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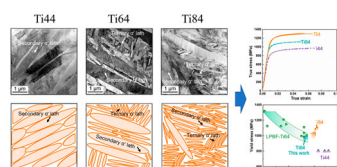
Laser additive manufacturing of titanium alloys with various Al contents

Zhe Song^a, Xiaoqin Zeng^{a,b} and Leyun Wang^{a,b,c}

^aNational Engineering Research Center of Light Alloy Net Forming, Shanghai Jiao Tong University, Shanghai, People's Republic of China; ^bState Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University, Shanghai, People's Republic of China; ^cZhangjiang Institute for Advanced Study, Shanghai Jiao Tong University, Shanghai, People's Republic of China

ABSTRACT

Laser powder bed fusion (LPBF) is an additive manufacturing technology for metals. Ti-6Al-4V (Ti64) is a frequently used material for LPBF, but as-built LPBF-Ti64 has poor ductility that limits its usage. Herein, we study how the Al content will affect the microstructure and mechanical properties of LPBF Ti-xAl-4V alloys ($x = 4, 6, 8$, wt.%). The materials' strength increases with Al%. Both Ti-4Al-4V (Ti44) and Ti-8Al-4V (Ti84) alloys exhibit higher work hardening and uniform elongation values than Ti64. This outcome is attributed to activation of multiple slip modes in Ti44 and a more heterogeneous microstructure in Ti84.



IMPACT STATEMENT

This paper reports that work hardening and uniform elongation values of LPBF Ti-xAl-4V alloys do not monotonically change with the Al content. Instead, the conventional Ti-6Al-4V exhibits the least values.

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KEYWORDS

Titanium alloys; laser powder bed fusion; ductility; work hardening

1. Introduction

Laser Powder Bed Fusion (LPBF) is an additive manufacturing technology for making metal components [1,2]. It uses a fine laser beam to selectively fuse regions of a metal powder bed to build the component layer-by-layer. LPBF Ti-6Al-4V (Ti64) features a hierarchical structure of acicular martensitic α' needles within columnar prior- β grains [3], which is drastically different from the two-phase $\alpha + \beta$ microstructure (fully lamellar, bi-modal, or fully equiaxed) typically found in this commercial alloy. Compared to Ti64 by traditional manufacturing processes (e.g. rolling, forging, etc.), LPBF Ti64 usually exhibits lower work hardening and ductility [4–6]. These issues significantly limit the usage of LPBF Ti64 in the aerospace industry (the biggest consumer of titanium), which has stringent safety standards.

Post-processing heat treatment can increase the ductility of LPBF Ti64, during which the brittle α' phase will decompose into a two-phase $\alpha + \beta$ microstructure

[4–8]. However, heat treatment increases the overall processing time and cost. Designing new titanium alloy compositions specifically for LPBF is a more challenging but also more rewarding strategy. Zhang et al. [9] showed that, powders from partial homogenization of Ti-6Al-4V and a small amount of 316L stainless steel melts can be used for LPBF processing, and the as-built material exhibits both high work hardening and high ductility. Recently, we reported that the microstructure and mechanical properties of LPBF titanium alloys can be engineered by adjusting the aluminum (Al) content in Ti-Al-V alloys [10].

In the present work, we further study the effect of Al content on the microstructure and mechanical properties of LPBF Ti alloys. Ti-xAl-4V alloys ($x = 4, 6, 8$, wt.%) were fabricated using pre-alloyed powders under the same LPBF building parameters. The alloys' strength increases with the Al content. Interestingly, both Ti44 and Ti84 exhibit higher work hardening and uniform

CONTACT Leyun Wang leyunwang@sjtu.edu.cn National Engineering Research Center of Light Alloy Net Forming, Shanghai Jiao Tong University, Shanghai 200240, People's Republic of China; State Key Laboratory of Metal Matrix Composites, Shanghai Jiao Tong University, Shanghai 200240, People's Republic of China; Zhangjiang Institute for Advanced Study, Shanghai Jiao Tong University, Shanghai 201203, People's Republic of China

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elongation values than Ti64. This outcome can be attributed to the activation of multiple slip modes in Ti44 and a more heterogenous microstructure in Ti84, respectively.

