Microstructure of titanium deformed by warm extrusion with forward-backward rotating die

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Abstract. The principal KoBo device is a press with a forward-backward rotating die, enabling the extrusion of ingots under conditions of constant destabilization of their substructure. Polycrystalline grade 2 titanium was subjected to warm KoBo type extrusion. Microstructure of the material was investigated by means of Electron Backscatter Diffraction (EBSD) in the scanning electron microscope. It clearly shows deformation-induced grain fragmentation. The EBSD maps reveal heterogeneous microstructure built of ribbons curled about the extrusion direction (ED) and some equiaxed or cigar-like grains. Sizes of grains vary in the range 70 – 1500 nm for the minor axis and 350 – 20000 nm for the major axis. The material has a relatively sharp nearly axial texture with the <0001> axis perpendicular to ED. In misorientation angle distribution, besides the peak at low angle boundaries, there are three other peaks at about: 29.7deg, 89.7deg and 93.2deg. They do not correspond to any twin boundaries or low Σ coincidence site lattice misorientations.

1. Introduction
It is well known that mechanical properties of metallic materials can be considerably improved by refinement of microstructure, e.g. [1]. This concept has contributed to rapid progress in development of various methods of severe plastic deformation (SPD) by which ultrafine-grained materials are produced, e.g. [2, 3]. Application of well-established SPD techniques such as high-pressure torsion, e.g. [4], equal channel angular extrusion, e.g. [5], or hydrostatic extrusion (HE), e.g. [6], results in the formation of submicrorcrystalline structures. These methods have almost unlimited ability to accumulate deformation while maintaining the initial shape and size of the specimen (with exception for HE where sample crosssection is reduced). However, multi-pass deformation is costly and laborious. Therefore, technological processes capable of producing a high level of straining over a single deformation cycle, and thus reducing costs of production, are very attractive. Besides the relatively popular HE technique, processing in a single deformation step is characteristic for the less known KoBo method, e.g. [7, 8].

The KoBo device is a press with a forward-backward rotating die, enabling extrusion of ingots under conditions of constant destabilization of their microstructure. The technique allows for high-rate deformation, which favors the accumulation of lattice defects and thereby facilitates the microstructure refinement. The method involves a cyclic change of deformation path which increases the plasticity of the material and inhibits the formation and propagation of cracks. The paper describes some results of an ongoing project in which the KoBo method is used to modify the microstructure of commercially pure titanium.

2. Experimental procedures
Investigated material was grade 2 titanium received as a hot-rolled bar (ø 35 mm) with homogeneous, globular microstructure and a mean grain diameter of ~30 µm. Yield stress and tensile strength of initial material were 123 and 473 MPa, respectively. The ingot was heated before extrusion to 450 °C, but KoBo process was carried out at the temperature of about 400
C. The bar was extruded to a rod with the diameter of 8 mm at the rate of 0.5 mm/s. The extrusion die oscillated by the angle of ±6° with the frequency of 5 Hz. Microstructure of the extruded sample was investigated in the longitudinal and transverse cross-sections by means of Electron Backscatter Diffraction (EBSD) using scanning electron microscopy (SEM) and also using transmission electron microscopy (TEM).

3. Results
Mechanical properties of the material processed under above described conditions were significantly improved; the yield stress and tensile strength increased to 560 and 672 MPa respectively. SEM and TEM examination revealed a heterogeneous microstructure consisting of grains shaped like ribbons and curled about the extrusion direction (ED). Grains strongly elongated along ED, were separated by well-developed high-angle grain boundaries (HAGBs). Most of HAGBs were parallel to ED, Fig. 1 a, b. Besides the curly ribbons, there were also areas with equiaxed or cigar-like grains with major axes parallel to ED. Generally, sizes of grains varied in the range 70 – 1500 nm (average value ~200 nm) for the minor axis and from 350 nm to 20 µm (average value ~640 nm) for the major axis. The mean grain area was ~1 µm², Fig. 1e.

Figure 1. Microstructure of grade-2 titanium after Kobo extrusion. Example EBSD maps in transverse (a) and longitudinal (b) cross-sections; the color corresponds to ED in the inverse
pole figure shown in (c). Example bright-field TEM image from the transverse cross-section (d). Grain area distribution for the transverse cross-section (e).

TEM micrographs showed heterogeneous distribution of dislocations with some areas of relatively high dislocation density and other containing only isolated dislocations. In some grains, dislocation walls or low angle grain boundaries (LAGBs) formed a sub-grain structure, and the curly ribbons were often composed of sub-grain chains with LAGBs roughly perpendicular to HAGBs, e.g. Fig. 1 d.

The orientation distribution function (ODF) calculated from transverse cross-section revealed a relatively sharp nearly axial texture with the <0001> axis perpendicular to ED, Fig. 2. Moreover, within the axial texture, the ED was predominantly parallel to <1 -1 0 0> direction. Also the texture function obtained from the longitudinal cross-section had a sharp axial component with the <0001> direction perpendicular to ED. This time, however, some deviation from perfect axial symmetry was observed. The ED position was more scattered between <1 -1 0 0> and <2 -1 -1 0> directions; this is also visible in Fig. 1b. Moreover, some asymmetry between +ED and –ED was observed. These effects were likely caused by a deviation of the section from the center of the specimen. This indicates an inhomogeneity of the deformation process along the radius of the rod.

