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X-RAY STUDY OF YTTRIUM TEXTURE USING STANDARD AND EXTENDED DIALING POLE DENSITIES

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Various methods of experimental study of the texture of polycrystalline metals and alloys are considered. The texture formed upon plastic deformation or upon recrystallization annealing leads to anisotropy of the physical and mechanical properties of metallic materials. For express calculations of the values of anisotropic physical properties of polycrystals, methods based on the use of integral characteristics of textures are preferred. An X-ray method for calculating orientation factors for textured metals with a hexagonal close-packed structure is discussed. Calculation of the orientational factor of texture Δ_i assumes averaging over the representative volume of the polycrystalline sample under study. The basis for calculating the orientational factors of a textured polycrystal is the reverse pole figure. In the standard method for calculating the reverse pole figure of hcp metals, 17 crystallographic orientations are used. Yttrium texture analysis can be performed using an extended set of pole densities on the reverse pole figure. A method for calculating the reverse pole figure for hcp metals is proposed, which takes into account 24 crystallographic orientations. The data of X-ray diffraction studies of polycrystalline yttrium after deformation by cold rolling with a degree of deformation $\varepsilon = 45\%$ are discussed. On the basis of the obtained X-ray data, the reverse pole figures of deformed yttrium were constructed using the standard method and using an extended set of pole densities. The orientation factor of the texture Δ_i of the deformed yttrium polycrystal was determined. A comparative calculation of the resistivity ρ_i of textured yttrium has been performed using two experimental procedures. The extended set of pole densities on the reverse pole figure is most convenient when studying samples in which a sharp deformation texture is formed. The proposed technique accurately describes the dynamics of texture formation, describes more precisely the features of the pole density distribution on the reverse pole figure, and allows one to determine the anisotropic physical properties of materials.

Keywords: polycrystals, texture, anisotropy, structure, deformation, rare-earth metals, X-ray analysis, diffraction.

Introduction

An ordered distribution of crystallites in metals and alloys (textured state) can be formed during crystallization (ingot texture), with plastic deformation methods of different symmetry (rolling texture, upsetting texture, drawing texture, etc.), during heat treatment (recrystallization texture), during mechanical treatment of the sample surface (work hardening texture) and other methods of external impact on the materials under study [1, 2]. The consequence of the appearance of texture in polycrystalline materials is the anisotropy of their physical and mechanical properties [3–5].

In practice, the need often arises for a comparative analysis of the texture states of samples at different stages of thermomechanical processing, as well as for current assessments of the level of anisotropy of the physical properties of these samples. Known techniques for studying textures make it possible to analyze the processes of texture formation during deformation of polycrystalline materials. However, the accuracy of the description of the texture can be increased using an extended set of experimental data.

1. Methods and Materials

Various methods are used to analyze the texture state of metals and alloys [6–8]. The first to appear were the methods based on the calculation of the crystallite orientation distribution function (ODF). Such approaches were proposed in the works of Bunge [9] and Roe [10]. ODF recovery in most cases is performed on the basis of processing the experimental data of X-ray analysis of polycrystalline samples by the method

of straight pole figures [11]. This was followed by studies in which texture analysis was performed on the basis of the experimentally reconstructed function of crystallite misorientations in textured polycrystals. Such techniques, despite the clarity of the description of the texture and the use of universal approaches of the theory of probability, are difficult for practical application.

Methods based on calculations of the integral characteristics of the texture of the materials under study are more convenient and simpler for use in the conditions of factory laboratories (carrying out express analysis) [3, 12]. One of such methods for analyzing the texture state of metals is the method for calculating the orientation factors of texture Δ_i [4]:

$$\Delta_i = \langle \alpha_{i1}^2 \alpha_{i2}^2 + \alpha_{i2}^2 \alpha_{i3}^2 + \alpha_{i3}^2 \alpha_{i1}^2 \rangle,$$

where α_{ij} are the elements of the rotation matrix defining the position of the j-th axis of the coordinate system associated with the crystallite relative to the i-th axis of the laboratory coordinate system or the system associated with an orthotropic sample (i = 1, 2, 3).

