Practical applications of the method of generalized pole figures

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Abstract. Several actual practical applications of the new X-ray method of generalized pole figures are considered. Among them there are determination of c- and a-dislocation densities in shell tubes from Zr-1%Nb alloy, analysis of strain hardening at opposite sides of shell tubes from ferritic-martensitic steel with oxide disperse strengthening particles for high-temperature nuclear reactor and revealing of substructure non-uniformity in rods of Cu, subjected to equi-channel angular pressing.

1. Introduction.
The X-ray method of generalized pole figures (GPF), realizable by texture diffractometers of last generations, includes successive registration of profiles for the same X-ray line at each point of a texture pole figure in the course of its measurement [1]. The method of GPF gives the most complete information on substructure conditions of grains with different crystallographic orientations. By computer treatment of obtained data, distributions of various substructure parameters are calculated.

According to the theory of X-ray diffraction [2], a profile of X-ray line (hkl) characterizes the condition of crystalline lattice along the normal to reflecting planes {hkl}. The GPF method allowed to reveal the systematic difference in strain hardening of crystallites, corresponding to maxima and minima of the deformation texture, and to disclose experimentally the equilibrium of residual elastic microstress in the deformed metal matrix [3]. X-ray studies testify, that these rules are general lows for deformed textured metal materials. This paper contains some examples, where the GPF method was applied efficiently.

2. Dislocation density distributions in shell tubes from Zr-1%Nb alloy
Determinations of the dislocation densities \( \rho_c \) and \( \rho_a \) in \( \alpha \)-Zr matrix by X-ray method [3] gives reasonable, statistically significant results, devoid of possible errors, connected with preparation of samples for direct local observation of dislocations. This approach was developed by use of GPF method, so that GPF \( \rho_c \) and GPF \( \rho_a \) were constructed for as-rolled shell tubes from Zr-1%Nb alloy. The recalculation of the coherent domain size and the lattice distortion into the dislocation density \( \rho \) bases on the supposition that the total energy of dislocations is equal to the energy of lattice distortions, whereas the distance between neighboring dislocations is assumed to be equal to the coherent domain size. But X-ray diffraction allows to detect only those dislocations, which cause some rearrangements of atoms along the normal to reflecting planes. When taking into account that the dislocation produces a displacement of neighboring atoms along its Burgers vector, presence in \( \alpha \)-Zr crystallites of c-dislocations with the Burgers vector parallel to axis \(<0001>\) and a-dislocations with the Burgers vector parallel to axis \(<10\bar{1}0>\) can be revealed by features of X-ray reflections from basal and prismatic planes, respectively. When calculating the dislocation density \( \rho \) for all orientations \((\psi,\phi)\) of basal and prismatic planes, presented in the texture of the sample, we obtain GPF \( \rho_c(\psi,\phi) \) and GPF \( \rho_a(\psi,\phi) \).

X-ray study of tubes included measurements of three sections, perpendicular to radial, tangential and axial directions, denoted as R-, T- and L-directions, respectively. These sections were prepared from segments, cut out the tube with the help of electro-erosion.
method. Three partial texture pole figures (PF) with an angular radius of 70° were measured and sewed into complete PF with angular radius 90°. The sewing procedure consists in mutual adjustment of corrected incomplete PFs by leveling of distributions within their overlapping regions (Fig. 1-a).

![Fig. 1. X-ray determination of dislocation density in shell tube from Zr-based alloy: a) cutting of the tube into segments; b) PF(0001); c) GPF ρc; d) ν(ρc); (e) ν(ρa).](image)

Since each crystallite has only one axis <0001>, GPF ρc(ψ,ϕ) fully describes the correspondence between the density of c-dislocations and the orientation of α-Zr crystallites. A distribution of volume fractions of grains with different densities of c-dislocations ν(ρc) can be constructed, using values of pole density as weight coefficients by the statistical treatment of GPF ρc. In the case of α-dislocations the situation is more complicated. However in rolled products from Zr-based alloys there is a texture maximum, coinciding with rolling direction, to which each α-Zr grain gives one of its axis <1010>, and namely this maximum was used for measurement of X-ray line profiles, aimed to construction of GPF ρa and ν(ρa).

Complete PF(0001) and GPF ρa for α-Zr matrix of the as-rolled Zr-1%Nb tube ø=20 mm are shown in Fig. 1-b, c. Distributions of volume fractions ν(ρc) and ν(ρa) for the same as-rolled tube are presented in Fig. 1-d, e. Obtained results testify, that estimation of the dislocation density in tubes by the standard method is unrepresentative, since it does not take into account the inhomogeneity of the dislocation distribution. According to data of the used GPF method, in studied tubes the dislocation density varies within 4 - 5 orders of magnitude depending on grain orientations and attain value of 10¹⁷ m⁻².