2. Materials and methods

Pure Ti (99.9%), pure Al (99.9%), and an Al-58wt.% V master alloy were melted in a vacuum arc remelting (VAR) furnace to fabricate cast billets with nominal compositions of Ti-4Al-4V, Ti-6Al-4V, and Ti-8Al-4V (wt.%). The billets were forged and machined to produce a cylindrical rod with a diameter of 50 mm. The rods were atomized into powders using an ALD EIGA 50–500 equipment. All three types of powders were sieved to have diameters ranging from 15 μm to 53 μm . Powders of each alloy were loaded into an EOSINT M280 machine for LPBF processing with the following building parameters: laser power = 280 W, scan speed = 1200 mm/s, hatch spacing = 140 μm , layer thickness = 30 μm , and substrate preheating temperature = 100 °C. The scan path was rotated by 67° between each layer. For each alloy, bulk bars with the dimension of 12 × 12 × 80 mm³ were built, whose longitudinal axes were parallel to the building direction (BD). The chemical compositions of the as-built Ti44, Ti64, and Ti84 bars were measured by inductively coupled plasma atomic emission spectroscopy (ICP-AES) and shown in Table 1.

X-ray diffraction (XRD, step size = 0.02°, Mini Flex 600) was performed for phase identification. Electron backscatter diffraction (EBSD) were taken using a TESCAN GAIA3 scanning electron microscope (SEM) equipped with the HKL Channel 5 EBSD system (Oxford Instruments). The EBSD data was analyzed using the OIM™ 8.1 software (EDAX, Inc.) and an associated MATLAB plugin MTEX (Version 5.60). We further used a Talos F200X G2 transmission electron microscope (TEM) to characterize the finer microstructure.

Flat dog-bone tensile specimens with the gauge dimensions of 18 mm (Length) × 3.4 mm (Width) × 2.0 mm (Thickness) were extracted from the Ti44, Ti64 and Ti84 bars, respectively. Tensile tests were performed on a Zwick-100 kN instrument equipped with an extensometer at the strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$. Three Ti44 specimens, two Ti64 specimens, and three Ti84

specimens were tested. Loading-unloading-reloading (LUR) tests were additionally performed on an Instron 5982 machine with a video extensometer at a strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$. During the LUR tests, specimens were unloaded under load control at various strains to 20N and then reloaded under displacement control at a strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$ to produce stress-strain hysteresis loops.

3. Results

3.1. Phase identification

Figure 1 shows the XRD profiles of the three alloys in the as-built state. Each material shows α' peaks only. Ti₃Al precipitates are often observed in titanium alloys with Al > 6 wt% by conventional manufacturing processes [11,12]. Nevertheless, no Ti₃Al peaks are observed in LPBF Ti84, likely because of the ultra-high solidification speed. From the XRD profiles, lattice parameters and the c/a ratios of these three alloys are estimated: Ti44, $a = 2.933 \text{ \AA}$, $c = 4.673 \text{ \AA}$, $c/a = 1.593$; Ti64, $a = 2.930 \text{ \AA}$, $c = 4.670 \text{ \AA}$, $c/a = 1.594$; Ti84, $a = 2.926 \text{ \AA}$, $c = 4.670 \text{ \AA}$, $c/a = 1.596$. This result agrees with previous studies that reported the c/a ratios of Ti-Al alloys increasing with the Al content [11].

3.2. Microstructure of LPBF-Ti alloys

Figure 2(b) shows the EBSD maps of as-built Ti44, Ti64 and Ti84 alloys. All three alloys consist of α' laths. The α' laths were formed due to the martensitic transformation of the prior- β grains during the rapid cooling in LPBF. We reconstructed the prior- β grains from the crystal orientations of the α' laths using the MTEX code [13], and the reconstruction results are shown in Figure 2(a). The prior- β grains in Ti64 and Ti84 are large and columnar. The prior- β grains in Ti44, however, are apparently smaller and more equiaxed. Using the Image-Pro Plus software, the average widths of prior- β grains in Ti44, Ti64, and Ti84 are $33 \pm 25 \mu\text{m}$, $49 \pm 32 \mu\text{m}$, and $124 \pm 23 \mu\text{m}$, respectively.