The calculation of the texture orientation factor Δ_i assumes averaging over the representative volume of the sample under study. The basis for calculations of Δ_i is the experimental data of X-ray analysis of polycrystals by the method of inverse pole figures (IPF) [13]. In the practice of X-ray analysis, the construction of inverse pole figures for metals with a bcc structure is performed according to 6 crystallographic orientations, for metals with an fcc structure – along 18 orientations, and for metals with an hcp structure – along 17 orientations.

The relationship between the pole density of the k-th orientation of crystallites on the reverse pole figure and the value of Δ_i can be expressed as follows [14]:

$$\Delta_i = \iiint_{0\,0\,0}^{\pi\ 2\pi\ 2\pi} \left(\alpha_{i1}^2 \alpha_{i2}^2 + \alpha_{i2}^2 \alpha_{i3}^2 + \alpha_{i3}^2 \alpha_{i1}^2\right) \times \\ \times \sum_{k=1}^{m} P_k A_k \delta(\varphi - \varphi_k) \delta(\psi - \psi_k) \times \\ \times \delta(\vartheta - \vartheta_k) d\varphi d\psi d\vartheta,$$

A_k is the Morris coefficient for the reverse pole figure, P_k is the pole density on the reverse pole figure for the k-th crystallite orientation, the position of which is described by the angular coordinates [φ_k, ψ_k, ϑ_k] [6]:

$$P_k = \frac{I_k / I_{k\,ref}}{\sum_{k=1}^{m} (A_k I_k / I_{k\,ref})},$$

I_k is the intensity of the X-ray line for the k-th orientation of crystallites in a textured polycrystal, $I_{k\,ref}$ is the intensity of the X-ray line for the k-th orientation of crystallites in the reference sample (isotropic state), m is the number of poles on the reverse pole figure.

Having determined from the data of X-ray analysis the value of the orientation factor Δ_i for the initial and subsequent state of the sample, it is possible to make a conclusion about the direction of the processes of texturing in the course of thermomechanical processing of the metal. In addition, the known texture parameters Δ_i can be used to calculate the values of the anisotropic physical properties of the materials under study. For metals with a hexagonal close-packed crystal lattice (hcp metals), the relationship between monocrystalline constants and texture parameters Δ_i is expressed as:

$$S_i = S_{\perp}^{M} + \left[S_{\parallel}^{M} - S_{\perp}^{M}\right] \Delta_i,$$

S_i is the value of the physical quantity in the i-th direction of the investigated polycrystalline sample, $S_{\perp}^{M}$ is the value of a physical quantity measured in the direction perpendicular to the hexagonal axis of the single crystal, $S_{\parallel}^{M}$ is the value of a physical quantity measured along the hexagonal axis of the single crystal.

Thus, the accuracy of texture analysis and calculation of the anisotropic physical properties of materials is largely determined by the accuracy of determining the orientation factors of texture Δ_i. The accuracy of calculations Δ_i is influenced not only by errors in determining the intensities of X-ray lines I_k or pole densities P_k, but also by the number of measured poles m on the pole figure of the sample under study. For most hcp metals and alloys, the number of poles is limited to 17 orientations because the diffraction reflections of other crystallographic orientations go beyond the angular range available for an X-ray diffractometer. In this case, the limiting value diffraction angle is the crystallographic orientation $\{30\bar{3}2\}$. The calculations performed for the crystallographic orientations of the yttrium rare earth metal (hcp structure) show that there are seven additional lines in the angular range available for X-ray photography. Taking into account these features of yttrium, a scheme for calculating the OPF was developed for a larger number of poles (for 24 poles) compared to the standard number (17 poles). The texture orientation factors were also calculated taking into account 24 pole densities.

The object of the study was samples of technical purity yttrium (grade ИтМ-1). The degree of deformation by cold rolling was ε = 45%.

