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Micro-nano twins appearing in ultrafine-grained Ti–6Al–4 V alloy induced by high-pressure water jet technology

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Abstract
In this study, the surface of an ultrafine-grained Ti–6Al–4 V alloy was treated with high-pressure water jet technology (HPWJT). Then, the microstructure of the alloy was investigated using electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM). Three obviously different microstructural characteristics were observed from the treated surface to the matrix: ultrafine equiaxed grains with diameters from hundreds of nanometers to a few micrometers, micro-nano twins inside ultrafine equiaxed α grains, and an initial undeformed microstructure with ultrafine equiaxed grains. Formation of micro-nano twins in the subsurface was related to the special misorientation angle of neighboring grains. We proved the dependence of twinning on the crystallographic orientation and grain size of the α phase. In addition, the quantitative contributions of the micro-nano twins to the surface hardness of the alloy were calculated; micro-nano twins significantly improved the surface hardness of the alloy. This study provides a new easy method for introducing micro-nano twins on the surfaces of Ti and its alloys, and it may further improve the mechanical properties of these alloys.

1. Introduction

Nanotwinned metals have attracted extensive attention in the last ten years due to their excellent mechanical properties. Their attractive properties originate from nanoscale coherent twin boundaries, which have a strengthening effect similar to the classical Hall–Petch relationship. In addition, their excellent ductility is attributed to their fine nanoscale twins, which have ample room to accommodate the accumulation of dislocations. Lu et al. [1] formed high-density nano-twins in pure Cu via an electrodeposition process, and both the ultimate tensile strength and ductility of the pure Cu were significant. The ultimate tensile strength of pure Cu reached 1068 MPa, which was ten times greater than that of conventional annealed coarse-grained pure Cu and better than that of bulk nanoscale grains [2]. In addition, the ductility was comparable to that of conventional coarse-grained pure Cu. The bulk nanotwinned Cu fabricated by Zhao et al. via dynamic plastic deformation (DPD) exhibited a good combination of strength and ductility [3]. The nanotwinned Ni produced by Zhang et al. demonstrated excellent mechanical properties [4]. Cu–Zn alloys with a mixed structure of microscale recrystallization grains and nano-twins processed by DPD also had high strength and ductility [5]. Moreover, an excellent combination of strength and ductility was obtained in steel with high volume fraction of nano-twins [6]. Nano-twins in 316 austenitic stainless steel prepared by DPD or electrodeposition provided a good combination of strength and ductility [7]. However, all the abovementioned nano-twinned metal materials had face-centered cubic (FCC) structure, mostly in copper and steel, and they always had low or medium
The material was received as a hot-rolled bar with a diameter of 54 mm; the chemical composition of the alloy is listed in Table 1. The initial material was heat treated in the $\alpha+\beta$ phase region (950 °C), annealed for 60 min and then isothermally forged. After forging, the bar was water-quenched; the diameter of the final bar was 30 mm. The alloy was then heated at 800 °C and annealed for 20 min with air cooling to obtain refined $\alpha+\beta$ grains. The bar was cut into specimens with dimensions of 20 mm $\times$ 20 mm $\times$ 5 mm by an electric spark cutting machine. The surfaces of the test specimens were processed by HPWJT. The shot peening abrasive used in this test was $\text{SiO}_2$ with an average particle diameter of 100 $\mu$m. The detailed parameters of this experiment are listed in Table 2.

Figure 1 illustrates the shot peening path, schematic of the characterization section and processed surface. The microstructure of the sample prior to or after shot peening was characterized using a Supra 40 field emission scanning electron microscope equipped with an electron backscattered diffraction (EBSD) system. Oxford HKL Channel 5 software was used for data acquisition and post-processing. The samples were ground with 5000 silicon carbide papers and then vibration polished in silica solution for 12 h to relax surface residual stress and obtain higher-quality EBSD maps. The hardness measurements were conducted on an HV-1000 hardness tester produced by Shimadzu Corporation with a load of 2 g and a dwell time of 10 s. The detailed microstructure was stacking fault energy (SFE). Introducing a certain number of nano-twins in hexagonal close packed (HCP) metals has been challenging, especially for titanium, which has a high SFE. Recently, Zhao et al used gentle compression in liquid nitrogen to introduce a high volume fraction of nano-twins in HCP titanium. Tensile test results showed that both room temperature and cryogenic properties of the nano-twinned titanium were significantly improved [8]. The success in fabricating nano-twinned titanium may provide an approach for introducing nano-twins in other HCP metals to further improve their mechanical properties.

