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ORIGINAL REPORT



Strain hardening mechanism induced by basal/pyramidal dislocation reactions in a 3D-printed high-strength titanium alloy

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ABSTRACT

The high yield ratio and low strain hardening ability of titanium alloys significantly limit their engineering applications. In this work, a Ti₆₅Zr₃₀Cu₅ (at.%) alloy with nanoscale α laths was additively manufactured and achieved a high yield strength of 1274 MPa and a high strain hardening rate up to 40% of the shear modulus. Results show that universal basal/pyramidal dislocation reactions occur during the tensile deformation process. This triggers high-density sessile $\langle c \rangle$ dislocation junctions, which accumulate and pin other mobile dislocations. Consequently, abundant dislocation walls are induced, leading to a strong hetero-deformation-induced strain hardening effect.

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KEYWORDS

Laser direct energy deposition; titanium alloys; basal-pyramidal dislocation reactions; hetero-deformation-induced strain hardening; nanostructure

1. Introduction

The high-strength titanium alloys are [Q1] important structural materials for advanced engineering applications in aerospace, energy and power field. In general, the yield strength and yield ratio of titanium alloys exhibit a typical positive correlation, i.e. the Ti alloys with high yield strength generally exhibit a high yield ratio and low strain hardening ability [1–4], which are often associated with the sudden brittle failure during their engineering service.

Ti alloys with ultra-fine or nano α phase are a typical high strength Ti alloys. The refined α phase can lead to a strong Hall-Petch effects and a high yield strength [5]. However, these Ti alloys typically exhibit a low strain hardening ability, since the ultra-fine or nano α phase possesses low ability to store dislocations [6]. Besides, the ultra-fine or nano α phase may grow or slide along α phase boundaries during deformation, leading to strain softening.

One of the key factors in enhancing the strain hardening ability is to improve the ability to store dislocations in the ultra-fine or nano α phase. Recently, a few new methods to enhance the strain delocalization were proposed through inducing the interactions between basal

and pyramidal slip in the surface nanostructured layers of the shot peen-treated Ti alloys [6,7]. This phenomenon is positive for promoting the ability to entangle and accumulate dislocations, thus being able to enhance the strain-hardening ability of the Ti alloys. However, the basal-pyramidal interactions have been rarely found in bulk Ti alloys with ultrafine or nanoscale α laths microstructure. Especially, the effect of sessile basal-pyramidal dislocation junctions [7–9] on the strain-hardening mechanism is still unclear.

Another key factor to improve the strain hardening ability is to pin the ultra-fine or nano α phase boundaries during deformation. Inducing the precipitation with coherent interface structure with α phase is an effective way [10]. For this purpose, the eutectoid Ti alloys possess the intrinsic advantages because their eutectoid transformation will produce intermetallic compounds, that can provide the pinning sources at α phase boundaries, such as the Ti₂Cu phase in Ti-Cu alloys [11,12]. In previous research [13], we have obtained a novel nano bainite microstructure consisting of nano α laths with the dispersed (Ti,Zr)₂Cu (γ) nano particles distributing at α boundaries in a Ti₆₅Zr₃₀Cu₅ alloy. It is found that the nano γ particles can pin the nano α laths boundaries

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during deformation, thus enhancing the lath boundary stability, and contributing to the strain hardening ability. It is also noteworthy that the $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy is similar to the Ti-Zr-Cu metallic glass. However, the as-deposited $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy consists of crystalline phases including solid solution phase α and intermetallic phase γ . There is no evidence of any metallic glass phase in the deposit.

In this work, the bulk $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ (at.%) alloy deposits with a nanoscale α lath microstructure were prepared by a laser direct energy deposition (LDED) technology. A high yield strength of 1274 MPa combined with a low yield to strength ratio (~ 0.87) was achieved. A high level back stress component of flow stress was revealed from the loading-unloading-reloading (LUR) tests. A heterogeneous dislocation configuration, based on a universal basal/pyramidal interaction in the nano α laths leads to a strong hetero-deformation-induced hardening mechanism.