The intensities I_k were measured on an industrial X-ray diffractometer ДРОН-0,5. A copper anticathode was used (Cu-$K_{\alpha 1}$ + Cu-$K_{\alpha 2}$ radiation). For calculations, the average value of the X-ray radiation wavelength $\lambda_{K_\alpha} = 1{,}54178$ Å was used. The parameters of the yttrium crystal lattice used in the calculations at $T = 290$ K [15]: $a = 0.36482$ nm, $c = 0.57318$ nm.

2. Results of the X-ray experiment and their discussion

Fig. 1 shows a reverse pole figure obtained for cold-worked yttrium using 17 poles. Fig. 2 shows the results of constructing the reverse pole figure of yttrium using X-ray data for 24 crystallographic orientations.

It can be noted that both techniques equally reveal the nature of the texture formed in a polycrystalline sample and do not contradict the results of the study [16]. An acute slanted texture component is formed in the sample, the center of which is the pyramidal preferred orientation $\{10\bar{1}4\}$. Both techniques reveal the preferred orientation $\{10\bar{1}5\}$ in the yttrium texture, as well as the developed prismatic orientation of $\{12\bar{3}0\}$. A similar acute one-component deformation texture is also characteristic of polycrystalline gadolinium [17].

It can be noted that both techniques equally reveal the nature of the texture formed in a polycrystalline sample and do not contradict the results of the study [16]. A rather sharp oblique texture component is formed in the sample, the center of which is the pyramidal preferred orientation $\{10\bar{1}4\}$. In this case, both techniques reveal the preferred orientation $\{10\bar{1}5\}$ in the yttrium texture, as well as the developed prismatic orientation of $\{12\bar{3}0\}$. A similar sharp one-component deformation texture is also characteristic of polycrystalline gadolinium [17].

The middle angular area of the stereographic triangle does not contain pronounced preferential orientations and is depicted in the same way according to the calculations of both methods. However, the technique using 24 crystallographic orientations describes in more detail the distribution of pole density on the reverse pole figure, which is more preferable for studying the regularity of texture formation mechanisms.

For comparison of the degree of texture detail, carried out by two methods, the most remarkable is the region of small angles on the reverse pole figure. Fig. 1b shows that the pole density is blurred from the center of the formed texture component $\{10\bar{1}4\}$ to the region of oblique orientation $\{11\bar{2}6\}$. In addition, it can be noted that in the OPF in Fig. 1b, the region of increased pole density is blurred in the radial direction with respect to the basic orientation (0002) and captures the $\{12\bar{3}5\}$ orientation,