Two-phase Ti–6Al–4 V alloy is widely used for structural applications because of its low density/strength ratio, biocompatibility and good corrosion resistance. The main form of failure always starts from the surface of the alloy because it is used in applications where it is under friction and fatigue loads [9, 10]. Therefore, it is essential to fabricate a surface-modified layer on the alloy. A surface-modified layer can be obtained by refining the initial surface microstructure into nanograins by severe plastic deformation methods, such as shot peening [11], ultrasonic surface rolling [12], and mechanical attrition surface treatment [13]. However, these processes usually required several hours, which increases energy consumption. High-pressure water jet technology (HPWJT) is a new technology for surface treatment that has been widely used in industrial fields. The HPWJT technology has the advantages of high efficiency, low cost, good flexibility, no pollution and no thermal damage [14]. The process is realized by applying a high-pressure jet of water laden with particles of specific size and chemistry to the surfaces of metallic components. The surface hardness and fatigue properties of the components can be increased because this treatment can refine the microstructure, reduce roughness, and introduce residual stress at the surface [15, 16].

The plastic deformation mode of the $\alpha$ phase plays a very important role in accommodating the overall plastic deformation and mechanical properties of the alloy. The most common mode of plastic deformation of the $\alpha$ phase is dislocation slip. However, due to the limited slip systems of the hexagonal crystal structure of the $\alpha$ phase, deformation twinning may play a crucial role in accommodating plastic deformation. It is generally believed that deformation twinning can be more easily activated in low-SFE metals, and it is difficult to generate deformation twinning in fine-grained Ti–6Al–4 V alloys with high SFE. It has been reported that deformation twinning can be easily activated under plastic deformation at high strain rates [17]. Because HPWJT may induce a high strain rate plastic deformation on the surfaces of metals, it is expected that HPWJT can introduce a certain number of micro-nano twins on the surface of Ti–6Al–4 V alloy. Based on this assumption, we successfully introduced a certain number of micro-nano twins at the surface of Ti–6Al–4 V alloy. Due to formation of micro-nano twins, the surface hardness of the alloy reached 396 HV. This paper introduces a novel method to fabricate micro-nano twins in an ultrafine-grained Ti–6Al–4 V alloy. In addition, this study may provide a new method for introducing micro-nano twins in other metals with high SFE, and it may improve their mechanical properties.

| Table 1. The chemical components of Ti–6Al–4 V (wt%). |
|----------------|---------|-----|-----|-----|-----|-----|-----|
| Ti             | V       | Al  | Fe  | C   | Si  | Cr  | Zr  | Pb  |
| Balance        | 4.226   | 5.926 | <0.0004 | 0.011 | 0.024 | 0.0017 | 0.020 | 0.013 |

2. Experimental

The material was received as a hot-rolled bar with a diameter of 54 mm; the chemical composition of the alloy is listed in Table 1. The initial material was heat treated in the $\alpha+\beta$ phase region (950 °C), annealed for 60 min and then isothermally forged. After forging, the bar was water-quenched; the diameter of the final bar was 30 mm. The alloy was then heated at 800 °C and annealed for 20 min with air cooling to obtain refined $\alpha+\beta$ grains. The bar was cut into specimens with dimensions of 20 mm $\times$ 20 mm $\times$ 5 mm by an electric spark cutting machine. The surfaces of the test specimens were processed by HPWJT. The shot peening abrasive used in this test was $\text{SiO}_2$ with an average particle diameter of 100 $\mu$m. The detailed parameters of this experiment are listed in Table 2.

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characterized via a Tecnai G2F20 S-TWIN (200 KV) field emission transmission electron microscope (TEM) produced by the FEI Corporation.

