Z. Kristallogr. Suppl. **26** (2007) 333-338

Reconstruction of high-temperature deformation process by texture of the low-temperature phase as applied to Zr-based alloys

M. Isaenkova, Yu. Perlovich, V. Fesenko, M. Grekhov

Moscow Engineering Physics Institute (State University)
Kashirskoe shosse, 31, Moscow 115409, Russia
e-mail: isamarg@mail.ru , perlovich@hotmail.com

Keywords: crystallographic texture, Zr-Nb alloys, deformation mechanisms, phase transformation, intergranular slippage.

Abstract. As applied to Zr-based alloys the deformation process at temperatures of β- and (α+β)-regions of the phase diagram Zr-Nb is considered by texture features of α-phase, revealed by X-ray study at the room temperature. Plastic deformation results in violation of the phase equilibrium and intensification of α↔β phase transformations, whereas these phase transformations are favorable to development of the additional deformation mode, consisting in slippage by interphase boundaries.

Introduction

The deformation behavior of metals with phase transformations (PT) varies in wide limits depending on the temperature-rate regime of deformation. But in most cases a direct observation of structure formation processes in the course of high-temperature deformation is impossible, so that the most data concerning these processes are obtained by means of their reconstruction on the basis of structure parameters, measured at room temperature by the low-temperature phase. The problem consists in the choice of measurable parameters, giving clear and reliable information on the high-temperature prehistory of material. As opposed to various structure characteristics, which sharply change by cooling and PTs, the texture of the material keeps an undistorted memory about previous deformation and therefore is used by its reconstruction.

By the technological treatment of Zr-based alloys for nuclear industry an important role belongs to deformation at temperatures of the ($\alpha+\beta_{Zr}$)-region of phase diagrams, which in the case of Zr-Nb system corresponds to the temperature interval 610°-860°C (figure 1) [1]. However, passing through this interval is accompanied by complete replacement of the phase composition: the low-temperature α-phase with HCP crystalline lattice is replaced by the

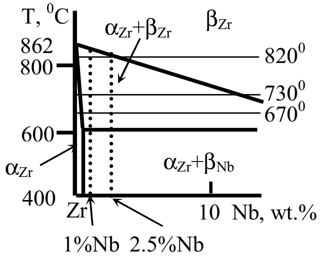

Figure 1. The fragment of Zr-Nb phase diagram.

high-temperature β-phase with BCC crystalline lattice. Deformation mechanisms as well as final structure and texture features of the product change also. There are reasons to suppose that under plastic deformation PT α↔β influence the mechanisms of deformation, whereas the latter, in its turn, has an effect on the development of these PT.

A specific character of the undertaken study consists in the preferred usage of X-ray methods and, in particular, the texture analysis, because of the high sensitivity of crystallographic texture relative to changes in plastic deformation mechanisms.

Experimental procedure and data treatment

As a deformation mode, imitating to some degree real technological procedures of forging and pressing, uniaxial compression was chosen. Cylindrical billets ∅16 mm and 24 mm in high were cut from ingots of Zr-1%Nb and Zr-2,5%Nb alloys. Within ($\alpha+\beta_{Zr}$)-region of the phase diagram Zr-Nb (Fig. 1) billets were preheated for deformation at 670°, 730°, 820°C, and within β-region – at 910°, 980°C; the rate of compression was equal to 0.1, 0.4, 0.7, 1.0 sec^{-1} by integral deformation degree of 43÷47%. After deformation the obtained samples were cooled in a salt solution.

For X-ray studies the diffractometer DRON-3M of Russian manufacture, supplied with an automated texture set, was used. Treatment of measured data, that is construction of texture pole figures (PF) and determination of their parameters, was carried out by a software, including a number of specially elaborated programs. Direct pole figures PF(0001)$_\alpha$ with angular radius up to 80° were constructed on the basis of experimental data. Some typical PF for both alloys are shown in figure 2. By presentation of obtained data the reference coordinate axes C, R1, R2 were used, so that axis C coincides with the direction of compression, whereas identical axes R1 and R2 lie in the perpendicular section of the cylindrical sample.

The texture of the predominant α-Zr phase in products from Zr-based alloy, measured at room temperature, depends on the phase composition at the temperature of deformation. In the general case this texture is the sum of textures, corresponding to several fractions with different prehistories. Among them there are: the fraction, experienced deformation in β-phase and the subsequent β→α PT; fractions, deformed either in β- and α-phases or only in α-phase; the fraction, deformed by means of non-crystallographic slippage along intergranular or interphase boundaries. The orientation relationship between β- and α-phases [1] controls the texture of α-Zr, arising by the PT β→α. Obtained texture data allow to refine details of the deformation development in Zr-based alloys and to reveal some additional effects, connected with interaction of plastic deformation and PTs.

