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Use of generalised pole figures in the X-ray study of textured metal materials

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Abstract. The regular non-uniform substructure of deformed textured metal materials is characterized by the new X-ray method of Generalized Pole Figures, including repeated registration of the same X-ray line for near 1000 groups of grains with different orientations. For each measured profile integral intensity I, physical half-width β and peak position 2θ are determined. Diagrams of correlation between obtained massifs of diffraction parameters I, β and 2θ are constructed. The typical interconnection of these parameters, conditioned by joint development of deformation texture and substructure in polycrystal, was revealed and interpreted in terms of involved physical processes.

Introduction

According to the kinematical theory of X-ray diffraction as applied to polycrystalline materials [1], main parameters of X-ray line (hkl), i.e. integral intensity I, true physical half-width β and peak position 2θ, are mutually independent, except for effects of stacking faults, which in cases of some X-ray lines cause both their additional broadening and shifts of the peak position. This result follows from theoretical consideration of diffraction by a separate grain with more or less distorted crystal lattice. But another approach to this question is rightful as well. It bases on the analysis of statistically significant massifs of experimental data, including measured parameters of the same X-ray line for the same material in different substructure conditions. When varying the substructure condition of material, one can trace mutually conjugated changes of parameters of the same X-ray line.

The optimal way to realize this approach consists in application of the recently developed X-ray method of Generalized Pole Figures [2,3]. This method allows to make certain that a deformed sample is characterized by the very wide spectrum of substructure conditions depending on the grain "biography" under plastic deformation. Such a biography includes, in particular, initial and final orientations of the grain, active plastic deformation mechanisms, affiliation of the grain to that or another part of the texture maximum.

Experimental procedure and data treatment

The used experimental procedure consists in recording of the same X-ray line (hkl) by successive positions of the sample in the course of texture study, so that the sample is characterized by the multitude of line profiles, corresponding to different orientations (ψ,φ) of reflecting planes {hkl}. All X-ray measurements were fulfilled by two techniques: (1) the use of a position-sensitive detector (PSD); (2) the computational method, involving the treatment of several texture files, obtained by different positions 2θ of the usual detector. SIEMENS texture diffractometers (D500/TX, D5000) and diffractometers of Russian manufacture (DRON-3, DRON-3M) with an automated texture set were used.

The treatment of measured data includes correction for the defocalization effect, approximation of X-ray line profiles with pseudo-Voigt functions, calculation of their parameters in view of results obtained for the annealed standard. This treatment is aimed to construction of so called Generalized Pole Figures (GPF), i.e. distributions of diffraction or substructure parameters in the stereographic projection of the sample [2,3].

Among diffraction parameters to be presented in GPF, along with integral intensity of X-ray line I (the corresponding normalized GPF is the usual texture pole figure PF), there are its true physical half-width β and peak position 2θ. Since a physical half-width β_{hkl} of X-ray line is determined by the size of coherent domains and by distortion of the crystal lattice along axes <hkl>, the physical half-width of X-ray line can be considered as a generalized characteristic of the lattice condition along the normal to reflecting planes: as coherent domains become smaller and lattice distortions increases, the physical profile of X-ray line widens [4]. The distribution of peak position 2θ(ψ,φ) can be recalculated into the distribution of lattice elastic deformation $\Delta d(\psi,\varphi)/d_{av}$ along crystallographic axes <hkl>, where d_{av} - average weighted interplanar spacing and $\Delta d(\psi,\varphi)=d(\psi,\varphi)-d_{av}$ with signs "+" or "-" for cases of local elastic extension or contraction, respectively.

Interconnection of different diffraction parameters was considered by use of the correlation diagrams, constructed by corresponding GPF. By construction of these diagrams as applied to GPF P_1 and GPF P_2, where P_1 and P_2 are different diffraction parameters, values P_1 and P_2 are determined for each orientation (ψ,φ) of reflecting planes (hkl), that is for each point (ψ,φ) in both GPF. Then they are used as ordinates and abscissas of points in the correlation diagram. Thus, the total number of points in obtained diagrams is equal to that in each GPF or in its some region of interest. The usual measurement procedure allows to construct incomplete GPF with an angular radius of $60^o \div 70^o$, containing near 1000 experimental points. The same number of points is contained in the correlation diagrams.