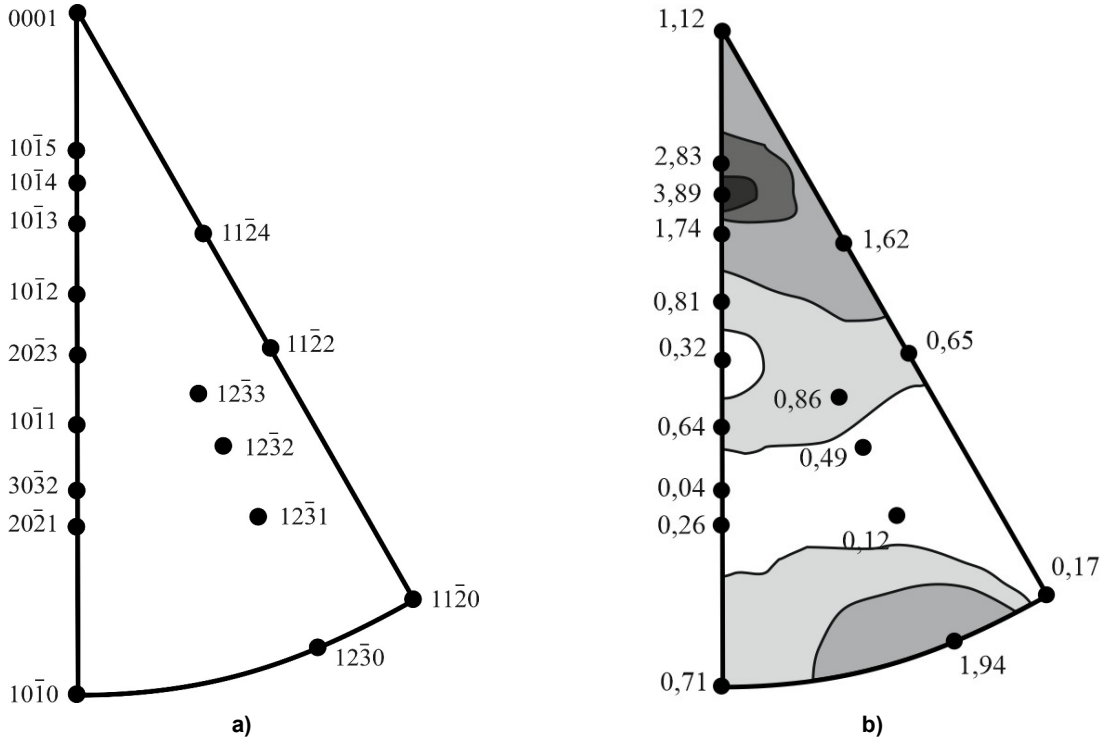
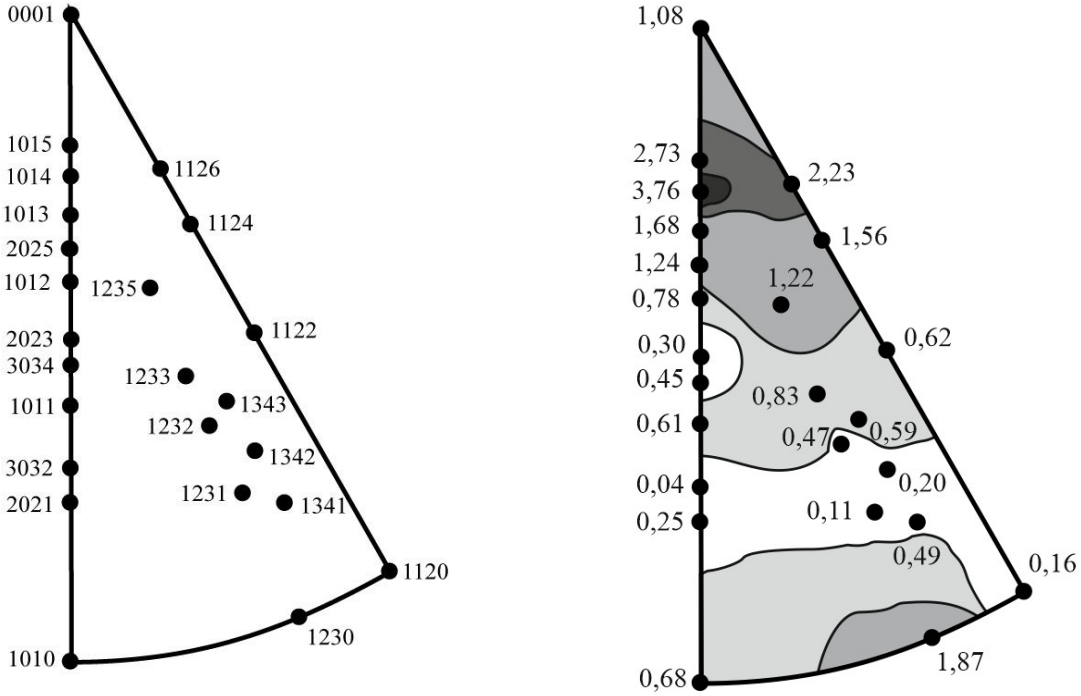


Fig. 1. Reverse pole figure of yttrium obtained using 17 poles



a) b)

Fig. 2. The reverse pole figure of yttrium obtained using 24 poles

Table 1

Number of poles on a reverse pole figure	Δ_i	$\rho_i \cdot 10^{-8}$ Ohm · m	$\frac{\lvert\rho_i - \langle\rho\rangle\rvert}{\langle\rho\rangle} \cdot 100\%$
17	0,5482	61,98	33,20
24	0,5656	62,69	34,25

which is not detected by the technique using only 17 orientations.