3. Results and discussion

3.1. Fabrication of refined equiaxed microstructure in Ti-6Al-4V and determination of critical conditions for micro-nano twins

The initial microstructure of Ti-6Al-4V is shown in figure 2(a); it was composed of a large amount of the $\alpha$ phase and a small amount of the $\beta$ phase. High-angle boundaries ($>15^\circ$) are denoted by black lines. Figure 2(b) shows an EBSD all Euler angle map corresponding to figure 2(a) wherein various colors represent different crystallographic orientations. The as-received microstructure was dominated by macrozones with approximately the same crystallographic orientation. In some regions, large macrozones were mostly fragmented into small segments, which indicated that the initial material was subjected to deformation, and small $\beta$ grains were present at the interfaces of equiaxed $\alpha$ grains. By analyzing the neighboring crystallographic orientation of the $\alpha$ phase and $\beta$ phase, we found that their crystallographic orientations obeyed the Burgers relationship ($\alpha\{0001\} || \beta\{110\}, \alpha<11\overline{2}0> || \beta<1\overline{1}\overline{2}1>$). This induced crystallographic orientation inherited from the high-temperature parent $\beta$ phase of the hot-rolled Ti–6Al–4 V alloy that gradually formed the room temperature $\alpha$ phase during cooling [18]. A previous study proved that the microstructure of Ti–6Al–4 V alloy can be refined by using thermomechanical processing [19]. Figure 2(c) shows a phase map of the refined microstructures of the alloy. As shown in figure 2(c), the initial microstructure of the alloy was refined into many fine equiaxed $\alpha$ and $\beta$ grains via thermomechanical processing. The fraction of the $\alpha$ phase decreased relative to that of the as-received material, which illustrated that the high-temperature $\beta$ phase did not fully transform into the $\alpha$ phase during the thermomechanical and subsequent air cooling processes. Figure 2(d) shows an EBSD all Euler angle map corresponding to figure 2(c). The average $\alpha$ grain size was approximately a few microns according to measurements using HKL Channel 5 software with the line interception method. The fine equiaxed

Table 2. The detailed experimental parameters used in this study.

| Experimental parameter | Numerical value |
|------------------------|-----------------|
| nozzle diameter        | 0.33 mm         |
| nozzle spray angle     | 90°             |
| shot peening pressure  | 80 MPa          |
| shot peening distance  | 10 mm           |
| nozzle velocity        | 500 mm min$^{-1}$|
| abrasive flow rate     | 160–200 ml min$^{-1}$ |
| nozzle spacing         | 0.1 mm          |
| shot peening area      | 10 × 10 mm      |
α grains and small amount of β grains were the typical recrystallized microstructure for Ti–6Al–4 V alloy. When the Ti–6Al–4 V alloy was thermomechanically processed in the high-temperature α+β region and then water-quenched, the material always had deformation storage energy because of severe plastic deformation. This stored energy provided a driving force for recrystallization at high temperature and finally resulted in the formation of refined equiaxed α and β grains [19].

3.2. HPWJT induced micro-nano twins at the subsurface

A cross-sectional EBSD map of a specimen after shot peening-treated is exhibited in figure 3(a); high-angle boundaries are depicted by black lines. Three distinct differentiable zones can be discriminated, as indicated by the red dashed lines. Zone I has low resolution due to grain refinement and severe strain at the processed surface. Micro-nano twins formed in Zone II, which was from 2 to 20 μm below the surface. To demonstrate the type of twins, {0001}, {1120}, and 1012 pole figures were created to understand the twin relationships in grain A. As shown in figure 3(b), purple indicates the crystallographic orientation of the grain A matrix in the 1012 pole figure, and green indicates the crystallographic orientation of the 1012 tensile twins in grain A. A pair of coinciding points are marked with a red circle in the 1012 pole figure, which indicates that the matrix and twins share the .. crystallographic plane. Two proximal points in the {1120} pole figure also prove that the matrix and twins share an identical <1120> rotation axis. The misorientation angles between adjacent pixels along the red arrow in grain A are shown in figure 3(c), which demonstrates four distinct boundaries with high misorientation angles of approximately 85°. Figures 3(b) and (c) fully demonstrate that the parallel lamellar microstructures in grain A are micro-nano 1012 tensile twins, which are the most common twins in Ti [20], Mg [21] and Zr [22] alloys. The misorientation angles between adjacent pixels along the arrows in grain B and grain C are shown in figures 3(d) and E, respectively. The results prove that the lamellae in grain B and grain C were micro-nano 1012 tensile twins. Only one 1012 tensile twin variant was observed in grain A and grain C. However, two types of 1012 tensile twin variants formed in grain B. The evolution of the texture became complex due to the sliding reorientation of the twinned part.