Authors employed the GPF method in studies of many metal materials, deformed by rolling. The presented diagrams of correlation between parameters of X-ray line are rather typical and characterize their actual interconnection in cases of concrete real materials. Intervals of variation of X-ray line half-width β and peak position 2θ cover values of these parameters, which are not arbitrary, but can be met in studied samples. It is meant, that changes of X-ray line half-width β and peak position 2θ can be interconnected not only through the mechanism of diffraction, but also due mechanisms of texture formation, predetermining both the distribution of grains in the orientation space and the distribution of their substructure conditions.

Dependence of substructure condition on grain orientation

Consideration of obtained GPF β testifies that the structure of deformed metals includes an extremely wide spectrum of substructure conditions. Within GPF for rolled metal materials the true half-width β of X-ray lines by intermediate Bragg angles varies usually from 0.2°÷0.6° up to 2.0°÷2.5°, i.e. covers the whole interval of practically observable values. The lower boundary of this interval corresponds to rather coarse coherent domains (60-70 nm) by insignificant lattice distortions (10^{-6}). However, its upper boundary, corresponding to the extreme fragmentation of coherent blocks and highest lattice distortions, is beyond reach for detection, since the wider is the X-ray line the lower becomes its intensity.

In figure 1 [5] the diagrams of correlation between GPF β_{hkl} and GPF I_{hkl} or PF(hkl) are presented for cases of: (a) the cold-rolled Nb foil, X-ray line (002); (b) α-Zr of Zr-1%Nb alloy after cold transverse rolling, X-ray line (0004). Though Nb and α-Zr have different crystal lattices (BCC and HCP, respectively) and were rolled by different regimes, both diagrams have a typical descending character, showing the general rule, which controls the substructure inhomogeneity of textured materials: residual deformation effects are minimal in texture maxima and increase up to highest values by passing to texture minima. Therefore minima of line broadening coincide with texture maxima, whereas maxima of line broadening are localized within texture minima. By passing from texture maxima to texture minima a slow growth of the line broadening within texture maxima gives place to its jump within texture minima.

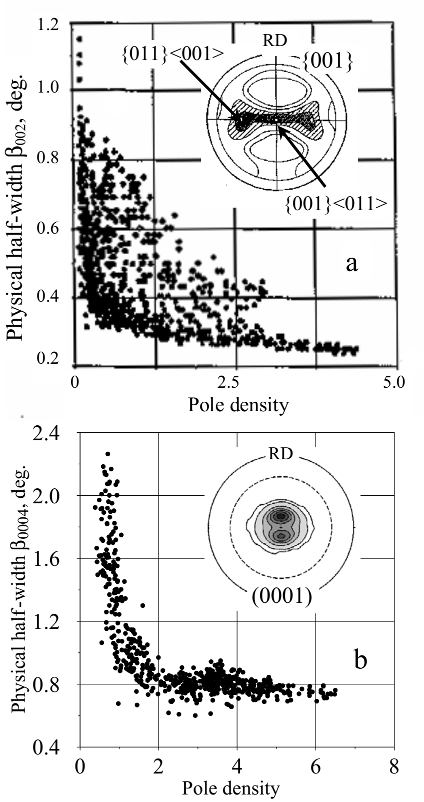

Figure 1. Correlation diagrams: (a) cold-rolled Nb foil, GPF β_{002} – PF{001}; r_{PF}=70°; (b) α-Zr, alloy Zr-1%Nb, transverse rolling, GPF β_{0004} - PF(0001), r_{PF}=60°.

In the case of Nb foil (figure 1-a) the rolling texture contains two main groups of components, close to {001}<011> and {011}<001>. In the correlation diagram the points, corresponding to these components, form two distinct branches: the lower one relates to {001}<011>, the upper – to {011}<001>, characterized by the higher strain hardening [3]. Thus, besides substructure differences between texture maxima and minima, there are differences between main texture components, developing by operation of different deformation mechanisms.

In the case of α-Zr (figure 1-b) the intermediate quasi-stable texture with main components (00.1)±15÷20°ND-RD<11.L> was formed under transverse rolling, so that basal axes of all crystallites are localized in the central part of PF(0001) and acting mechanisms of their plastic deformation are similar [6]. Owing to the latter circumstance the correlation diagram is formed by the single branch.