The difference between the two methods for describing the texture of a polycrystalline sample is strongly pronounced in the region of small angles of the stereographic triangle. The yttrium texture detail is higher when using 24 crystallographic orientations to construct the reverse pole figure.

Fig. 2 shows that the pole density is blurred from the center of the formed texture component $\{10\bar{1}4\}$ to the region of oblique orientation $\{11\bar{2}6\}$. In addition, in the reverse pole figure (Fig. 2), the region of increased pole density is blurred in the radial direction with respect to the basic orientation (0002) and captures the $\{12\bar{3}5\}$ orientation, which is not detected by the technique using only 17 orientations.

3. The calculation results

As an example of calculating the anisotropic physical property of textured yttrium, we used the resistivity value. Single-crystal parameters for yttrium at $T = 300$ K are [15]:

$\rho_{\parallel} = 39{,}5 \cdot 10^{-8}$ Ohm·m,

$\rho_{\perp} = 80{,}5 \cdot 10^{-8}$ Ohm·m,

$\langle\rho\rangle = 67{,}7 \cdot 10^{-8}$ Ohm·m.

The main calculated values obtained by the two methods used are shown in Table 1.

Conclusions

The yttrium texture can be investigated based on the use of 24 crystallographic orientations. An extended set of pole densities increases the accuracy of constructing a pole figure and allows a more accurate analysis of the texture state of a polycrystalline sample, more accurately describing the dynamics of texture formation during metal deformation. The extended set of pole densities on the reverse pole figure is most convenient when examining specimens in which a sharp deformation texture is formed. The proposed method for calculating inverse pole figures makes it possible to more accurately take into account the inhomogeneity of the pole figure density when calculating the anisotropic physical properties of polycrystalline materials.

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РЕНТГЕНОГРАФИЧЕСКОЕ ИССЛЕДОВАНИЕ ТЕКСТУРЫ ИТТРИЯ С ИСПОЛЬЗОВАНИЕМ СТАНДАРТНОГО И РАСШИРЕННОГО НАБОРА ПОЛЮСНЫХ ПЛОТНОСТЕЙ

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В статье рассматриваются различные методы экспериментального исследования текстуры поликристаллических металлов и сплавов. Сформированная при пластической деформации или при рекристаллизационном отжиге текстура приводит к анизотропии физических и механических свойств металлических материалов. Для экспресс-расчетов величин анизотропных физических свойств поликристаллов предпочтительными являются методики, основанные на использовании интегральных характеристик текстур. Обсуждается рентгеновская методика расчета ориентационных факторов для текстурованных металлов с гексагональной плотноупакованной структурой. Расчет ориентационного фактора текстуры Δ_i предполагает усреднение по представительному объему исследуемого поликристаллического образца. Основой для расчетов ориентационных факторов текстурованного поликристалла является обратная полюсная фигура. Стандартная методика построения обратной полюсной фигуры ГПУ-металлов требует учета 17 полюсных плотностей. Текстурный анализ иттрия возможно выполнить по расширенному набору полюсных плотностей на обратной полюсной фигуре. Предложена методика построения обратной полюсной фигуры для ГПУ-металлов на основе 24 полюсных плотностей. Обсуждаются данные рентгенографического исследования поликристаллического иттрия после деформации холодной прокаткой со степенью деформации $\varepsilon = 45$ %. На основе полученных рентгенографических данных построены обратные полюсные фигуры деформированного иттрия с использованием стандартного метода и с использованием расширенного набора полюсных плотностей. Определены ориентационного фактора текстуры Δ_i деформированного поликристалла иттрия. Выполнен сравнительный расчет удельного сопротивления ρ_i текстурированного иттрия по двум экспериментальным методикам. Расширенный набор полюсных плотностей на обратной полюсной фигуре наиболее удобен при исследовании образцов, в которых сформирована острая текстура деформации. Предлагаемая методика позволяет точнее описывать динамику текстурообразования, описывать неоднородность распределения полюсной плотности на обратной полюсной фигуре и определять анизотропные физические свойства материалов.

Ключевые слова: поликристаллы, текстура, анизотропия, структура, деформация, редкоземельные металлы, рентгеновский анализ, дифракция.

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