Microstructure evolution of pure titanium during hydrostatic extrusion

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Abstract

Regarding severely deformed materials of potentially high applicability in various industry branches, their microstructure evolution during processing is of vast significance as it enables to control or adjust the most essential properties, including mechanical strength or corrosion resistance. Within the present study, the microstructure development of commercially pure titanium (grade 2) in the multi-stage process of hydrostatic extrusion has been studied with the use of the well-established techniques, involving electron backscatter diffraction as well as transmission electron microscopy. Microstructural deformation-induced defects, including grain boundaries, dislocations, and twins, have been meticulously analyzed. In addition, a special emphasis has been placed on grain size, grain boundary character as well as misorientation gradients inside deformed grains. The main aim was to highlight the microstructural alterations triggered by hydroextrusion and single out their possible sources. The crystallographic texture was also studied. It has been concluded that hydrostatically extruded titanium is an exceptionally inhomogeneous material in terms of its microstructure as evidenced by discrepancies in grain size and shape, a great deal of dislocation-type features observed at every single stage of processing and the magnitude of deformation energy stored. Twinning, accompanied by grain subdivision phenomenon, was governing the microstructural development at low strains; whereas, the process of continuous dynamic recrystallization came to the fore at higher strains. Selected mechanical properties resulting from the studied material microstructure are also presented and discussed.

Keywords  Hydrostatic extrusion · Severe plastic deformation · Titanium · Microstructure · Electron backscatter diffraction · Transmission electron microscopy · Mechanical tests

Abbreviations

ASD  As delivered
BF  Bright field
CS  Cross section
DRX  Dynamic recrystallization
EBSD  Electron backscatter diffraction
ECAP  Equal channel angular pressing
ED  Extrusion direction

FG  Fine-grained
GAM  Grain average misorientation
GOS  Grain orientation spread
HAGB  High-angle grain boundary
HE  Hydrostatic extrusion
IPF  Inverse pole figure
KAM  Kernel average misorientation
LAGB  Low-angle grain boundary
LCS  Longitudinal cross section
OIM  Orientation imaging microscopy
PF  Pole figure
SAED  Selected area electron diffraction
SPD  Severe plastic deformation
TCS  Transverse cross section
TEM  Transmission electron microscopy
UFG  Ultrafine-grained
UTS  Ultimate tensile strength
YS  Yield strength
1 Introduction

It is well known that grain refinement is an effective approach for strengthening of polycrystalline materials [1]. Apart from mechanical behavior, nanostructured, ultrafine-grained (UFG) as well as fine-grained (FG) metals and alloys have proven to demonstrate excellent functional and biological properties [2]. The improvement in these characteristics, in comparison with coarse-grained counterparts, originates in large part from a substantial share of multi-level microstructure defects, e.g., fine grains, high-angle grain boundaries (HAGBs), low-angle grain boundaries (LAGBs) or high-density dislocations, generated while heavy strains are exerted on bulk samples [3]. Over the decades, severe plastic deformation (SPD) methods have dominated the research of plastic working processes as they enable for a significant refinement of ordinary, coarse-grained microstructures and, as a result, fully dense, porosity- and contamination-free materials with high mechanical properties are fabricated. Up till now, equal channel angular pressing (ECAP), high-pressure torsion (HPT), accumulative roll-bonding (ARB), and multidirectional forging (MDF) have been established as the most promising SPD methods with respect to potential applicability [4]. On the other hand, a great deal of the unconventional SPD techniques, including hydrostatic extrusion (HE) technique, also known as hydroextrusion, have been greatly optimized so the strength of various materials could be increased [5].

The HE technique has been especially useful in producing long-length rods, wires, and tubes of various structural and functional materials as well as biomaterials, composites and polymers [6]. However, the manufactured high-strength elements are known to be characterized by a distinctive anisotropy of microstructure and strong texture [7]. In addition, owing to restraints in geometry, hydrostatically extruded materials might be applied to a limited number of applications, including small-sized medical implants or electrical wires [2]. Even though the process of HE is not a novel manufacturing technique, the reports concerning microstructural evolution of cold-worked, hydrostatically extruded pure metals are scarce. Lewandowska et al. [8] investigated the impact of HE on aluminum and stated that due to its stacking fault energy, high ability to recover as well as that of cross-slip, microstructure evolution proceeds through a gradual increase of grain boundary misorientation without a significant drop in grain size. Lee et al. [9] inspected the changes in microstructure of pure copper as a function of extrusion ratios. They claimed discontinuous dynamic recrystallization to be a major mechanism responsible for the heterogeneous microstructures as the observed grain growth had its origin in recrystallized, dislocation-free grains that consumed their work-hardened surroundings. In another study [10], the same authors applied the HE method to pure niobium and praised the role of interactions among dislocation-type defects in the overall evolution of microstructure.

So far, in terms of pure titanium, its microstructural changes during ECAP have been the most often reported. It was demonstrated that the rebuilt, substantially refined microstructure originated from the LAGB-to-HAGB transformation in the process associated with continuous dynamic recrystallization (cDRX) [11]. However, the deformed microstructures were also greatly affected by twinning [12]. In the case of undergoing the ARB deformation, a lamellar microstructure, filled with equiaxed grains, was formed in titanium. Twin boundaries, generated at lower reduction, gradually evolved into HAGBs, surrounding small-sized grains within shear bands. Twinning was proven to have a minor effect on the final ultrafine microstructure, whereas the processes of recovery, micro shear banding at regions of lamellar boundaries as well as macroscopic shear banding were believed to dominate during ARB [13]. When it comes down to the HPT-treated titanium, its highly disordered microstructure was proven to be the resultant of dislocations-involved processes since an increase in density of dislocations tended to be balanced by dislocations adsorption by HAGBs. At low shear stresses, the deformation of titanium during HPT was governed by twinning and slip, while with an increase in strain, it became exclusively slip-dominated [14]. With respect to the MDF-ed titanium, twinning was enlisted as the main deformation mechanism taking place during the very first stage of processing. A steady transformation of LAGBs into HAGBs was, obviously, identified as well. In general, dynamic recovery and cDRX or dynamic recrystallization (DRX) were thought to be the most probable grain refinement mechanisms during MDF [15].

