Texture analysis of zirconium samples deformed by uniaxial tension using neutron and X-ray diffraction

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Abstract. Seven zirconium samples were studied by neutron and X-ray diffraction after deformation on uniaxial tensile machine INSTRON 5882 from strain 5% to strain 30% (strain step was 5%). Preferred orientation parameters were determined by using pole figures and inverse pole figures. The X-ray measurements were performed at theta/theta X'Pert PRO diffractometer with Cr X-ray tube. Observed data were processed by software packages GSAS and X'Pert Texture. Our results can be summarized as follows: (i) Samples prefer orientation of planes (100) and (110) perpendicular to rolling direction. (ii) The position of the basal poles is tilted by 30° from the normal direction toward the transverse direction. (iii) Samples prefer orientation of planes (102) and (103) perpendicular to normal direction. (iv) Level of resulting texture increases with deformation. The obtained results are characteristic for zirconium.

1. Introduction

The textured material usually exhibits anisotropic mechanical, physical and chemical properties. Preferred orientation occurs in both powder samples (shape texture) and compact samples (caused by mechanical or heat treatment of material) [1]. Looking on diffraction methods to measure textures mainly three different radiations are in use. These are X-rays produced by conventional X-ray tube or synchrotron, electrons and thermal neutrons. Basic differences in analytical use of these radiations follow from their different interaction with matter [2].

This article deals with the use of neutron an X-ray diffraction in texture analysis of alpha zirconium.

Zirconium has very low absorption cross-section of thermal neutrons, high hardness, ductility and corrosion resistance. Therefore, zirconium and its alloys are used in the nuclear industry as fuel rod cladding, especially in water reactors.

In the alpha zirconium slip takes place usually on the $\{10\bar{1}0\}$ first order prism planes along the $\langle12\bar{1}0\rangle$ direction. Slip can occur also on the (0002) basal plane in the same direction. In regions of high stress concentration, the {10-11} slip is observed. Activation of slip with $\langle c \rangle$ component was noticed on first and second-order pyramidal planes $\{10\bar{1}1\}$ and $\{11\bar{2}1\}$ in $\langle c + a \rangle$ direction [3].

Most common twinning modes at room temperature are $\{10\bar{1}2\}\langle\bar{1}011\rangle$ tensile twins and $\{11\bar{2}2\}\langle\bar{1}\bar{1}23\rangle$ compressive twins [3].
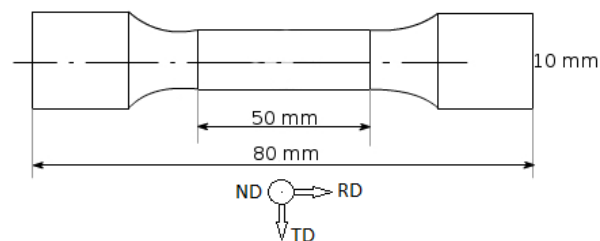
2. Experimental

2.1. Samples

The alpha zirconium plate was forged and hot rolled to the thickness 3.3 mm. Then it was annealed at 664 °C and the tensile specimens were made from the plate. Fig. 1 shows the shape, dimensions and sample coordinate system (ND means Normal Direction, RD is Rolling Direction and TD is Transverse Direction). Tab. 1 shows the amount of the admixtures in the samples.

**Table 1.** Chemical composition of investigated material.

Zr	Hf	Ca	Mg	O	H
[at%]	[at%]	[at%]	[at%]	[at%]	[at%]
99.39	0.41	0.044	0.096	0.055	0.0011

Figure 1. Shape and dimensions of zirconium samples.

2.2. Deformation experiments

The samples were deformed on uniaxial tensile machine INSTRON 5882 from strain 5% to strain 30% (strain step was 5%) at room temperature. The structure of the initial (non-deformed by uniaxial tension) sample observed by using light microscope Zeiss Axio Imager ZM1 is in Fig. 2. The grain size is about 20 μm.
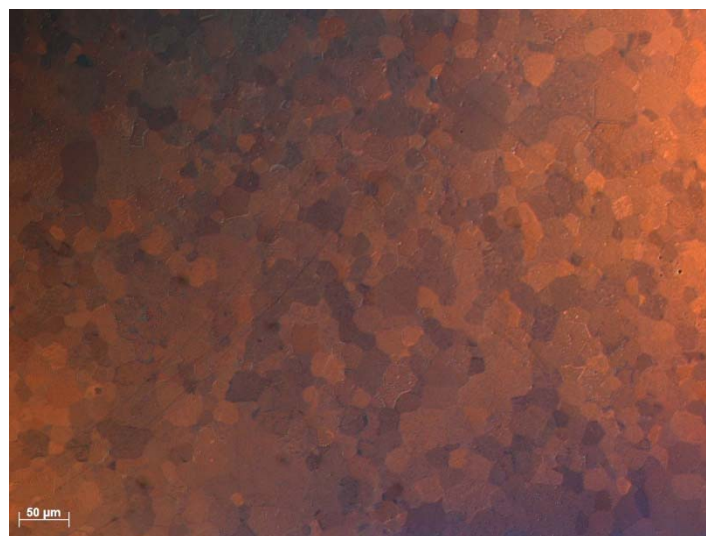
Figure 2. Structure of non-deformed sample.

