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Microstructure and texture of zinc deformed by extrusion with forward-backward rotating die (KoBo)

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Abstract. The KoBo device is a press with a cyclically rotating die enabling extrusion of ingots with the permanent destabilization of their substructure. The method involves a cyclic change of deformation path that increases the plasticity of the material and inhibits formation and propagation of cracks. The possibility of using KoBo for obtaining zinc wires with high mechanical properties was explored. A polycrystalline zinc ingot of purity of 99.995% was subjected to extrusion at room temperature. The microstructure of the material was investigated primarily via high-resolution electron backscatter diffraction using a scanning electron microscope. The microstructure is heterogeneous and consists of grains elongated slightly in the extrusion direction (ED). The material has a relatively sharp nearly axial texture with the ED scattered between the <1 0 -1 0> and <2 -1 -1 0> directions. More than 95% of high-angle grain boundaries do not correspond to any twin or low Σ CSL boundaries. The dimensions of the grains range from 10 - 20 micrometers down to the sub-micron scale. Despite the relatively large grains, the final product exhibits very good mechanical properties, which could not be explained by the Hall - Petch relation. It was found that besides grain refinement two other effects could affect the material mechanical properties. These effects are formation of nano-grains inside the large, micrometer-sized grains and formation of broad areas of high-density crystal lattice defects extended along the HAGBs.

1. Introduction

A particularly effective way to strengthen metallic materials proved to be grain refinement up to nanometric sizes, which can be realized by various methods of severe plastic deformation (SPD), e.g., [1-3]. The goal of the classical SPD methods, such as Equal Chanel Angular Pressing, Accumulative Roll Bonding, or High Pressure Torsion, is manufacturing of a material with "only" strong modification of its microstructure. In addition to the classical SPD methods, methods were developed that also change the product shape. One of these methods is the KoBo extrusion, [4, 5]. The KoBo device is a press with a forward-backward rotating die, enabling extrusion of ingots under conditions of permanent destabilization of their microstructure. The technique allows for a high-rate deformation, which favors the accumulation of lattice defects and thereby facilitates the microstructure refinement.

Polycrystalline zinc exhibits very poor mechanical properties; for example, with the grain size of approximately 40 μm, the yield strength $R_{0,2}$ reaches approximately 40 MPa and the tensile strength Rm is approximately 110 MPa. SPD methods enable a significant improvement of these properties, with R0,2 and Rm able to reach ~ 160 MPa and ~ 170 MPa, respectively. This enhancement is obtained by grain refinement to the nanometer scale, practically in the absence of dislocation and with a negligible concentration of point defects, e.g. [6, 7]. Using the KoBo device, one can obtain material with high mechanical properties [8, 9], similar to those after SPD, wherein the average grain size does not fall below of 10 - 20 μm. Thus, the grain refinement interpretation fails. This result indicates that during the

KoBo deformation of zinc, besides grain refinement, other effects that modify the microstructure strengthen the material. In this work, we seek to identify these effects.

2. Experimental procedures

The investigated material was pure zinc (99,995%) received as ingots with a diameter of 40 mm and a length of 50 mm and a typical for casting products microstructure of long columnar grains. The KoBo process was performed at room temperature. The bars were extruded to wires using different sets of KoBo process parameters. Samples for microstructure investigations were prepared from wires extruded with extrusion ratios of λ=100, λ=400 and λ=490 from longitudinal and transverse cross sections.
Microstructures were investigated using scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

3. Results and discussion

The microstructure of zinc after the KoBo process was heterogeneous, with the degree of heterogeneity being dependent on selected process parameters. Nevertheless we could specify certain characteristics of the microstructure that were common for all samples, at least qualitatively. Each of the investigated microstructures was composed of slightly elongated ED grains of a varying fragmentation degree, Fig. 1. The size of grains varied from a few hundred nanometers to several tens of micrometers. The highest refinement of grains was presented in the outer, most distorted, layers of the rod. In such microstructure, there were two effects that, although occurring with varying intensity, could strengthen the material.
Fig. 1 shows the chains of recrystallized fine grains extending through the entire cross section of the extruded rod. These chains are probably effects of recrystallization in shear bands produced by extrusion with forward-backward rotating die. The change in orientation topography between the transverse cross-section taken from the region close to the band and the longitudinal cross-section exhibits characteristic differences in the orientation of the grains in the bands and in the matrix. The matrix orientation distribution function (ODF) revealed a relatively sharp nearly axial texture with the <0001> axis perpendicular to the ED which was scattered between the <1 0 -1 0> and <2 -1 -1 0> directions., as shown in Fig. 2a.

