD of DPF(0001) (Figure 2), this variant of determining the defocus coefficients comprises preparing a three composite samples, normals to the studied surfaces are oriented over each thirty degrees along section DPF (0001) as shown in figures 2 and 3, where RD - rolling, ND - normal and TD - transverse directions in sheet, correspondingly. Exits of normal to studied sample surfaces shown in Figure 3 are built in DPF (0001) in Figure 2 and are marked as a, b and c, correspondingly.

Assuming that at the optimal geometry of measuring DPFs, defocusing effect does not affect the intensity of the diffracted beam up to the tilt angle of the sample 50° , we will use parts of the tilt to ψ

= 30° as reference (Figure 4). Calculation of the complete cross section of DPF (0001) TD-ND as a result of the construction of reference parts of tilt curves for all samples a, b, c, shown in Figure 3 and the presence of the tilt curve for the sample a recorded up to 80° (Figure 4, curve a) allows us to calculate the defocusing coefficients of the diffracted X-ray beam. The variation of defocusing coefficients with increasing tilt angle is shown in Figure 5. Defocusing coefficients K_i shows how many times is it necessary to increase the intensity of the measured value to obtain its true meaning.

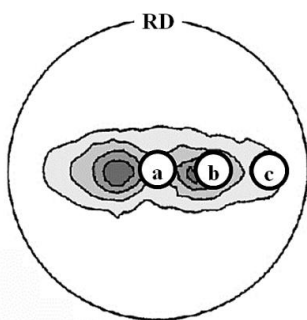


Figure 2. Typical DPF (0001) for rolled sheets from Zr-based alloys.

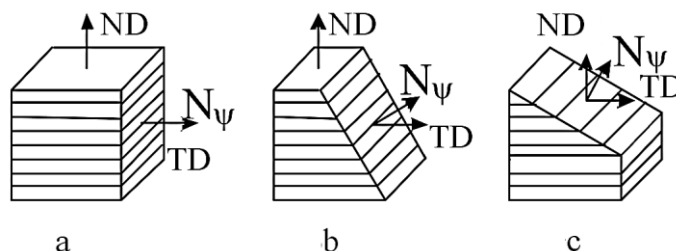


Figure 3. Composite samples used for construction of coefficients of diffracted beam defocusing: a – sample a, b – sample b, c – sample c.

The advantage of this technique is to obtain correction factors for the bulk samples with a density which coincides with the density of the material.

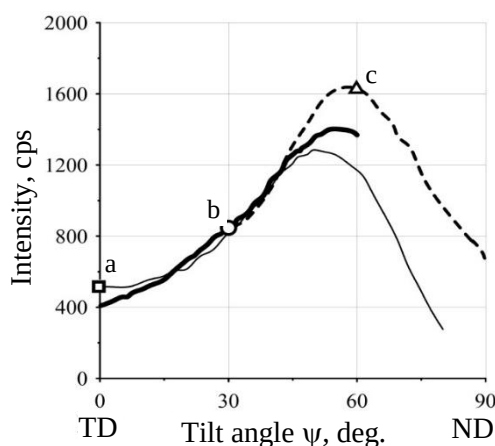


Figure 4. Tilt curves measured for composite samples presented in Figure 3. Exits of normal to studied section are marked by white points.

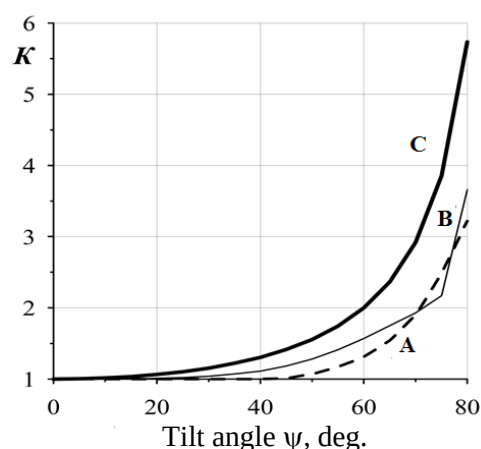


Figure 5. Defocus coefficients for different tilt angles: A - measured for composite samples; B – measured for textureless powder standard; C) calculated for small sample.