2. Materials and methods

The $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ (at.%) alloy deposits were prepared by a LMR-III LDED system equipped with an IPG-3000W fiber laser and a numerical control robotic arm (Figure S1). A block deposit with dimension of $65 \times 50 \times 40 \text{ mm}^3$ was fabricated using the argon gas atomized $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy powder with the size of $75 \sim 150 \mu\text{m}$ (Figure S2). The gauge section of the tensile specimens is $22 \times 6 \times 2 \text{ mm}^3$, and a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ is applied for the uniaxial tensile and LUR tests. Microstructure characterization was performed using a Tescan Clara GMH field emission scanning electron microscope (SEM) equipped with an OXFORD electron backscattered diffraction (EBSD) system and Transmission Kikuchi diffraction (TKD) system, a FEI Helios G4 CX transmission electron microscope (TEM) equipped with Energy Dispersive Spectrometer (EDS) and scan transmission electron microscope (STEM). X-ray diffraction (XRD) patterns are acquired on a D8 DISCOVER A25 systems equipped with $\text{Cu K}\alpha$ radiation.

3. Results and discussion

Figure 1 shows the as-deposited microstructure of $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy. It can be seen from Figure 1(a) that the as-deposited microstructure exhibits coarse columnar prior- β grains, indicating an epitaxial growth along the deposition direction. There also exist a uniform nano α laths distribution in the prior- β grains (Figure 1(b)). The average width of the α laths is about 102 nm (Figure S3). It is interesting to note that the nano α lath boundaries are decorated by dispersed nano γ particles (indicated by the yellow arrows in Figure 1(b)). The average width

of nano γ particles is about 23 nm (the TEM image in Figure 1(c)). The EDS mapping indicates the γ nanoparticles are enriched in Cu and Zr (Figure S4). Meantime, the nano γ particles exhibit an orientation relationship with α laths of $0002_\alpha || 013_\gamma$, $\langle 11\bar{2}0 \rangle_\alpha || \langle 331 \rangle_\gamma$, as revealed by the EBSD pole figures shown in Figure 1(d).

Figure 2(a) illustrates the variation of tensile true stress (σ_t) versus true strain (ϵ_t) and the LUR hysteresis loops of $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy deposits. The $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy deposits exhibit high yield strength of 1274.12 ± 11.54 MPa (at 0.2% strain offset), high ultimate strength 1434.79 ± 26.32 MPa and an acceptable tensile elongation of $5.98 \pm 0.75\%$. Meantime, the as-deposited alloys exhibit a low yield ratio comparing to other typical Ti alloys [1–4,12,14–17], as shown in Figure 2(b). The yield ratio of the so-called high strength Ti alloys (with yield strength over 1.2 GPa [18]) are typically exceeding 0.93. However, the as-deposited alloys in this work exhibit a low yield ratio of about 0.87. This should be attributed to a high strain hardening ability in $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy deposits. The evolution of the strain hardening rate (θ) is shown in Figure 2(c), which exhibits three characteristic stages. In stage I, θ presents the high values, slowly decreasing from 0.4μ to 0.17μ as the ϵ_t increases from 1.5% to 2.9%. In stage II, θ exhibits a sudden rise to 0.2μ with increasing ϵ_t from 2.9% to 3.4%. Finally, a monotonic decrease of θ till fracture can be seen in stage III. To explore the strain hardening mechanism, the flow stress (σ_f) was partitioned into two main parts, i.e. back stress (σ_b) and effective stress (σ_{eff}) (Figure 2(d)), using the method described in Ref. [19]. It can be seen that σ_b is the major part of σ_f during the entire deformation process. σ_b significantly increases with ϵ_t in stage I and reaches to maximum value of 803 MPa in stage II, meanwhile, the value of σ_{eff} decreases. This indicates a dominant back stress hardening or the so-called hetero-deformation-induced hardening effect [20] in stage I and II. In stage III, σ_b begin to decrease while σ_{eff} significantly increases with further increasing ϵ_t , suggesting that the increment of σ_f in the final stage is determined by σ_{eff} , which reveals the effects of short-distance resistances resulted from lattice friction stress and solute atoms [21].