Interestingly, the HE technique proved itself very efficient in improving the mechanical characteristics of pure titanium which, produced by the multi-stage HE process combined with rotary swaging, outperforms the very popular Ti–6Al–4 V alloy in terms of mechanical characteristics, as discussed by the authors in [6]. The finding is especially promising in the light of potential biomedical purposes due to elimination of the possibly harmful influence of the alloying additions. Moreover, in comparison to its coarse-grained, unprocessed counterpart, the HE-produced titanium offers superior corrosion resistance and biocompatibility as well as slightly lower elastic modulus, a property of critical importance with respect to eliminating the stress-shielding phenomenon in orthopedics [2]. Nevertheless, for the hydrostatically extruded pure titanium, typically obtained in several steps, there has been a lack of literature data about the microstructure changes after each extrusion pass. Pachla et al. [16] examined titanium processed in 20 steps of HE with the cumulative true strain of 5.47, the
highest ever reported. However, with respect to microstructure investigations, the authors focused only on the final material as shown to be comprised of equiaxed and elongated grains on transverse (TCS) and longitudinal cross sections (LCS), respectively. Contrarily, Sitek et al. [17] studied both the initial and processed microstructures, yet arrived at similar findings. In fact, Topolski et al. [18] revealed the impact of the initial state on the resultant microstructures to be of less significance. Despite tremendous advancements in the field of HE [19], the intermediate stages of processing have not been studied in detail. It is thought that such information will be useful with regard to the efficient production of high-strength titanium and making use of its properties for future applications. Therefore, the main objective of the present study was to examine pure titanium (grade 2) in terms of its microstructure evolution triggered by the multistep HE method.

2 Materials and methods

2.1 Material development

Pure titanium (grade 2), the chemical composition of which is given in Table 1, was the investigated material.

The billet, in its as-delivered (ASD) condition, had 50.6 mm in a diameter and got hydrostatically extruded to a diameter of 26.7 mm. Afterwards, the obtained rod was machined to a diameter of 24.8 mm and recrystallized at 700 °C for 2 h. Such a material is considered as the initial one within the present study. Upon cooling in air, the material got sliced and processed in five consecutive stages of the HE technique and two steps of rotary swaging. Each of the processing step generated a portion of strain within the material, according to the following formula:

$$\varepsilon = \ln \left( \frac{A_0}{A_e} \right) = \ln R,$$

where $A_0$ and $A_e$ denote the initial and the final cross-sectional area of an obtained sample, whereas $R$ stands for an area reduction. The reductions in diameter and true strains exerted during the whole process of deformation are listed in Table 2.

To eliminate adiabatic heating effects, the fabricated rods were water-quenched right after exiting the extruder chamber. Subsequently, they were left air-cooled and stored.

2.2 Samples preparation

To assure the accuracy of the collected data during microstructural experiments, the samples used for the studies described herein were carefully prepared. Two orthogonal cross sections, i.e., perpendicular to the extrusion direction (ED), denoted as TCS, and parallel to the ED, designated as LCS, were investigated. With respect to EBSD measurements, the first step included grinding with a set of silicon carbide (SiC) foils, ranging in grit from 220 to 4000. Every time a SiC paper was changed, the samples were ultrasonically cleaned in acetone and, consequently, in isopropanol so as to remove abrading-related residues. Then, the specimens were etched using a Struers Electropolisher and the A3 electrolyte (Struers). In doing so, the polishing time of 15 s and the applied voltage of 35 V were applied, while the electrolyte temperature was controlled at 5 °C. In the case of TEM analyses, the samples were initially thinned to a thickness of 100 μm by using the aforementioned metallographic procedures related to abrading. Consequently, the A3 electrolyte, kept at the temperature below 0 °C, was employed for preparation of foils, having approximately 100 nm in thickness.

2.3 Microstructure and texture examinations

A FEI Quanta 3D FEG scanning electron microscope supplied with the EDAX/OIM/EBSD collecting system was used for EBSD investigations. Prior to collecting the data, a step size as well as the overall size of a map were adjusted so at least 1.8 million diffraction points could be recorded. Due to the fact that grain refinement in the studied materials strongly varied, different step sizes were assumed (0.5, 0.2, 0.15, 0.1 and 0.06 μm for the materials deformed at the strain of 0, 0.9, 1.54, 2.44 and 3.23, respectively). Upon gathering of the diffraction patterns, meticulous analyses of the obtained data were performed using the TSL 7.0 software. Orientation maps were color-coded based on an Inverse Pole Figure (IPF) triangle along the normal direction of a sample and cropped so the examined areas were of the same dimension. Any points having

![Table 1](attachment:table1.png)

| O   | Fe  | C   | N   | H   | Ti    |
|-----|-----|-----|-----|-----|-------|
| 0.139 | 0.110 | 0.020 | 0.013 | 0.0007 | Balance |