Figure 5 also shows the defocus coefficient changing at depending on the tilt angle obtained for the powder sample (B) and sample of small size (C). Comparison of curves A and B shows significant differences in the magnitude of the coefficients of defocusing obtained for textureless standard and composite samples for the same reflection (0002).

4. The methods of construction complete DPFs

For a complete description of the crystallite orientation of hexagonal system it is enough to know the orientation of the basal normal [0001] and rotation around it. That is, for evaluation of the crystallographic texture polycrystal it is necessary to construct two complete DPF (0001) and the

$\{10\bar{1}0\}$ or $\{11\bar{2}0\}$. Having regard to the features of the X-ray spectrum of α -Zr, the texture of products from Zr-based alloys was characterized by DPF (0001) and $\{11\bar{2}0\}$.

In the study of the texture of α -Zr, to quantify integral texture Kearns parameters or the so-called f -parameters, it is necessary to construct CDPF. In the case of the measuring of a single section of the sample, only IDPF can be constructed, due to the geometry of a standard method of measuring texture "by reflection". It is filled by following various methods of restore of CDPF from some IDPFs:

- 1) the completing of peripheral regions of the PF by using a result of the "transmission" measuring;
- 2) the coincidence, or "sewing" of three IDPF obtained for three mutually perpendicular surfaces;
- 3) the using of equi-tilted sample;
- 4) extrapolation of data of IDPF in the uncharted area of stereographic projection;
- 5) the using of orientation distribution function (ODF) to reconstruct CDPF by several IDPF recorded for the same surface.

Using the method (1) connects with considerable difficulties associated with the preparation of X-ray transparent foil of the same thickness over the entire area of the sample. Method "sewing" (2) is simpler, but requires three times more the initial data and sufficiently complicated software. In the case of "sewing", precision cutting of products and preparation of composite samples are required. In addition, the method of "sewing" cannot be used in the presence of a layerwise sample inhomogeneity or large grains. The complexity of preparation of exactly oriented equi-tilted plane for complex products in the method (3) leads to the fact that it is impossible to distinguish between the methodological error and the existential asymmetry of the sample texture. For the channel and especially for cladding tubes, the method of equi-tilted sample does not apply because of the fundamental impossibility of preserving the external orientation of the axes of the sample across the surface prepared. The quality of the data obtained by the method (4) depends on the accuracy of determining the coefficient of defocusing, the correction of extrapolation algorithm and the location of texture maxima.

It is widely used at present mathematical method reconstruction CDPF using ODF [7, 8] for the products of Zr-based alloys is unacceptable because of significant differences integral Kearns' parameters calculated from the experimentally built CDPF and restored from ODF. In addition, developers ODF indicate the possibility of the formation of additional false peaks in the reduction CDPF [9, 10]. Based on the above as the main methods of CDPF reconstruction are generally used methods (2) and (4) which are effectively applied to the analysis of the crystallographic texture of zirconium products depending on its features and problems solved. The most express method in the study of thin-walled tubes is an extrapolation technique of IDPF on uncharted area of the stereographic projection. In this case the accuracy of the data depends mainly on the accuracy of determining the coefficients of defocusing. The best results are achieved if the orientation of the basal normals is near the normal to the surface under investigation. In the case of thick layers of homogeneous products the method of "sewing" are used.

4.1. "Sewing" of IDPF obtained for three mutually perpendicular cross-sections of the sample.

For three mutually perpendicular planes of the sample (Figures 6, 7a), the normal to which is designated as ND (R), TD (T) and RD (L) are recorded three IDPFs, where R, T, L – radial, tangential, and longitudinal directions of the tube. Scheme of sample preparation from tubes for X-ray study of the texture is shown in Figure 6, for sheets - in Figure 7 a.