2.3. Diffraction experiments

The neutronographic texture measurements were performed on the KSN-2 neutron diffractometer situated at the horizontal channel of the research reactor LVR-15 in the Nuclear Research Institute, plc. Rez, Czech Republic. The monochromatic neutrons having wavelength 0.1362 nm were used. The single-crystal Cu(200) was used as monochromator. The KSN-2 diffraction device offers good intensity and the best resolution value of Δd/d = 0.007 in the region d ~ 1.0÷0.1 nm (d is interplanar spacing). All the obtained neutronographic patterns were corrected for non-linear background and then evaluated using the Rietveld method implemented in the software package GSAS [Von Dreele, 2000]. The samples were mounted in reflection geometry with its normal parallel to the direction of interest - usually RD, TD, or ND - and the reflected intensities were measured depending on Bragg angle. The resulting peak intensities were normalized to the intensities of a standard sample with random texture by using Mueller formula [4]:

$$p_{hkl,q} = \frac{\frac{I_{hkl,q}}{I_{R,hkl}}}{\frac{1}{n}\sum_n \frac{I_{hkl,q}}{I_{R,hkl}}} \ ,$$

where $I_{hkl,q}$ is measured intensity of hkl reflection for directions q = TD, ND and RD, $I_{R,hkl}$ is intensity of hkl reflection for non-textured sample, n is number of reflections measured.

The X-ray texture measurements were performed at theta/theta X'Pert PRO diffractometer with Cr X-ray tube. Full pole figures for planes (10$\bar{1}$0), (0002), (10$\bar{1}$1) and (11$\bar{2}$0) were calculated from incomplete pole figures using orientation distribution function (ODF). Observed data were processed by software package X'Pert Texture, PANalytical.

3. Results

The intensity ratios $p_{hkl,q}$ calculated (for q=ND, RD and TD) by using Mueller formula from neutronographic data are in Tab. 2. Pole figures measured by X-ray diffraction for planes (10$\bar{1}$0), (0002), (10$\bar{1}$1) and (11$\bar{2}$0) for samples deformed at 5%, 15% and 30% are given in Fig. 3.

Table 2. Inverse pole figures calculated from neutronographic measurements of deformed zirconium samples.