Figure 2(c) shows magnified images of the black boxes in Figure 2(b). The lenticular α' laths are called primary α' [14]. We measured the widths (d) of indexed primary α' laths in Ti44, Ti64, and Ti84 alloys, and their distributions are shown in Figure 2(d). The width distributions can be fitted by a lognormal function: $f =$

$\frac{A}{d \cdot B \cdot \sqrt{2 \cdot \pi}} e^{-\frac{(\ln d - C)^2}{2 \cdot B^2}}$, where A , B , and C are constants. From the fitting, the average widths of primary α' laths in Ti44, Ti64, and Ti84 are $2.8 \pm 1.3 \mu\text{m}$, $2.4 \pm 1.0 \mu\text{m}$, and $2.3 \pm 0.9 \mu\text{m}$, respectively.

Table 1. Chemical compositions (wt%) of the as-built Ti44, Ti64, and Ti84 bars.

	Al	V	Fe	C	N	H	O	Ti
Ti44	3.80	3.94	0.026	0.0096	0.017	0.0016	0.112	bal
Ti64	5.87	3.99	0.21	0.0088	0.011	0.0022	0.087	bal
Ti84	7.55	4.02	0.042	0.0078	0.024	0.0030	0.109	bal

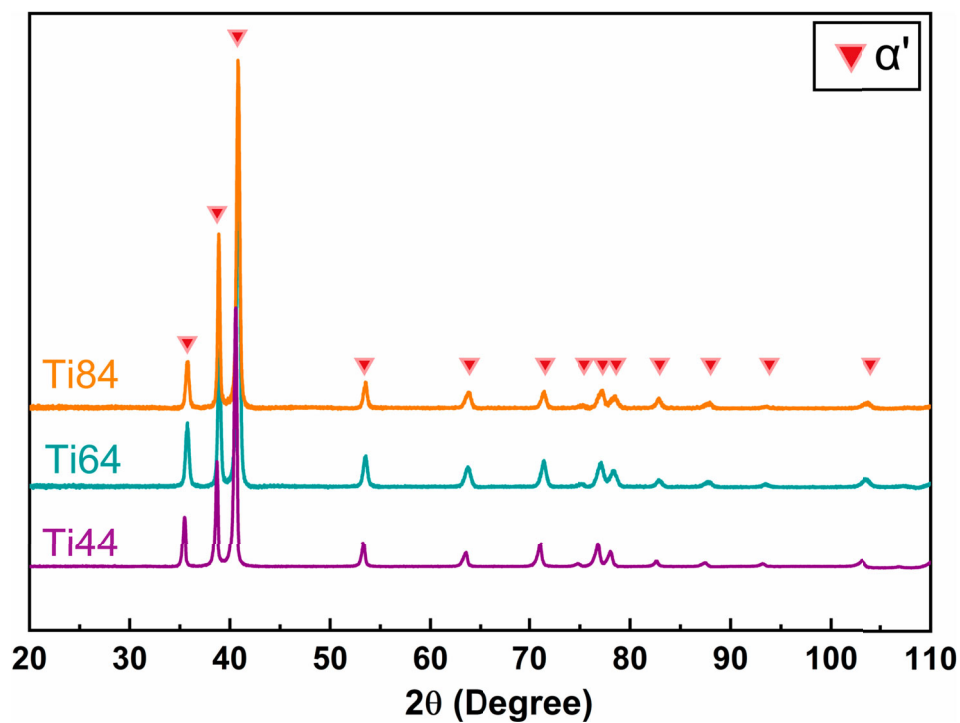


Figure 1. XRD profiles of the as-built Ti44, Ti64, and Ti84.

