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## TEXTURE INHOMOGENEITY IN TITANIUM DEFORMED BY ECAP

### NIEJEDNORODNOŚĆ TEKSTURY W TYTANIE ODKSZTAŁCONYM METODĄ ECAP

Titanium of commercial purity was deformed by equal channel angular pressing (ECAP) with back-pressure at 380°C using 4 passes of route B<sub>C</sub>. Billets of 120 mm length were pressed at 0.4 mm s<sup>-1</sup> through square channels (14 mm × 14 mm) intersecting sharply at an angle of 90°. The global texture measurements of the starting and ECAP processed material were done by neutron diffraction. To investigate the texture inhomogeneity after ECAP the local texture was measured with high-energy synchrotron radiation at different positions in the cross-section of the billet. The texture gradient with regard to intensity and orientation of the dominant texture component is characterized and discussed.

*Keywords:* Titanium, Equal Channel Angular Pressing (ECAP), Microstructure, Texture, Inhomogeneity

Tytan o czystości przemysłowej (klasa 2) został odkształcony w procesie wyciskania w kanale kątowym (ECAP) z przeciwnością w temperaturze 380°C w 4 przepustach wg. drogi BC. Wlewki 120 mm długości były wyciskane przy prędkości 0.4 mm s<sup>-1</sup> przez prostokątny kanał kwadratowy (14 × 14 mm<sup>2</sup>). Pomiary tekstury globalnej w materiale wejściowym i po odkształceniu wykonano metodą dyfrakcji neutronów. Aby zbadać niejednorodność tekstury po odkształceniu zmierzono teksturę lokalną przy wykorzystaniu promieniowania synchrotronowego o wysokiej energii w różnych obszarach przekroju poprzecznego wlewka. Wskazane zostały zalety tej metody ujawnienia gradientu tekstury. Omówione zostały intensywności i odchylenia dominujących składowych tekstury w odniesieniu do ich idealnych położenia wynikających z prostego ścinania.

## 1. Introduction

Equal channel angular pressing (ECAP) is one of the most promising methods of severe plastic deformation (SPD) leading to bulk ultrafine to nanocrystalline materials. Compared to the conventional coarse-grained counterparts such materials possess a much higher strength simultaneously keeping sufficient ductility [1]. During SPD the materials develop a pronounced texture, which in ECAP due to predominantly simple shear in the intersection plane of the two channels (Fig. 1) is a shear texture. This texture may produce a strong anisotropy of certain properties, like mechanical and magnetic properties. Moreover, it has been shown that in ECAP processed materials there exists a texture gradient with respect to intensity and deviation from the ideal texture components from top to bottom of the billets [2, 3]. It is the aim of the present paper to study in detail the texture inhomogeneity of ECAP processed pure titanium. In

contrast to Ti alloys, strong and ductile pure Ti because of its excellent biocompatibility and corrosion resistance is of particular interest for biomedical and orthopaedic applications such as implants and miniaturized medical instruments.

## 2. Experimental

A billet (14 × 14 × 120 mm<sup>3</sup>) cut from a hot-rolled plate of commercially pure titanium (VT1-00, supplied by Verkhnyaya Salda Metallurgical Production Association, Russia, impurities in wt. %: Fe < 0.2, Si < 0.08, C < 0.05, O < 0.1, N < 0.04, H < 0.008) was deformed by ECAP using four passes of route B<sub>C</sub>, i.e. after each pass the billet was rotated first 90° clockwise about the transverse direction (TD) in order to insert the billet front first into the channel (along the normal direction (ND)) and then 90° clockwise about the rotated extrusion direc-

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tion (ED) axis. The intersection angle of the square channels was  $90^\circ$  without rounding of the corners. ECAP was performed isothermally at  $380^\circ\text{C}$  ( $T/T_m = 0.34$ ,  $T_m$  melting temperature) using a pressing speed of  $0.4\text{ mm s}^{-1}$ . In order to approach the simple shear condition along the intersection plane as good as possible the contact friction was minimized by lubrication with a mixture of flaked graphite,  $\text{MoS}_2$  and engine oil. Additionally, a back-pressure of  $240\text{ MPa}$  (0.2 of forward pressure) was applied [4]. The accumulated shear strain is about 8.

