



Severe plastic deformation of an additively manufactured Ti–6%Al–4%V alloy: Effect of post-processing heat treatment and hot isostatic pressing on the behavior over a wide range of strain

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ABSTRACT

Ti–6Al–4V (wt.%) alloy containing 0.14 wt.% trace Fe and 1 wt.% yttria-stabilized zirconia (YSZ) was manufactured by laser powder bed fusion. In addition to the as-built samples (AB), some samples received post-heat treatments, either by conventional heat treatment (HT) alone or by a combination of HT and hot isostatic pressing (HIP). The HT and HIP processing increased the grain size and the secondary β -phase fraction, and decreased the as-grown dislocation density, leading to a decrease in hardness from about 4500 to 4100 MPa. All three sample types were severely deformed by high-pressure torsion (HPT) to an equivalent strain of ~ 208 . The differences in the initial microstructures of the three states gradually disappeared as strain increased. The dislocation density increased with plastic deformation, leading to a decrease in the crystallite size and the β -phase fraction. The saturation values of the crystallite size and the dislocation density were about 20 nm and 10^{16} m^{-2} , respectively, while the β phase practically disappeared at a strain of about 26. Instead, V/Fe-rich regions developed at the grain boundaries of the α phase. The strong initial texture components of $\langle 10\bar{1}2 \rangle$ and $\langle 11\bar{2}0 \rangle$ changed to an almost random texture with a weak $\langle 10\bar{1}1 \rangle$ component during HPT. Due to these changes in the microstructure, the hardness of the three types of samples saturated at similar values within the range of 4500–4700 MPa. The low hardness increase of 5–12% after HPT can be attributed to texture softening which moderated the hardening caused by the defects and small grain size.

1. Introduction

The Ti–6Al–4V alloy, also known as Ti64, is an $\alpha+\beta$ titanium alloy containing 6 wt.% Al and 4 wt.% V [1]. This is the main type of alloy, accounting for almost 40% of the total titanium tonnage supplied annually [2]. The Ti–6Al–4V alloy is used in various applications: in the aerospace industry for fan and compressor discs and blades [3–5], in the automotive industry for valves and connecting rods [6], in the biomedical applications for bone plates, screws, fasteners, and orthodontic brackets [7]. This widespread use is due to its combination of high specific strength, high corrosion resistance, and increased forgeability. However, its relatively low hardness and poor wear resistance

limit its performance in demanding tribological environments. To improve the properties, reinforcement particles can be incorporated into the titanium matrix to produce a metal matrix composite (MMC). The reinforcing materials in MMCs are usually ceramic-based, such as SiC, Al_2O_3 , TiC, TiB_2 , Si_3N_4 , ZrO_2 , and TiO_2 [8,9]. Yttria-stabilized zirconia (YSZ) is a promising ceramic reinforcement material due to its outstanding wear resistance, high fracture toughness, good biocompatibility, high chemical stability, and high melting temperature [10–15]. Partially stabilized zirconia (PSZ) with a metastable tetragonal structure can be obtained by adding 4–5 wt.% of Y_2O_3 to ZrO_2 , whereas a higher concentration of 8–10 wt.% of Y_2O_3 is required for full stabilization to form YSZ with a stable cubic fluorite structure at all temperatures [16,

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17]. The metastable tetragonal phase can transform to monoclinic due to stresses developing at crack tips during deformation, and the associated volume expansion can hinder crack propagation. This effect is the strongest when 3 mol% (5.3 wt.%) Y_2O_3 is added to zirconia [18,19].

Ti-YSZ composites have been widely studied for biomedical applications. For instance, the composites based on Ti-xMo ($x = 23, 27, 35$ wt.%) alloys with the addition of 3, 5 and 10 wt.% YSZ, and 1 wt.% Y_2O_3 were fabricated by mechanical alloying and hot-pressing. The results demonstrated that the hardness increased with YSZ addition, and the Ti-35Mo matrix material containing 10 wt.% YSZ achieved the highest hardness value of 6920 MPa [20]. In another study, Ti-YSZ composites with YSZ contents of 0, 1, 3, 5, and 10 wt.% were fabricated via electric discharge sintering. The results demonstrated a significant enhancement of the mechanical properties with increasing YSZ content. The microhardness increased from 860 MPa for pure Ti to 5990 MPa for the composite containing 10 wt.% YSZ. The yield strength and ultimate tensile strength also increased from 366 MPa to 743 MPa for pure Ti to 922 MPa and 1944 MPa for the composite containing 10 wt.% YSZ [21].

The conventional production process of MMCs includes powder metallurgy (PM), compocasting and thixoforging, infiltration and casting methods [22,23]. These established methods are effective; however, there are some limitations including non-uniform reinforcement distribution and potential interfacial reactions between the reinforcement phase and the molten matrix [24]. To overcome some of these constraints, additive manufacturing (AM) has emerged as a revolutionary technology for producing MMCs. AM has many benefits, including minimal material waste, design flexibility, superior mechanical properties, reduced lead time and cost [25–29]. It is important to note that AM processes are characterized by rapid cooling rates and repeated heating and cooling cycles which substantially affect the evolution of the microstructure. This results in a hierarchical microstructure, typically with a strong crystallographic texture, coarse columnar grains, fine cellular/sub-grain structures, metastable phases, and dense dislocation networks [30–38].

Due to superior properties, MMCs are well-suited for applications where components are subjected to extreme mechanical loading, wear, and high temperatures [28,39]. To ensure reliability under such demanding conditions, it is imperative to understand their deformation behavior at high strains. The high-strain behavior has been studied only on Ti MMCs produced by conventional methods [40–42], and the deformation behavior of the AM-processed MMCs at high strains remains unestablished. It remains unclear whether the unique microstructure produced by AM leads to distinct deformation mechanisms during severe plastic deformation (SPD) compared with those observed in conventionally produced MMCs.

The present work investigates the high-strain deformation behavior of Ti-6Al-4V (wt.%) alloy containing YSZ nanoparticles and produced by laser powder bed fusion (L-PBF). A prior study showed that when YSZ was added to the Ti alloy matrix and the blend was sintered by SPS, the densification was significantly improved when nanoscale YSZ powder was used instead of the micron-scale counterpart, owing to a more homogeneous distribution of the powder particles in the matrix [43]. Thus, in the present study, nanocrystalline YSZ (nYSZ) powder containing 3 mol% Y_2O_3 was used as a ceramic reinforcement in Ti-6Al-4V matrix. In a previous work [44], the tensile properties of Ti-6Al-4V matrix composites incorporating 0.5, 1 and 2.5 wt.% of nYSZ particles were investigated. These studies showed that the best compromise between strength and ductility was achieved with 1 wt.% nYSZ. Therefore, this composition was investigated in the present study. It is noted that the effect of YSZ addition on the mechanical behavior of the Ti-6Al-4V matrix at low strains has already been studied in previous works [44,45], and in this present investigation, YSZ-free and YSZ-added samples are not compared. In addition, a recent work by Choi et al. [44] demonstrated the decomposition of YSZ nanoparticles during additive manufacturing of Ti64-YSZ composites. Therefore, in this study, the original YSZ phase is not expected to persist after L-PBF

processing, and its role during subsequent HPT deformation could not be systematically analyzed. Rather, the 3D printed Ti-6Al-4V alloy with 1 wt.% nYSZ was plastically deformed to high strains (up to ~ 208) via high-pressure torsion (HPT) technique. The primary objective is to understand how the unique AM microstructures evolves under SPD and how post-processing treatments influence this evolution. To evaluate the influence of post-processing on deformation mechanisms, three initial material conditions were considered: as-built, heat-treated and hot isostatically pressed (HIP). The microstructural evolution with increasing applied strain is characterized using electron microscopy and X-ray line profile analysis (XPLA), and the results are related to hardness variation and the evolution of crystallographic texture. The role of heat treatment and HIP in governing the high-strain deformation behavior is elucidated.

2. Materials and methods

2.1. AM-processing and subsequent post-treatment of Ti6Al4V/YSZ composite samples

The YSZ-reinforced Ti-6Al-4V (wt.%) alloy was manufactured from a powder blend consisting of Ti64 powder produced via Electrode Induction-melting Gas Atomization (EIGA) with an average particle size of about 25 μm and 1 wt.% YSZ nanopowder with an average particle size of 40 nm. The nominal chemical composition of Ti64 and YSZ powder can be found in Ref. [46]. In addition to Al and V, the Ti64 powder also contained 0.14 wt.% Fe. The ZrO_2 powder contained 5% Y_2O_3 . The powder blends were prepared using a T2F Turbula blender with 6 h of mixing time under an argon-protected environment. The blending protocol and L-PBF process were developed and carried out at Z3DLAB SAS company, Chaumontel, France.

The bulk samples were fabricated on an SLM 125HL machine (SLM Solutions Group AG, Germany) using a 200 W ytterbium fiber laser. The L-PBF platform was preheated to 200 °C to reduce residual stresses. Specimens were printed using the chess scanning strategy with the following parameters: laser power of 200 W, scan speed of 700 mm/s, layer thickness of 30 μm , and hatch spacing of 80 μm . These parameters resulted in a volumetric energy density (VED) of 119 J/mm³. The initial material was printed as cubes with dimensions of 10 × 10 × 10 mm³ and cylinders with a diameter and height of 10 mm.

Selected samples were subjected to stress relief heat treatment at 600 °C for 2 h in an argon-protected environment and in accordance with AMS 2801 standards. A separate subset of heat-treated specimens was subjected to a HIP post-treatment (920 °C/100 MPa/2 h), in accordance with F3001 standards. The HIP post-treatment was carried out in three stages, as described in Ref. [46].

Sample designations in this study are as follows: AB for as-built specimens, AB+HT for specimens receiving conventional heat treatment alone, and AB+HT+HIP for specimens receiving both HT and HIP.

2.2. Plastic deformation for extremely high strains using HPT technique

HPT-processing was conducted using a conventional HPT facility with a quasi-constrained set-up [47]. The HPT disc surfaces were oriented perpendicular to the additive manufacturing building direction. For the HPT processing, the cylindrical AB, AB+HT, and AB+HT+HIP specimens were cut into discs with a cross-section diameter of 10 mm and a thickness of ~ 0.85 mm. Before torsional deformation, the discs were loaded to supply the frictional force needed for slippage-free torsion. To avoid potential slippage issue during HPT processing [48], the anvils used in this study were designed with reasonably rough surfaces. The processing was conducted at room temperature (RT) under 6.0 GPa at the rotational speed of 1 rpm for $\frac{1}{2}$, 1, 5 and 10 turns. The disc thicknesses were approximately 0.7 mm after HPT across all numbers of turns. The equivalent strain was calculated using the following formula [49]:

$$\varepsilon = \varepsilon_0 + \frac{2\pi r N}{\sqrt{3}t} \quad (1)$$

where ε_0 is the compressive strain introduced by the initial loading in the HPT facility, which is 0.08, r is the distance of the studied location from the disc center, N is the number of turns, and t is the thickness of the HPT-processed disc.

The microstructure of the HPT-processed samples was studied in the center and at the edge of the discs. The largest dimension of the XRD spot on the sample surface was approximately 2 mm. Therefore, the measurements at the 'center' and 'edge' locations correspond to average distances of $r = 0.5$ mm and 4 mm from the disc center, respectively. The equivalent strain values for these distances from the disc center for the applied numbers of turns were calculated using eq. (1) and listed in Table 1, where the items in the left column are arranged in order of increasing calculated equivalent strain. To illustrate the calculation, for the center of the disc processed by $N = 1/2$ turn, using the values $t = 0.7$ mm and $r = 0.5$ mm, the equivalent strain was obtained as 1.38.