Heterogeneous dislocation configurations are found in the α nano laths after deformation. Figure 3 and Figure S5 depict the morphology of dislocations in an α lath under double beam mode along different diffraction vectors (g). The dislocation patterns are determined according to the criterions proposed by Lee [22]. When the absolute values of $|g \bullet b|/gb \rangle^{1/3}$ (b is burgers vector) the dislocation lines are visible otherwise invisible. A summary of the values of $|g \bullet b|/gb$ of the dislocations seen in Figure 3 is shown in Table S2. According to Table S2, Figure 3(b) observed under

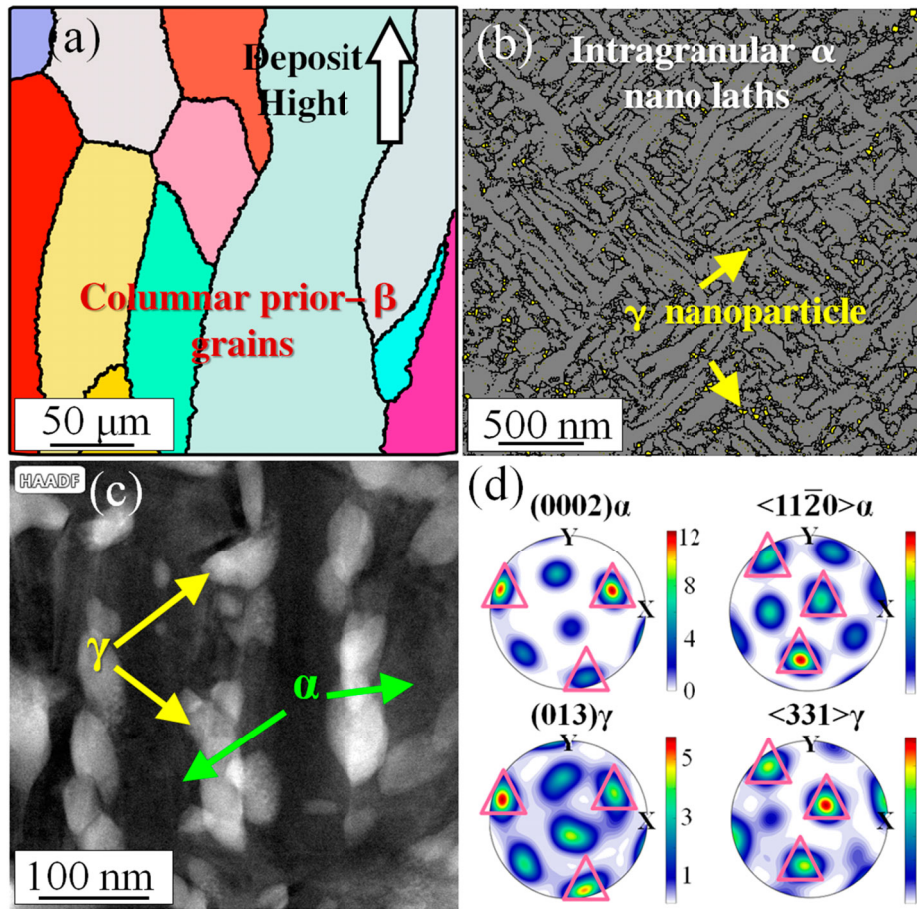


Figure 1. (a) An EBSD image of the macrostructure morphology of the as-deposited microstructure. (b) An TKD phase mapping of the intra-grain microstructure, the α and γ phases are colored gray and yellow, respectively. (c) An STEM-HAADF image of the α laths and the γ particles distributing on the α laths boundaries. (d) Pole figures of the α and γ phases.

$g1 = [2\bar{1}10]$ shows all the $\langle a \rangle$ dislocations and some of the $\langle c + a \rangle$ dislocations, while all the $\langle c \rangle$ dislocations are invisible. Figure 3(c) observed under $g2 = [2\bar{1}\bar{1}2]$ shows all the $\langle a \rangle$ and $\langle c \rangle$ types of dislocations and some of the $\langle c + a \rangle$ dislocations. Figure 3(d) observed under $g3 = [0002]$ shows all the $\langle c \rangle$ and $\langle c + a \rangle$ types of dislocations, while all the $\langle a \rangle$ dislocations are invisible. Therefore, the dislocations marked by the cyan lines seen in Figure 3(b and c) have their $b = \langle 11\bar{2}0 \rangle$, because they are visible under $g1$ and $g2$ but invisible under $g3$. Besides, these cyan dislocation lines are parallel to the basal planes, suggesting that they are basal $\langle a \rangle$ dislocations, as schematically illustrated in Figure 3(e-i). The yellow single lines and purple single lines seen in Figure 3(b) indicate $\langle c + a \rangle$ dislocations, because they are all visible under $g1$ (Figure 3(b)) and $g3$ (Figure 3(d)), suggesting they are pyramidal $\langle c + a \rangle$ 2nd dislocations, as schematically illustrated in Figure 3(e-ii). The yellow double lines seen in Figure 3(c and d) indicate some dislocation lines parallel to the $\bar{2}112$ planes, which are visible under $g2$ (Figure 3(c)) and $g3$ (Figure 3(d))

