Regularities of recrystallization in rolled Zr single crystals

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Abstract. Experiments by rolled single crystals give a more visible conception of the operating mechanisms of plastic deformation and the following recrystallization, than experiments by polycrystals. Studies by usage of X-ray diffraction methods were conducted by Zr single crystals. It was revealed, that regions of the α-Zr matrix, deformed mainly by twinning, are characterized with decreased tendency to recrystallization. Orientations of recrystallized α-Zr grains correspond to “slopes” of maxima in the rolling texture, where the level of crystalline lattice distortion is maximal and the number of recrystallization nuclei is most of all.

1. Introduction
The use of single crystals (SC) in experiments, investigating mechanisms of plastic deformation and following recrystallization, allows to reveal preferential modes of texture formation in polycrystalline zirconium and its alloys under main technological processes. Earlier studies of the plastic deformation mechanisms were carried out by coarse-grained polycrystalline zirconium [1-2]. A series of papers [3-4], performed on SCs, were devoted to estimation of critical shear stresses for different slip and twinning systems in zirconium, deformed by uniaxial tension or compression. In this paper, plastic deformation mechanisms by cold rolling and subsequent recrystallization of deformed Zr SCs were studied by use of X-ray diffraction methods.

2. Samples and methods of investigation
The studied plates 7x10x3 mm³ in size were cut out by the electro-erosion method from cylindrical SC of pure Zr, obtained by solid-phase over-crystallization. SCs with different initial orientations were rolled on a laboratory mill up to various deformation degrees from 5 to 98% with ~5% reduction per pass. X-ray measurements of SCs were conducted after each stage of its plastic deformation. SC reorientation under rolling was analyzed by direct pole figures (PF). These PFs were constructed on the basis of data, obtained by the X-ray diffractometer DRON, equipped with an automatic texture attachment and utilizing the filtered radiation Cr. Distributions of strain hardening in rolled SCs were studied by X-ray method of generalized pole figures (GPF) [5], measured by diffractometer D8 Discover with the filtered Cu radiation and self-developed software for a description of the line profile at each point of the stereographic projection. All rolled samples were annealed in a vacuum furnace at 580°C for 3 h, which is a regime, used usually for recrystallization of products from Zr-based alloys.

3. Regularities of rolling texture formation
Development of texture in polycrystalline zirconium and its alloys during deformation and heat treatment is described in detail in [2]. The same steps of rolling texture formation as in polycrystalline zirconium-based alloys are observed in rolled SCs. Repeatedly described formation in α-Zr of the final stable rolling texture (0001) ± (30-40 °) ND-TD <1010> (texture T2), where ND and TD - normal and transverse directions in the rolled plate (Fig. 1-d), is preceded by the texture T1, stable at intermediate deformation degrees (50-70%) (0001) ±15-20°ND-RD <112L> (Fig. of 1-c), characterized by the deviation of the basal axes by 15-20 ° from ND to RD. Variation of the initial SC orientation with respect to the RD is conditioned by mechanisms, underlying the formation of rolling texture at successive stages.
of its development. Incomplete PF \{11\overline{2}0\} with an angular radius of 80° for the investigated SC is shown in Fig. 1-a. The axis of the initial cylindrical SC, which coincides with the ND of a plate, cut of it, is in the center of the PF. The output of basal axis [0001] is shown in the same PF near the circumference of the stereographic projection with an angular radius of 80°. It is represented by circular contours and spaced at 90° from outputs of prismatic axes <11\overline{2}0>.

This investigation was performed for SC plates of three initial orientations, differing only in the RD. These orientations correspond to the following angular coordinates of basal axes: N 1 - (70°, 33°); N 2 - (70°, 152°); N 3 - (70°, 87°). Three columns of PFs in Fig. 1 demonstrate successive stages of rolling texture development in SCs of indicated orientations: N 1 – (a-d), N 2 – (e-h), N 3 – (i-l). First stages of all three successes in Fig. 1 correspond to deformation degree \(\varepsilon\approx5\%\) in order to create mutually differing distributions of SC fragments due to various positions of twinning systems relative to the RD.

4. Twinning as the main mechanism of deformation in SCs of \(\alpha\)-Zr
When the strain is only 5-7%, the texture maximum of PF (0001), representing the initial orientation of the SC, is divided into 5 – 9 new maxima, corresponding to reorientation of the initial SC by twinning in planes \(\{10\overline{1}2\}\), \(\{10\overline{1}1\}\) and \(\{11\overline{2}1\}\) (red, green and blue arrows in Fig. 1-b, e, i, respectively). These planes are identified by the characteristic angles of reorientation: 85, 57, 123 or 35°. At that, maxima, situated near ND, have the greatest intensity. Additional weak texture maxima, marked with figures 1-4 in Fig. 1-e, i, j, k, form due to twinning of the initial single crystal by planes \(\{10\overline{1}1\}\) (Fig. 1-e). Twinning by planes \(\{10\overline{1}1\}\)
previously did not consider by analysis of rolling texture formation in $\alpha$-Zr [2-6]. Described above variations of basal axis orientations under cold rolling of SCs N 1 and N 2 are quite consistent with the diagram of Hobson [6], indicating mechanisms of plastic deformation, which are most active in $\alpha$-Zr grains with different orientations of basal axis relative to ND, RD and TD. Twinning process continues as long as there are SC areas, having its initial orientation. At subsequent stages of deformation of SC N 2 it become possible to detect activation of twinning in planes $\{11\bar{2}1\}$. In Fig. 1-f, along with the initially formed texture maxima, new maxima appear. They are conditioned by secondary twinning on planes $\{11\bar{2}1\}$ in grains with orientation B (Fig. 1-f, g, blue arrows). Such twinning apparently also continues until there are grains with orientation corresponding to texture T1. Twinning is responsible for transition from texture T1 to texture T2, which is manifests in a shift of texture maxima from quasi-stable positions near the diameter of the stereographic projection ND-RD to its final stable positions at the diameter ND-TD. Such shift of texture maxima has an evident jump-like character along with slower flow of pole density in the same direction. This indicates, that along with pyramidal and basal slip, causing gradual flow of texture maxima, twinning by planes $\{11\bar{2}1\}$ is an active mechanism of $\alpha$-Zr deformation, which is not suppressed by lattice distortions in separate blocks or coherent domains of the rolled SC. Twinning in planes $\{10\bar{1}2\}$ and $\{11\bar{2}1\}$ operates also in SC N 3, where the basal axis is oriented along TD. The effect of twinning in plains $\{10\bar{1}2\}$ and $\{11\bar{2}1\}$ is seen especially at first stages of rolling. According to [6], in SC of this orientation prismatic slip systems work, but, due to equilibrium of these systems, the orientation proves to be stable and the basal axis does not deviate from TD (Fig. 1-i, j).