2.3. X-ray diffraction study on the evolution of phase composition and texture

The phase composition and crystallographic texture of the additively manufactured and deformed Ti–6Al–4V/YSZ samples were characterized by X-ray diffraction (XRD). Patterns were acquired using a Smartlab diffractometer (Rigaku, Tokyo, Japan) with $\text{CuK}\alpha$ radiation ($\lambda = 0.15418$ nm) in Bragg-Brentano geometry over a 2θ range of 30 – 155° . Before XRD experiments, the sample surfaces were mechanically polished to 4000-grit paper, followed by polishing with a $1\text{-}\mu\text{m}$ colloidal alumina suspension. Finally, these surfaces were etched with a 40% hydrofluoric acid (HF) solution for 20 s to remove the uppermost surface layer affected by mechanical polishing. The XRD intensity fraction of the retained β -Ti phase was calculated as the ratio of the sum of the integrated peak areas of the β phase to the sum of the integrated peak areas of all reflections in the pattern.

The crystallographic texture was characterized with the $\langle 10\bar{1}0 \rangle$, $\langle 10\bar{1}1 \rangle$, $\langle 10\bar{1}2 \rangle$, $\langle 11\bar{2}0 \rangle$ and $\langle 10\bar{1}3 \rangle$ XRD pole figures (PFs) measured with parallel beam optics. The orientation distribution functions (ODFs) were calculated from the PFs, using a 5° resolution and triclinic specimen symmetry in the MTEX MATLAB plugin [50]. The texture index was subsequently calculated from the ODF.

2.4. Characterization of the microstructure via X-ray line profile analysis

The microstructure of the samples was investigated by X-ray line profile analysis (XLPA). The patterns were measured using a high-resolution rotating anode diffractometer (RAMultiMax9, Rigaku, Tokyo, Japan) operating at 40 kV and 80 mA with $\text{CuK}\alpha_1$ radiation ($\lambda = 0.15406$ nm). Measurements were conducted using a parallel X-ray beam with a width of 0.2 mm and a height of 2 mm. The surface preparation was the same as described in the previous section.

The X-ray diffraction peak profiles were evaluated by the convolutional multiple whole profile (CMWP) fitting procedure [51]. In this

method, the diffraction pattern was fitted by the sum of a background spline and the convolution of the instrumental peaks with theoretical line profiles for crystallite size and dislocations. The instrumental peaks were measured on a NIST SRM660a LaB₆ peak profile standard material. The following microstructure parameters were obtained from the CMWP fitting: the area-weighted mean crystallite size ($\langle x \rangle_{\text{area}}$) and the dislocation density (ρ). The value of $\langle x \rangle_{\text{area}}$ is calculated as $\langle x \rangle_{\text{area}} = m \cdot \exp(2.5\sigma^2)$, where m is the median and σ is the lognormal variance of the crystallite size distribution.

2.5. Microstructure study performed by electron microscopy

The microstructure and crystallographic orientation of the AB, AB+HT, and AB+HT+HIP samples were characterized by electron backscatter diffraction (EBSD) in multiple states: undeformed condition, after compression in the HPT facility, and after HPT processing to 0.5 turns.

Prior to EBSD examination, the samples were mechanically ground using SiC papers of progressively finer grit (up to 4000 grit), followed by polishing with 3 μm and 1 μm diamond suspensions. Final surface finishing was performed by electrolytic polishing using the Struers A3 electrolyte solution in a LectroPol-5 unit, operated at a potential of 30 V for 15 s. EBSD data were acquired using an EDAX Velocity EBSD camera mounted on a FEG SUPRA 40VP field-emission scanning electron microscope (FE-SEM). The EBSD maps were captured using EDAX APEX software with a step size of 0.1 μm . The acquired Kikuchi patterns were indexed using the hexagonal α -Ti phase database provided within the APEX software. Subsequent data post-processing and visualization, including inverse pole figure (IPF) mapping and grain orientation spread (GOS) mapping, were conducted using OIM Analysis™ v9 software.

Transmission electron microscopy (TEM) was used to investigate the nanostructure of AB, AB+HT, and AB+HT+HIP samples formed at the highest applied strain achieved near the edge of the HPT-processed discs, up to the highest number of turns (10). Thin sections were cut perpendicular to the radius and the circular surface of the disc using a dual-beam FIB/SEM instrument Scios II (Thermo Fisher Scientific, Waltham, MA, USA) with a focused Ga^+ beam. The final steps in FIB preparation were made at 2 kV. The TEM investigations were performed in a Cs-corrected Themis microscope (Thermo Fisher Scientific, Waltham, MA, USA) equipped with four EDS detectors in Super-X geometry. The analysis was performed at an accelerating voltage of 200 kV with a point resolution of 0.08 nm. High-angle annular dark-field (HAADF) images and EDS elemental maps were recorded in scanning transmission electron microscopy (STEM) mode. The grain dimensions were determined from dark-field (DF) images, as the orientation differences are better resolved in these images than in BF micrographs. As most grains were elongated, for the characterization of grain dimensions the average grain length and width were measured using software ImageJ [52]. It is noted that the crystallite size obtained by XLPA is usually smaller than the grain dimensions determined by microscopic methods, and the former quantity measures rather the subgrain size in severely deformed metallic materials [53].

2.6. Hardness test

The Vickers microhardness was measured on the AB, AB+HT and AB+HT+HIP samples both before and after deformation at different strains. A Zwick Roell ZH μ microindenter (manufacturer: ZwickRoell LP, Kennesaw, GA, USA) was used with an applied load of 500 g and a dwell time of 10 s. For the HPT-processed specimens, the hardness was measured along the disc diameter at RT with an indent spacing of 0.5 mm. At each radial position, ten indents were made, and the mean hardness value was calculated for each distance from the disc center.

Table 1

The deformation methods and the corresponding equivalent strain values.

Deformation method, measured location within disc	Equivalent strain
Compression in HPT facility	0.08
½ turns of HPT, close to center	1.38
1 turns of HPT, close to center	2.67
½ turns of HPT, close to edge	10.5
5 turns of HPT, close to center	13.0
1 turns of HPT, close to edge	20.8
10 turns of HPT, close to center	26.0
5 turns of HPT, close to edge	103.8
10 turns of HPT, close to edge	207.5

3. Results

3.1. Characterization of the microstructure before plastic deformation

Fig. 1 shows SEM micrographs of the electropolished surface of specimens a) AB, b) AB+HT, and c) AB+HT+HIP. These images reveal that the as-processed AB sample contains pores with the typical size of 2–3 μm , as indicated by the yellow circles in Fig. 1a. Similar pores still exist in specimen AB+HT, as shown in Fig. 1b. On the other hand, HIP processing after HT led to the disappearance of the pores, as shown in Fig. 1c.

XRD patterns taken on samples AB, AB+HT and AB+HT+HIP before HPT-processing are shown in Fig. 2. It is revealed that the main phase is an hcp α -Ti alloy; however, the peak that appeared at the two-theta value of 57.3° with a lattice spacing value of about 0.161 nm can be related to a bcc β -Ti secondary phase. The presence of a single reflection of the β -Ti phase with the indices 200 is attributed to the strong crystallographic texture inherent to the L-PBF. In bcc metals, $\langle 100 \rangle$ directions are the preferred growth directions during solidification, promoting a strong $\langle 100 \rangle$ fiber texture. Consequently, reflection 200 became dominant for the β -Ti phase, while the other reflections were low in intensity.

The amounts of the β -Ti phase in the different sample conditions are characterized by the integrated intensity fraction of the peaks on the diffractograms. Thus, the fractions of the β phase are 2 ± 0.4 , 9 ± 2 and $11 \pm 2\%$ in samples AB, AB+HT, and AB+HT+HIP, respectively. Therefore, it can be concluded that HT and HIP processes after AM increased the amount of the bcc phase. The partial α -to- β transformation can be attributed to the high temperatures used in both treatments and to the β -stabilizing elements V and Fe in the alloy. Although in HIP the applied temperature was higher than that in HT, the increase in the β -phase fraction is marginal. Most probably, after HIP during cooling to RT, most of the β -Ti transformed back to the α phase, thereby resulting in a similar phase composition to that after HT. It is noted that the hcp phase may contain both α and martensitic α' phases. Hereafter, the hcp phase is referred to as α phase. YSZ-related peaks were not detected in any of the XRD patterns. Similar observations have been reported in the literature for Ti64-YSZ composites processed by directed energy deposition, where YSZ nanoparticles were found to decompose during

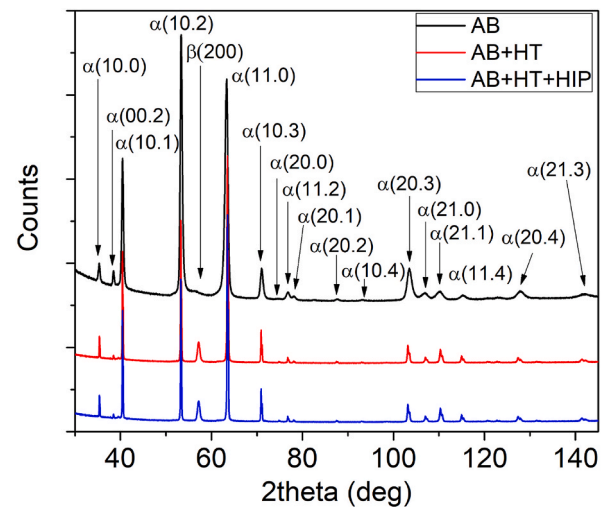


Fig. 2. XRD patterns taken on samples AB, AB+HT, and AB+HT+HIP before HPT-processing. α and β indicate hcp and bcc Ti alloy phases, respectively. The indices of reflections can be found in parentheses.

processing [44]. Specifically, it was shown that YSZ nanoparticles completely dissolved in the melt pool during laser-based AM, and upon solidification, Zr and O remain in solid solution in the Ti64 matrix due to their high solubility in both α and β phases, while Y_2O_3 nanoparticles precipitate at temperatures below 1553 K due to the low solubility of Y in Ti. If this scenario occurred in our case as well, the Zr atoms are in solid solution, and the yttria phase is present in very low amount (0.05 wt.%), which is not detectable by XRD. Therefore, the absence of the YSZ phase in our XRD and TEM analyses is consistent with this decomposition behavior. It is noted that other techniques used in this work (EBSD, TEM, EDS) also did not detect YSZ phase.

Fig. 3 shows $\langle 10\bar{1}0 \rangle$, $\langle 10\bar{1}1 \rangle$, $\langle 10\bar{1}2 \rangle$ and $\langle 11\bar{2}0 \rangle$ XRD PFs for the major hcp α phase in specimens AB, AB+HT, and AB+HT+HIP. Sample AB exhibits strong texture components of $\langle 10\bar{1}2 \rangle$ and $\langle 11\bar{2}0 \rangle$ parallel to the building direction. HT and subsequent HIP treatments did not alter the components of the crystallographic texture, as revealed by the PFs.

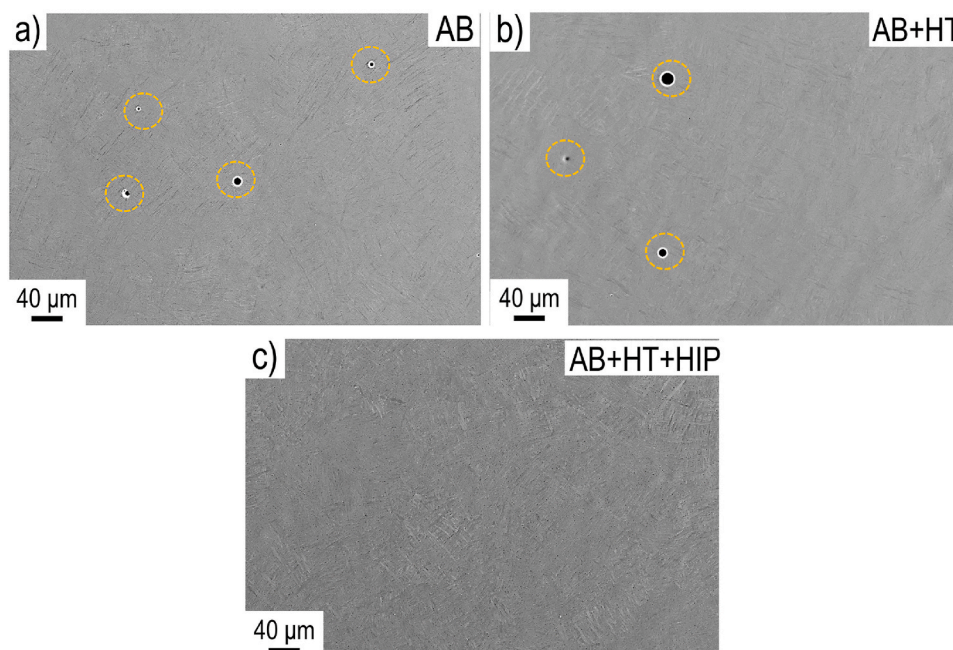


Fig. 1. SEM micrographs taken on samples a) AB, b) AB+HT, and c) AB+HT+HIP before deformation. The pores are indicated by circles in the images.