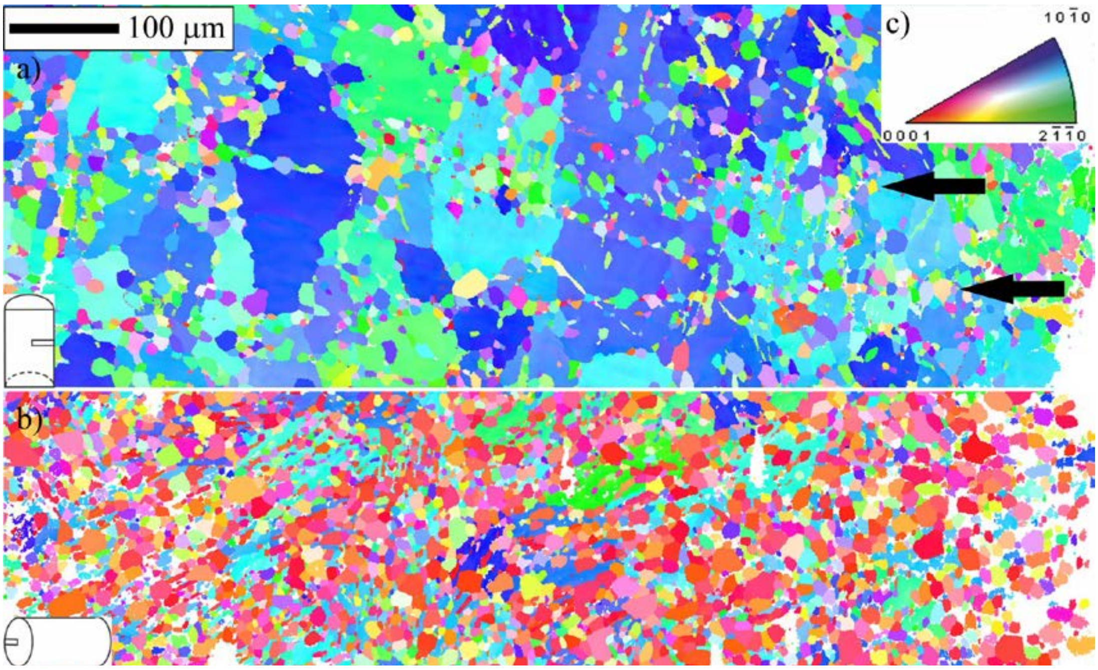

Fig. 1. Microstructure of zinc after KoBo extrusion (λ =400). Example EBSD maps in the longitudinal (a) and the transverse (b) cross-sections, with examples of recrystallized shear bands areas marked; the colors correspond to the ED in the inverse pole figure (c).

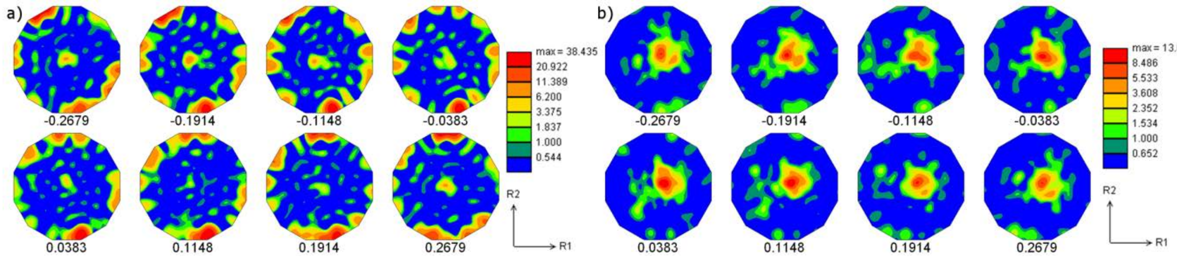

Fig. 2. ODFs of the matrix grains (a) and the grains in the bands (b); calculated from single orientations measured at transverse cross-section (fig.1. b) . Rodrigues' representation R_1 R_2 R_3, with cross-section R_3 = constant; asymmetric domain of D_6 symmetry.

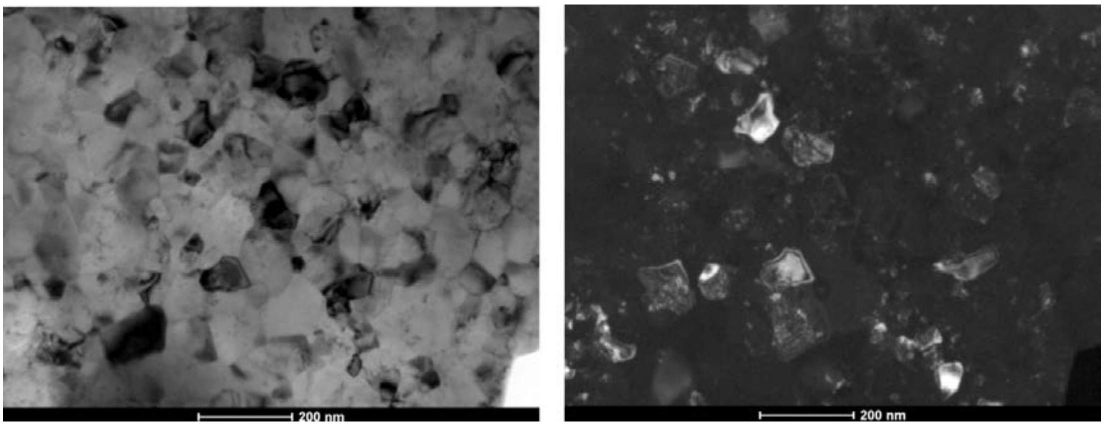

Fig. 3. An example of the zinc microstructure after KoBo deformation (λ =100), transverse cross-section; bright- and dark-field micrograph, respectively, TEM.