According to the texture formation theory [2], operation of the crystallographic deformation mechanisms (intragranular slip and twinning) results in regular rotations of the crystalline lattice, directed reorientation of grains and sharpening of the texture. On the contrary, mutual non-crystallographic displacements of grains by intergranular boundaries are accompanied by accidental lattice rotations and scattering of the texture, as it occurs under conditions of the superplastic flow in metal materials and, in particular, in Zr-based alloys with ultra-fine grains [3]. These mutually opposite tendencies can be compared quantitatively only by texture pole figures, containing statistically significant information on the deformation behavior of grains within polycrystalline material.

For quantitative characterization of the texture scattering the following parameters were determined by PF$(0001)_\alpha$:

- averaged angular width of texture maxima $B_{0.75}$, measured along PF radius at the level, corresponding to 0,75 of their height;
- volume fractions ν_{min} and ν_{max} of α–Zr crystallites with basal axes within texture minima and maxima, respectively, by the boundary between maxima and minima, going along the pole density contour p=1, so that for random material $\nu_{min} \approx \nu_{max}$, whereas for textured material $\nu_{min} < 0.5$ and $\nu_{max} > 0.5$;
- volume fraction ν_o of the random component, formed by crystallites with basal axes, uniformly distributed by all possible orientations, and numerically equal to minimal pole density p_{min}.

Figure 3 demonstrates changes of these parameters depending on the nominal deformation temperature for both alloys; separate curves correspond to different used deformation rates.

Discussion

Consideration of the texture development in studied samples allows to reconstruct plastic deformation processes and their interaction with PT under compression at different temperatures. By this consideration it is necessary to take into account an initial condition of samples before deformation. A microstructure of initial samples is inherited from the ingot and consists of coarse β-Zr grains, transformed by cooling into several families of parallel α-Zr lamellae. There are only a few β-grains with occasional orientations within a volume of ~1 mm^3, studied by the X-ray diffractometric texture measurement of the initial sample, so that one can not determine its texture as a statistically significant characteristic (figure 2-a,f).

Obtained results show several variants of the texture development depending on the temperature of their preheating for deformation.

By preheating in the β-region, the whole alloy turns into β-Zr phase with subsequent additional growth of β-grains, so that their deformation develops by rules, typical for single crystals with BCC crystalline lattice. It rotates under compression to the nearest final stable orientation, by which axis C coincides alternatively with crystallographic axes <001>, <111>, <112> and sometimes <011> [2]. According to the texture formation theory, stability of these orientations is connected with operation of several mutually symmetric slip systems. Since at high temperatures the deformation of β-Zr proceeds without noticeable strain hardening, its texture has an ideal character with very intense and narrow maxima. By the next cooling, as a result of β→α PT, according to the actual orientation relationship $\{011\}_\beta || (0001)_\alpha$, derivative families of α-Zr lamellae inherit the texture features of β-Zr, so that PF$(0001)_\alpha$ proves to be similar to PF$\{011\}_\beta$ (figure 2-b,g). But whereas in PF$\{011\}_\beta$ these maxima correspond to mutually connected axes $<011>_\beta$ of the single texture component, after β→α transformation in derivative PF$(0001)_\alpha$ they become mutually independent and belong to different texture components of new-formed α-Zr.

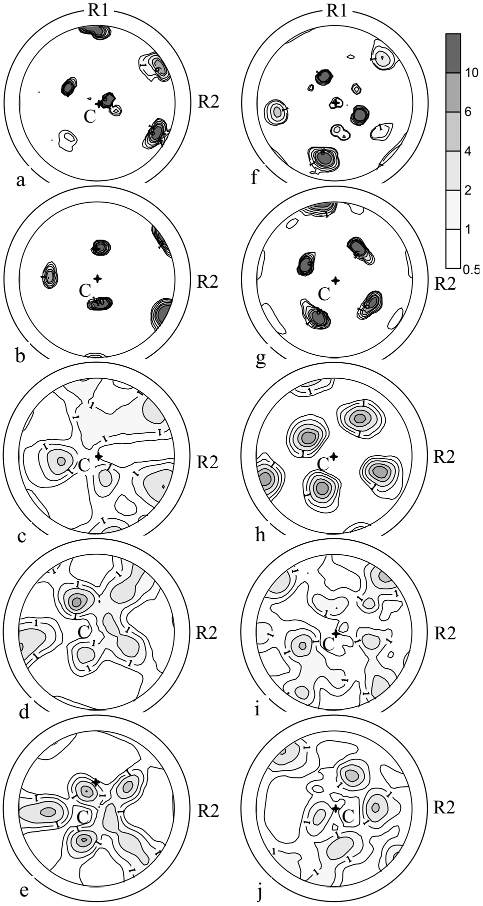

Figure 2. Typical PF(0001)$_{\alpha}$ for samples from Zr-1%Nb (a-e) and Zr-2.5%Nb (f-j) alloys in initial condition (a,f) and after deformation at different temperatures: b,g) 910^{o}C; c,h) 820^{o}C; d,i) 730^{o}C; e,j) 670^{o}C.