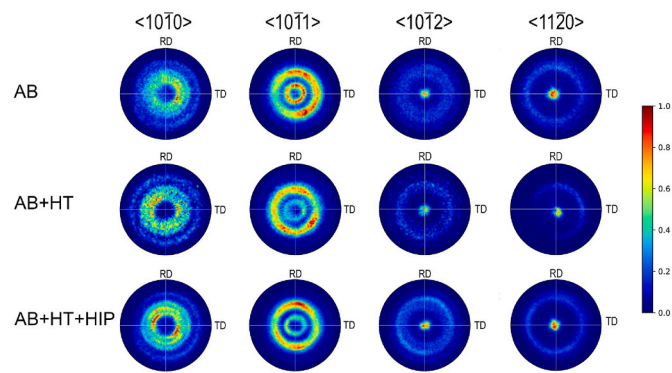


Fig. 3. XRD pole figures for the major hcp α phase in specimens AB, AB+HT, and AB+HT+HIP before deformation.

This was also confirmed by the ODFs in Fig. S1 in the Supplementary material. The overall texture strength, quantified by the texture index, decreased slightly after heat treatment, from 2.9 for sample AB to 2.6 for sample AB+HT, and increased significantly to 5.3 after HIP. Accordingly, the intensities of the corresponding XRD peaks (10.2 and 11.0) on the diffraction patterns of the three samples shown in Fig. 2 have higher relative intensities than the values characteristic for random crystallographic orientation (about 12% for both peaks).

The microstructures of samples AB, AB+HT, and AB+HT+HIP are illustrated in the EBSD images shown in Fig. 4a–c, and their characteristic parameters are listed in Table 2. All three specimens exhibit a lath microstructure. The average length and width of lath-shaped grains were measured as 4.7 μm and 0.7 μm for sample AB. HT increased these values to 5.7 μm and 0.9 μm , respectively. Subsequent HIP further increased the length and width to 7.7 μm and 1.3 μm , respectively. The fraction of low-angle grain boundaries (LAGBs) in specimen AB is 21%, which decreased to 6% in samples AB+HT and AB+HT+HIP. The change in the average GOS during HT and HIP is less pronounced; it decreased from 2.6 to 1.9–2.0°.

The decrease in LAGBs and GOS in specimens AB+HT and AB+HT+HIP may be due to structural relaxation; therefore, the dislocation density in the three samples was determined by XLP. As an example, Fig. 5 shows the CMWP fitting of the XRD pattern taken on the

Table 2

Characteristic parameters of the microstructure for samples AB, AB+HT, and AB+HT+HIP before and after deformation at low strains. The strain values of $\epsilon = 0.08$ and 1.38 correspond to the compression in the HPT facility and the deformation close to the center of the disc processed by $\frac{1}{2}$ turn of HPT. The relative errors of the listed parameters are about 10%. LAGB: low-angle grain boundary. Values in parentheses are the standard deviations (SD).

Sample	Average length (μm)	Average width (μm)	LAGB fraction (%)	Average GOS ($^\circ$)
AB	4.7 (5.4)	0.7 (0.4)	21	2.6
AB, $\epsilon = 0.08$	4.1 (4.7)	0.5 (0.4)	31	2.2
AB, $\epsilon = 1.38$	4.2 (4.7)	0.6 (0.4)	32	2.9
AB+HT	5.7 (5.5)	0.9 (0.6)	6	2.0
AB+HT, $\epsilon = 0.08$	6.4 (6.7)	1.1 (1.1)	7	2.1
AB+HT, $\epsilon = 1.38$	3.1 (2.7)	0.7 (0.4)	12	3.0
AB+HT+HIP	7.7 (6.8)	1.3 (1.2)	6	1.9
AB+HT+HIP, $\epsilon = 0.08$	7.1 (6.6)	1.1 (0.8)	9	2.2
AB+HT+HIP, $\epsilon = 1.38$	3.7 (3.1)	0.8 (0.4)	11	2.9

AB+HT sample. Specimen AB contains a high dislocation density of $(34 \pm 4) \times 10^{14} \text{ m}^{-2}$. These dislocations may be introduced by the stresses caused by the temperature gradient developed during laser scanning in AM processing. Subsequent HT resulted in a reduced density of dislocations to $(2.6 \pm 0.3) \times 10^{14} \text{ m}^{-2}$, while additional HIP did not yield further reduction in the dislocation density. This observation is in accordance with the change of the LAGB fraction and the average GOS value observed by EBSD. The crystallite size obtained by the CMWP fitting for sample AB is $79 \pm 9 \text{ nm}$. This value is much lower than the length and width determined by EBSD ($\sim 4.7 \mu\text{m}$ and $\sim 0.7 \mu\text{m}$), which suggests a hierarchical microstructure in the AM material. Namely, the grains are fragmented into subgrains and/or dislocation cells, which scatter X-rays incoherently due to the small misorientation between them. Therefore, XLP measures the sizes of these subgrains and cells, which are much smaller than the dimensions of grains bounded by high-angle grain boundaries (HAGBs). Similar observations have been made for other AM-processed metallic materials [54,55]. For samples AB+HT and AB+HT+HIP, the crystallite size was approximately 1000 nm, which is close to the detection limit of the XLP method. Therefore, these values are used only for qualitative comparison. The coarsening of

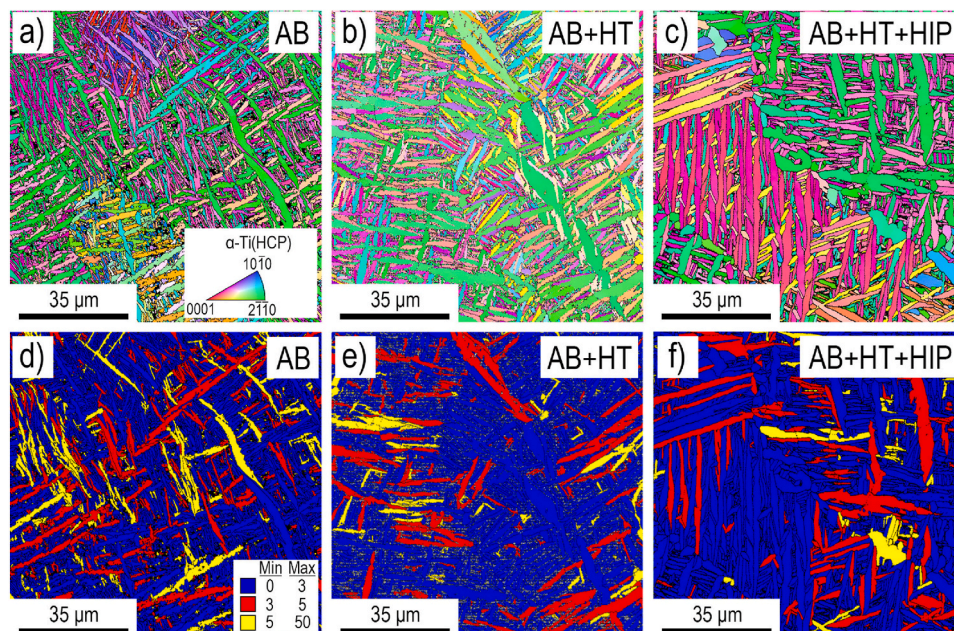


Fig. 4. IPF (a–c) and GOS (d–f) maps for samples AB, AB+HT, and AB+HT+HIP before HPT-processing.

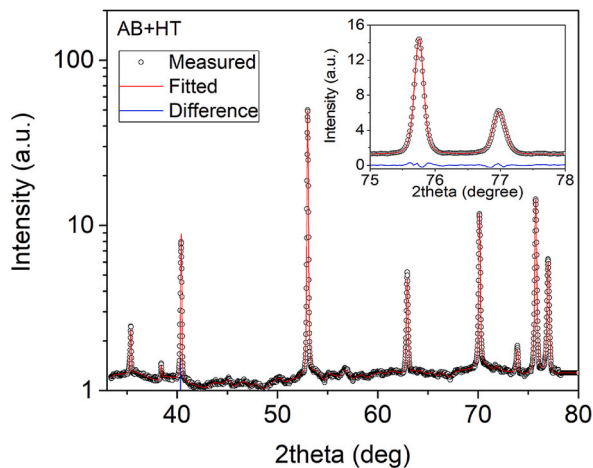


Fig. 5. CMWP fitting on the diffraction pattern taken on the AB+HT sample before deformation. The open circles and the red solid line represent the measured and fitted diffractograms, respectively. The intensity is plotted on a logarithmic scale. The inset shows a smaller part of the fitted pattern at higher magnification, with a linear intensity scale to better estimate the quality of the fit. In the inset, the difference between the measured and fitted patterns is shown by the blue curve.

crystallites is a part of the structural relaxation caused by the high temperatures applied during HT and HIP.

3.2. Microstructure evolution with increasing strain up to 208

The lowest strain (0.08) by HPT was imposed by compression under 6 GPa on all three samples. These deformed samples were studied by EBSD, and the corresponding IPF a)-c) and GOS maps d)-f) are shown in Fig. 6. The microstructural parameters obtained by the analysis of the maps are listed in Table 2. Measured dislocation density (a), crystallite size (b), and β -phase fraction (c) versus equivalent strain are summarized in Fig. 7 for samples AB, AB+HT, and AB+HT+HIP.

Considering the relative errors of $\sim 10\%$ in these estimated parameters, the present EBSD analysis reveals minor changes in the microstructure at the compression stage of HPT. In addition, the dislocation

density measured by XLPA increased with compression in all three samples. For instance, in specimen AB the dislocation density increased from $(34 \pm 4) \times 10^{14} \text{ m}^{-2}$ to $(59 \pm 7) \times 10^{14} \text{ m}^{-2}$, and in samples HT and AB+HT+HIP the values increased from 2.5 to $2.6 \times 10^{14} \text{ m}^{-2}$ to 12 – $14 \times 10^{14} \text{ m}^{-2}$ during compression. These changes are shown in Fig. 7b, where the dislocation density versus equivalent strain is plotted. The lack of observable changes in the EBSD images suggests that, during compression up to a strain of 0.08, statistically stored dislocations formed and did not cause considerable misorientations within the grains. In sample AB, the crystallite size decreased from 79 ± 9 to 51 ± 6 nm due to compression by HPT (see Fig. 7d). Similar quantitative values are not reported for the specimens AB+HT and AB+HT+HIP, as their crystallite sizes remain near the detection limit of ~ 1000 nm, suggesting reliable quantification is uncertain. Accordingly, these values are used only for qualitative comparison. Fig. 7f reveals that the β -phase fraction determined by XRD did not change considerably during compression up to a strain of 0.08.