The microstructure was investigated in scanning electron microscopes (SEM, Zeiss DSM 962 and LEO 1530) with orientation imaging microscopy (OIM) based on electron backscatter diffraction (EBSD) using HKL software.

Global texture measurements of the starting and ECAP processed material were done by neutron diffraction [5]. The pole figures measured are (10-10), (10-11), (11-20) and (0002). To examine the homogeneity of ECAP deformation, the local texture was analyzed by high-energy synchrotron radiation (100 keV) using beam line BW5 at DESY-HASYLAB in Hamburg, Germany. The sample for texture measurements with synchrotron radiation was a pin of  $(2 \times 2 \times 14)\text{ mm}^3$  taken from the centre as well as 3 mm from each side wall of the billet with the long sample axis directed from the top to the bottom. The texture was measured at 7 positions along this direction with an aperture of  $(2 \times 0.5)\text{ mm}^2$  in volumes of about  $2\text{ mm}^3$ . Details about synchrotron texture measurements as well as texture representation are given in [2, 6]. The pole figures constructed from the Debye-Scherrer rings are the same as for neutron diffraction. These pole figures have been taken to calculate the orientation distribution function (ODF) using the program LABOTEX. The intensity and Euler angles of the main texture component have been determined from the ODF sections at  $\varphi_2 = 25^\circ$  or  $\varphi_2 = 30^\circ$ . The sample and crystal coordinate systems used (ED, TD, ND and 1, 2, 3, respectively) to describe the textures are shown in Fig. 5.

### 3. Results and discussion

The starting rolled material is partially recrystallized with a grain size smaller than about  $100\text{ }\mu\text{m}$  (Fig. 1a). The initial texture is  $(0001)\langle 10\text{-}10\rangle$  with a  $\pm 40^\circ$  rotation about the rolling direction (Fig. 2a). This texture is generally observed for hot-rolled Ti [7]. After 4 passes of ECAP route  $B_C$  the material has a grain size smaller than  $1\text{ }\mu\text{m}$ . The largest grains which could be measured by EBSD probably are dynamically recrystallized (Fig. 1b). The global texture may be characterized by a predominant elliptical component located around

$\varphi_1 = 225^\circ$ ,  $\phi = 45^\circ$  and  $\varphi_2 = 30^\circ$  with  $\Delta\varphi_1 = 20^\circ$ ,  $\Delta\phi = 40^\circ$  and  $\Delta\varphi_2 = 20^\circ$  (Fig. 2b). Its volume fraction is about 20%. The texture sample symmetry is triclinic. Local texture measurements by synchrotron radiation reveal that within the cross-section of the billet there is a texture gradient with respect to intensity and orientation of the main component. On the left and right side of the billet the intensity slightly decreases from the top to the bottom except in the lower 20% where it increases again (Fig. 3). The texture gradient with respect to  $\varphi_1$  and  $\phi$  is about  $-1^\circ/\text{mm}$  and  $1^\circ/\text{mm}$ , respectively (Fig. 4). A local variation of  $\varphi_2$  is within  $5^\circ$ . The intensity also decreases from the left to the right side. The angular spread from the left to the right side at a given position is smallest in  $\varphi_1$  when it is largest in  $\phi$  and vice versa. The texture type observed compares quite well with that measured by Yu et al. [8] after the same ECAP process. The texture inhomogeneity of a Ti sample billet processed 8 passes ECAP route  $B_C$  through a round die has been investigated by Bonarski and Alexandrov [9]. They observed a decrease of the maximum ODF intensity from the centre to the edge of the billet. However, a direct comparison of the texture type is not possible.