When the temperature of preheating corresponds to the upper part of the (α+β)-region, which is not sufficiently high for growth of β-grains, compression of samples results in formation of the texture with the same main components, typical for BCC metals, but with much wider maxima (figure 2-h). Since preheating does not result in the complete $\alpha \rightarrow \beta$ PT, new-arising grains of β-Zr are separated from each other by streaks of α-Zr and therefore the sample deforms as a polycrystal, characterized by some mutual mismatching in behavior of neighboring grains. Rotations of the crystalline lattice in these grains are directed in the similar way, but are not quite identical, so that texture maxima widen.

When by heat removal and resulting cooling the deformation continues in the α-phase, the PT texture changes in such a way that its maxima tend to approach final stable positions, typical for deformation textures of α-Zr. Under compression of α-Zr crystallites, stable positions of their basal axes are distanced by ~20^{o} from the direction of compression [4]. Therefore, a shift of texture maxima in PF(0001)$_{\alpha}$ from their positions, inherited from β-Zr, is an undoubted evidence of continuation of deformation in α-phase.

In those cases, when under compression at PT temperatures contents of α- and β-phases in the sample are mutually comparable and their grains are sufficiently fine-dispersed, the intragranular crystallographic slip proves to be restrained due to interaction of dislocations with grain boundaries. Such conditions stimulate operation of an alternative deformation mechanism, consisting in displacements of crystallites along interphase boundaries, which becomes noticeable by suppression of texture development or by sharp weakening of the texture, formed earlier (figure 2-c,i). This variant is of most interest in the context of the given work, and the above-introduced parameters are used for more accurate estimation of an actual input of interphase slippage into deformation.

In samples, compressed at lowest temperatures within ($\alpha+\beta_{Zr}$)-region, the texture development must be determined by predominant deformation in the α-phase. Then, by the used deformation degree the texture must be rather distinct and its components – close to typical components of the α-Zr rolling texture. However, many samples after deformation at 670° and 730°C display textures, clearly originating from ideal orientations of β-Zr grains, as if the majority of initial α-Zr grains experienced PT α↔β in spite of a relatively low deformation temperature (figure 2-d,e,j).

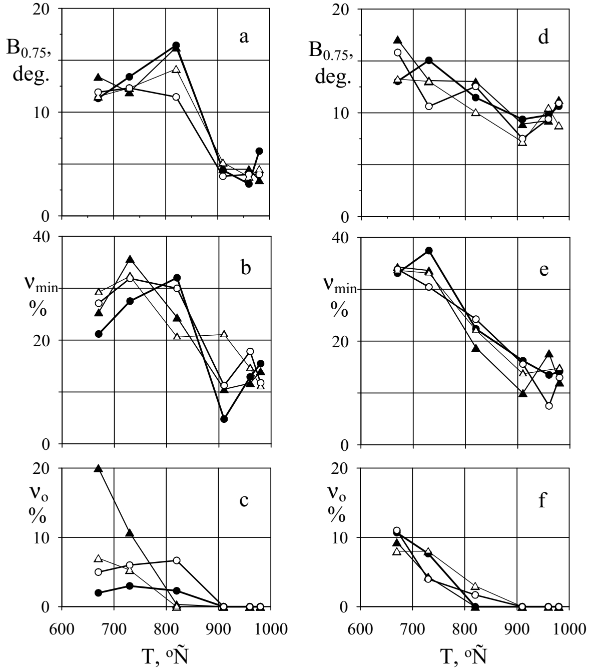

Figure 3. Changes of texture parameters of model samples from Zr-1%Nb (a-c) and Zr-2.5%Nb (d-f) alloys depending on deformation temperature: a, d) $B_{0.75}$, b,e) ν_{min}, c,f) ν_o. Shown curves correspond to different deformation rates:

— • — *0.1 s^{-1};* — o — *0.4 s^{-1};*
—▲— *0.7 s^{-1};* — Δ — *1.0 s^{-1}.*

The following factors can be responsible for this effect:

(1) By preheating of samples for deformation, intergranular lamellae of residual β-phase in accordance with Zr-Nb phase diagram transform from β-Nb into β-Zr, so that an initial Nb content in β-phase decreases by several times with the corresponding upgrowth of its volume fraction.