After the initial compression, the samples were torsionally deformed in the HPT facility. The effect of the lowest number of torsion of $\frac{1}{2}$ turn, which provides an equivalent strain of 1.38, on the microstructure was studied near the centers of the disc specimens. Fig. 8 shows the IPF a)-c) and GOS maps d)-f) obtained by EBSD for the three samples under the deformation state. The corresponding microstructural parameters are listed in Table 2. In the AB sample, grain length and width remained essentially unchanged, whereas in AB+HT and AB+HT+HIP, both dimensions decreased as strain increased from 0.08 to 1.38. At the same time, in both AB+HT and AB+HT+HIP samples, the LAGB fraction and the average GOS increased as strain increased from 0.08 to 1.38. For specimen AB, these changes are less pronounced or negligible since the fine and distorted microstructure existed even in the initial state. This observation is in accordance with XLPA results, which reveal that with a strain increase from 0.08 to 1.38, the dislocation density increased and the crystallite size decreased only slightly in sample AB, as shown in Fig. 7b and d. On the other hand, in specimens AB+HT and AB+HT+HIP, the dislocation density increased by a factor of 2–3, while the crystallite size reduced considerably between strains of 0.08 and 1.38. A further increase in strain yielded an additional enhancement of the dislocation density and a decrease in the crystallite size until saturation, which occurred at the equivalent strain of about 26. The saturation values of the dislocation density and the crystallite size were

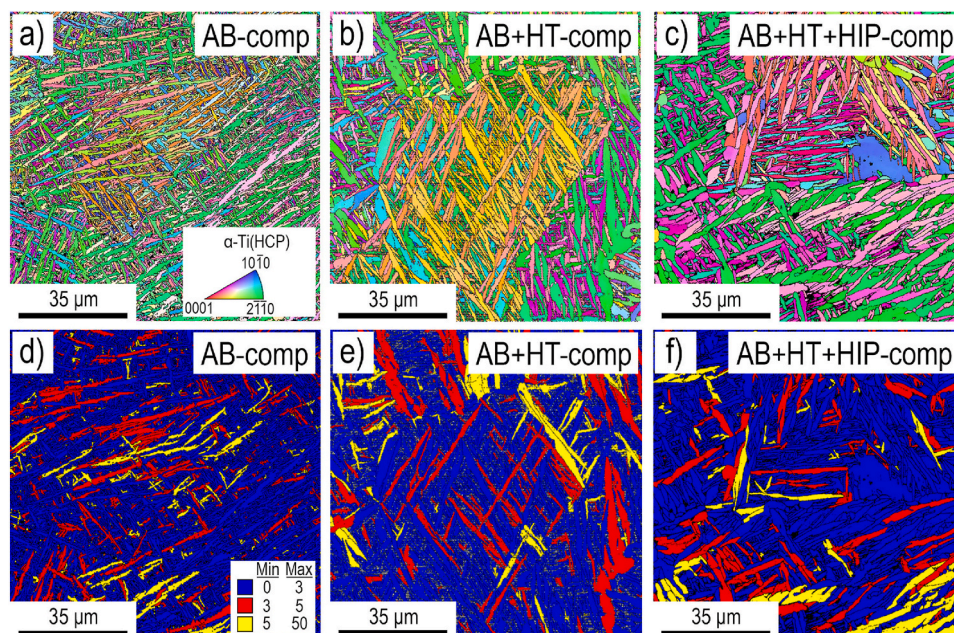


Fig. 6. IPF (a-c) and GOS (d-f) maps for samples AB, AB+HT, and AB+HT+HIP after compression in the HPT facility (equivalent strain: 0.08).

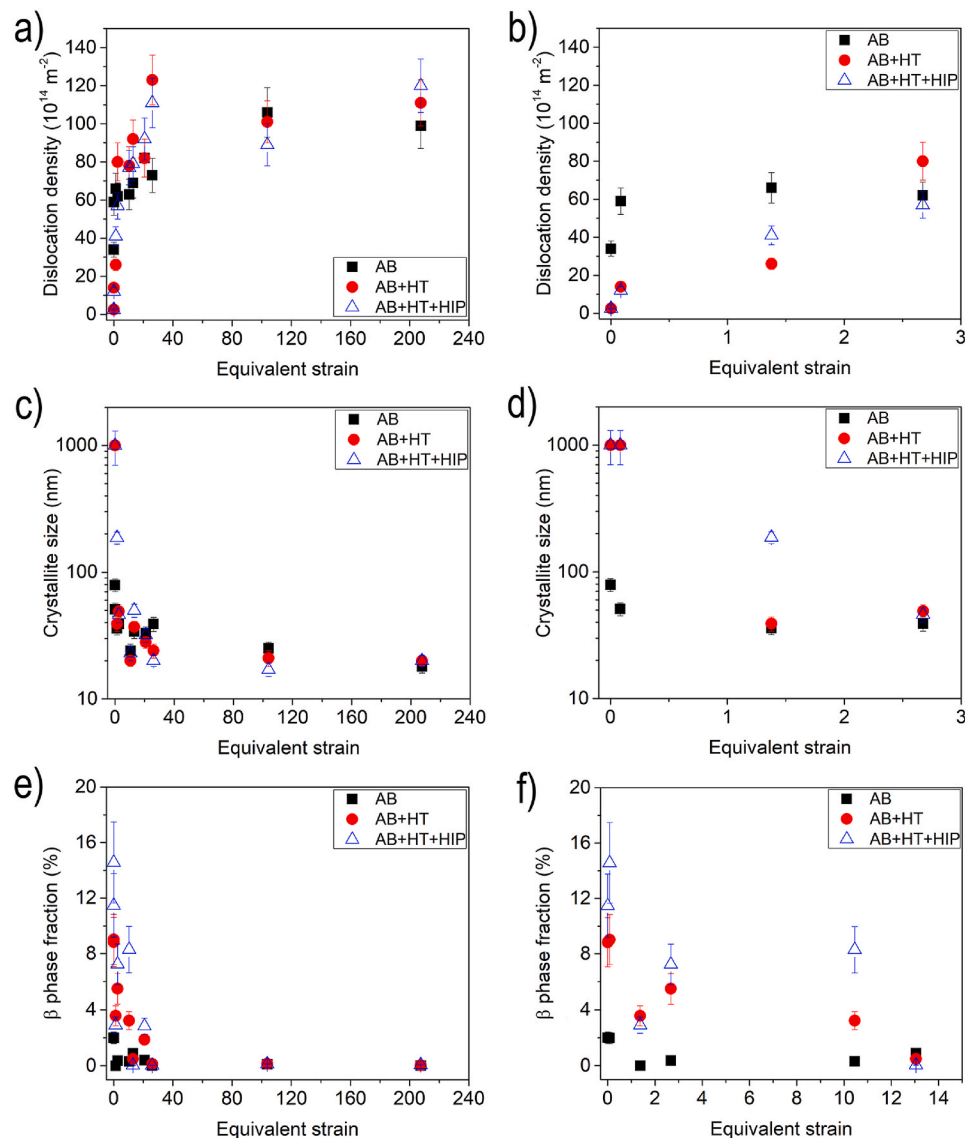


Fig. 7. Dislocation density (a, b), crystallite size (c, d), and β -phase fraction (e, f) versus the equivalent strain for samples AB, AB+HT, and AB+HT+HIP. Panels (b, d, f) show the low strain region. The crystallite size is plotted on a logarithmic scale due to the orders-of-magnitude difference between its values in the initial state and after SPD.

$\sim 100\text{--}120 \times 10^{14} \text{ m}^{-2}$ and $\sim 20 \text{ nm}$, respectively, for all three samples. No additional change in the microstructure was observed up to the highest studied equivalent strain of 208, corresponding to the strain at the disc edge for 10 turns of HPT.

Fig. 7e shows the evolution of the β -phase fraction as a function of the equivalent strain. With increasing strain, the secondary phase fraction decreased, and above a strain of ~ 26 , the β phase almost completely transformed into α -Ti. XRD PFs are shown in Fig. 9 for the major hcp α phase in specimens AB, AB+HT, and AB+HT+HIP after 10 turns of HPT. Besides the microstructure and the phase content, the crystallographic texture also changed significantly due to SPD via HPT. Although the three initial samples have strong $\langle 10\bar{1}2 \rangle$ and $\langle 11\bar{2}0 \rangle$ texture components parallel to the building direction, after 10 turns of HPT, the orientation is more randomly distributed and only a weak $\langle 10\bar{1}1 \rangle$ texture component is observed parallel to the former building direction in the IPFs of Fig. 8. However, ODF analysis (see Fig. S2 in the Supplementary material) revealed the presence of both weak $\langle 10\bar{1}1 \rangle$ and $\langle 10\bar{1}2 \rangle$ fiber components, indicating that the $\langle 10\bar{1}2 \rangle$ component is too weak to produce a distinct maximum in the corresponding PF. Moreover, the texture index confirms the weak texture, with values close to that of a random

orientation distribution (1.0): 1.42, 1.46 and 1.2 for samples AB, AB+HT, and AB+HT+HIP.

To complement the XLPAs study of the microstructure at very high strains, the disk edge processed by 10 turns of HPT corresponding to the equivalent strain of ~ 208 was investigated by TEM. Fig. 10a shows a bright-field (BF) TEM micrograph of the microstructure developed in sample AB at a strain of ~ 208 . The corresponding SAED pattern is presented in Fig. 10b, which confirms that the material has a single-phase hcp structure. The black circles indicate the segments of the diffraction rings that were used for obtaining the dark-field (DF) images shown in Fig. 10c and d. These micrographs reveal that the material contains ultrafine, elongated grains. The distribution of the length and width of the grains are shown in Fig. S3 in the Supplementary material. The mean length and width of grains were measured as 203 (92) nm and 61 (24) nm, respectively. The values in the parentheses show the standard deviation. The grain dimensions determined by TEM, together with the crystallite size and the dislocation density obtained by XLPAs for the samples deformed to the highest applied strain of ~ 208 are listed in Table 3. Inside the grains, bright spots are visible in the DF images, which scatter electrons mostly to the selected part of the SAED pattern.

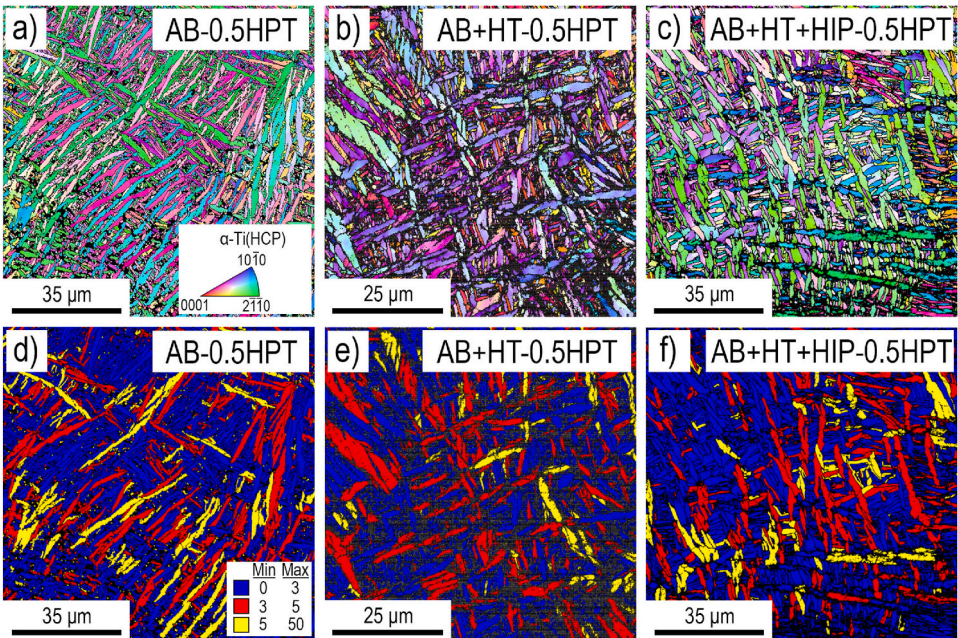


Fig. 8. IPF (a-c) and GOS (d-f) maps for samples AB, AB+HT, and AB+HT+HIP taken in the center of the discs processed by ½ turn of HPT (equivalent strain: 1.38).