Further, in the designation of the surface under study and DPF the name of the appropriate normal will be used. To match the results of the three measuring IDPF, two of them - $IDPF_{TD}$ and $IDPF_{RD}$ - should be brought to the axes of the first $IDPF_{ND}$ by applying to spherical coordinates when they turn on the 90° around one of the axes. Using the formula given in [11], we can obtain the relation between the coordinates of $IDPF_{TD}$ (ψ, φ) and coordinates of $IDPF_{ND}$ (ψ', φ'):

$$\begin{aligned} \operatorname{tg} \varphi' &= (1/\operatorname{tg} \psi / \cos \varphi) \\ \operatorname{tg} \psi' &= -1/\operatorname{tg} \varphi / \cos \varphi', \end{aligned}$$

and also between the coordinates of $IDPF_{RD}(\psi, \phi)$ and coordinates of $IDPF_{ND}(\psi', \phi')$:

$$\text{tg}\phi' = \text{tg}\psi \cdot \sin\phi$$

$$\text{tg}\psi' = -\text{tg}\phi / \sin\phi'.$$

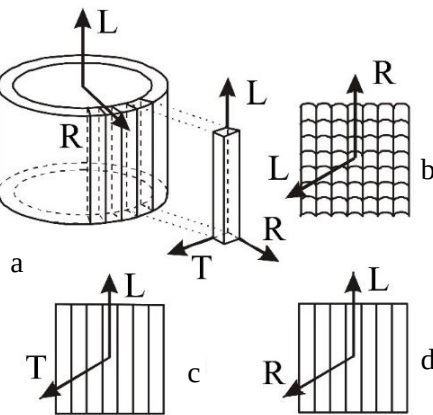


Figure 6. The preparation of the composite samples from the tube for X-ray study of its texture: a – cutting off segments from the tube; b – L-sample; c – T-sample; d – R-sample. Studied surfaces of composite samples are perpendicular to corresponding tube axes.

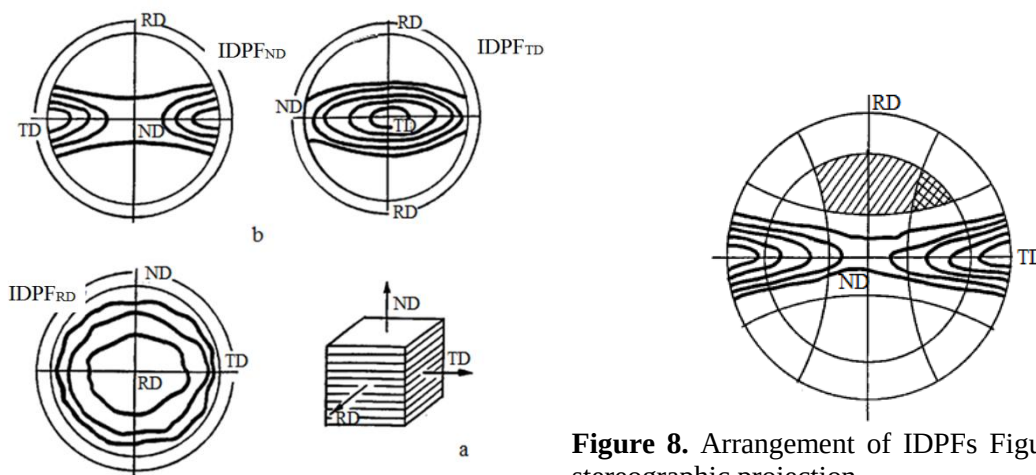


Figure 8. Arrangement of IDPFs Figure 7 on the stereographic projection.

Figure 7. IDPF (0001) (b) built for the three mutually perpendicular planes (a).

Taken together DPFs cover all areas of the stereographic projection (Figure 8), which allows you to calculate CDPF. If the limit tilt angle of the sample at measuring IDPF is more than 54.7° , besides the double areas of overlapping, triple areas appear, i.e. areas in which the value of the pole density (or intensity proportional to it) at the each point of stereographic projection can be determined from the two or three IDPFs recorded for mutually perpendicular surfaces. "Sewing" of IDPFs is carried out by comparing the intensity of X-ray reflection at equivalent points of different pole figures (Figure 8). Since the intensity measured for different IDPFs at equivalent points belongs to different volumes of the material (as different sections of the sample are explored, even in layerwise uniform sample), and registration of the intensity occurs with an error of pulse count obeying Poisson's distribution [12] you can not expect a perfect coincidence of intensity values at equivalent points. Therefore, to adjust the values of $IDPF_{TD}$ and $IDPF_{RD}$ by using overlapping areas, the average values of the coefficient $\bar{X} = P_k/P_l$, where k and l are indices of one of the directions of ND, RD or TD, are found. If the average value of this ratio is 1, this corresponds to an ideal "sewing" of IDPF.