but invisible under $g1$ (Figure 3(b)), suggesting they are basal $\langle c \rangle$ dislocations, as schematically illustrated in Figure 3(e-iii). Besides, the purple double lines indicate some dislocation lines parallel to the $\bar{2}110$ planes, as seen in Figure 3(c and d), which are visible under $g2$ and $g3$ but invisible under $g1$ (Figure 3(b)), suggesting they are also basal $\langle c \rangle$ dislocations, as schematically illustrated in Figure 3(e-iv).

In general, the $\langle c \rangle$ edge dislocations in hcp alloys are sessile and are normally generated through dislocation reactions [7]. The present dislocation configurations suggest the occurrence of basal-pyramidal dislocation reactions in the $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy deposits, as described as follows: $0001\langle 2\bar{1}10 \rangle$ (basal $\langle a \rangle$) + $\bar{2}112\langle \bar{2}113 \rangle$ (pyramidal $\langle c + a \rangle$ 2nd) = $1/3\bar{2}113\langle 0001 \rangle$ (sessile $\langle c \rangle$) (schematic illustrated in Figure S6).

As a result, high density of sessile $\langle c \rangle$ dislocations are produced in the α nano laths, as shown in Figure 3(c and d), which are strong barriers to dislocation movements [7,9]. These sessile $\langle c \rangle$ dislocations pile up inside the α nano laths and entangle other mobile dislocations,