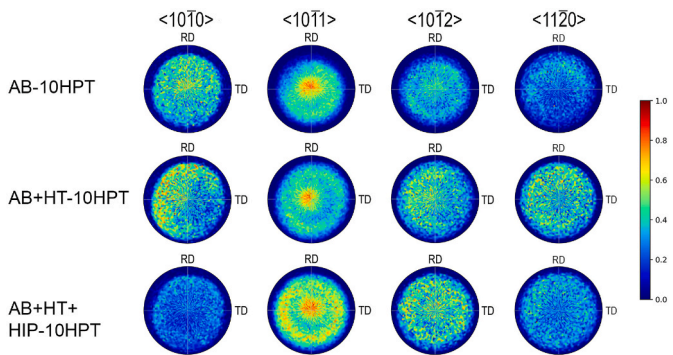


Fig. 9. XRD PFs for the major hcp α phase in specimens AB, AB+HT, and AB+HT+HIP after 10 turns of HPT.

The intensity scattered by the surrounding darker volumes to this part of the diffraction pattern is lower due to their slightly different crystallographic orientations. Therefore, the bright spots can be considered as subgrains in the microstructure. The area-weighted mean size of these subgrains (~ 20 nm) agrees well with the area-weighted mean crystallite size obtained by XLP (see Table 3), suggesting that the crystallites correspond to the subgrains in this HPT-processed sample, in accordance with previous studies on other SPD-processed materials [53,56]. This agreement between the crystallite and subgrain sizes is valid for all three samples deformed up to the highest strain in this study.

Fig. 11a shows an HAADF image of sample AB deformed up to a strain of ~ 208 . A higher-magnification portion of this micrograph is shown in Fig. 11b, which also confirms the elongated shape of the grains, with a width of about 30 nm. The corresponding element maps in Fig. 11c–f reveal that the long parallel grain boundaries (GBs) are enriched in V and Fe at the expense of Ti. The chemical compositions at the GBs and in the grain interiors obtained by TEM-EDS are listed in Table 4. Since the V/Fe-enriched GB regions are very narrow, their composition determined by EDS is uncertain and the effect of this segregation on the chemical composition of the grain interior cannot be detected.

Fig. 12a and b show a BF-TEM micrograph and the corresponding SAED pattern obtained from sample AB+HT at the highest applied strain

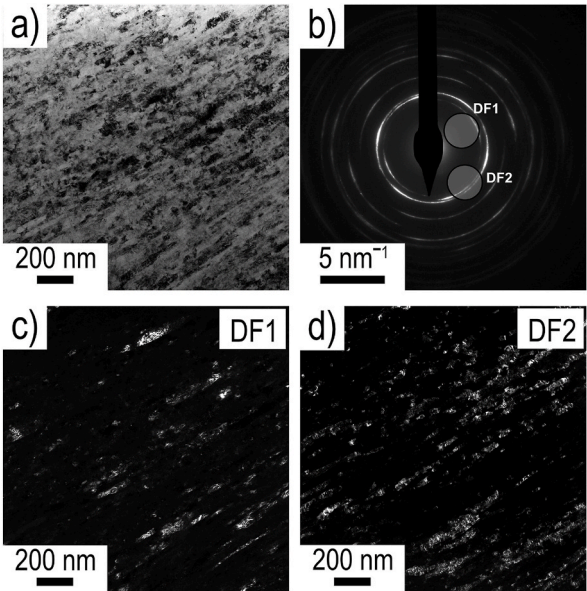


Fig. 10. TEM images obtained for sample AB at the edge of the disc processed by 10 turns of HPT (equivalent strain: ~ 208). (a) BF-TEM micrograph. (b) SAED pattern taken on the area shown in (a). The DF images shown in (c) and (d) were obtained using the diffraction ring parts marked by black circles in (b).

Table 3

The grain dimensions determined by TEM, together with the crystallite size and the dislocation density obtained by XLP for the samples deformed to the highest applied strain of ~ 208 . The values in parentheses next to the grain dimensions indicate the standard deviation.

Sample	Average length (nm)	Average width (nm)	Crystallite size (nm)	Dislocation density (10^{14} m^{-2})
AB	203 (92)	61 (24)	18 ± 2	99 ± 13
AB+HT	261 (227)	93 (68)	20 ± 2	110 ± 16
AB+HT+HIP	212 (132)	68 (29)	20 ± 2	120 ± 18

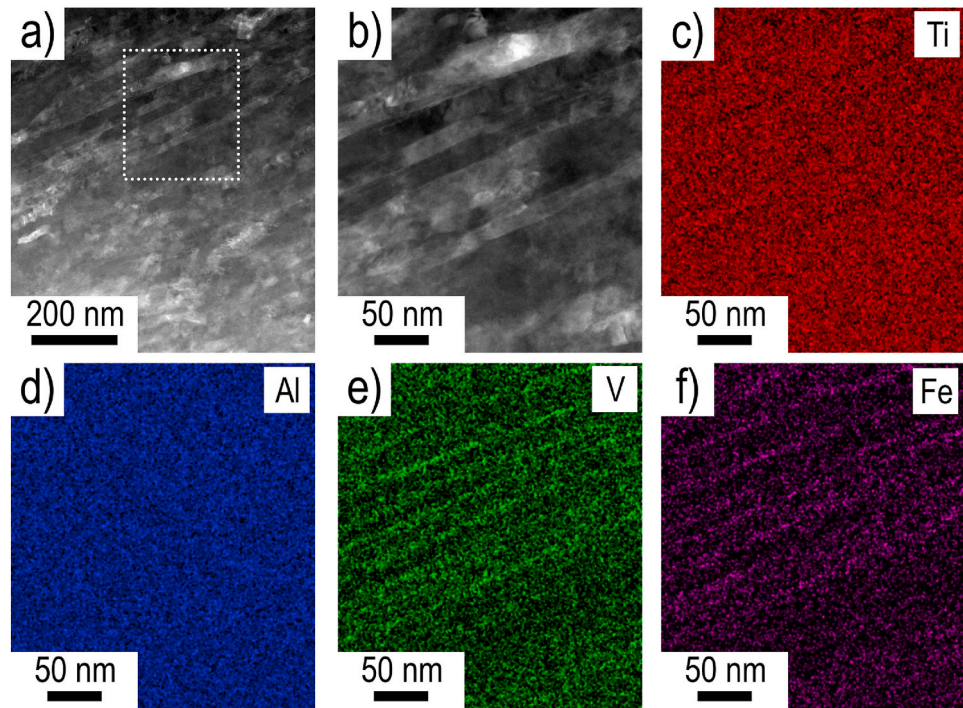


Fig. 11. (a) HAADF image taken on specimen AB at the edge of the disc processed by 10 turns of HPT (equivalent strain: ~ 208). The area marked by the white rectangle is shown at higher magnification in (b). The corresponding elemental maps for Ti (c), Al (d), V (e) and Fe (f) are also shown.

Table 4
The average elemental concentrations for samples AB, AB+HT, and AB+HT+HIP after HPT-processing (equivalent strain: ~ 208), as determined by TEM-EDS. The same is presented for the GB regions and the grain interiors. The relative errors of the latter values are similar to those given for the average concentration values.

Sample	Element	Average (at.%)	GB (at.%)	Grain interior (at.%)
AB	Ti	86.0 ± 2.1	81.6	86.1
	Al	10.1 ± 1.5	10.0	10.0
	V	3.7 ± 0.7	7.5	3.7
	Fe	0.2 ± 0.1	0.9	0.2
AB+HT	Ti	85.8 ± 2.1	80.7	86.4
	Al	10.3 ± 1.5	6.0	11.0
	V	3.7 ± 0.7	12.0	2.5
	Fe	0.2 ± 0.1	1.3	0.1
AB+HT+HIP	Ti	85.4 ± 2.1	66.0	86.0
	Al	10.3 ± 1.5	4.0	11.0
	V	4.0 ± 0.8	27.0	2.9
	Fe	0.3 ± 0.1	3.0	0.1

of ~ 208 . Only hcp diffraction rings are visible in the SAED pattern. The DF-TEM image in Fig. 12c was taken using the part of the diffraction pattern marked by the black circle in Fig. 12b. The length and the width of the grains were determined as 261(227) nm and 93(68) nm, respectively. The values in the parentheses show the standard deviation. Compared with the AB sample, both the grain length and width were larger in the AB+HT sample.

Fig. 13a shows an HAADF image of sample AB+HT deformed to a strain of ~ 208 . The area indicated by the white rectangle is shown at higher magnification in Fig. 13b while the corresponding EDS elemental maps are presented in Fig. 13c–f. Enrichment of GBs in V and Fe can also be observed, similar to sample AB. On the other hand, in specimen AB+HT, the concentrations of these elements in the enriched GB regions are higher as revealed by the data listed in Table 3. Moreover, the depletion of Al and Ti in these volumes is also evident. In addition, in specimen AB+HT, the V/Fe-rich lines are thicker and their spacing is wider than in sample AB.

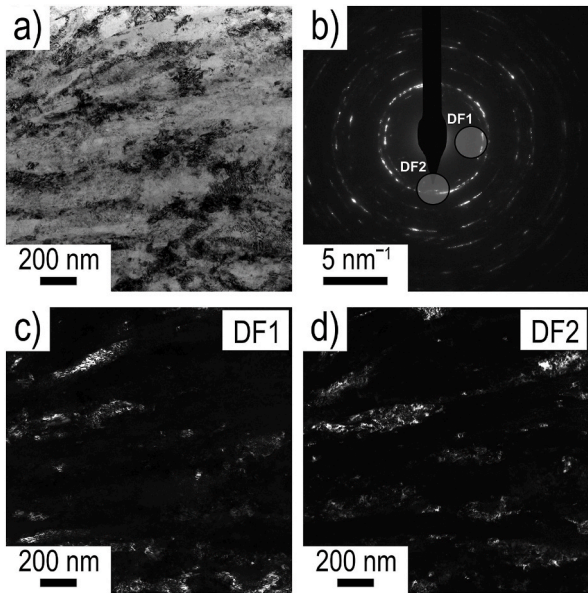


Fig. 12. TEM images obtained for sample AB+HT at the edge of the disc processed by 10 turns of HPT (equivalent strain: ~ 208). (a) BF-TEM micrograph. (b) SAED pattern taken on the area shown in (a). The DF images shown in (c) and (d) were obtained using the diffraction ring parts marked by black circles in (b).

The microstructure in sample AB+HT+HIP after deformation up to the strain of ~ 208 is illustrated in the BF-TEM image in Fig. 14a. The corresponding SAED pattern is shown in Fig. 14b, which suggests that the structure is fully hcp. The grain size was determined from the DF micrographs shown in Fig. 14c and d, which were obtained from the SAED regions indicated by the black circles in Fig. 14b. The length and width of the grains were 212(132) nm and 68(29) nm, respectively. The values in the parentheses show the standard deviation.