The measurement error is inversely proportional to the intensity of $I^{1/2}$, then the error of intensity ratio I_1/I_2 is expressed as $E = \sqrt{1/I_1^2 + 1/I_2^2}$. The higher the recorded X-ray reflection intensity, the greater the accuracy of the measured value (weight). Weight of measured intensity is directly proportional to I_1/I_2 , and then the weight of calculated ratio I_1/I_2 is expressed as $W = \sqrt{\frac{I_1 \cdot I_2}{(I_1 + I_2)}}$. Since the calculation of the coefficient of "sewing" is conducted in the areas of DPF with different pole density, it is necessary to take into account the weight of the calculated values, and hence to carry out the calculation of the average value considering weight. This method of calculating the weighted average coefficients $\bar{X}$ can significantly reduce the error of their determination. The closer the ratio $\frac{\bar{X}_1 \cdot \bar{X}_3}{\bar{X}_2}$ to the unit, the more accurate the coefficients of "sewing". If sample is produced accurate and its installation at the X-ray measuring in the texture holder of attachment is also accurate, and if the layerwise inhomogeneity is absent, then deviation of calculated ratio $\frac{\bar{X}_1 \cdot \bar{X}_3}{\bar{X}_2}$ from unity must be minimum.

4.2. Extrapolation of the data of IDPF in the uncharted areas of stereographic projection.

The measurement of experimental IDPF is carried out to tilt angle 80° , IDPF is completed by the extrapolation of the data in uncharted area of the stereographic projection. Let us consider the distribution of pole density (intensity) in section IDPF specified by some angle φ . Given the equivalence of DPF points with coordinates $(\psi = 90^\circ, \varphi)$ and $(\psi = 90^\circ, \varphi + 180^\circ)$, the problem of extrapolation can be reduced to the problem of interpolation. We get a one-dimensional distribution function of the intensity with three uncertain points with coordinates $\psi = 85, 90, -85^\circ$. Using the Lagrange interpolation formula [11], we can calculate the value of the intensity at an intermediate point ψ :

$$I(\psi) = \frac{(\psi - \psi_1)(\psi - \psi_2) \dots (\psi - \psi_N)}{(\psi_0 - \psi_1)(\psi_0 - \psi_2) \dots (\psi_0 - \psi_N)} I_0 + \frac{(\psi - \psi_0)(\psi - \psi_2) \dots (\psi - \psi_N)}{(\psi_1 - \psi_0)(\psi_1 - \psi_2) \dots (\psi_1 - \psi_N)} I_1 + \\ + \dots + \frac{(\psi - \psi_0)(\psi - \psi_1) \dots (\psi - \psi_{N-1})}{(\psi_N - \psi_0)(\psi_N - \psi_1) \dots (\psi_N - \psi_{N-1})} I_N.$$

Note that the Lagrange interpolation formula gives satisfactory results only in the case of sufficiently "smooth" of the initial data. Therefore, the interpolated function is pre-smoothed using the methods of median and deskewing. CDPF calculation is impossible without accurate knowledge of the correction coefficients K_d , taking into account the deviation of the sample from the focusing circle, which leads to the scattering of the diffracted beam and the intensity loss of X-ray reflection.

In case the location of sharp texture maximum outside the study area restored by extrapolation DPF (0001) does not correspond the texture actually existing in the sample. In case of a deviation of the maxima in distribution of basal normals from the radial direction at an angle more than 60° (which can also be assessed by the location of texture maxima on DPF $\{11\bar{2}0\}$ or $\{10\bar{1}0\}$ near the radial direction) the calculation of f -parameters must be carried out either by extrapolating DPF (0001), built for the T (TD)-surface, or use the technique of "sewing".

5. The method of calculation of the integral texture Kearns' parameters by CDPF (0001)

Materials with a hexagonal structure characterized by a significant anisotropy of physical and mechanical properties. For example, the Young's modulus for a Zr single crystal changes from 1.25 GPa in the direction of [0001] to 0.99 GPa along $\langle 10\bar{1}0 \rangle$ [13]. For the polycrystalline materials Kearns [1] introduced the integral textural f -parameters which are widely used in the calculation of the properties of materials with a hexagonal lattice and called as f -parameters or Kearns' parameters [1, 14].