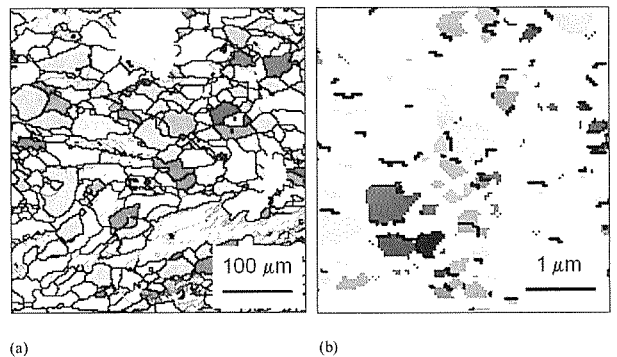


Fig. 1. Grain structure reconstructed from OIM. Thin and thick lines represent low and high angle boundaries with misorientation angles between  $3^\circ$  and  $15^\circ$  and  $> 15^\circ$ , respectively. (a) initial microstructure, (b) microstructure after 4 passes ECAP route  $B_C$ . White areas in (b) could not be indexed. (Horizontal axis = transverse direction (TD), vertical axis = normal direction (ND))

The texture of route A ECAP processed face-centred cubic metals is quite inhomogeneous from the top to the bottom of the billet [2, 3]. With increasing number of passes it becomes more homogeneous. The non-uniform texture in ECAP processed Ti is in line with these observations. However, in route  $B_C$  ECAP processed Ti a texture gradient also exists from the left to the right side of the billet. The texture heterogeneity may be explained by Tóth's flow line model [10] yielding an increasing flow line coefficient from the top to the bottom of the

billet and here, due to a  $90^\circ$  rotation about ED, also from the left to the right side.

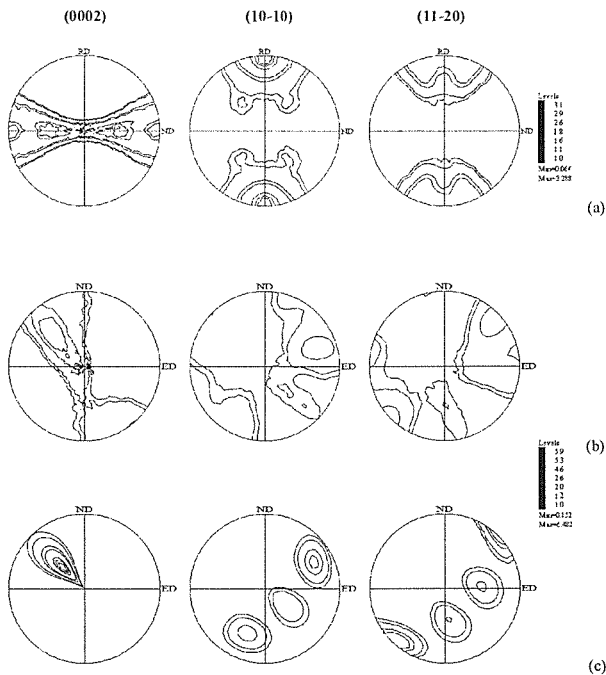


Fig. 2. (a) Initial texture of hot-rolled plate (ND = normal direction of rolling plane, RD = rolling direction). (b) Experimental texture and (c) main texture component after 4 passes ECAP route B<sub>C</sub> (ND = normal of ECAP channel, ED = extrusion direction)

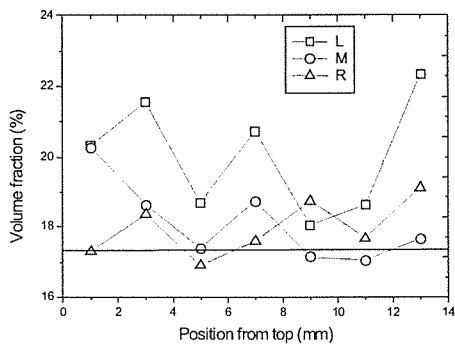


Fig. 3. Volume fraction of the main texture component as a function of position from the top to the bottom of the billet measured at the left (L), middle (M) and right (R) side. Dotted line gives the global intensity measured with neutrons