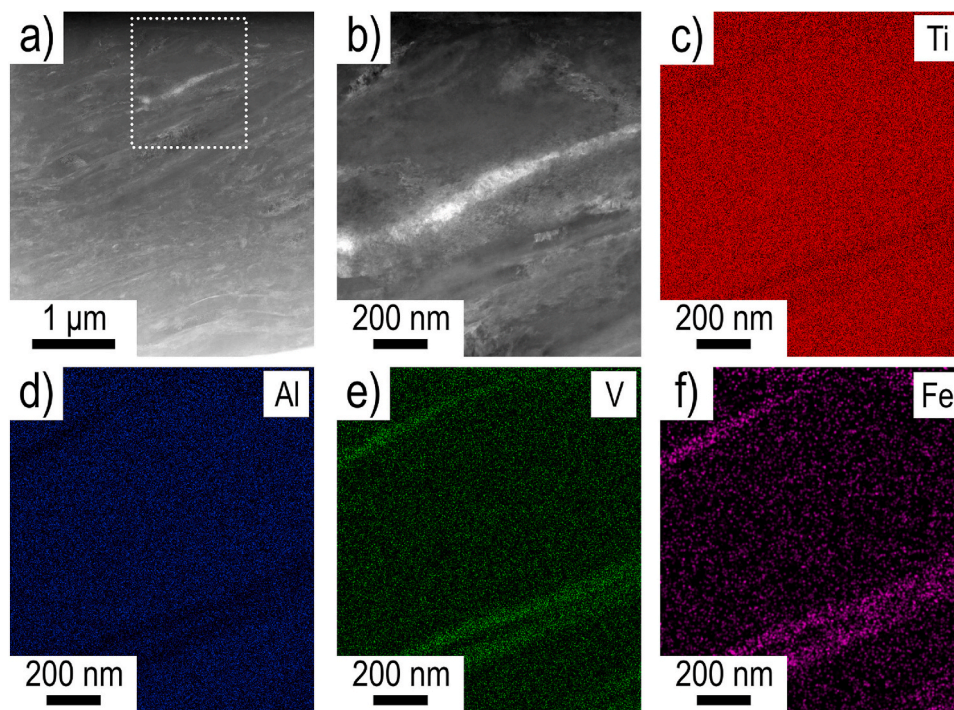


Fig. 13. HAADF image taken on specimen AB+HT at the edge of the disc processed by 10 turns of HPT (equivalent strain: ~ 208). The area marked by the white rectangle is shown at higher magnification in (b). The corresponding elemental maps for Ti (c), Al (d), V (e), and Fe (f) are also shown.

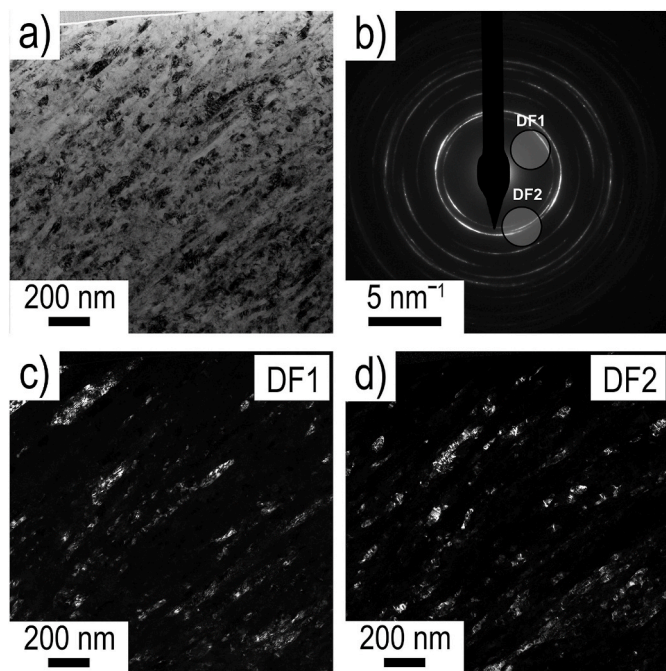


Fig. 14. TEM images obtained for sample AB+HT+HIP at the edge of the disc processed by 10 turns of HPT (equivalent strain: ~ 208). (a) BF-TEM micrograph. (b) SAED pattern taken on the area shown in (a). The DF images shown in (c) and (d) were obtained using the diffraction ring parts marked by black circles in (b).

The microstructure of the AB+HT+HIP specimen deformed by HPT up to a strain of ~ 208 is also shown in the HAADF image in Fig. 15a. The area indicated by the white rectangle is shown in Fig. 15b at higher magnification. The corresponding element maps in Fig. 15c–f reveal similar chemical inhomogeneities as have already been observed for the

other two samples. Namely, the GBs of the elongated grains are enriched in V and Fe and depleted of Al and Ti. On the other hand, the deviation of the average elemental concentrations is much higher than for the other two samples. The V/Fe-rich GB regions are thicker and more densely spaced than in specimen AB, but thinner and less densely spaced than in sample AB+HT.

3.3. Evolution of hardness due to deformation

The hardness values for the initial AB, AB+HT, and AB+HT+HIP specimens were 4470 ± 130 , 4100 ± 120 , and 4070 ± 120 MPa, respectively. The lower hardness values in the latter two samples are attributed to lower dislocation density and larger grain size resulting from the high temperatures used in the HT and HIP processes. Compression in the HPT facility yielded no significant change in hardness, as shown in Fig. 16a–c, although the dislocation density considerably increased, as seen in Fig. 7b. This apparent contradiction can be explained by the deformation caused by the hardness testing itself. Namely, when the Vickers tip is pressed into the material, plastic deformation occurs, which corresponds to an average strain of 0.08 [57]. Therefore, the hardness value reflects the mechanical strength of a material deformed additionally to a strain of 0.08 rather than the initial state before compression by HPT. Thus, if the strain hardening of a material is not very high in the strain range between 0.08 and 0.16, the hardness values measured on the initial state and the sample deformed to a strain of 0.08 are only slightly different.

The hardness versus the distance from the disc center is plotted for different numbers of HPT turns in Fig. 16a–c for samples AB, AB+HT, and AB+HT+HIP. The hardness increases with increasing distance from the disc center due to the higher applied shear strain, and this effect remains significant even for high numbers of turns. Since the XLPA study was performed near the center and edge of the discs, hardness was analyzed only for these regions. First, the hardness values obtained for the same distances in the two parts of the disc diameter were averaged and then plotted as a function of the equivalent strain in Fig. 16d. The hardness increases when the strain is enhanced during HPT and

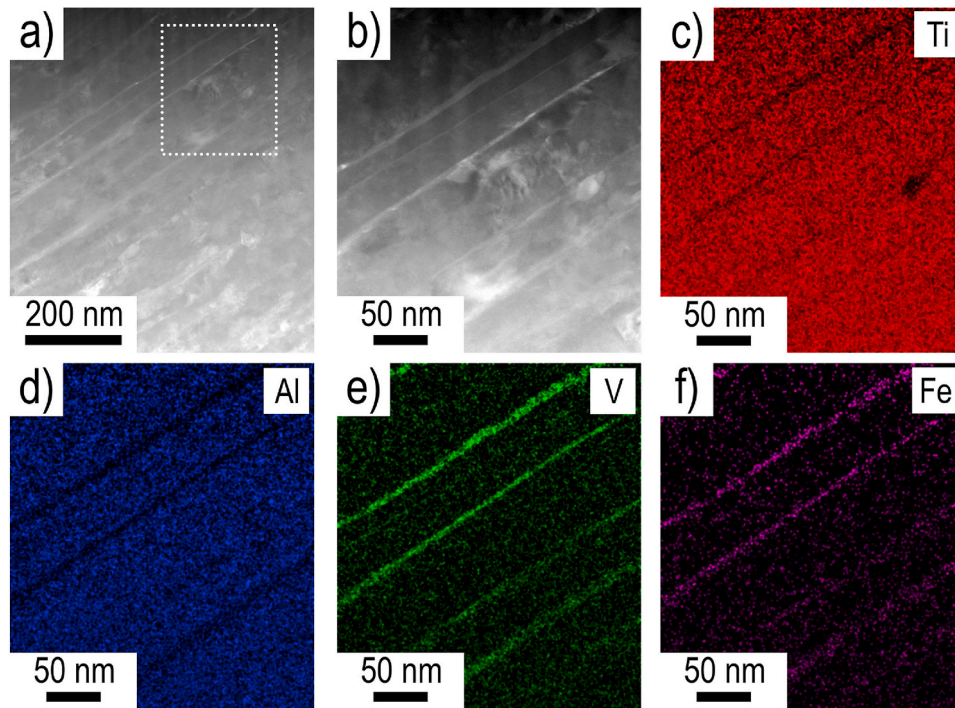


Fig. 15. HAADF image taken on specimen AB+HT+HIP at the edge of the disc processed by 10 turns of HPT (equivalent strain: ~ 208). The area marked by the white rectangle is shown at higher magnification in (b). The corresponding elemental maps for Ti (c), Al (d), V (e), and Fe (f) are also shown.

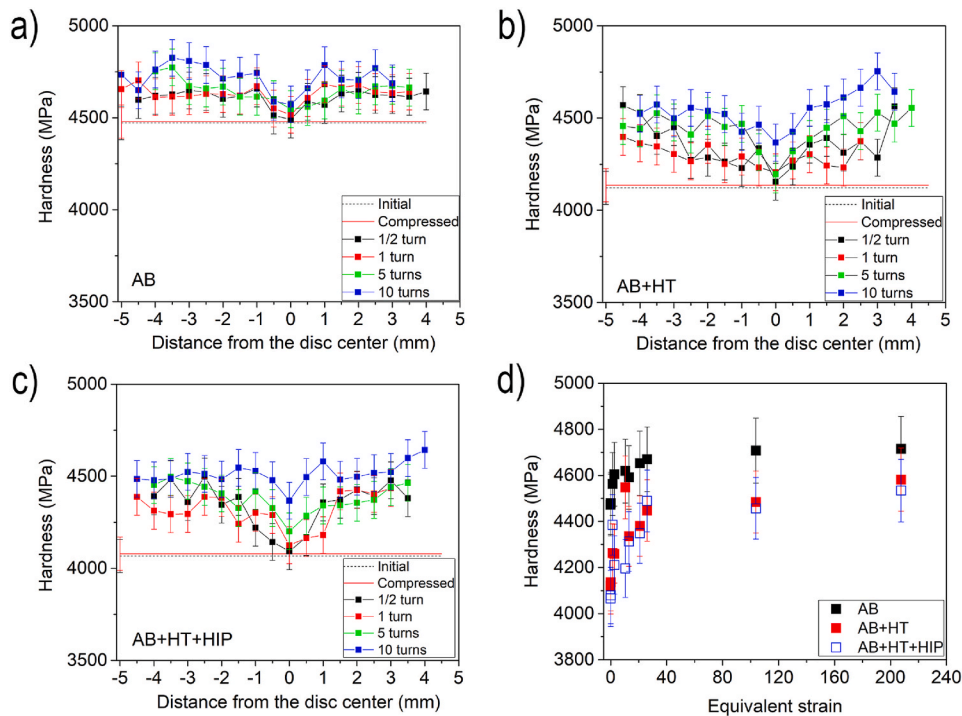


Fig. 16. The hardness as a function of the distance from the HPT disc center for samples AB (a), AB+HT (b), and AB+HT+HIP (c). The hardness values for the initial specimens and the samples compressed in the HPT facility are also shown. The hardness versus the equivalent strain for the three samples is shown in (d).

saturates at a strain of about 26. The saturation value is as high as 4600–4700 MPa. However, it should be noted that the increase in hardness caused by HPT processing up to a strain of ~ 208 is very moderate. Namely, the relative hardness increase varies only between 5 and 12% despite the very large increase of the dislocation density and decrease of the grain size. The lower bound (5%) of this range is obtained for sample

AB, while the upper bound is valid for specimens AB+HT and AB+HT+HIP. The reasons for this very low increase in hardness will be discussed in the next section.