![Table 2](attachment:table2.png)

| Reduction [mm] | 50.06 → 26.70 | 24.80 → 15.94 | 15.94 → 11.58 | 11.58 → 9.04 | 9.04 → 7.41 | 7.41 → 6.14 | 6.14 → 5.00 |
|---------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| $\varepsilon$ [-] | 1.24 | 0.90 | 0.64 | 0.50 | 0.40 | 0.38 | 0.41 |
| $\varepsilon_{\text{cum}}$ [-] | – | 0.90 | 1.54 | 2.04 | 2.44 | 2.82 | 3.23 |
the Confidence Index (CI) value below 0.1 were removed from the examinations as they correspond to the nano-sized structures, which cannot be resolved using the EBSD method. Subsequently, the grain dilation cleanup procedure was implemented under the assumption that a grain is defined as an area containing at least 5 points, surrounded completely by a boundary segment with a misorientation angle of $5^\circ$. The size of a single grain was specified by diameter, established by calculating the total area of a grain having an assumed circular shape. The mean grain size was calculated based on the weighted mean grain diameter approach, assuming that the weight is the actual grain size, according to the formula:

$$d = \frac{\sum_{i=1}^{n} A_i d_i}{\sum_{i=1}^{n} A_i},$$

where $d_i$ stands for a diameter of an $i$-th grain, $n$ is the number of grains, while $A_i$ denotes the area of the $i$-th grain. Moreover, distributions of the grain size, misorientation angle profiles, grain boundary densities as well as a series of parameters commonly examined in the case of the SPD-treated materials were also characterized. Grain boundary density was measured by dividing the total length of a particular grain boundary, either LAGB or HAGB, by the orientation map size. In addition to orientation maps, crystallographic textures were obtained based on the EBSD data. In doing so, (0001) and (10$\overline{1}$0) pole figures (PFs) were calculated. Furthermore, the IPFs for the extrusion direction (ED) were also revealed in order for an unambiguous determination of the preferred orientations to be possible. The convention of directions used to depict PFs and IPFs is shown in Fig. 1.

To execute a detailed, comprehensive microstructure analysis, a FEI TECNAI SuperTWIN G2 FEG200 kV microscope, combined with the HAADF-STEM/EDS attachments, was additionally utilized. The microstructures of the HE-fabricated titanium specimens were examined by setting the bright field (BF) mode. Moreover, selected area electron diffraction (SAED) patterns were inspected to provide the complementary information about the phase composition and poly/nanocrystalline character of the samples. The obtained diffraction patterns were indexed by using the CSpot software, delivered by courtesy of the CrystOrient company.

![Fig. 1 The convention of directions used to depict PFs](image)

2.4 Mechanical properties

To link the changes in microstructure of titanium induced by HE with physical properties, a series of static tensile and compression tests was performed for each of the analyzed materials. In doing so, a Zwick/Roell Z250 machine was used. Uniaxial tensile and compression tests were carried out at room temperature, employing at least three ‘dog-bone shaped’ and cylindrical specimens, respectively. The obtained samples were CNC-machined (CNC; Computerized numerical control) from the longitudinal cross section of deformed rods. The overall length, width and gage length of the specimens utilized for tensile tests were 15 mm, 4 mm and 3.3 mm, respectively, while the applied strain rate was 0.008 s$^{-1}$. The samples used for compression testing had 7.5 mm in height, 5 mm in diameter and the applied strain was kept at 5 MPa·s$^{-1}$. While executing the compression tests, the maximum capacity of the testing machine was reached, therefore it was impossible to measure the ultimate compressive strength for the analyzed materials and so only the yield strengths in compression were facilitated. Overall, due to a vast amount of data gathered, only the most representative curves were plotted, and the results were averaged. Strains were recorded using an automatic extensometer equipped with a Cardan shaft. In addition, microhardness based on the Vickers HV0.2 method was measured using a Zwick indenter. Only the surface parallel to TCS was tested. The impressions were made along the diameter of a sample with a load of 200 g.

3 Results

3.1 EBSD examinations

Orientation maps taken from the TCS and the LCS of the ASD, the initial as well as the HE-produced titanium samples are illustrated in Fig. 2 and Fig. 3, respectively. The ASD material was used here as reference, mostly to stress out how remarkable changes are made by subjecting a bulk specimen to the HE method. It may be seen that the ASD sample had clearly heterogeneous microstructure as it was composed of large grains mixed with significantly smaller ones. In addition, substructure should also be highlighted as demonstrated by a high fraction of LAGBs i.e., 0.54 and 0.62 on the TCS and the LCS, respectively, as well as substantial differences in crystallographic orientation between the regions constituting the same grains. The mean grain size measured on the TCS was $79.3 \, \mu m \pm 41 \, \mu m$, while that on the LCS equaled $66.2 \, \mu m \pm 42.1 \, \mu m$. Strikingly, the initial material was characterized by the mean grain size of $33.3 \, \mu m \pm 13 \, \mu m$ and $33.8 \, \mu m \pm 14 \, \mu m$ evaluated on the TCS and the LCS, respectively. An exceptional grain refinement
Fig. 2 Orientation maps taken from the TCSs of the a ASD material, b initial material, and Ti deformed at the strain of c ε = 0.9, d ε = 1.54, e ε = 2.44, f ε = 3.23

Fig. 3 Orientation maps taken from the LCSs of the a ASD material, b initial material, and Ti deformed at the strain of c ε = 0.9, d ε = 1.54, e ε = 2.44, f ε = 3.23
was accomplished already at this stage of processing, although the specimens tested were in a recrystallized state. Two-fold drop in the mean grain size has to be highlighted. In general, the initial material was markedly homogeneous since coarse, equiaxed grains were bordered mainly by HAGBs. At the same time, the fraction of LAGBs decreased considerably (0.22 on the TCS and 0.19 on the LCS) with comparison to the ASD. In fact, the only difference between the analyzed CSs was crystallographic orientation of grains (a complete presentation of crystal orientations can be given, in general, by orientation distribution function—see, e.g., [20]).