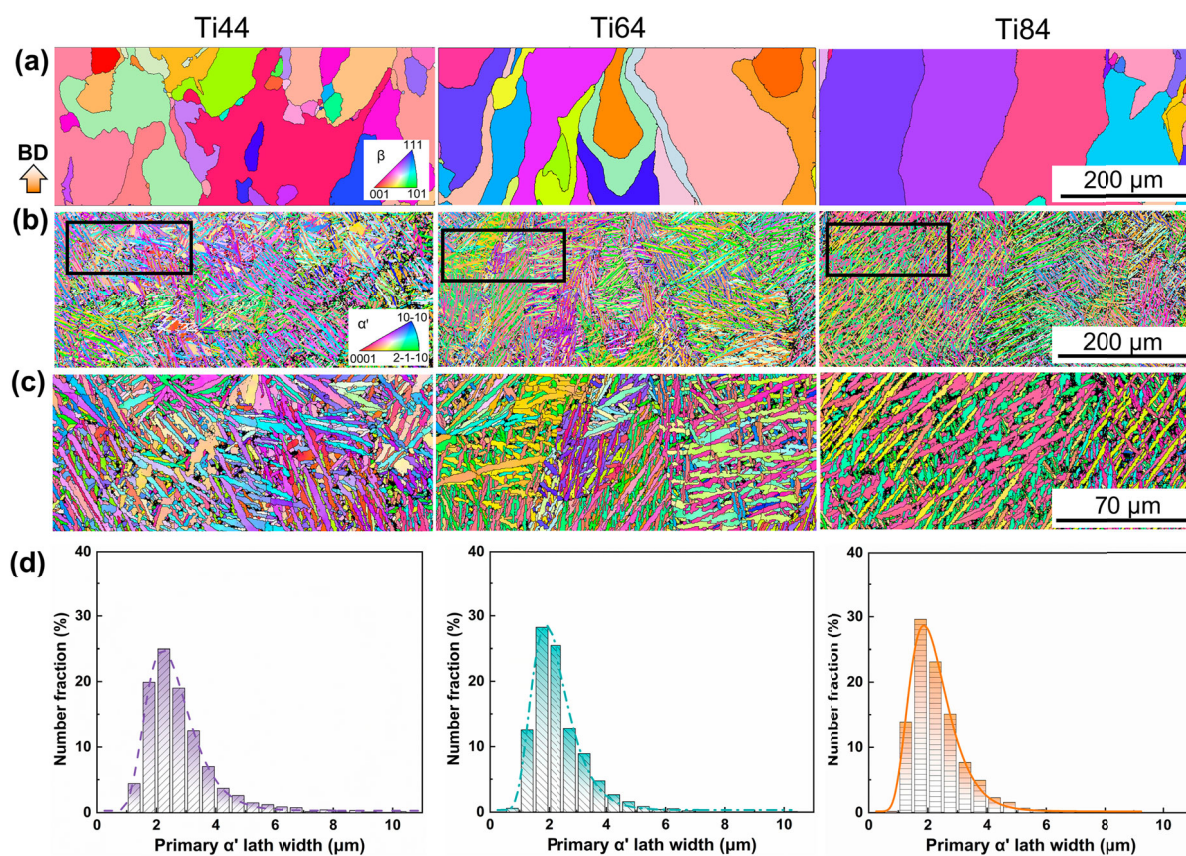


Figure 2. Comparison of the microstructures in as-built Ti44, Ti64, and Ti84. (a) Reconstructed prior- β grains from the (b) EBSD inverse pole figure maps of α' laths. (c) Magnified images of the black boxes in (b). (d) Width distributions of the primary α' lath in the three alloys.