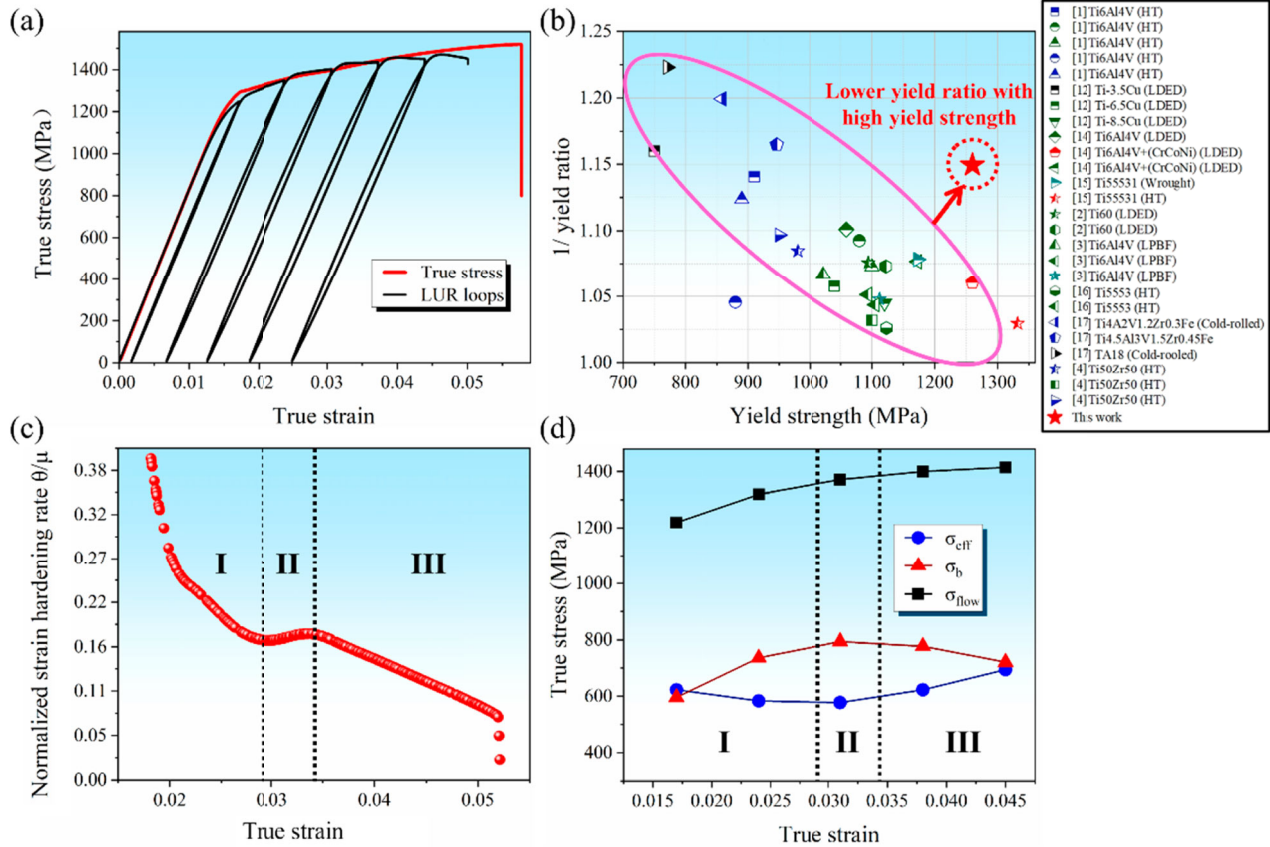


Figure 2. (a) Tensile true strain-true stress curve and LUR hysteresis loops of the as-deposited alloy. (b) Statistics of yield ratio versus yield strength of Ti alloys in literatures [1–4,12,14–17]. (c) The strain hardening rate normalized by shear modulus μ (37.09 GPa [13]) (d) Stress partitioning results induced from the LUR loops.

forming a local region with high density dislocations accumulations, as indicated by the red dot-dashed circles in Figure 3(b–d). These high density of dislocation accumulations are termed substructures or dislocation walls (DWs). A schematic depiction of the DWs formed in the $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy deposits are seen in Figure S7.

Figure 4(a) indicates the kernel average misorientation (KAM) map of the as-deposited alloy. The KAM inside the α laths is relatively low and uniform, indicating low geometrically necessary dislocations (GNDs) density. There are higher KAM regions near the boundary of some α laths, as indicated by the yellow arrows in Figure 4(a), which indicates that the alloy undergoes slight heterogeneous deformation induced by the residual stress during the LDED process, and a small amount of GNDs accumulate near the α boundaries to accommodate the heterogeneous strain gradient in the surrounding α laths. The KAM map of the deformed alloy after 5.8% tensile strain is seen in Figure 4(b). The KAM is significantly higher than in the as-deposited alloy, which indicates a much severe lattice distortion caused by the higher density of GNDs after deformation.

A large number of ‘nanobands’ with the higher KAM can be seen inside the α laths, as indicated by the white arrows in Figure 4(b), suggesting higher local lattice curvatures induced by the pile-up of sessile(c) edge dislocations at the core of DWs. The maximum KAM at the DWs is about 0.8 degree, which is more than 3 times the average KAM inside the α laths. The large amount of the ‘nanobands’ suggests a widespread distribution of the DWs induced by the universal basal/pyramidal dislocation reactions in the α laths during the tensile deformation. These heterogeneous dislocation configurations build up a high level of back stress (σ_b) in the alloy, which will lead to a prominent hetero-deformation-induced strain hardening effect [20]. This explains the significantly increased σ_b and high θ in stages I and II as shown in Figure 2(c).

In stage III of the deformation process, the easy basal and pyramidal slip systems are depleted and the number density of DWs gradually becomes saturated. The increasing density of dislocations on both sides of the DWs partially offset the internal stress, leading to a decrease of σ_b . Meanwhile, it is necessary to activate the residual slip systems with the unfavorable orientation or