The microstructures were characterized in further detail using TEM. Figure 4(a) displays a bright-field microstructure image of the treated surface. The grains were more refined than the initial equiaxed grains, but this refinement was not obvious because the surface had undergone instantaneous deformation, which did not
allow sufficient time for the dislocations to slip. In contrast, Liu et al treated the Ti–6Al–4 V surface with steel shot for several dozen minutes, and a gradient structure developed from the surface to the interior [11]. In this case, the dislocations had sufficient time to slip, making the grain refinement more obvious than that in this experiment. Figure 4(b) displays a cross-sectional image showing the micro-nano twin microstructures. The twin relationship can be further proven by the corresponding selected area electron diffraction pattern (figure 4(c)). The twin type was determined to be micro-nano $\bar{1}012$ tensile twin.

It has been proven that the most common slip systems of the $\alpha$ phase in Ti–6Al–4 V alloy are the $\{10\bar{1}0\}$ (prismatic plane), $\{0001\}$ (basal plane), $\{1\bar{1}0\bar{1}\}$ (pyramidal plane) gliding along the $<$a$>$ direction, and the $\{1\bar{1}01\}$ (pyramidal plane) gliding along $<$c+a$>$ direction [23, 24]. The most common twinning systems of the $\alpha$ phase in Ti–6Al–4 V alloy are $\{10\bar{1}2\}$, $\{11\bar{2}1\}$, $\{10\bar{2}6\}$, $\{11\bar{2}2\}$, $\{11\bar{2}3\}$, $\{11\bar{2}4\}$, and $\{2243\}$ [25]. However, in this study, in addition to dislocation slip, only the $\{10\bar{1}2\}$ $<$10$\bar{1}$1$>$ type twin system was triggered.

Figure 3. (a) Cross-sectional Euler angle orientation map of the Ti–6Al–4 V alloy after shot peening treatment. (b) $\{0001\}$, $\{11\bar{2}0\}$, and $\{10\bar{1}2\}$ pole figures showing a twin crystallographic orientation between the matrix and twins in grain A. (c–e) Misorientation angles along the red arrows in (c) grain A, (d) grain B, and (e) grain C.
in some α grains. Rather than other types of twin systems, the prevailing {1012} \text{<} 10\overline{1} \text{l} \text{>} type twin system may be attributed to its relatively low critical resolved shear stress [26, 27]. Not all α grains can trigger {1012} \text{<} 10\overline{1} \text{l} \text{>} type twin system. Further clarification of this behavior is needed. It has been proven that twins play an important role in coordinating the plastic deformation of neighboring grains [20]. Thus, the formation of twins must be connected with the misorientation angle of neighboring grains. In some cases, grain boundaries may accumulate high local stress during plastic deformation due to the special misorientation angle between the neighboring grains. To release the high local stress, twinning must be triggered. Releasing the local high stress through twinning also appears in the same alloy that was processed after high-energy shot peening [28]. Because the misorientation angle of neighboring grains varies, some areas form twins and some areas cannot form twins. The twins in grains (A, B and C) originated from grain boundaries (the enlarged figures marked by the yellow lines in figure 3(a)). The twins terminated in the interior of grain A, which may further prove that these twins originated from the grain boundary, and the formation of the twins was attributed to high local stress at the grain boundary.

The equiaxed α grains in the subsurface underwent transient plastic deformation. Therefore, these α grains in the subsurface maintained their grain size, and the initial α grain size that formed micro-nano 10\overline{1} \text{2} tensile twins was analyzed statistically. The original α grain that stimulated micro-nano 10\overline{1} \text{2} tensile twins had a grain size between 0.8 and 6 μm. Previous reports showed that formation of many micro-nano 10\overline{1} \text{2} tensile twins in α grains with grain sizes from 0.8 to 6 μm can hardly be observed in Ti-6Al-4V alloys. Sun et al produced nano-twins in pure Ti via dynamic plastic deformation at high strain rates [29, 30]. The initial grain size in their experiment was approximately 86 μm, which was much larger than the α grain size of the Ti-6Al-4V alloy in this experiment. It is expected that the hardness of the metal can be increased because the twins were restrained in these refined α grains.