5. Recrystallization of rolled $\alpha$-Zr single crystals

Analysis of texture changes in rolled Zr single crystals by recrystallization was carried out in detail for SC N 1, deformed by 53%. PF (0001) of rolled SC is shown in Fig. 1-c, and PF (0001) after its recrystallization - in Fig. 2-a. For rolled single crystals X-ray method of generalized pole figures (GPF) was used. This method consists in measuring of X-ray line profiles in each point of texture PF, so that the true width at half-height of X-ray line profile (the line broadening) characterizes strain hardening of corresponding grains [5]. The line broadening of X-ray line (0002) is denoted as $\beta_{0002}$. It is used as a measure of grain distortion, increasing by strain hardening and removing by recrystallization. At that, separate fragments of rolled SC respond to a wide spectrum of substructure conditions, i.e. values of $\beta_{0002}$, distribution of which in the stereographic projection of sample is depicted by GPF $\beta_{0002}$. For the studied rolled SC GPF $\beta_{0002}$ is presented in Fig. 2-b.

Fig. 2-c represents the subtraction diagram $[PF(0001)_{\text{recr}}-PF(0001)_{\text{def}}]$, where differences of texture pole densities, calculated for each point ($\phi$, $\psi$) of stereographic projection were drawn; pole densities were measured for basal axes distributions both in recrystallized and rolled samples. The subtraction diagram is presented within the region, limited by a dotted line and corresponding to pole density values above 0.5 in the PF (0001) for the rolled sample. This subtraction diagram visually demonstrates the location of maximal changes in PF (0001) as a result of recrystallization. Gradations of the red color in the difference diagram $[PF(0001)_{\text{recr}}-PF(0001)_{\text{def}}]$ indicate its positive values and correspond to an increase in the pole density due to recrystallization, whereas the lack of color agrees with a decrease in the pole density upon recrystallization and characterizes grains, deformed with predominant participation of twinning.

Due to the use of rolled $\alpha$-Zr SCs, rolling textures, formed mainly by twinning, were obtained. Namely twinning is responsible for formation of the rolling texture with maxima, which, in spite of relatively high deformation degrees, take up transition positions between maxima of stable textures T1 and T2. The difference diagram shows, that by recrystallization of the deformed SC in PF regions, formed by twinning, the pole density does not grow (Fig. 2-c, e).
In grains, corresponding by their orientation of basal axes near normal direction (ND), only pyramidal slip can be activated under rolling, since values of shear stress for pyramidal slip in these grains are high. Other slip systems in these grains are suppressed because of low values of Schmidt factor. Therefore, in α-Zr grains, reoriented by predominant participation of twinning, strain hardening, i.e. distortion of crystalline lattice and fragmentation of substructure elements, is relatively small, so that in the course of subsequent annealing recrystallization nuclei do not form in these grains. As a result, they are swallowed up by grains, which are growing more actively and correspond by their orientations to regions of PF with increased strain hardening. At the same time difference diagrams, constructed for the rolled SC, show, that its texture maxima are bordered with zones of intensive recrystallization (Fig. 2-e). Namely in these regions of stereographic projection the increased distortion of grains is observed also (Fig. 2-b, d) due to above-shown simultaneous operation of pyramidal and basal slip. According to GPF $\beta_{0002}$, slopes of texture maxima respond to regions of the greatest fragmentation and distortion of crystalline structure [5]. Thus, in the considered case the main mechanism of α-Zr recrystallization consists in the growth of regions within the deformed matrix, characterized by increased energy of residual distortions.

6. Conclusions

1. The main plastic deformation mechanism in α-Zr SCs under rolling is twinning in planes $\{10\bar{1}2\}$, $\{10\bar{1}1\}$ and $\{11\bar{2}1\}$, but calculated diagrams, predicting active twinning systems by values of Shmidt factor, are correct not for all grain orientations, - in particular, within the region of prismatic slip the twinning by planes $\{10\bar{1}2\}$ and $\{11\bar{2}1\}$ operates.

2. Regions of α-Zr matrix, deformed by predominant participation of twinning, show a low strain hardening and a decreased tendency to recrystallization.

3. Recrystallization of α-Zr SCs, rolled up to $\varepsilon=50\%$, develops by growth of new grains at slopes of texture maxima, showing that slip participated in deformation along with twinning.

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