	$p_{002,\,ND}$	$p_{002,\,TD}$	$p_{100,\,RD}$	$p_{101,TD}$	$p_{110,\,RD}$	$p_{102,\,RD}$	$p_{103,\,RD}$
0%	2.82	1.27	1.43	1.49	1.72	1.74	1.65
5%	2.67	1.73	1.51	1.10	1.45	1.58	1.41
10%	2.60	1.76	2.19	1.22	1.42	1.62	1.55
15%	2.77	2.01	2.49	1.01	1.45	1.36	1.34
20%	2.91	2.12	1.58	0.83	0.72	0.81	1.20
25%	2.85	2.27	2.34	1.31	0.83	0.34	1.52
30%	2.77	3.30	2.67	1.22	0.87	0.47	1.42
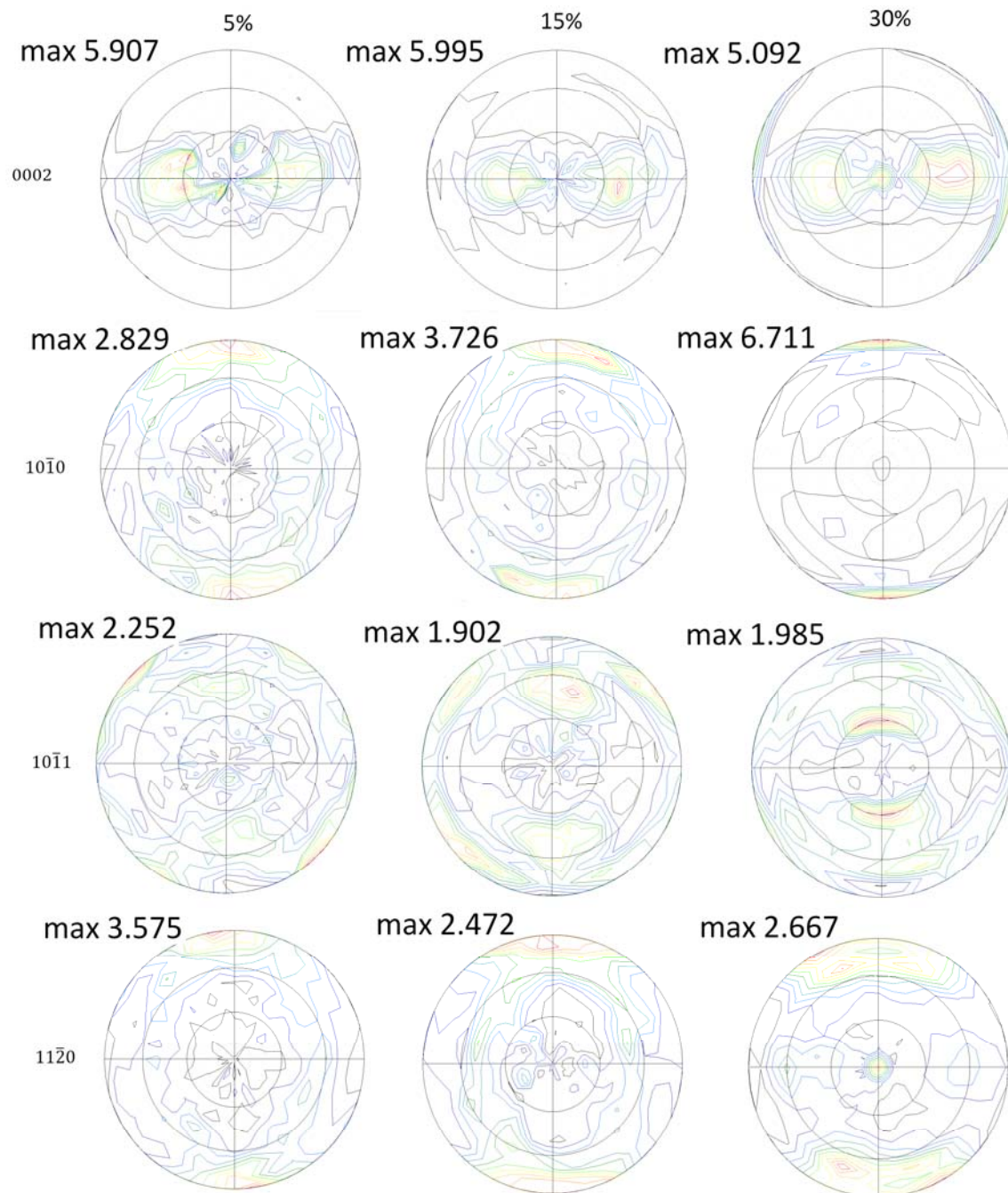
Figure 3. Pole figures measured by XRD for planes (10$\bar{1}$0), (0002), (10$\bar{1}$1) and (11$\bar{2}$0) for samples deformed at 5%, 15% and 30%.

4. Discussion

The position of basal poles is tilted from the normal direction toward the transverse direction by $\pm 20°$ for 5% deformation and by $\pm 45°$ for 30% deformation samples (Fig. 3). This rotation indicates twinning. Similar results can be seen from calculated intensity ratios in Tab. 2. The value of pole density $p_{002,ND}$ does not significantly change during the deformation. On the other hand, the value of pole density $p_{002,TD}$ shows a remarkable growth (from 1.27 to 3.30, see Tab. 2) during the deformation.

Planes (10$\bar{1}$0) are oriented perpendicularly to the rolling direction. An analogous situation is observed for planes (11$\bar{2}$0) - see Fig. 3 and Tab. 2. From (11$\bar{2}$0) pole figure (Fig. 3) obtained

for deformation 30% it is obvious that it shows maxima along the normal direction. Such texture component was not observed in the inverse pole figures.

Planes ($10\bar{1}2$) and ($10\bar{1}3$) are mostly oriented perpendicular to the normal direction.

5. Conclusions

From the results reported above it can be seen that samples prefer orientation of planes ($10\bar{1}0$) and ($11\bar{2}0$) perpendicular to the rolling direction.

The results show that, even at low deformations, twinning causes the basal poles orient from the normal direction toward the transverse direction. The split of basal poles toward the transverse direction can be explained by pyramidal slip with a ($c + a$) Burgers vector on $\{11\bar{2}1\}$ and $\{10\bar{1}1\}$ planes [5].

Furthermore, samples prefer orientation of planes ($10\bar{1}2$) and ($10\bar{1}3$) perpendicular to the normal direction. Texture become stronger with increasing the level of deformation (Tab. 2). The obtained results are characteristic for zirconium [5,6].

Acknowledgement

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