### 3.3. Effect of crystal orientation on formation of micro-nano twins

To understand which crystallographic orientation of the α phase tends to form micro-nano twins, the orientation distribution function (ODF) map was created. Figure 5 shows an ODF map of an area where micro-nano twins formed. The Euler angles (\(\varphi_2\), Φ, \(\varphi_2\)) of the grains that were prone to form micro-nano twins were (126°, 90°, 0 ~ 60°), (143°, 14°, 0 ~ 60°), and (82° 17°, 0 ~ 60°). Because of the range of the Euler angles \(\varphi_2\) was very large. Therefore, it did not fully illustrate which crystallographic orientation of the α phase tended to form micro-nano twins. A more detailed pole figure analysis was performed to understand the effect of crystal orientation on formation of micro-nano twins. As shown in figure 6, twins were activated when the c-axis of the initial α phase tilted from the ND at an angle between approximately 30° and 90°. Most c-axis α grains tilted from the ND with an angle from approximately 50° to 90°.

According to the von Mises criterion, the homogeneous plastic deformation of a metal or alloy requires at least five independent slip systems. In the case of the α phase in the Ti-6Al-4 V alloy, the prismatic and basal slip systems provide only four independent slip systems [31]. Thus, plastic deformation of the α phase needs twinning to accommodate plastic strain in addition to dislocation slip. The activation of the different types of twins is connected with the crystallographic orientation of the initial grains. The most commonly employed criterion for the activation of this type of twin in hexagonal materials is the Schmidt criterion. That is, the type of activated twins is dependent on the angle between the twins systems and the external force. In this study, twins were activated when the c-axis of the initial α grain tilted from the ND at an angle between approximately 30°
and 90°. The surface of the alloy was subject to compressive stress, and the direction of the compressive stress was approximately parallel to the ND. In this case, the c-axis of most of α grains were subject to tensile stress. Thus, the 1012 tensile twins were prone to be triggered. Additionally, a small number of 1012 tensile twins formed in α grains tilted from the ND at an angle between approximately 30° and 50°. It did not favor the formation of 1012 tensile twins for these α grains. It has been proven that 1012 tensile twins have relatively low critical resolved shear stress among all twin systems [26, 27]. In addition, it has also been proven that more 1012 tensile twins with various crystallographic orientations can be obtained when the material is deformed under a high strain rate [32]. Since HPWJT may induce a very high strain rate plastic deformation at the surface of metals. It is reasonable to observe that some twins that form in α grains tilt from the ND at an angle between approximately 30° and 50°. The evolution of twins in pure polycrystalline Ti during rolling was studied by Bao et al [33]. They demonstrated that 1012 tensile twins were activated when the c-axis of the α grain tilted from the
ND with an angle approximately in the range of $70^\circ$–$90^\circ$. Compared to the current study, this angle range was relatively narrow. This result indicated that it was possible to introduce more $\text{10\bar{1}2}$ nano-twins into $\alpha$ grains with a wide range of crystallographic orientations in Ti-6Al-4V alloy by HPWJT.

3.4. Effect of grain size on formation of micro-nano twins

To clarify the effect of grain size on formation of micro-nano twins, the distributions of all the initial $\alpha$ grain sizes and the initial $\alpha$ grain sizes that formed $\text{10\bar{1}2}$ tensile twins were proposed. The average grain size in figure 7(a) is obviously smaller than that in figure 7(b). In figure 7(a), the volume fraction of grains with grain size between 0.3 $\mu$m and 1.0 $\mu$m accounts for 29.4%. However, in figure 7(b), the volume fraction of the grains with sizes between 0.8 $\mu$m and 1.0 $\mu$m accounts for only 5.7%. The results showed that deformation micro-nano $\text{10\bar{1}2}$ tensile twins were more likely to form in large grains than small grains.