(2) Heat generation, accompanying any elementary acts of plastic deformation, causes a local rise of temperature, resulting in local violation of the phase equilibrium; therefore, within shear bands additional α→β PT takes place and crystallites of β-Zr appear.

(3) Under specific conditions of plastic deformation by an increased content of defects at temperature close to that of α→β PT, the crystalline lattice of α-Zr looses its stability and changes into the lattice of β-Zr with an increased probability.

Because of above reasons new nuclei of β-grains arise in each region of localized deformation. As a result, the fraction of β-phase under deformation proves to be so significant, that it determines the final character of developing texture. At the same time, by analogy with the above-considered case, deformation in α-Zr phase causes deviation of texture maxima from positions, inherited by β→α transformation, to axis C.

When comparing deformation textures, formed in alloys with different Nb contents (figure 2), it is seen, that the maximal texture scattering in Zr-1%Nb alloy takes place at a higher deformation temperature (820°C), than in Zr-2.5%Nb alloy (730°C). This experimental fact agrees with the phase diagram of Zr-Nb (figure 1), where within the ($\alpha+\beta_{Zr}$)-region a decrease of the Nb content in alloy involves a rise in the temperature of equi-phase composition, when slippage by interphase boundaries is most probable.

Texture scattering, characterized by changes of parameters in figure 3, is an evidence of deformation-induced PT. When judging from values of parameters $B_{0.75}$ (figure 3-a,d) and ν_{min} (figure 3-b,e), samples from Zr-1%Nb alloy have the most scattered texture after compression at temperatures 730°-820°C, whereas samples from Zr-2.5%Nb alloy - after compression at temperatures 670°-730°C. The character of determined temperature dependences indicates, that at 670°C the same deformation mechanism of interphase slippage is certainly active, as at higher temperatures within $(\alpha+\beta_{Zr})$-region. And what is more, the samples, compressed at 670°C, contain the greatest fraction of random oriented grains ν_o (figure 3-c,f). Thus, scattering of the deformation texture, accompanying development of $\alpha\rightarrow\beta$ PT, proves to be more intense, than it is anticipated on the basis of equilibrated phase diagram. Evidently, the deformation, beginning in α-phase, quickens $\alpha\rightarrow\beta$ PT, so that this PT covers the greater part of initial α-matrix at a lower temperature in comparison with predicted one. As the temperature of deformation within $(\alpha+\beta_{Zr})$-region increases, β-grains begin to grow in size and an input of the interphase slippage in deformation is reduced, so that effects of texture scattering weaken.

In the course of compression the effect of cooling by heat sink gradually increases and at some moment can exceed the effect of deformative heating by operation of slip systems in crystallites of β-phase, so that its lattice becomes locally instable and deformation-induced $\beta\rightarrow\alpha$ PT develops. The lower the deformation temperature the higher is the probability of $\beta\rightarrow\alpha$ PT. At the next stage the plastic deformation of newly-formed α-Zr crystallites begins, initiating local $\alpha\rightarrow\beta$ PT once more. This cycle, including crystallographic slip and deformation-induced PT, can repeat in both phases and promotes deformation by interphase slippage.

Summary

In model samples from Zr-1%Nb and Zr-2.5%Nb alloys, subjected to uniaxial compression at temperatures within the $(\alpha+\beta_{Zr})$-region of the phase diagram Zr-Nb, a wide spectrum of deformation mechanisms, including crystallographic slip in α- and β-phases as well as non-crystallographic displacements of grains along interphase boundaries, is observed. Crystallographic slip in grains of α-Zr causes local heating, instability of crystalline lattice and intensification of $\alpha\rightarrow\beta$ PT, which embraces the whole α-matrix at lower temperatures, than those foreseen by the phase diagram. Next local cooling due to heat sink, accompanying further deformation, can induce $\beta\rightarrow\alpha$ transformation. Repeated cycles of $\alpha\leftrightarrow\beta$ PTs create favorable conditions for operation of the non-crystallographic deformation mechanism of interphase slippage.

References

1. Douglass, D.L., 1971, *The Metallurgy of Zirconium* (International Atomic Energy Agency, Vienna).
2. Wassermann, & Grewen, J., 1962, *Texturen metallischer Werkstoffe* (Springer-Verlag, Berlin / Heidelberg)
3. Holt, R.A. & Aldridge, S.A., 1985, *J. Nucl. Mat.* **135** , 246.
4. Tenckhoff:, E., 1988, *Deformation Mechanisms, Texture and Anisotropy in Zirconium and Zircaloy*, ASTM, STP 966 (Philadelphia, PA).