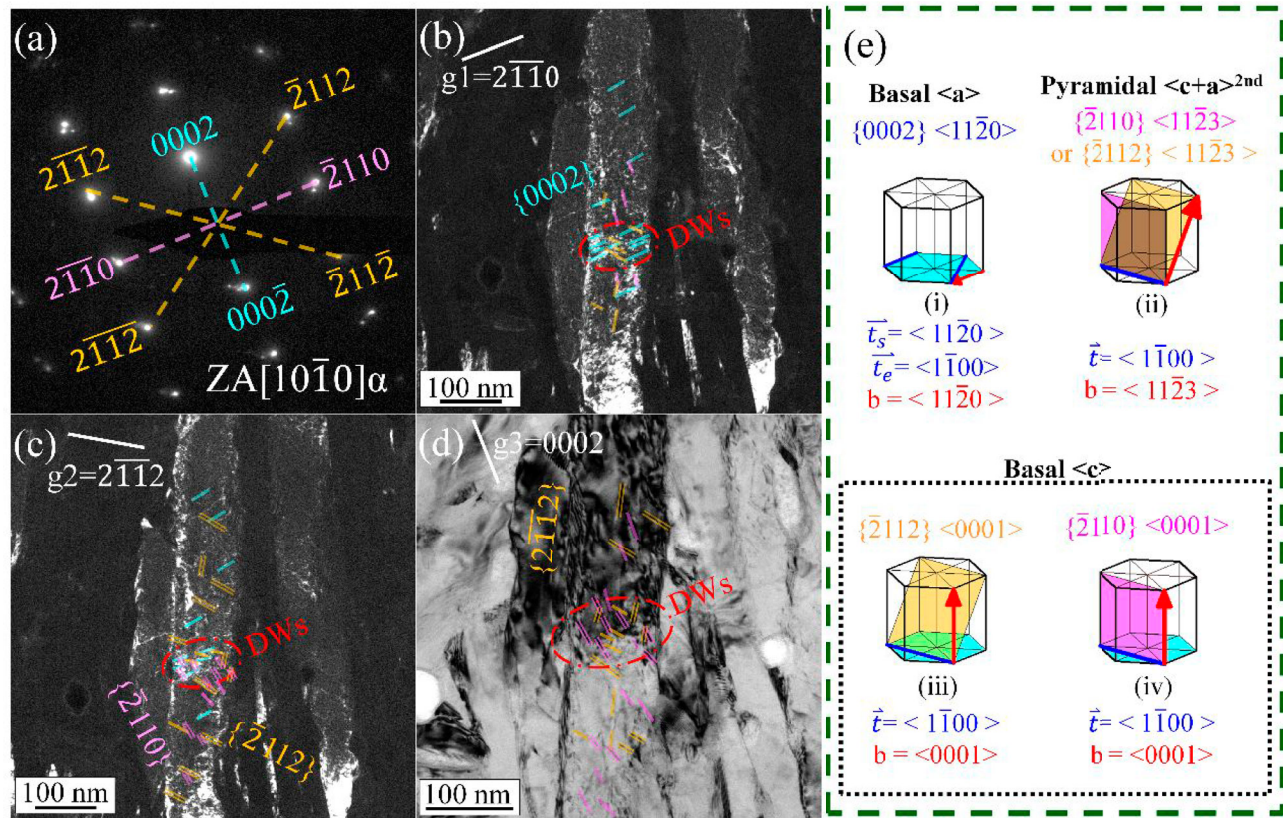


Figure 3. (a) Selected area electron diffraction (SAED) of a α lath along $[10\bar{1}0]$ zone axis. Dark field TEM image of the α lath under diffraction vector $g_1 = [2\bar{1}10]$ (b) and $g_2 = [2112]$ (c). (d) Bright field TEM image of the α lath under diffraction vector $g_3 = [0002]$. (e) Schematic depiction of the dislocations observed in hexagonal cells. Red arrows indicate the burgers vector of dislocations. Blue lines indicate the dislocation lines. Note: $\rightarrow t_s$ suggests the trace vector of screw dislocation line while $\rightarrow t_e$ of edge dislocation line.