Besides, among factors, controlling the condition of crystal lattice along that or another crystallographic axes, there is the substructure anisotropy within each grain, consisting in different substructure conditions along crystallographic axes of the same type.

The most evident physical reason for the revealed difference in substructure conditions of grains in texture maxima and minima is the following. By sufficiently high deformation degrees the central part of texture maximum corresponds to the stable orientation of grains under the used loading. The symmetric stable orientation is maintained by simultaneous operation of several mutually balanced slip systems. When distortion of the crystal lattice within some fragment proves to be too high because of a high content of defects, balanced operation of several slip systems within this fragment becomes impossible and its orientation shifts from the symmetric stable position. Therefore at the periphery of texture maxima and in texture minima there are grains with "spoiled" crystal lattice, whereas grains with relatively perfect lattice remain at the center of texture maximum and retain the symmetric stable position. Grains with the too "spoiled" crystal lattice lose the ability to deform by means of crystallographic mechanisms, so that their final orientations prove to be occasional; texture minima contain only such grains.

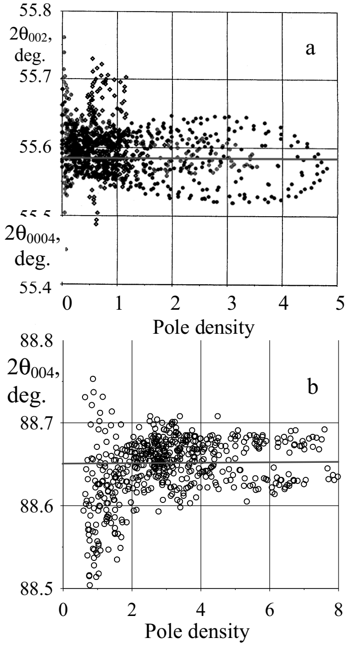

Figure 2. Diagrams of correlation: (a) cold-rolled Nb foil, GPF $2\theta_{002}$ – PF{001}; $r_{PF}=70^o$; (b) α-Zr, alloy Zr-1%Nb, transverse rolling, GPF $2\theta_{0004}$ - PF(0001), $r_{PF}=60^o$.

Equilibrium of residual elastic microstresses

In figure 2 diagrams of correlation between GPF $2\theta_{hkl}$ and GPF I_{hkl} are presented for: (a) the cold-rolled Nb foil, X-ray line (002) [5]; (b) α-Zr of Zr-1%Nb alloy after cold transverse rolling, X-ray line (0002). Their symmetry about the central line testifies, that each point (ψ,φ) of PF{002} for Nb or PF(0001) for α-Zr has its pair (ψ,φ)*, characterized by the same integral intensity I_{002} or I_{0002} and by the elastic deformation $\Delta d/d_{av}$ of the same absolute value with the opposite sign. Hence, measured GPF 2θ are formed by X-ray reflections from grains (subgrains, coherent domains and the like), whose crystal lattice experiences alternatively elastic deformations of opposite signs relative to the average level, determined by elastic macrostress.

This result is an evidence of the equilibrium of residual elastic micro-stresses within the reflecting volume of material. Since elastic microstresses, or residual stresses of 2-nd kind, by definition are equilibrated within several neighbou-ring grains [4], the volume irradiated by texture diffractometric measurements is suffi-ciently large to satisfy conditions for this equilibrium. Therefore, the symmetric shape of correlation diagrams between GPF $2\theta_{hkl}$ and GPF I_{hkl} seems to be quite natural by any concrete distribution of elastic microstrains both in usual and orientation spaces. The main principles of the latter are seen directly in GPF $2\theta_{hkl}$ by its comparison with PF{hkl}.

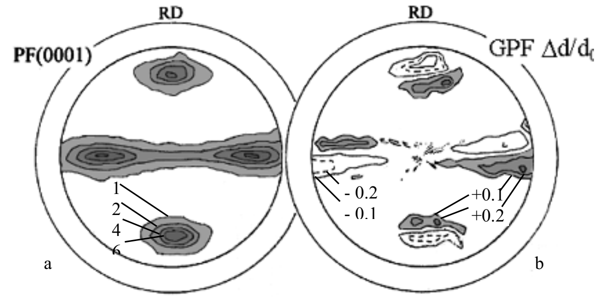

Figure 3. The distribution of lattice elastic microstrains along axes <0001> in the ω-phase of the quenched rolled alloy Zr-20%Nb: (a) PF(0001), (b) GPF $\Delta c/c_{av}$.