On the contrary, microstructure of the hydrostatically extruded titanium (see Fig. 2 c, d, e and f and Fig. 3 c, d, e and f) was distinctly heterogeneous. Regardless of the strain introduced, large grains were accompanied by finer ones and a progressively growing fraction of UFG grains (i.e., having the size in the range from 100 nm to 2 μm [21]) as deformation proceeded was noticed. For instance, the material deformed at the strain of 1.54 contained 13.2% and 5.5% of ultrafine grains on the TCS and the LCS, respectively, whereas that processed at the strain of 3.23 had 31.6% of ultrafine grains on the TCS and 17.2% on the LCS. At the same time, the shape of grains was often hard to distinguish. Nonetheless, the tendency of wavy grains to be present on the TCS and elongated ones on the LCS could be seen, especially while considering the materials deformed at intermediate (>2) or high (>3) strains. Even though the process of grain refinement took place successively, the occurrence of equiaxed grains was confirmed by means of the EBSD technique only for those materials, which were subjected to intermediate strains. In crude terms, as deformation continued, grains were forming more refined clusters and elongated bands on the TCS and the LCS, respectively. For the material deformed at the strain of 3.23, thin, narrow, and clearly lengthened grains could be observed. Generally, while inspecting the LCSs, the propensity of grains to align with the characteristic direction, i.e., ED, is a phenomenon typical of the materials processed by the HE method. In addition, as indicated by the black areas (related to the features in size less than those assumed by the grain definition in EBSD analysis) on the OIM maps, it may be declared that elongated bands alternated with UFG or even nanocrystalline structures. It is also worth noting that the higher the strain was applied, the greater the coverage of unindexed points (i.e., CI < 0.1) on the OIM maps was observed. What deserves an additional pointing out is that considerable differences in the crystallographic orientation between the areas encompassing the same grains may be easily viewed at every single stage of processing. It confirms the substructure to pertain within the microstructure of the HE-fabricated titanium. This claim may be supported by considering how the fraction of grain boundaries alters with an increase in steps of HE. The share of LAGB and HAGB for all the analyzed CSs is displayed in Fig. 4. As expected, the initial deformation introduced a vast amount of LAGBs into the material, irrespective of the CS examined. When consecutive extrusion operations were performed, the fraction of HAGBs grew steadily, reaching 0.26 and 0.51 of TCS for the materials fabricated at the strain of 1.54 and 3.23, respectively. However, constantly high share of LAGBs on the LCSs, e.g., 0.80 for a material deformed at the strain of 1.54 and 0.76 for that processed at \( \varepsilon = 3.23 \) needs to be emphasized. In fact, the number of deformation-induced LAGBs observed on the LCSs was always higher than that on the TCSs. A special attention should be also devoted to a material deformed at the strain of 3.23 since the increment in HAGB fraction on the TCSs was the greatest during the last stage of processing. The effect may be substantiated by a significantly increased volume of fine and ultrafine grains with comparison to the materials deformed at lower strains. Overall, the differences in grain boundary share between the characterized CSs proves the HE-produced titanium to be a highly nonhomogeneous material in terms of its microstructure. As seen

Fig. 4 The fraction of LAGBs and HAGBs on the a TCSs, b LCSs of the hydrostatically extruded titanium
from the grain boundary fractions, the LAGB-to-HAGB transformation occurred when a material was subjected to the additional stages of HE. Such a phenomenon is believed to be related to the DRX processes, leading eventually to the formation of small-sized grains bordered by HAGBs. At the same time, one has to bear in mind that a great deal of grain boundaries were trapped inside coarse grains, suggesting that grain subdivision mechanism might also be taking place as deformation proceeded.

Grain size distributions combined with misorientation angle profiles plotted for the TCS and the LCS of the investigated materials are shown in Fig. 5 and Fig. 6, respectively. Irrespective of the examined cross section, the ASD material had a bimodal microstructure, filled with a variety of defects as manifested by misorientation angle profiles, skewing towards low angles. In general, the process of grain refinement during HE could be easily noticed by analyzing how grain size distributions and misorientation angle profiles change on the TCSs. The former, with an increase in the applied plastic strain, became narrower, proving the grain size to be exceptionally reduced, whereas the latter evidenced a tremendous fraction of LAGBs at every step of the processing. On the other hand, grain size distributions drawn for the LCSs confirmed the HE-fabricated microstructures to be bimodal as elongated bands were always accompanied with fine grains.

Interestingly, grain coarsening was observed for the material manufactured at the strain of 0.9. Such a phenomenon could result from high surface activity of a manufactured sample as well as the overall complexity of deformation. In addition, the presence of the misorientation peak around $86^\circ$, stemming from the $\{10\overline{1}2\}$ twinning, ought to be noted for the materials deformed at the strain of 0.9 and 1.54, indicating that twinning mechanism has been initiated. It got suppressed during the later stages of deformation, though its role cannot be overlooked. Simultaneously, no peak around $56^\circ$ was detected for all of the analyzed materials, albeit $\{10\overline{1}1\}$ twinning is more likely to get activated when pure titanium gets processed.

Naturally, any stress induced in the material leads to a substantial increase in the deformation energy stored. OIM-derived characteristics, including Kernel Average Misorientation (KAM) and Grain Average Misorientation (GAM) serve as good indicators of the energy conserved in a sample subjected to any SPD method. For a recrystallized, undeformed specimen, the value of these parameters remains below $0.5^\circ$. The overall change in the KAM and GAM with an increase in plastic strain during HE is illustrated in Fig. 7. A steady increase in both of the parameters could be seen (with final values of around $2^\circ$ and $3^\circ$), implying that hydrostatically extruded titanium is a highly deformed material. Surprisingly, no significant discrepancies in deformation energy stored between the examined cross sections were observed.