The misorientation angle distributions obtained from the transverse and longitudinal sections were similar. Both showed a very large number of orientation fluctuations characterized by small misorientation angles (e.g. Fig. 3), and a significant part of these fluctuations corresponded to LAGBs. Besides the peak at LAGBs, there were three other peaks at about 29.7°, 89.7° and 93.2°. (The 89.7° peak in the longitudinal section was much weaker than that in the transverse section.) The peaks did not correspond to any twin boundaries or low Σ coincidence site lattice misorientations. The comparison of correlated and uncorrelated misorientation angle distributions demonstrates that, besides the above-mentioned peaks, the character of the misorientation angle distribution in the range 15 – 90°
is mainly determined by the texture of the material, and the probability of occurrence of any misorientation angle between grains is similar.

Misorientation distribution functions (MDFs) from both sections were also similar. Besides a sharp maximum at zero misorientation, there was an intensive misorientation angle fiber along $<2\ -1\ -1\ 0>$ axis and a peak at the “corner” corresponding to the largest possible misorientation - $93.8^\circ$ about $<50\ -13\ -37\ 13>$, Fig. 4. The fractions of misorientations corresponding to twin boundaries were negligible.

Figure 3. Misorientation angle distributions obtained from the transverse cross-section. (Figure based on pixel-to-pixel misorientations with angles exceeding $5^\circ$).

Figure 4. Correlated MDF from the transverse cross-section. Rodrigues’ representation $R_1\ R_2\ R_3$, cross-section $R_3$=const, asymmetric domain (D$_6$, D$_6$).
4. Discussion

Besides titanium, the only hexagonal materials examined after KoBo processing were zinc and AZ31 magnesium alloy [9]. Their microstructures are composed of grains slightly elongated in ED and they differ significantly from that of the KoBo processed titanium.

Curly microstructure of titanium is similar to those observed in heavily drawn wires of bcc metals, e.g. [10]. An explanation of development of this microstructure was given by Hosford in [11, 12]. He showed that with \( <110>_{\text{bcc}} \) axial texture, there is a tendency of individual grains to deform in the plane-strain regime. That would lead to elliptical shapes of grains (in the transverse cross-section) incongruent to the axial symmetry of the wire. To maintain compatibility, neighboring grains have to bend around one another, and this consequently leads to curled grain shapes. The same mechanism seems to be applicable to the considered case. In KoBo deformed titanium, the \( <110>_{\text{bcc}} \) axial texture is replaced by \( <1 -1 0 0> \) and slip systems are different. A change in length along the \( c \) axis cannot be achieved by the \( <2 1-1 0> \) slip on \( \{0001\}, \{1 0 -1 0\} \) and \( \{1 0 -1 1\} \) planes [13]. Thus, analogously to the case considered by Hosford [11], with the \( c \) axis perpendicular to ED, these slips would lead to elliptical grains, so the grains have to curl. This requires a slip direction lying out of these planes. Such a slip, along \( <1 1 -2 3> \), has been reported in commercially pure titanium [14].

In principle, the compression along the \( c \) axis can also be accommodated by twinning. However, twinning becomes less important as temperature increases. At the temperatures corresponding to that of the KoBo extrusion, twinning on \( \{1 0 -1 1\} \) planes was observed [13], but that was in a Ti single crystal compressed along the \( c \) axis. Moreover, twinning is also suppressed by high dislocation density. It is likely for these reasons (elevated temperature plus relatively high dislocation density) that no twins were observed after deformation by KoBo.

Transformations of a globular microstructure in FCC and BCC materials are associated with the creation of the so-called deformation-induced boundaries (DIBs) [15]. DIBs create a lamellar microstructure, in which geometrically necessary boundaries (GNBs) separate regions deformed by different slip combinations. This happens only when dislocation density becomes high enough to suppress mechanical twinning [16]. A similar mechanism may be responsible for distinct HAGB peaks clearly visible in the MDF of titanium after KoBo.

Unlike the microstructures, the textures of all KoBo extruded hexagonal materials were very similar. The textures measured in zinc and AZ31 magnesium alloy along the line of the plastic flow show that during the KoBo extrusion the \( \{0001\} \) planes remain constantly tangential to that line independently of the state of material and its initial texture [9]. As a result, in all these materials, the sharp nearly axial texture with the \( <0001> \) axis perpendicular to ED is formed.

Gusak et al [17] claim that the material behaves like a liquid changing the flow direction without changes the local texture. They have proposed a model describing this process. The model assumes presence of zones of reduced viscosity with highly increased concentration of points defects. These zones make the high extrusion rate possible. The evolution of the texture along the line of the plastic flow and some specific features of the microstructure (like the curly ribbons in titanium, or extraordinarily thick areas of high defect concentration adjacent to HAGBs in zinc [18]) seem to support the hypothesis that the metal becomes superplastic in narrow layer near the rotating die, and it is squeezed out like viscous liquid [19].
5. Conclusions
The KoBo extrusion enables plastic forming of titanium in a single operation at high strain. The obtained material has a relatively strong <1 -1 0 0> fiber texture. The microstructure is built mainly of ribbons curled about the ED and equiaxed or cigar-like grains. With such microstructure, titanium has improved mechanical properties. There are sharp peaks in the misorientation (angle) distribution but they do not correspond to twins. A full explanation of the mechanism of the microstructure formation in the KoBo process will require further studies with better spatial and angular resolutions.

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