### 3. Shell tubes for high-temperature nuclear reactors

The GPF method was used for the study of residual deformation effects in tubes from ferritic-martensitic steel with oxide dispersed strengthening (ODS) particles. Such steels presently are most prospective materials for production of shells and covers for high-temperature nuclear reactors. This is conditioned by their small induced activity, low vacancy swelling, high stability relative to high-temperature embrittlement and stability to high-temperature creep [5]. By manufacture of concrete products from these steels a structure inhomogeneity inevitably arises, and its analysis promotes understanding of mechanisms, responsible for properties, which distinguish these steels from other reactor materials.

Studied shell tubes of 13.0 mm in diameter with wall thickness 1.0 mm were made from ferritic-martensitic ODS steel EP-450, containing 0.35 mass.% of yttrium oxide Y₂O₃.
Application of the GPF method allows to estimate the strengthening caused by disperse oxide particles, which were added in steel at the stage of powder mechanical alloying. Features of plastic deformation mechanisms in steel in the presence of strengthening disperse phase can be revealed as well. Changes of substructure condition of matrix in the presence of disperse strengthening particles helps to estimate indirectly the influence of additions on the steel structure, though direct observation of the oxide phase by diffraction pattern proves to be impossible.

Since manufacture of tubes is connected with large plastic deformations, tube material proves to be strongly textured and, as in any metal material with a developed deformation texture, significant substructure inhomogeneity arises in it. This inhomogeneity consists in sharp differences of strain hardening in different grains depending on their place in the tube texture. At that, additions of disperse strengthening particles into steel can result in redistribution of strain hardening, typical for the initial matrix, which did not contain these particles. As an illustration of possibilities of the used approach to study tubes from ODS steel, the following results are considered. Fig. 2 shows direct PF\{001\} for inward (a) and outward sides (d) of tube wall, GPF $\beta_{002}$, presenting distributions of true half-width for X-ray line for both sides of the same tube (b and e, respectively), and diagrams of correlation between GPF $\beta_{002}$ and PF\{001\} for inward and outward sides, where abscissa of each point (\(\phi, \psi\)) in the correlation diagram is equal to pole density in this point of PF\{001\}, while ordinate is equal to angular half-width $\beta_{002}$ in the same point of GPF $\beta_{002}$ (c and f, respectively). PF and GPF, constructed for opposite sides of tube wall, are characterized by significantly different scattering. At that, the mean level of values $\beta_{002}$ for outward side of the tube wall significantly exceeds that for its inward side.

Near outward surface of the rolled tube the texture is more scattered. This fact testifies that some factors prevent grains from attainment of texture maxima, make the crystallographic slip more difficult and less regular. An evident reason of observed differences between inward and outward tube surfaces consists in increased strain hardening of the latter one, connected with greater quantity of ODS particles due to their redistribution by extrusion and cold rolling. Thus, considerations of PF and GPF for opposite sides of steel ODS shell tubes lead to the mutually agreed conclusion concerning the non-uniform distribution of ODS particles within the tube.

4. Substructure non-uniformity in rods of Cu, subjected to ECAP

Though structure and texture formation under equal-channel angular pressing (ECAP) is now the main topic of numerous studies, still a number of questions remains to be considered more accurately on the basis of additional experimental data. A new experimental approach, based on X-ray GPF methods, makes clear crucial aspects of ECAP deformation.
process. Cu rods had a rectangular cross-section 8 x 8 mm in size and initially were cut out from rolled and annealed slabs. The ECAP axis coincided with the transversal direction of slabs. The temperature of ECAP was 20°C. For X-ray study of ECAP rods cubic samples 3x3x3 mm in size were cut out by the electro-sparkle method from different regions of the cross-section (Fig. 3-a). Complete texture PF were constructed by sewing of incomplete PF, measured for three facets of cubic samples (Fig. 3-b).

By X-ray study it was assumed, that any textured metal material can be adequately characterized only by distributions of substructure parameters instead of their single values. From this point of view the new X-ray GPF method was used. On the basis of obtained GPF, distributions of coherent domain size $D$, lattice distortion $\varepsilon$, dislocation density $\rho$, characterizing the condition of crystalline lattice in reflecting grains along axes $\langle hkl \rangle$, were constructed (Fig. 3-c).

In Cu rods the substructure anisotropy decreases with the number of ECAP passes and in most grains some perfection of the crystalline lattice takes place. In Cu rods within M-region most often the coherent domain size $D_{111}$ varies in limits, typical for highly deformed metal materials – from 5 nm to 20 nm. At the same time there are minor fractions with the coherent domain size $D$ up to 40 nm and more. After 4th ECAP pass the Cu rod becomes more isotropic and homogeneous, so that everywhere $D$ is equal 7-8 nm with probability ~60%.

The substructure of ECAP Cu rods shows an evident correlation with the texture non-uniformity through the rod section: weak disordered textures in T- and B-regions after 1st ECAP pass are accompanied by increased intervals of lattice distortion. Hence, irregular texture and relatively increased lattice distortion are conditioned by the same mode of deformation process, when the material cannot form the specific ECAP texture due to turbulence of the plastic flow.

5. Summary

By use of X-ray GPF method grains of deformed metal polycrystals can be split into fractions, differing by substructure parameters and, in particular, by strain hardening.

References
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