4. Discussion

4.1. Effect of AM-processing on the microstructure evolution during SPD

In accordance with former studies on AM-processed Ti–6Al–4V alloys, e.g., Ref. [46], the samples manufactured by L-PBF have a microstructure consisting of a lamellar α -Ti phase and a secondary β phase. Previous experiments on the same materials have revealed that the grains of the latter phase were equiaxial, with sizes between 200 nm and 1 μm , and were located between the α lamellas [58,59]. The size of the β grains was smaller than the width (0.5–2 μm) and the length (1–5 μm) of the α lamellae. A previous study [60] indicated that globular β grains formed between α -phase laths with very differently oriented longitudinal axes. In addition, thin lamellar regions with β structure may also develop at the boundaries of neighboring α grains with parallel longitudinal axes. In contrast, the as-cast Ti–6Al–4V alloys typically consist of equiaxed α grains and lamellar $\alpha + \beta$ dual-phase regions due to the slow cooling rate after casting [61–63]. The grain sizes of these materials usually vary between 7 and 10 μm , whereas the microstructures are coarser than those formed using AM techniques. A coarse and mostly α phase microstructure with a grain size of 15 μm was also obtained when a Ti–6Al–4V powder was consolidated by spark plasma sintering [64]. Furthermore, the dislocation density in the present AM-processed sample AB is significantly higher ($\sim 34 \times 10^{14} \text{ m}^{-2}$) than in the as-cast and wrought Ti–6Al–4V alloy (10^{11} m^{-2} [65]) due to the thermal stresses developed as a result of the large temperature gradients during laser scanning in the L-PBF process. Moreover, the dislocation density is very sensitive to the AM processing route. It has been shown that electron beam PBF (EB-PBF) yielded one order of magnitude lower dislocation density ($1\text{--}2 \times 10^{14} \text{ m}^{-2}$) than L-PBF ($\sim 43 \times 10^{14} \text{ m}^{-2}$) in a Ti–6Al–4V alloy [66,67]. This difference can also be explained by the much lower temperature gradients in EB-PBF compared to L-PBF. During the former procedure, the powder bed is preheated to high temperatures of 600–1100 $^{\circ}\text{C}$ depending on the material and the entire build chamber is kept hot. On the other hand, in L-PBF, the powder bed is typically near room temperature or sometimes mildly preheated only to ~ 200 $^{\circ}\text{C}$, resulting in very steep thermal gradients. In Ti–6Al–4V alloy processed by direct metal laser sintering (DMLS), the observed dislocation density of $\sim 38 \times 10^{14} \text{ m}^{-2}$ [68] was very similar to that observed for L-PBF in this study.

The present analysis also revealed that L-PBF yielded a considerable crystallographic texture in the AM-processed samples, with components of $\langle 10\bar{1}2 \rangle$ and $\langle 11\bar{2}0 \rangle$. A previous study [69] showed that during the L-PBF process, the Ti–6Al–4V alloy solidified in a bcc structure (β phase), with $\langle 001 \rangle$ texture parallel to the building direction. Upon cooling, the β phase transformed to α' phase following the Burgers orientation relationship (BOR), where $\{110\}\beta \parallel \{0001\}\alpha'$ and the $\langle 111 \rangle\beta \parallel \langle 11\bar{2}0 \rangle\alpha'$ [70]. A single β grain can transform into 12 different α' variants during cooling. If all variants form with equal probability, then the texture is weak. The α' variant type depends on the prior β grain orientation, the annealing temperature, and the cooling rate [71,72]. In accordance with the BOR, the $\langle 10\bar{1}2 \rangle$ fiber texture of the α' phase observed in this study could form from the $\langle 001 \rangle$ texture of the prior β phase. BOR also suggests that the $\langle 11\bar{2}0 \rangle$ texture component of the present α' phase might develop from the $\langle 111 \rangle$ texture component of the prior β phase [73]. Indeed, a previous study demonstrated that the prior β phase exhibited both $\langle 111 \rangle$ and $\langle 100 \rangle$ orientations in an AM-processed Ti–6Al–4V alloy [70]. It is worth noting that similar α' phase texture components were reported in Ti–6Al–4V alloys produced by direct metal deposition and electron beam melting techniques [73, 74]. On the other hand, an as-cast Ti–6Al–4V alloy showed ND|| $\langle 0001 \rangle\alpha'$ texture, which was caused by the transformation of β phase with $\langle 110 \rangle$ texture to α' according to the BOR [75]. The texture strength evolved differently during subsequent annealing and HIP after AM processing. While the texture index slightly decreased from 2.9 to

2.6 after heat treatment, it increased significantly after HIP to 5.3. The slight decrease due to HT can be attributed to the decomposition of α' martensite to α and β phases, which introduces new orientations and slightly weakens the existing texture. The higher texture index in the AB+HT+HIP sample can be explained by the nature of the phase transformation during heating to and cooling from the high-temperature HIP process at 920 $^{\circ}\text{C}$. Namely, upon heating, the β -phase fraction increased mostly by growing the β grains existing after HT. At 920 $^{\circ}\text{C}$, the equilibrium fraction of the primary α phase in Ti–6Al–4V alloy decreased to about 30–40% [76]. During furnace cooling, the hcp phase fraction increased rather by growing of the α grains existing at 920 $^{\circ}\text{C}$ than the nucleation of new grains from the β phase [77]. This effect can cause a sharpening of the initial crystallographic texture due to HIP. The texture enhancement observed in this study is consistent with that reported for EBM-processed Ti–6Al–4V alloy subjected to a subsequent HIP treatment [78].

Fig. 4 and Table 2 show that both HT and HT+HIP post-treatments caused an increase in length and width of the main α -Ti phase lamellae and a decrease in the dislocation density. Simultaneously, the β -phase fraction increased from about 2 to 9–11%. These changes in the microstructure were caused by the high temperatures applied in HT and HIP post-treatments. The β -phase contents in samples AB+HT and AB+HT+HIP are close to the equilibrium value at RT of $\sim 5\text{--}10\%$ [79–81]. Therefore, in sample AB, the β phase fraction is lower than the equilibrium value. In general, the L-PBF-processed microstructure is far from equilibrium, which is also indicated by the very high dislocation density of $\sim 34 \times 10^{14} \text{ m}^{-2}$. The high temperature and low cooling rate used in HT and HIP processes moved the microstructure closer to thermal equilibrium, as a result, the grain size increased, the dislocation density decreased, and the β -phase fraction approached the equilibrium values. In contrast, this study revealed that the crystallographic texture was not influenced by the post-processing HT and HIP.

Although the differences in the initial states caused different evolutions of the microstructure at relatively low strains, a saturation with the same microstructural parameter values in samples AB, AB+HT, and AB+HT+HIP occurred at the equivalent strain of about 26. Thus, HPT-processing yielded a saturated microstructure with a crystallite size of 20 nm and a dislocation density in the range of $100\text{--}120 \times 10^{14} \text{ m}^{-2}$. After HPT at an equivalent strain of approximately 208, the grain length and width of the three AM-processed samples decreased to 200–260 nm and 60–90 nm, respectively. These values are similar to the saturation limit typically observed in severely deformed as-cast Ti–6Al–4V alloy, despite differences in the initial microstructures of the as-cast and the AM-processed alloys. For instance, in a Ti–6Al–4V alloy obtained by casting and subsequent HIP, HPT at RT yielded a comparable saturated crystallite size of about 25 nm and a dislocation density of $114 \times 10^{14} \text{ m}^{-2}$ [82]. Furthermore, the reported average grain width after 10 turns of HPT in the spark plasma sintered Ti–6Al–4V alloy was about 49 nm [64], which is close to our measured average grain width values (60–90 nm). The convergence of the different initial microstructures (AB, AB+HT, AB+HT+HIP) to similar microstructural parameters at high strains is a direct consequence of reaching a steady state in SPD-processing. During deformation, the continuous generation of lattice defects (dislocations, grain boundaries, vacancies) is balanced by their annihilation through recovery processes. Recrystallization may also occur, depending on the material's chemical composition and the HPT conditions (temperature, pressure, torsional rate, number of turns). Eventually, a dynamic equilibrium is established where the rate of defect production equals the rate of annihilation. The parameters of the steady-state (saturation) microstructure are basically determined by the fundamental properties of the material (e.g., melting point, stacking fault energy, atomic bond energy, crystal structure) and the conditions of SPD processing (e.g., temperature, strain rate) [83,84]. Since these materials' properties and processing conditions are identical for all three samples investigated in this study, their saturation microstructural parameters must be similar after 10 turns of HPT. However, differences in

the phase composition and the crystallographic texture before HPT may cause slightly different microstructures and hardness values after HPT-processing as revealed in former publications [62,63,85]. For instance, in Ti–6Al–4V alloy, the volume fractions of equiaxed α and lamellar $\alpha+\beta$ phases in the as-cast state influence the saturation microstructure and hardness after HPT. As the volume fraction of the lamellar $\alpha+\beta$ structure increased, the grain size in the HPT-processed material decreased. However, this effect is marginal here due to the very low initial fraction of the secondary β phase (2–11%), leading to a similar saturation microstructure during HPT.

The present TEM-EDS investigation revealed the formation of V/Fe-rich regions in the vicinity of GBs of the ultrafine hcp grains at a very high strain of ~ 208 for all three studied samples. Simultaneously, the β -phase fractions became practically zero. These changes suggest that the β phase grains were transformed to α during HPT processing and their high V and Fe contents in the prior β phase are found in these GB regions having hcp structure. This effect can be explained by the increase in the solubility limit of V and Fe in the hcp phase due to the large defect density developed during HPT. Indeed, in SPD processing, many non-equilibrium GBs are formed, where the dislocation density is high in the pile-ups. Since V and Fe atoms are much smaller than Ti or Al, the former elements can be stored with lower lattice distortions in the compressed zones of edge dislocations in the hcp Ti matrix. The decrease in the V and Fe contents in the β grains destabilizes this phase. The present results suggest that it is energetically more favorable if the V and Fe were stored in the highly deformed GB region of the hcp phase instead of the β particles. The high pressure applied during HPT also decreases the stability of the β phase relatively to the α phase. A previous study [86] showed that due to the lower atomic packing density of the bcc structure (0.68) compared to the hcp phase (0.74), the Gibbs free energy of the β phase increases more rapidly with pressure than that of the α phase. Thus, under the pressure of several GPa applied during HPT, the phase equilibrium shifts toward the denser hcp phase, resulting in the β to α transformation. It is noted that former first-principles calculations have also confirmed a thermodynamic preference for V and Fe enrichment and Al depletion on model α/α' grain boundaries [87]. The transformation from β to α phase and the corresponding solute segregation at grain boundaries may have an impact on the material properties. Since the β phase is more ductile than the α phase, a decrease in ductility is expected. On the other hand, the thermal stability of the UFG microstructure obtained by HPT may increase since the grain boundaries are enriched in alloying elements, which usually reduces boundary mobility, thereby hindering grain growth at elevated temperatures. The experimental investigation of the effect of this change in the microstructure on ductility and thermal stability is a subject of further studies.

It should be noted that a previous study found that enrichment of V and Fe at the GBs of martensitic hcp Ti grains can also occur during AM processing [60]. These V/Fe-rich regions were detected in the top layer of a Ti–6Al–4V sample produced by L-PBF. During AM processing, mostly α' martensitic laths formed, and the development of V/Fe-rich regions between them can be considered as the first step of α' decomposition. In semi-top and middle layers, the partitioning of V and Fe to lath boundaries led to the formation of α_{HME} (high in Mo_{eq}) with an hcp structure, while in the bottom layer the V/Fe-rich regions appear in the form of β phase [60]. However, in the present case, at least for the two studied materials, AB+HT and AB+HT+HIP, they were annealed at elevated temperatures of 600 and 920 °C for 2 h; therefore, the microstructures evolved toward an equilibrium state. Indeed, the β -phase fraction was close to the equilibrium value, as discussed above. Thus, a martensite decomposition was not expected during HPT processing. Furthermore, the AB sample, which initially contained a detectable amount of β phase, also exhibited complete β dissolution after HPT, confirming that this process is universally driven by the SPD itself rather than by the initial microstructure. Similar SPD-induced dissolution of the β phase in Ti–6Al–4V alloy has been observed in Refs. [86,88–92].