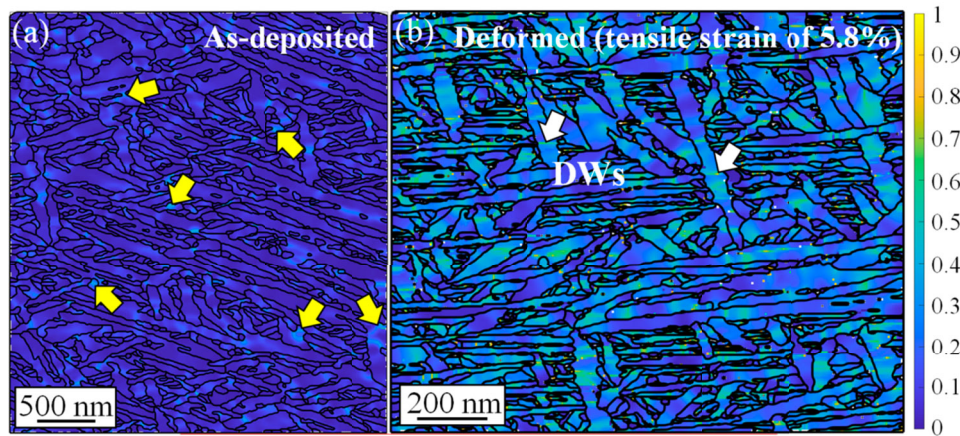


Figure 4. The KAM maps of the as-deposited alloy (a) and the deformed alloy (b). The yellow arrows indicate the higher KAM regions inside the α laths before deformation. The white arrows indicate the DWs in the α laths after deformation.

the higher critical resolved shear stress to continue plastic deformation, which leads to a significant increase in σ_{eff} . Therefore, a significant rise of σ_{eff} is seen in stage III which offsets the decrease in σ_b and continues to strengthen the alloy.

The XRD patterns of the $Ti_{65}Zr_{30}Cu_5$ alloy deposits before and after deformation are shown in Figure 5. It can

be found that all the α diffraction peaks shift to higher angles after deformation on comparing Figure 5(b) with Figure 5(a). Figure 5(c) provides a comparison of the $(10\bar{1}0)_\alpha$ peaks in more detail. Figure 5(d) gives the lattice constants of α phase along a-axis ($\langle 11\bar{2}0 \rangle$) and c-axis ($\langle 0001 \rangle$), showing a decrease of α lattice constants after deformation. Besides, the volume fraction of γ

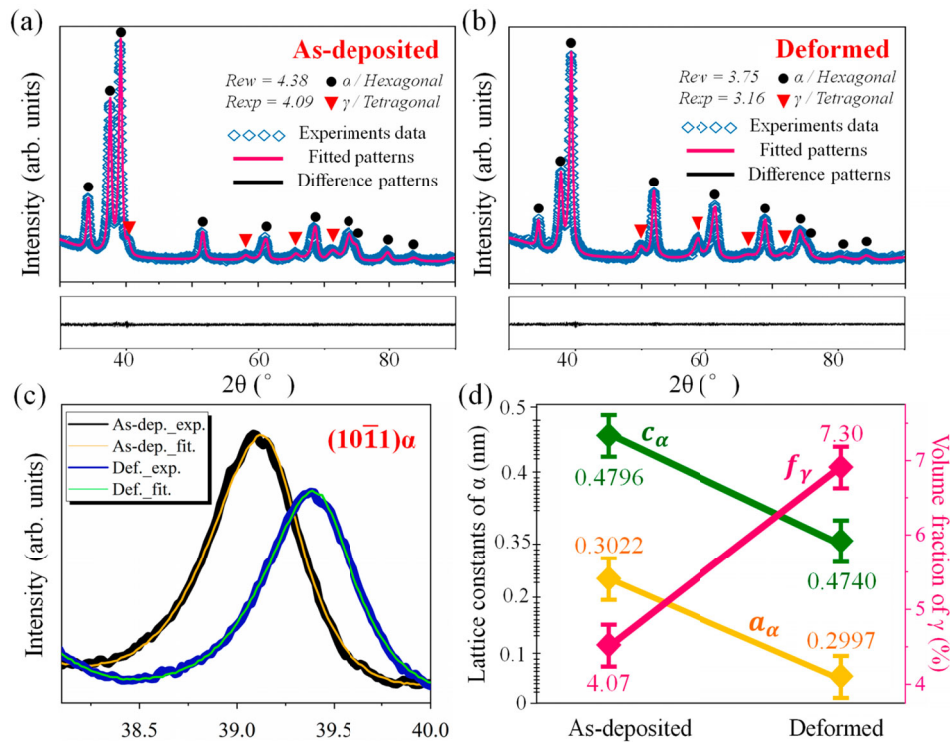


Figure 5. XRD pattern of the as-deposited (a) and deformed (b) alloy. (c) is the comparison of the $(10\bar{1}0)_\alpha$ peaks of the as-deposited and the deformed alloy. The black line is the experimental data of the as-deposited alloy (As-dep._exp.), the yellow line is its fitting pattern (As-dep._fit.), the blue line is the experimental data of the deformed alloy (Def._exp.), and the green line is its fitting pattern (Def._fit.). (d) shows the lattice constants of the a axis and c axis in the α phase and volume fraction of γ phase in the as-deposited and deformed alloys, respectively.