If polycrystalline is represented as a set of grains with different orientations, i.e. their interaction with each other is ignored, the property value in the selected direction of the polycrystalline material is the sum of the properties of i -th crystals [1, 14-16]:

$$P(\psi) = P_c \sum_i V_i \cos^2 \psi_i + P_a \sum_i V_i (1 - \cos^2 \psi_i) = f \cdot P_c + (1-f) \cdot P_a,$$

where P_c and P_a are properties along the crystal lattice axes c and a , respectively, and ψ_i - the deviation angle of the selected direction of i -th grain from the normal to the basal plane; from axis c . Then the Kearns' parameters are defined as the sum of effective volume fractions of grains $\sum_i V_i \cos^2 \psi_i$, where $V_i = p(\psi, \varphi) \cdot \delta\omega = p(\psi, \varphi) \cdot \sin\psi \delta\psi \delta\varphi$ - volume fraction of grains calculated from

DPF (0001) by pole density value $p(\psi, \varphi)$ registered in solid angle $d\omega$ limited by sections $\psi \div \psi + \Delta\psi$ и $\varphi \div \varphi + \Delta\varphi$ (Figure 9).

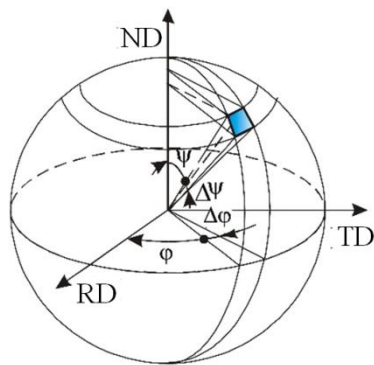


Figure 9. To the question of calculation of f -parameters: angular element on the unit sphere.

The value of f -parameter is represented as the sum (integral) of volume fractions across the all stereographic projection [1, 14-16]:

$$f = \int_0^{\frac{\pi}{2}} \int_0^{2\pi} p(\psi, \varphi) \cdot \sin\psi \cdot \cos^2 \psi \cdot d\psi \cdot d\varphi$$

Normally, f -parameters are calculated for the three main directions of products ND, TD and RD in a sheet or R , T and L in the tube [16]:

$$f_R = \int_0^{\frac{\pi}{2}} \int_0^{2\pi} p(\psi, \varphi) \cdot \sin\psi \cdot \cos^2 \psi \cdot d\psi \cdot d\varphi$$

$$f_T = \int_0^{\frac{\pi}{2}} \int_0^{2\pi} p(\psi, \varphi) \cdot \sin^3 \psi \cdot \sin^3 \varphi \cdot d\psi \cdot d\varphi$$

$$f_L = \int_0^{\frac{\pi}{2}} \int_0^{2\pi} p(\psi, \varphi) \cdot \sin^3 \psi \cdot \cos^2 \varphi \cdot d\psi \cdot d\varphi$$

As seen from the above formulas, the calculation of f -parameters is carried out by means of the complete PF (0001), besides the sum of them should be equal the unit: $f_R + f_T + f_L = 1$.

Since the precision of construction of DPF depends on the intensity recorded in its various areas, so far the texture measurement error is characterized by the orientation distribution of the normals to the reflecting planes in the stereographic projection of the test sample as described in the works [17, 18]. Building such distribution for the instrumental errors by comparing the results of multiple PF

recording of the same sample shows that they increase in minima of PF and decrease in texture maxima. Textured heterogeneity is seen as a factor which introduces a mistake in the description of the texture of the material based on the results of the measuring of single sample.

Error of constructing of CDPFs (0001) also affects the values of integral texture parameters. The analysis of the calculated integral f -parameters [19] indicates that the error of their calculation, is not more than 0.02 provided that all recommendations by the construction CDPF (0001) are adhered.

6. Conclusions

Construction of a complete direct texture pole figures, even with the modern development of X-ray equipment, and computer processing of the results is very time-consuming task, especially in those cases when it is necessary to define the integral texture parameters with high accuracy as in the analysis of products from Zr-based alloys for nuclear technology. The article discusses ways to correct for defocusing, experimental techniques of construction of complete direct pole figures using the "sewing" texture data for different cross sections of the product and by extrapolation, and method of determining the integral texture parameters. The algorithms of computer processing of the results of the texture measurements used in the study of products from zirconium alloys are described.

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