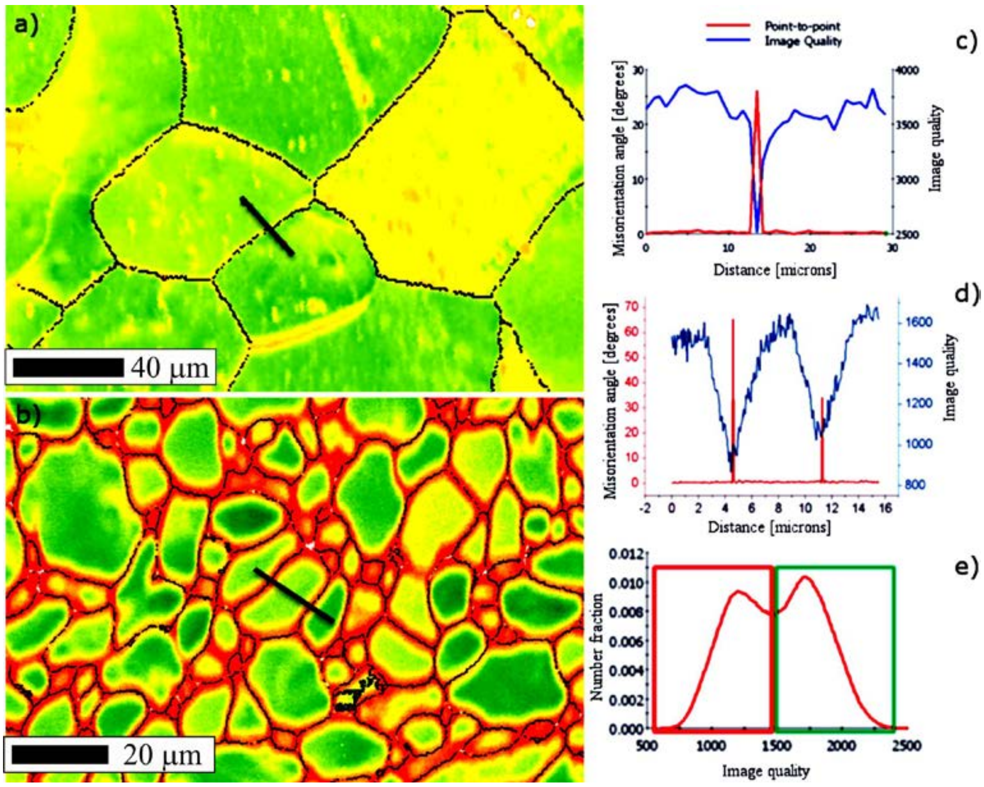

Fig. 4. Exemplary IQ maps (a and b) in conventionally extruded zinc and zinc after KoBo extrusion (λ =490); black - HAGBs, red/yellow - the areas of highly defected crystal lattice, green – areas of low lattice defect density. c) and d) misorientation angle (point to point) and IQ changes along the line segments shown in (a) and (b); number fraction with low values of

IQ – high density of lattice defects and number fraction with high values of IQ – low density of lattice defects.

Quite different situation was found in the recrystallized shear bands, where the ED was predominantly parallel to the <0001> direction, Fig. 2b. Immediately after the extrusion process is completed, nano-grains, an example of which is shown in Fig. 3 were found in microstructure. Those nano-grains are suspected to nucleate and recrystallize over time in the vicinity of shear bands and form chains of micrometer size grains observed in EBSD measurements (Fig. 1a).
The second microstructural effect that results in the strengthening of zinc after KoBo extrusion could be the formation of the unusually thick areas of the deformed crystal lattice extended mainly along the HAGBs, Fig. 4b [9]. Detection of these areas was enabled by the use of image quality (IQ) maps. The IQ of the EBSD pattern is strongly affected by the local concentration of the lattice defects. The areas with a high density of lattice defects occupied up to 30% of the volume of material within the sample and up to 60% in the surface layer.

4. Conclusions
The improvement of the mechanical characteristics in zinc after KoBo extrusion cannot be explained only on the basis of grain refinement because an average grain size does not fall below a value in the range of 10 to 20 μm, and the mechanical properties correspond to those obtained for nanocrystalline materials.
During the KoBo deformation process, besides the grain refinement, which is not as intense as that in the SPD methods, two other effects are generated:

- The first effect is the formation of nano-grains within of large, matrix grains. The nano-grains were generated during the shearing of the material due to the extrusion with forward-backward rotating die.
- The second effect is the formation of the unusually broad areas of higher density of crystal lattice defects along HAGBs.

Both of these effects require further study via High-Resolution Transmission Electron Microscopy.

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