In addition to KAM and GAM characteristics, Grain Orientation Spread (GOS) parameter was also deeply studied as

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**Fig. 5** Grain size distributions combined with misorientation angle profiles characterizing the TCSs of the a) ASD material, b) initial material, and Ti deformed at c) $\varepsilon = 0.9$, d) $\varepsilon = 1.54$, e) $\varepsilon = 2.44$, f) $\varepsilon = 3.23$
it enables not only to deliberate about intra-granular lattice distortion and the overall microstructural heterogeneity, but also to distinguish DRX-ed grains from those un-recrystallized. The change in GOS with an increase in strain during HE is illustrated in Fig. 8. It is seen that a tremendous rise in the parameter was caused by low strains, and it reached a maximum value for a material deformed at $\varepsilon = 1.54$. The consequent, small drop in GOS evidences defects saturation stemming from a decreasing dislocation density, an increased volume of fine grains as well as the ongoing DRX-related processes e.g., dynamic recovery. Interestingly, at the entire strain range, GOS evaluated on the LCS was always substantially higher than that on the TCS, indicating that the density of deformation-induced defects was greater and...
more complex for LCSs. The observation confirms yet again the material’s inhomogeneity.

To reveal misorientation fluctuations inside single grains, point-to-point as well as point-to-origin misorientation angle distributions within typically observed grains in a sample processed at the strain of 3.23 are depicted in Fig. 9. The grains were chosen randomly and, afterwards, two different profiles were meticulously analyzed to gain an insight into misorientation angle gradients and the overall inhomogeneity of the material. The L1 line was plotted parallel to the elongation of a grain, whereas the L2 line was drawn perpendicular to the L1. In the case of the TCS, plenty of point-to-origin misorientation jumps, varying in magnitude from 0.5° to 13.6° were observed, regardless of the examined profile. Such a phenomenon originates from the presence of sub-boundaries, cell structures and subgrains in a grain. Overall, a continuous, steady rise of the point-to-origin misorientation, as seen for most of the plotted lines, confirms that a substantial amount of LAGBs was accumulated over an analyzed distance. However, a sharp increase in the point-to-origin misorientation and its subsequent decline (as seen along the L1 line in the TCS) proves the strain inhomogeneity to be a characteristic feature of the material. For the TCS, in general, the point-to-point misorientation remained below 5°, although a couple of misorientation jumps, surpassing 10° might also be seen on the L1 profile. Such a behavior indicates that the newly forming boundaries align parallel to the already-existing ones. With respect to the LCS, a plenty of similarities to the TCS could be distinguished except for a distinct plateau visible on the L1 line. Such a course of the profile refers to the presence of a banded region exhibiting a nearly constant misorientation. Overall, the analysis of long-range misorientation gradients enables to confirm that a vast number of deformation-induced defects are present within single, deformed grains.

### 3.2 TEM examinations

TEM images collected under the BF mode for consecutive stages of the hydrostatically extruded specimens are depicted in Fig. 10. A host of different microstructural defects was generated at every single stage of processing. In fact, highly diversified dislocation-type structures, including dense-dislocation walls, cells or tangles may be easily distinguished while inspecting the material processed at the strain of 0.9. Rather surprisingly, upon the very first pass of HE, some of the defects were already arranged into straight, parallel sets of grain boundaries, clearly visible subgrains as well as cell blocks, i.e., subgrains containing high-density dislocation cells. At the same time, substantially refined, dislocation-bearing ultrafine subgrains were also observed, especially on the LCS. They were mostly irregular in shape.

![Fig. 9](image_url)  
**Fig. 9** Misorientation angle profiles taken for a typical grain on **a** the TCS, **b** the LCS, of the material processed at the strain of 3.23. Distributions shown in the middle refer to the L1 lines and these on the right side were plotted for the L2 lines.
Fig. 10 TEM/BF images taken from the transverse (left side) and longitudinal (right side) cross sections of the HE-processed Ti deformed at the strain of a, $\varepsilon = 0.9$, c, $\varepsilon = 1.54$, e, f $\varepsilon = 2.44$, g, h $\varepsilon = 3.23$

and enclosed by fuzzy, poorly lineated LAGBs. The SAED pattern, embedded in Fig. 10b, confirms the occurrence of FG/UFG structures within the investigated material. Characteristic blurring of spots indicates dislocation accumulation introducing the lattice distortions that affect the diffraction conditions. As deformation proceeded, the nonhomogeneous contrast, resulting from various crystallographic orientation of grains/subgrains and the higher share of lattice defects, became a typical characteristic of the microstructure. Moreover, some areas of grains featured local bending contours which are believed to be the remnants of heavily straining. For a material deformed at the strain of 1.54, an abundance of different microstructural defects, including triple points or substructure-containing lenticular twins, was seen. The fact that the latter were full of microstructural defects was proven by the SAED pattern, inserted in Fig. 10d. In some of the coarse grains, dislocation-type defects filled the entire grain interior, confirming the overall dislocation density to be extremely high. With an additional increase in strain, i.e., up to 2.44, the fraction of submicron-sized or nanocrystalline features grew considerably.

While most of the grains/subgrains contained dislocations and their ramifications, some of the fine subgrains were almost defects free. It might be substantiated by the fact that thermally driven processes have already been activated. Such a claim is additionally supported by the tendency of deformation-induced defects to cluster near the well-defined HAGBs, regardless of the strain exerted. For the material deformed at the $\varepsilon = 3.23$, a vast amount of ultrafine, close-to-equiaxed and nearly defects-free grains/subgrains was
viewed. The most intensely refined grains were shorn of microstructural defects as these were possibly absorbed into the neighboring HAGBs. The actual shape of grains/subgrains was barely discernable since most of them were contained within downscaled clusters, surrounded by LAGBs. Without a doubt, the role of the DRX-related mechanisms was of great significance during the later stages of HE. Furthermore, SAED patterns taken for the material processed at the strain of 2.44, proved that microstructure of this particular specimen is of strictly polycrystalline nature. In general, spots organized in regular circles imply refined microstructure as well as considerable misorientation differences between grains [22].