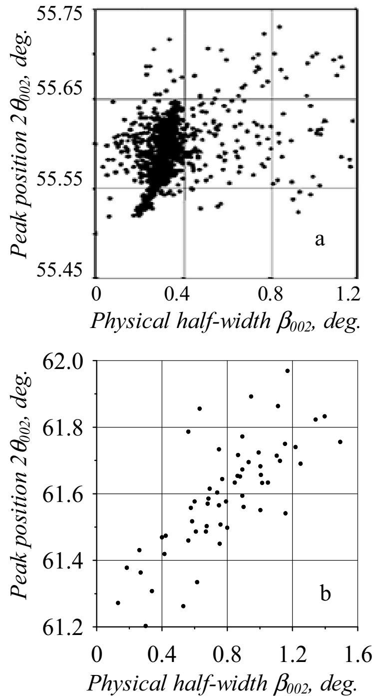

Figure 4. Correlation diagrams: (a) cold-rolled Nb foil, GPF $2\theta_{002}$ - GPF β_{002}; $r_{PF}=35^o$; (b) rolled single crystal Ti-Ni, phase B2, GPF $2\theta_{002}$ - GPF β_{002},, $r_{PF}=70^o$.

The equilibrium of residual microstresses realizes in the studied sample in connection with the observed distribution of elastic microstrans. Realization of microstress equilibrium is seen most distinctly in GPF $\Delta d/d_{av}$ for HCP metal phases with developed rolling textures. For example, in figure 3 PF(0001) and GPF $\Delta c/c_{av}$ are presented for the ω-Zr phase in the rolled alloy Zr-20%Nb [7]. In this case elastic microstrains are distributed in such a manner, that zones of tension and compression are aligned parallel with texture maxima at their opposite slopes and form the cross-wise pattern, whereas main diameters of PF separate these zones with opposite signs of residual elastic deformation. In the general case of rolled polycrystalline material, tensile and compressing microstresses prove to be mutually equilibrated about PF diameters, corresponding to symmetry planes of the deformation scheme by rolling. As a result, for high symmetry axes <hkl>, quadrants with predominant tension and compression of the crystal lattice along these axes alternate in the stereographic projection of the rolled sample, so that residual elastic microstresses prove to be equilibrated about symmetry planes of the loading scheme.

Correlation between line broadening and peak position

Diagrams of the correlation between physical half-width $\beta(\varphi,\psi)$ and peak position $2\theta(\varphi,\psi)$ of the X-ray line, measured for grains with different crystallographic orientations as applied to rolled metal materials, are presented here for the first time. A character of these diagrams varies depending on material and indexes (hkl) of reflecting planes. In figure 4 the diagrams are shown for (a) cold-rolled Nb foil; (b) Ti-Ni single crystal, rolled in B2 phase by 77% with initial orientation {011}<011>; in both cases X-ray lines (002) were measured. The tendency, revealed in both cases, is very simple: the wider is the X-ray line, the higher is angle 2θ. Since the rolling texture of Ti-Ni single crystal is very sharp [8], its GPF contains a relatively small number of experimental points, but the indicated tendency is seen distinctly.

The most evident interpretation of this tendency is the following. By passing from residual tension of the crystal lattice to its compression, that is by exceeding of the average peak position $2\theta_{av}$, fragmentation of crystallites and distortion of their lattice become stronger. When representing the profile of X-ray line as a superposition of several profiles, corresponding to fragments with different values of elastic microstrain, we can establish, that tensile elastic deformation is more uniform than compressing one. The character of revealed correlation between β and 2θ is determined by fundamental properties of crystalline solids and can be the basis for the new method to study these properties.

Summary

For a number of rolled metal materials with developed deformation textures the diagrams of correlation between integral intensity I, physical half-width β and peak position 2θ are considered as applied to the extensive massif of the same X-ray line profiles, measured for grains with different orientations. Correlation between β and I indicates to the regular substructure inhomogeneity of textured materials, which involves differences in substructure of crystallites, belonging to texture maxima and minima. Correlation between 2θ and I is conditioned by the equilibrium of residual elastic microstresses, whereas correlation between β and 2θ – by different reactions of the crystal lattice to tension and compression.

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