increased after deformation. This indicates that the solute concentration of α phase is decreased and a mechanically induced precipitation of γ phase is achieved in the $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy deposits during the deformation process. The rapid solidification and cooling during the LDED process lead the α solid solution to be supersaturated with Zr and Cu atoms. During deformation process, the increased defect density accelerates the atom diffusion rate, which promotes the excess Cu and Zr atoms to discharge from the supersaturated α phase, and precipitation of γ phase will occur. As a result, the lattice friction stress in α phase declines, which explains the drop of σ_{eff} in stage I.

It can be seen from Figure 2 that the alloys exhibit limited tensile plasticity despite high strain hardening ability. The effect of the unique as-deposited microstructure on the mechanical properties and fracture mechanisms can be revealed by analyzing the tensile fractographies [23–25]. The fracture surfaces are seen in Figure S8. The macroscopic morphology presents rough layered cracks, with a flat fracture surface of about 90° to the loading direction, as seen in Figure S8(a) and (c). This indicates a brittle fracture mechanism. Microscopically, the fracture surface is covered by fine and shallow dimples, as

seen in Figure S8(b), which are consistent with the high strength but limited plastic storage capacity of the nano α laths. Additionally, many pits with a lath morphology are observed on the fracture surface, as indicated by the cyan arrows, which indicates the separation of the alloy along the α laths boundaries during tensile deformation. The surfaces inside the pits are very smooth, suggesting the α laths underwent no deformation before being torn apart. This reveals that the transfer of dislocation slip between different α laths is hindered and the plastic deformation is locally concentrated in a small fraction of α laths with favored orientations. The poor slip transfer ability is induced by the high density of γ nanoparticles distributing along the α laths boundaries, which exhibit strong blocking effect on the dislocations slip. As a result, premature cracks nucleate in the heavily deformed α laths, and the α laths with unfavored orientations directly fracture as the cracks propagate along their boundaries. In our future work, revealing the interaction mechanisms between dislocation slip and γ nanoparticles and enhancing the slip-transferring ability will be the research focus.

Moreover, to further improve the elongation and strain hardening ability of the alloy, the volume fraction

and size of the γ nanoparticles at the α laths boundaries should be reduced in the as-deposited microstructure. This can be achieved by optimizing the LDED deposition method to increase the cooling rate during solidification and cooling processes, thereby limiting the diffusion and partition of Cu atoms, suppressing the precipitation of γ nanoparticles, and increasing the supersaturation degree of the metastable α laths. These can facilitate deformation transfer in the early stages of plastic deformation and enhance the dynamic $\alpha \rightarrow \gamma$ phase transformation during deformation, contributing to improved elongation and strain hardening ability.

4. Conclusions

In conclusion, a bulk $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy with nanoscale α laths microstructure was prepared by LDED. The mechanical properties of the as-deposited alloy exhibited a high yield strength of 1274 MPa combined with a low yield ratio of 0.87 and a high strain hardening ability. It is found that a universal basal/pyramidal dislocations reaction will occur in the nano α laths in the as-deposited alloy during the deformation process, which generates a high density of sessile $\langle c \rangle$ dislocations. These sessile dislocations pile up inside the nano α laths and trap other mobile dislocations, prompting the formation of a large number of DWs. These heterogeneous dislocation configurations lead to a strong hetero-deformation-induced strain hardening effect in as-deposited $\text{Ti}_{65}\text{Zr}_{30}\text{Cu}_5$ alloy, that significantly increases the strain hardening ability of the alloy. The results of this work shed light on designing and preparing high strength titanium alloys with high strain hardening ability and low yield ratio.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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Data availability statement

The authors confirm that the data supporting the findings of this study are available within the article and its supplementary materials.

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