The texture observed compares well with the stable orientations found in simulations on simple shear deformation of hexagonal metals [11, 12] (Fig. 5). In the present case a P fibre texture is observed with a strong elliptical component at  $\varphi_1 = 225^\circ$ ,  $\phi = 45^\circ$  and  $\varphi_2 = 30^\circ$ . The P fibre is due to prismatic and pyramidal slip [11], indicating that these slip modes predominate the texture development in Ti. Surprisingly, there exists some similarity of the main texture component found with that observed in magnesium deformed in the same way, although this material primarily deforms on the basal system. However, in contrast, in magnesium

the component has a pronounced spherical shape and is more rotated towards the shear plane normal. An explanation of this behaviour may be given by the texture simulations planned.

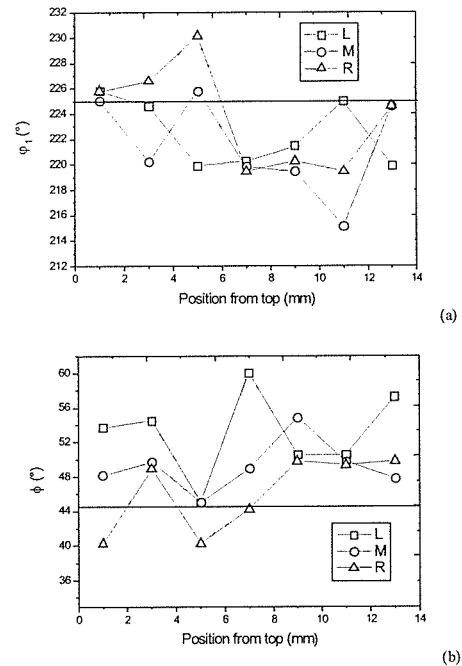


Fig. 4. Euler angles of the main texture component as a function of position from the top to the bottom of the billet measured at the left (L), middle (M) and right (R) side. Dotted lines give the average angles measured with neutrons

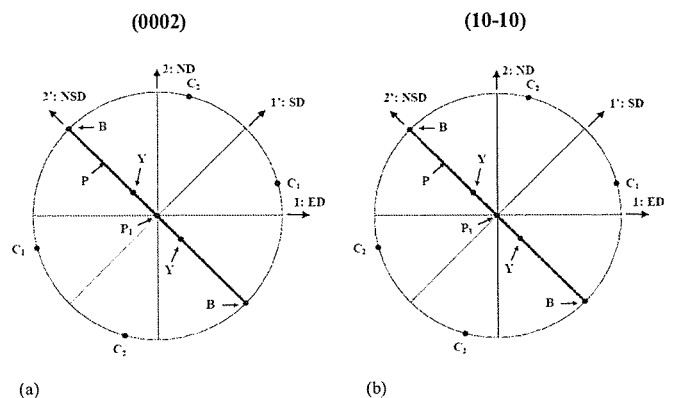


Fig. 5. Main stable orientations in hexagonal metals displayed as fat lines and dots in the (0002) (a) and (10-10) (b) pole figure with respect to the ECAP (ED, TD, ND) and shear (SD; TD; NSD) sample reference system. The crystal coordinate system is 1 = [10-10], 2 = [11-20] and 3 = [0001]. (Ideal fibres in  $\varphi_1$ ,  $\phi$ ,  $\varphi_2$  are B:  $45^\circ$ ,  $90^\circ$ ,  $0^\circ-60^\circ$ ; P:  $45^\circ$ ,  $0^\circ-90^\circ$ ,  $30^\circ$ ; Y:  $45^\circ$ ,  $30^\circ$ ,  $0^\circ-60^\circ$ ; C<sub>1</sub>:  $105^\circ$ ,  $90^\circ$ ,  $0^\circ-60^\circ$ ; C<sub>2</sub>:  $165^\circ$ ,  $90^\circ$ ,  $0^\circ-60^\circ$ ), taken from [12])

#### 4. Conclusions

- 1) Route B<sub>C</sub> ECAP processed Ti develops a P type fibre texture with a strong elliptical component.

- 2) There exists a texture gradient not only from the top to the bottom of the billet but also in the transverse direction.

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