3.3 Crystallographic texture determination

(0001) and (10 1 0) PFs as well as IPFs for the ED are illustrated in Fig. 11. In general, the analysis of crystallographic texture confirmed the nature of the investigated materials to be highly anisotropic. The texture of the ASD material was relatively weak as a single one predominant component was not revealed while inspecting the (0001) and (10 1 0) PFs. However, the IPF for the ED (Fig. 11a) confirmed that orientations of some grain clusters fall towards the center of this IPF. Obviously, upon plastic deformation, the initial texture was markedly altered as the formation of preferred orientations took place. Hydrostatically extruded titanium exhibits strong fiber texture, typical for extruded, drawn and swaged metals [23]. Interestingly, the fiber component had already formed during the first stage of HE and the consecutive extrusion operations strengthened it as the material deformed at the strain of 3.23 demonstrated axial texture. The examination of the PFs enables to confirm that grains are aligned such that <10 1 0> directions are parallel to the ED (or equivalently, {10 1 0} planes are perpendicular to the ED). On the other hand, the presented PFs show that grains are oriented such that <0001> directions, forming a radial distribution, are perpendicular to the ED, which proves the fiber character of crystallographic texture. Finally, while inspecting the IPFs, it can be summarized that the texture of the material deformed at the strain of 3.23 is comprised between two fiber components with respect to the ED: the main component <10 1 0> and the weaker one <11 2 0>. Also the weak <0001> component is visible at the final strain.

3.4 Mechanical behavior

The most representative engineering stress–strain curves plotted for the HE-processed samples are illustrated in Fig. 12. It may be seen that after reaching the ultimate tensile strength (UTS), the initial material underwent gradual flow softening, resulting in the elongation to failure of nearly 25%, typical for unprocessed, coarse-grained titanium. The similar behavior may be observed for the ASD samples. On the other hand, all of the HE-fabricated specimens exhibited flow softening quickly without demonstrating the apparent strain hardening mechanism.

In general, severely deformed titanium samples are characterized by low ductility (less than 10%). Interestingly, elongation at break did not diminish considerably with a subsequent increase in cumulative plastic strain as it persisted in the order of 7–8%.

In addition to tensile properties, representing the most typical way of revealing how a material behaves under a mechanical stimulus, compressive strength was also investigated. However, since the maximum capacity of the testing machine had been reached and the materials were not destroyed, only YS in compression is discussed. The change in YS with an increase in plastic strain is illustrated in Fig. 13. For comparison, YS in tension was also added. Tension–compression asymmetry, frequently reported for titanium-based materials, could be clearly seen. Interestingly, as deformation progressed, the difference between YS in tension and that in compression became greater, e.g., 188 MPa and 305 MPa for the samples obtained at the strain of 0.9 and 2.44, respectively. Nonetheless, a noteworthy value of YS in compression, i.e., 753 MPa, was achieved for the material deformed at the strain of 3.23.

Finally, hardness of the investigated materials, a property assessing the resistance of a material to plastic deformation, was determined. The change in Vickers hardness with an increase in plastic strain is depicted in Fig. 14a. The results prove that the consecutive extrusion operations caused hardness to slowly, almost linearly, rise and that the material deformed at the strain of 3.23 was approximately 40% harder than that in the initial state. The increase in hardness was not that spectacular as in the case of YS or UTS, but it is worth highlighting how hardness is distributed over a diameter of a sample (Fig. 14b). It may be seen that the HE process provoked hardness to be more homogeneous.

To better visualize the occurring changes in hardness, its distribution was illustrated in a form of radar plots, displayed in Fig. 15. Generally, the more blurred the graph, the more consistent the hardness. Even though there were a few violent fluctuations seen for the HE-processed sample, the general tendency of a material to become more uniform in terms of hardness after having been plastically deformed could be, without a doubt, noticed.

4 Discussion

Owing to its mechanical characteristics as well as excellent biocompatibility, titanium produced via multi-pass HE followed by rotary swaging could be potentially applied in
medicine for orthopedic or dental implants [24]. In fact, the attractiveness of a material from the point of view of biomedical purposes has been demonstrated in its superior corrosion resistance [25], enhanced protein adsorption on the surface [26] as well as improved cell–material interactions [27]. As a great advantage of the multi-pass HE one may regard the possibility of the mechanical properties adjustment for a certain application of the final product by applying different process parameters such as reduction diameter, number of passes, operating pressures etc. Therefore, inspecting the microstructure changes after each extrusion pass and correlating them with the mechanical properties is
of great interest. The importance of this issue was recently shown for other hcp material, i.e., zinc and its alloys [28]. Hence, the knowledge of microstructure evolution during HE opens new paths for titanium development.

Generally, by means of HE, a tremendous volume of deformation-induced defects is generated in pure titanium leading to high microstructural inhomogeneity, as confirmed within the present study by broad distributions of the grain size and considerable misorientation gradients inside single grains. Regarding the grain size distributions, although they got significantly reduced with an increase in plastic strain, the log-normal plots, characteristic of the severely deformed materials, have never been obtained and the overall fraction of coarse grains have always outbalanced that of the fine or ultrafine ones. Microstructure heterogeneity can be also witnessed by examining not only the grain size but also the grain shape. At every single stage of deformation large grains were mixed with medium-sized, ultrafine and nanocrystalline, either elongated, wavy or highly irregular in morphology. Furthermore, the claim could be also supported by a host of localized deformation regions such as in-grain strain contours, triple points, or dislocation tangle zones. Finally, high deformation energy stored in the processed materials, demonstrated by the constantly growing values of the KAM and GAM parameters as deformation proceeded, is also a definite proof of the HE-processed titanium microstructure to be heterogeneous. Such a finding stands in stark contradiction to that reported by Topolski et al. [29], who examined homogeneity of nanostructure and hardness in two hydrostatically extruded rods, varying in diameter, the number of HE stages and the accumulated strain. The authors concluded that owing to the minimized friction during processing, the obtained specimens are homogeneous as manifested in hardness measurements, TEM examinations and stereological analyses. In fact, our previous study [6] also showed that Vickers hardness is consistent if measured over a diameter of a titanium sample. Nevertheless, uniform mechanical properties of a material do not reflect on the homogeneity of its microstructure.