The spacing between the V/Fe-rich regions is higher in sample

AB+HT compared to specimens AB and AB+HT+HIP (see Figs. 11, 13 and 15), which is in accordance with the larger grain dimensions of the former material since V and Fe were segregated to the GBs. Thus, the spacing between the V/Fe-rich thin regions is the highest for specimen AB+HT. The less pronounced grain refinement in the latter sample can be attributed to the relatively weaker $\langle 10\bar{1}2 \rangle$ texture component, since this texture can facilitate extension twinning $\{10\bar{1}2\}\langle 10\bar{1}1 \rangle$ during HPT, as the twinning shear plane is parallel to the shear stress in HPT processing. Twinning can contribute to grain refinement in samples AB and AB+HT+HIP, resulting in a finer grain size at the highest applied strain compared to specimen AB+HT.

It should be noted that in the V/Fe-rich hcp regions at GBs, the lattice constants most probably differ from the values characteristic of the α grain interiors due to the different chemical compositions. As a consequence, the X-rays scattered by the GB region and the grain interior have slightly different Bragg angles, which yields a diffraction peak broadening. Moreover, in the CMWP fitting, only the peak broadening contributions caused by crystallite size and dislocations are considered. Therefore, due to the chemical inhomogeneities, the crystallite size and the dislocation density were slightly underestimated and overestimated, respectively. However, due to the low fraction of V/Fe-rich regions, this effect could not cause a large deviation from the values determined in the present study and plotted in Fig. 7.

The PFs measured on the three samples indicate that the initial $\langle 10\bar{1}2 \rangle$ and $\langle 11\bar{2}0 \rangle$ texture components were considerably weakened, and an almost random crystallographic texture developed during HPT. Only weak $\langle 10\bar{1}1 \rangle$ and remaining $\langle 10\bar{1}2 \rangle$ fiber components were observed at a strain of ~ 208 . The formation of the $\langle 10\bar{1}1 \rangle$ texture can be explained by the activation of pyramidal $\langle c+a \rangle$ slip system which becomes necessary to accommodate c-axis strains during HPT processing [93]. This explanation is supported by a former study on severe shot peening of Ti–6Al–4V alloy with an initial rolling texture $\{11\bar{2}0\}\langle 10\bar{1}0 \rangle$, where the same $\langle 10\bar{1}1 \rangle$ texture component formed at low strains [94]. In that study, the initial configuration with $\{11\bar{2}0\}$ planes perpendicular to the loading direction necessitated the activation of pyramidal $\langle c+a \rangle$ dislocations to accommodate the c-axis strain. The subsequent operation of the pyramidal slip caused the orientation of $\{10\bar{1}1\}$ planes perpendicular to the loading direction [94], and the consistent configuration was observed in the present HPT specimens. The overall weakening of the texture can be attributed to the necessary operation of different dislocation types during HPT [95,96]. In shear deformation during HPT processing, the grains tend to adapt orientations that maximize the resolved shear stress in the active slip systems, i. e., the slip planes become perpendicular to the disc normal. That is the main reason of the formation of $\{111\}$ texture in the HPT-processed face-centered cubic (fcc) alloys. On the other hand, in hcp structures the activated dislocation types are diverse, since they are basal and prismatic $\langle a \rangle$ types as well as pyramidal $\langle c+a \rangle$ slip systems. Therefore, HPT yields rather an orientation scattering than a convergence in the case of Ti alloys, i. e., the initial texture formed during AM processing is weakened.

4.2. Influence of deformation-induced microstructure change on the hardness

The HT and HIP post-treatments yielded an 8–9% decrease in hardness due to reduced dislocation density and increased grain size. In addition, the increase of the β -phase fraction during HT and HIP processing can also contribute to the observed softening, since the hardness of α/α' hcp phases in Ti–6Al–4V alloys was found to be about 20% higher than that of the bcc β phase [97]. The hardness of the as-built sample of ~ 4470 MPa is higher than the values obtained formerly on Ti–6Al–4V specimens manufactured by other methods, such as casting of 2890–4020 MPa [88,98,99]), which can be caused by the high

dislocation density and the relatively fine grain structure in the α' martensite in specimen AB. The $\langle 11\bar{2}0 \rangle$ texture component can also contribute to the high initial hardness of the as-built material, since in most of the basal and prismatic $\langle a \rangle$ type easy glide slip systems the dislocation glide is not feasible; therefore, other slip systems with considerably higher critical resolved shear stress should be activated at the onset of plastic deformation. Indeed, Table 5 shows that for four slip systems of the six basal and the prismatic $\langle a \rangle$ type ones, the Schmid factors are zero if the load is parallel to the $\langle 11\bar{2}0 \rangle$ direction. For the other texture component that appeared in sample AB of $\langle 10\bar{1}2 \rangle$, the Schmid factors are non-zero and only slightly higher or lower than those calculated for the randomly orientated hcp Ti–6Al–4V alloy. Thus, the texture component $\langle 10\bar{1}2 \rangle$ does not contribute considerably to the high hardness of specimen AB.

The Schmid factors listed in Table 5 reveal that the texture evolution from $\langle 11\bar{2}0 \rangle$ to a nearly random orientation with a weak $\langle 10\bar{1}1 \rangle$ component during HPT is expected to promote softening. Namely, the Schmid factors in four slip systems increase considerably, while for the remaining two systems the Schmid factors only slightly decrease. If we consider the other initial texture component $\langle 10\bar{1}2 \rangle$, the randomization of the crystallographic orientation during HPT yielded a large increase in the Schmid factors in two slip systems, and only a slight decrease was found in the remaining four systems. Thus, based on the overall Schmid factor analysis, the texture change induced by HPT facilitated an easier activation of the most important $\langle a \rangle$ -type slip systems. This texture softening has also been observed in previous studies on Ti and its alloys [100], and in the present case, this effect can moderate the hardening caused by grain refinement, increased dislocation density, and reduced β -phase fraction. Thus, the increase in hardness is only 5–12% during HPT of the studied AM-processed Ti–6Al–4V alloy. Softening caused by texture weakening during HPT has also been observed for other hcp metallic materials such as AM60 magnesium alloy [101]. It should be noticed that the initial texture in hcp Ti and the HPT-processing conditions (pressure, temperature, and number of turns) influence the texture evolution during HPT [93,102]. The initial texture may also affect the phase composition of Ti alloys prone to forming the ω phase during HPT, thereby indirectly influencing hardness [103]. It is also worth noting that a recent study revealed that a significant proportion of grains in hcp Ti–6Al–4V alloy deform by slip systems other than those with the highest Schmid factor [104]. Instead, slip tends to occur on systems that better accommodate the overall macroscopic shear pattern. This finding indicates that interactions with neighboring grains play an important role in determining slip system activation.

It is worth noting that the increase in hardness induced by HPT in sample AB processed by AM is lower than that previously observed for the same composition manufactured by non-AM methods. For instance, in a previous study as-cast Ti–6Al–4V alloy was cold rolled and heat treated to produce varying fractions of equiaxed α and lamellar $\alpha+\beta$ phases [63]. The hardness increased with the fraction of lamellar $\alpha+\beta$ phase. After HPT up to 10 turns, however, the hardness increase was similar between 21 and 25% regardless of the initial phase content. In another study, as-cast Ti–6Al–4V alloy was hot rolled and extruded, with

different initial microstructures achieved by annealing [62]. Subsequent HPT increased hardness by 24–33% across all microstructures. Similarly, a Ti–6Al–4V consolidated by spark plasma sintering had a predominantly α phase with minor β and showed a 28% increase in hardness after HPT [64]. Other studies on Ti–6Al–4V samples processed by non-AM techniques reported HPT-induced hardness gain of 11–38% [88,98,99], which is considerably higher than the $\sim 5\%$ increase observed for the sample AB in the present work. The relatively low increase in hardness for sample AB can be attributed in part to the specific microstructure produced by the L-PBF technique, which leads to a high initial hardness before HPT. Namely, the fine grain dimensions (length: 4.7 μm , width: 0.7 μm), the high dislocation density of $\sim 34 \times 10^{14} \text{ m}^{-2}$, the hard texture component $\langle 11\bar{2}0 \rangle$, and the highly alloyed main martensitic α' phase are those features of the microstructure that can yield an elevated hardness. Indeed, previous studies on non-AM Ti–6Al–4V alloys have shown that the acicular martensitic α' microstructure obtained by quenching from 1000 °C exhibited a much higher hardness of $\sim 4000 \text{ MPa}$ than the counterparts, consisting of a predominantly equiaxed α phase with a minor lamellar $\alpha+\beta$ dual phase, which showed 3000–3500 MPa [63,99]. The hard texture in the present AM-processed samples was weakened during HPT, which almost compensated for the hardening caused by the increase in dislocation density and grain refinement. A detailed quantitative description of the correlation between the changes of the microstructure and the hardness due to HPT would be very uncertain, since in addition to grain refinement and dislocation density increase, simultaneous phase transformation, solute redistribution, and texture evolution occurred.

5. Conclusions

Powders of Ti–6Al–4V (wt.%) alloy containing 0.14 wt.% trace Fe and 1 wt.% yttria-stabilized zirconia were blended and used for manufacturing samples by L-PBF technique. Additionally, specimens underwent subsequent heat treatment (HT) and HIP. The evolution of the microstructure and the hardness of these samples during plastic deformation up to the strain of ~ 208 was studied using the HPT method. The following conclusions were drawn from the results:

1. Sample AB exhibited a high dislocation density of $\sim 34 \times 10^{14} \text{ m}^{-2}$ and a low fraction of β phase due to the large temperature gradient and the fast cooling in L-PBF processing. The high dislocation density, the small dimensions of the elongated grains (length: 4.7 μm , width: 0.7 μm), the low β -phase fraction, the highly alloyed martensitic α' phase, and the strong $\langle 11\bar{2}0 \rangle$ texture component yielded a high hardness ($\sim 4470 \text{ MPa}$). HT and HIP processing decreased the dislocation density, coarsened the grain structure, and increased the β -phase fraction, resulting in softening. No fingerprint of the YSZ phase was observed in the microstructure analyses, most probably due to its dissolution during L-PBF processing.
2. The differences in the initial microstructures before deformation led to different microstructural and hardness evolutions during HPT up to a strain of ~ 26 . The microstructure saturated with the dislocation density of about $100\text{--}120 \times 10^{14} \text{ m}^{-2}$ and a crystallite size of $\sim 20 \text{ nm}$ for all three samples. With increasing strain, the secondary phase fraction decreased, and above the strain of ~ 26 the β phase fully transformed to α -Ti. The V and Fe solute atoms were segregated to the grain boundaries. The strong initial texture components of $\langle 10\bar{1}2 \rangle$ and $\langle 11\bar{2}0 \rangle$ changed to an almost random texture with a weak $\langle 10\bar{1}1 \rangle$ component.
3. Despite the different initial microstructures and hardness, the saturation hardness of the three samples was very similar (4600–4700 MPa) in accordance with the close dislocation densities and phase compositions. The hardness increase after HPT was only 5–12%. The relatively low increase in hardness for sample AB (5%) is attributed to the high initial hardness prior to HPT, resulting from the specific

Table 5

The Schmid factors for the most important basal and prismatic $\langle a \rangle$ -type easy glide systems in hcp Ti–6Al–4V alloy under different textures.

Slip system	Texture components			Random
	$\langle 11\bar{2}0 \rangle$	$\langle 10\bar{1}2 \rangle$	$\langle 10\bar{1}1 \rangle$	
$(0001)\langle 11\bar{2}0 \rangle$	0	0.37	0.30	0.32
$(0001)\langle 2\bar{1}10 \rangle$	0	0.18	0.15	0.32
$(0001)\langle 1\bar{2}10 \rangle$	0	0.18	0.15	0.32
$(10\bar{1}0)\langle 1\bar{2}10 \rangle$	0.43	0.40	0.38	0.38
$(01\bar{1}0)\langle 2\bar{1}10 \rangle$	0.43	0.44	0.33	0.38
$(1100)\langle 11\bar{2}0 \rangle$	0	0.44	0.33	0.38

microstructure produced by the L-PBF technique. Although HPT increases the dislocation density and reduces the β -phase fraction, the randomization of the texture had a softening effect which finally led to a low hardness increase.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jmrt.2026.07.208>.

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