Numerous studies have shown that the activation of deformation twins is closely related to the initial grain size [30, 34]. It is generally believed that deformation twins form more easily where large grains are present in the initial material. According to the Hall–Petch relationship, the dislocation slip has a large range in large grains, and more dislocations accumulate at grain boundaries, which leads to stress concentrations. Thus, the deformation twins are more easily induced due to the large stress concentrations at the grain boundaries. In contrast, the total volume fraction of grain boundaries increased as the grain size decreased. In this case, the
stress concentrations at grain boundaries were released by activation of non-basal plane slipping. Thus, it became difficult to achieve the stress required for activating deformation twins [35]. In addition, it has been proven that the propensity for deformation twinning in ultrafine-grained pure titanium decreases monotonically with decreasing grain size. In small grains, instead of deformation twins, \(<c+a>\) type dislocations are more easily triggered to coordinate plastic deformation along \(c\)-axis [30]. The deformation twinning propensity in ultrafine-grained pure titanium is similar to the case of Ti-6Al-4V alloy, which indirectly proves that \(<c+a>\) dislocations in Ti-6Al-4V alloy may be triggered more easily than deformation twins in small grains, which results in the dependence of deformation twins on grain size.

3.5. Hardness increase by formation of micro-nano twins at the subsurface of the alloy

Figure 8 shows the variation in hardness with depth from the processed surface to the matrix of the Ti–6Al–4V alloy after HPWJT treatment. As shown in figure 8, the depth of the surface-modified layer was approximately 15 \(\mu\)m, which coincided with the EBSD results. A hardness gradient formed from the treated surface to the matrix. At approximately 5 \(\mu\)m below the processed surface of the HPWJT-treated Ti–6Al–4V alloy, the hardness was approximately 396 HV, and this value gradually decreased with increasing depth from the processed surface to the matrix. The hardness tended to be the same as the matrix hardness at depths below 15 \(\mu\)m.

It has been proven that both grain size and refined structures (dislocations and twins) affect the hardness of metals because both interfaces (grain boundary and twin boundary) and dislocations act as strong obstacles to block the movement of dislocations [11, 36]. In this study, the grain size at the subsurface was approximately equal to that in the matrix. In this case, the increment of hardness at the subsurface must be attributed to the formation of dislocations and micro-nano twins. Thus, the increment of hardness can be calculated by the following equation:

\[
\Delta H = \Delta H_D + \Delta H_T
\]

where \(\Delta H_D\) and \(\Delta H_T\) are increments of hardness caused by dislocations and twins, respectively. According to the empirical equation [36]:

\[
\Delta H_D = \frac{1}{3} \Delta \sigma_D
\]

where \(\Delta \sigma_D\) is the increment of strength caused by dislocations. The increment of strength caused by dislocations can be determined by equation [37]:

\[
\Delta \sigma_D = \alpha Mg b \sqrt{\rho_s} - \alpha Mg b \sqrt{\rho_m}
\]

where \(\alpha\) is a constant for scaling the interaction strength between dislocations, \(M\) is the average Taylor factor, \(G\) is the shear modulus, \(b\) is the magnitude of the Burgers vector and \(\rho_s\) and \(\rho_m\) are the dislocation densities at the subsurface and matrix, respectively. The dislocation density can be calculated by the following equation [38]:

\[
\rho_s = \rho_m + \frac{1}{2} \Delta \sigma_D \rho_m \frac{1}{\alpha Mg b}
\]
where $\rho$ is the dislocation density at a certain pixel, the KAM (kernel average misorientation) is the local misorientation, $\mu$ is step size of the EBSD measurement, and $b$ is the Burgers vector. The local misorientation at a certain point can be calculated by the following equation [38]:

$$\text{KAM} = \frac{1}{n} \sum_{j=1}^{n} |\theta_1^{\text{sur}} - \theta_j|$$

where KAM stands for the local misorientation at pixel ‘$i$’ and $\theta_1^{\text{sur}}$ represents the misorientation at its neighboring point ‘$j$’. The KAM can be calculated by using EBSD local misorientation data. Figure 9 shows an EBSD local misorientation map of the subsurface and matrix of the alloy. It is clear that the local misorientation at the subsurface was larger than that in the matrix, which indicated that the subsurface had a higher dislocation density than the matrix. Based on these local misorientation data, the KAM value of the subsurface and the matrix were determined. The material constants and calculated results of the present study are summarized in Table 3.