Unlike in the case of fcc and bcc metals, deformation of hcp titanium is more complex because a smaller number of active slip systems is involved. Thus, twinning becomes a mode of vast significance so a material is to accommodate the heavy deformation. Zherebtsov et al. [30] declared that during low straining of pure titanium by means of the HE technique, twinning is to happen as demonstrated by a vast fraction of HAGBs and the existence of \{10\overline{1}0\}, \{10\overline{1}2\} and \{11\overline{2}3\} twins. Herein, the microstructure of a material

![Fig. 12](image12.png)

**Fig. 12** Typical stress–strain curves plotted for the investigated materials

![Fig. 13](image13.png)

**Fig. 13** Change in YS during HE

![Fig. 14](image14.png)

**Fig. 14** a change in Vickers hardness with an increase in plastic strain during HE, b measurements of hardness distribution over diameter for the analyzed specimens
deformed at the strain of 0.9 bore the stamp of twinning in shear-like bands, similar to those discovered in the HPT-treated titanium [14]. After all, it is commonly known that twinning generates a new orientation in a crystal by shear; thus, the observed bands are, indeed, its remains. In addition, the evidence of twinning was also confirmed by a deep analysis of misorientation angle profiles. The characteristic misorientation peak around 86°, originating from the \{1012\} twinning, was noticed for the materials processed at the strains of 0.9 and 1.54. In fact, substructure-containing twins were clearly visible while investigating the microstructure of a material deformed at 1.54 of strain with the use of TEM. However, twins were not observed further on, implying the suppression of twinning mechanism. The deformation at higher strains is believed to be ruled by slip. Similar conclusions were drawn when microstructures of the ARB-ed [13], the HPT-ed [14] and the MDF-ed [15] titanium were examined.

The process of HE is generally viewed as an unconventional SPD method [2], mostly due to the reduction in a diameter of a processed sample, yet a great deal of similarities between microstructures obtained by HE and those of the other classic techniques, e.g., ECAP, may be revealed, especially while inspecting on the microstructures produced at lower strains. The truth is that generation of high-density dislocations and the interactions they undertook were dictating the microstructure evolution of titanium during the first stages of HE. Already during the first step, a vast amount of dislocation-derived structures was formed (as proven by TEM images), confirming that limited, small strains (< 1) are required to accommodate various types of non-equilibrium microstructural defects in a material. The formation of dislocation walls and dislocation cells as well as LAGBs were the dominating processes in the beginning of deformation. The latter, as seen in Fig. 16, were assembling into arrays, localized nearby HAGBs. However, the remnants of unconnected segments of GBs, trapped inside coarse grains and extending across the grain interiors were also detectable. Such a behavior is a clear indication of grain segmentation/subdivision phenomenon [11]. The mechanism proceeds by a constant breaking down of parent grains into subgrains and is believed to be regulated by deformation-induced boundaries, i.e., geometrically necessary boundaries, such as twin boundaries or parts of dislocation slip, and incidental dislocation boundaries e.g., dislocation cells or bundles [31]. In general, increase in dislocation density leads to the

![Fig. 15 Radar plots and the corresponding distribution of hardness over a diameter (claret-colored line) plotted for the a initial material, b material deformed at $\varepsilon = 3.23$.](image)
formation of cell blocks and, with an additional rise in plastic strain imposed on a processed sample, sub-boundaries or LAGBs are eventually created [15]. Within the present study, in addition to LAGBs behavior, the occurrence of grain subdivision may be also backed up by the rapid increase in intra-granular orientation spread, the quick disappearance of twin-typical misorientations as well as the continuous presence of barely refined or slightly downsized grains and these containing high-density dislocations, even while high strains were exerted into a deformed material. In fact, as discussed by Wert et al. [32], in materials having larger grain sizes, microstructures are first divided into mesoscale, orientation domains, usually called deformation bands, that reflect discrepancies in the activity of various slip systems. The authors define these microstructural features as subvolumes of the original grains in which the crystal orientation is almost constant, but differs greatly from that localized in other domains originating from the same grain. Development of several distinct crystal orientations within a single grain is an indicative of the grain subdivision mechanism. Within the present study, the process may further substantiate the unusual existence of lengthened grains/subgrains on the TCSs (Fig. 10a), the features resembling deformation bands. Such phenomenon was observed for the material deformed at the strain of 0.9, i.e., the coarse-grained material characterized by considerable differences in the crystallographic orientation between the regions constituting the same grains. Interestingly, grain segmentation takes place easily, even at ambient temperatures, as a simple result of mechanical deformation. However, it generally induces a substantial increase in the fraction of HAGBs [33], what was not reported herein. The overall deficiency of well-developed HAGBs in the processed microstructures strongly suggests that another grain refinement mechanism must have taken place as well. Ma et al. [31] studied the changes in the microstructure of pure titanium triggered by shearing. The authors implemented precession electron diffraction and arrived at similar conclusion of LAGBs constituting most observed GBs, yet the main deformation mechanism was claimed to be grain subdivision. Hence, it might be postulated that the microstructure of the HE-processed titanium, specifically that analyzed at lower strains, was twinning- and grain-segmentation dominated.