The hardness of the subsurface increased by 23.8% compared to that of the matrix. The hardness of the subsurface increased by 15.0% and 8.8% as a result of the strength effects of micro-nano twins and dislocations, respectively. Obviously, the increment of hardness caused by micro-nano twins was larger than that from dislocations, which indicated that micro-nano twins had a positive effect on increasing the hardness of the alloy. A similar effect was also verified in Zr702, where micro-nano twins formed at the surface of the material after a pulsed laser surface treatment [36]. Liu et al [11] prepared a gradient structure in a Ti–6Al–4 V alloy using the metal shot peening method. The average grain sizes in the elongated ultrafine grain (EIU) layer and refined grain

![Figure 9. EBSD local misorientation map of the subsurface (a) and matrix (b).](image)
layer of their experiment were equal to the gradient-modified layer of this experiment. However, the hardness values in the EIU and RG layers were between 345 HV and 380 HV; they were less than the value (396 HV) near the surface-modified layer of this study. In addition, Luo et al.[41] successfully introduced a small number of micro-nano twins in 51CrV4 spring steel; the micro-nano twins increased the tensile strength by 264 MPa. These results indicated that introducing a certain number of micro-nano twins in metal is very significant. The strengthening mechanism of nano-twins has been fully proven in Cu[1]. Coherent twin boundaries and conventional grain boundaries are equally efficient at hindering dislocation motion, especially when the thickness of twin lamellae is on the nanometer scale. An extremely high critical stress is needed for a single dislocation to cross a nano-twins boundary, which is the reason that the subsurface of the alloy was as hard as 396 HV in this study.

### 4. Conclusions

In this study, the initial coarse-grained microstructure of a Ti–6Al–4 V alloy was transformed into refined equiaxed grains after the hot forging process. Then, HPWJT was applied to treat the alloy with refined equiaxed grains. The HPWJT treatment resulted in significant changes in the microstructure and hardness from the surface to the matrix. The main conclusions are as follows:

1. After HPWJT treatment, three obviously different types of microstructure formed in the Ti–6Al–4 V alloy from the treated surface to the matrix: ultrafine equiaxed grains with diameters from of hundreds of nanometers to a few micrometers, micro-nano twins inside ultrafine equiaxed α grains, and initially undeformed microstructural features with fine equiaxed grains.

2. Twins originated at the grain boundaries of neighboring grains, and they formed as the result of the release of high local stress between neighboring grains that had a special misorientation angle.

3. twins depended on the crystallographic orientation and grain size of the α phase. Twins were activated when the c-axis of the initial α grains tilted from the ND at an angle between approximately 30° and 90°; most of them were in the range between approximately 50° and 90°. Statistical results showed that twins were more likely to form in large grains than small grains.

4. The hardness reached 396 HV at the subsurface, which increased by 23.8% compared to that of the matrix. Micro-nano twins had a positive effect on the hardness of the alloy and increased the hardness of the subsurface by 15.0%.

### Table 3. The material constants and calculated results of the present study.

| Symbol | Description | Value |
|--------|-------------|-------|
| KAM_s | KAM value at the subsurface | 0.0182° |
| KAM_m | KAM value of the matrix | 0.0096° |
| μ | The step size of the EBSD measurement | 0.1 μm |
| b | Burgers vector | $2.95 \times 10^{-10}$ m[39] |
| α | Interaction between dislocations | 0.24[40] |
| M | Taylor factor | 2.75[40] |
| G | Shear modulus | 45 GPa[39] |
| $\rho_s$ | GND density at the subsurface | $1.23 \times 10^{15}$ m$^{-2}$ |
| $\rho_{0s}$ | GND density of the matrix | $6.49 \times 10^{14}$ m$^{-2}$ |
| $\Delta \sigma_{0s}$ | Increment of strength caused by dislocations | 85 MPa |
| $\Delta H$ | Increment of hardness | 76 HV |
| $\Delta H_s$ | Increment of hardness caused by dislocations | 28 HV |
| $\Delta H_t$ | Increment of hardness caused by twins | 48 HV |
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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

Conflict of interest statement

The authors declare that there are no conflicts of interest.

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