Obviously, with an increase in strain, the density of microstructural defects grows steadily. Within the present study, the phenomenon was reflected by a small decrease of the mean grain size measured on the LCS and a variety of dislocation cells or triple points observed within the deformed microstructures. Concurrently, not only dislocations generation, but also their rearrangement and annihilation took place as evidenced by the progressively, yet slowly increasing share of HAGBs. It could be assumed that dislocation walls initiated the process of microstructure rebuilding and, with the HE steps increasing, they were transformed
into dislocation cells. These were, consequently, gathering into subgrains and/or cell blocks, surrounded by poorly lineated, branched LAGBs. Eventually, yet another increase in strain caused the pre-existing LAGBs to turn into HAGBs as subgrains evolved into small-sized grains. Although individual, ultrafine grains/subgrains could be observed on every single stage of the HE processing, their actual formation was governed by an increase of misorientations between subgrains in the thermally facilitated process of cDRX. The fact is that the LAGB-to-HAGB transformation is the most typical evidence of the cDRX [11]. Herein, the absorption of dislocations into delineated, sharp boundaries and, thus, the continuously growing fraction of HAGBs on both examined CSs was undeniable. Moreover, the occurrence of cDRX could also be justified by the presence of small-sized, either lengthened or equiaxed, grains/subgrains, aggregating at HAGBs, the never-ending rise in the deformation energy stored with an increase in the applied strain as well as huge misorientation gradients inside individual grains of large size, providing the necessary driving force for the transformation of LAGBs into HAGBs [34].

For the whole strain range, the HE-processed titanium had clearly bimodal microstructure, whose evolution was, in fact, manifested by a progressive increase in the fraction of HAGBs and fine grains. Surprisingly, wealth of dislocation-type structures could have been observed within the microstructure, regardless of the strain introduced. What ought to be mentioned is that pure titanium is characterized by the extremely low thermal conductivity (17 Wm⁻¹ °C⁻¹) [13], so the metal’s tendency to local rise in temperatures while being processed as well as that to experience recovery is indisputable. In addition, the material exhibits high stacking fault energy (i.e., 300 and 150 mJ/m² for basal and prismatic planes, respectively [15]); therefore, it is expected for the cDRX mechanism to occur easily during deformation. To fully confirm that grain subdivision and cDRX actually occurred, the change in the grain boundary density with an increase in plastic strain is displayed in Fig. 17. Two stages may be distinguished while analyzing the grain boundary density–grain size plots. The first one up to the strain of 0.9 was characterized by a huge drop in the mean grain size (only while analyzing the TCS, because in the case of the LCSs, an increase was initially noted) as well as an almost unchanged density of boundaries. Clearly, original grains were subdivided into defects-containing fragments of different size. During the second stage, the mean grain size decreased substantially and a rapid increase in the share of grain boundaries was noted. The observed changes should be attributed to the presence of small-sized, fine, and ultrafine grains as well as nanograins. Naturally, it is cDRX which is responsible for their formation. It can be mentioned that another important insight into material microstructure can be obtained by the analysis of residual stress [35], what is foreseen in the future studies.

It could be expected that such tremendous changes in the microstructure of pure titanium triggered by HE have an influence on its physical properties, including mechanical behavior. As shown, both YS and UTS were significantly enhanced with an increase in HE steps. The material deformed at the strain of 3.23 exhibited extraordinary value of UTS, i.e., 1110 MPa, being almost twice as high as that of the initial state. Figure 18. illustrates how grain size and UTS change with an increase in cumulative strain. Without a doubt, substantial grain refinement led to the development of a high-strength material. Strengthening mechanisms were deeply examined in our previous report [6]. It has been revealed that strengthening due to HABGs and substructure have the greatest impact on mechanical properties of the HE-processed titanium. Herein, we have additionally proven that the obtained material exhibits tension–compression asymmetry. However, the impact of strong, fiber texture on mechanical properties ought to be taken into consideration.
as well. The fiber component had formed already during the first stage of HE and the subsequent extrusion operations strengthened it as the material deformed at the strain of 3.23 demonstrated axial texture. Interestingly, texture of this particular material was comprised between two fiber components with respect to the ED: the main component $<10\overline{1}0>$ and the weaker one $<11\overline{2}0>$. The activity of different slip systems at the later stage of processing is, therefore, evident and it might substantiate a slight increase in ductility of the material, as revealed by tensile tests.

5 Conclusions

Within the present study, pure titanium (grade 2) was processed by the multi-stage hydrostatic extrusion method with an aim to reveal the mechanisms governing its microstructure evolution. The use of the well-established techniques (electron backscatter diffraction and transmission electron microscopy) enabled to confirm the following:

- Hydrostatically extruded titanium is a highly inhomogeneous material in terms of its microstructure as proven by a great deal of dislocation-derived features (including dislocation walls, dislocation cells, cell blocks, dislocation tangles, twins) present therein, a variety and complexity of the grain size and shape (large, medium-sized, fine, ultrafine, nano-sized; wavy, elongated or irregular), high deformation energy stored in a material as well as tremendous misorientation gradients measured within single grains.

- At low strains, twinning and grain subdivision process are the main mechanisms dictating the evolution of titanium microstructure during hydrostatic extrusion. Twinning was evidenced by the presence of shear bands-like structures composed of dislocation-type defects, the actual existence of lenticular-shaped twins as well as the occurrence of some characteristic peaks around 86° on the misorientation angle profiles, stemming from the $\{10\overline{1}2\}$ twinning. Grain segmentation was confirmed by a significant number of grain boundaries, trapped inside coarse grains, and extending across their interiors, the sharp increase in intra-granular orientation spread, the presence of elongated grains/subgrains on various transverse cross sections as well as the rapid vanishing of twin boundaries misorientation.

- At higher strains, the role of continuous dynamic recrystallization in microstructure evolution is of profound significance as shown by the LAGB-to-HAGB transformation, the progressively growing share of ultrafine grains with an increase in plastic strain, the unremitting interactions between dislocation-type features, huge misorientation gradients inside deformed grains as well as aggregating of small-sized grains at high-angle grain boundaries.

- The observed changes in the microstructure, mainly substructure development, progressively smaller grain size and the increasing share of HAGBs, have a great impact on the mechanical behavior of the investigated material. With a decrease in the mean grain size, a steady rise in both yield strength and ultimate tensile strength was noticed.

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Fig. 18 Change in grain size (for TCS) and UTS with an increase in strain during HE
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