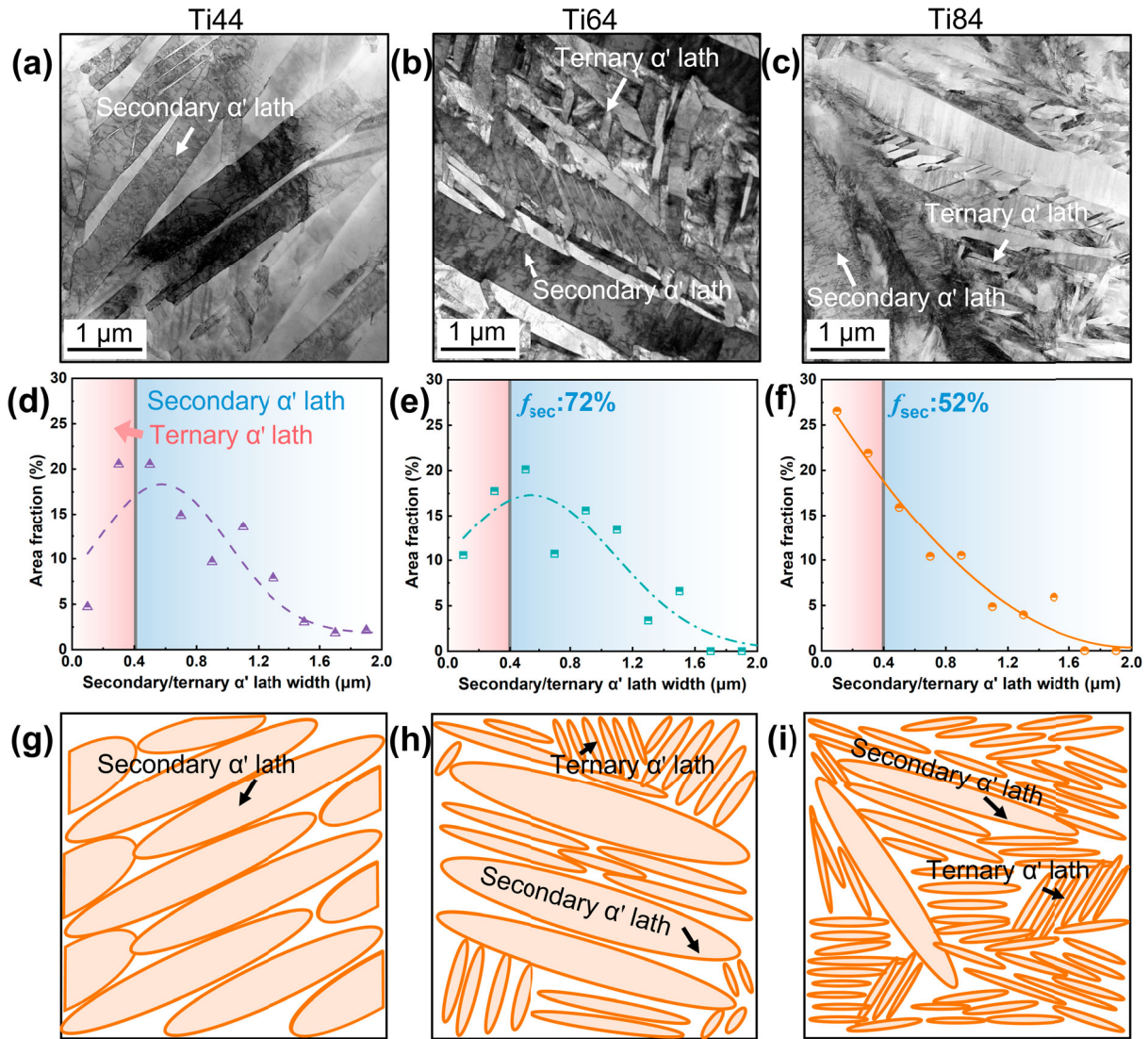


Figure 3. (a – c) Bright field TEM images of as-built Ti44, Ti64, and Ti84. (d – f) Width distributions of the secondary/ternary α' lath. (g – i) Schematic illustrations of the different microstructures in Ti44, Ti64 and Ti84.

It is known that LPBF titanium alloys often consist of a hierarchical martensitic microstructure [14]. Figure 3(a–c) shows bright field TEM images of Ti44, Ti64, and Ti84. At this scale, Ti44 shows sub-micron α' laths, which are called secondary α' . Ti64 and Ti84 also have secondary α' , but finer α' laths (i.e. ternary α') are also present in their microstructures. The average widths of secondary/ternary α' laths in Ti44, Ti64, and Ti84 are $0.40 \pm 0.29 \mu\text{m}$, $0.30 \pm 0.24 \mu\text{m}$, and $0.21 \pm 0.22 \mu\text{m}$, respectively. We use a value of $0.40 \mu\text{m}$ to distinguish between secondary and ternary α' laths. Figure 3(d–f) show the width distributions of secondary/ternary α' laths in the three alloys. The area fractions of secondary α' laths (f_{sec}) in Ti64 and Ti84 are measured to be 72% and 52%, respectively. The secondary α' laths in Ti84 are constrained by ternary α' laths. Figure 3(g–i) illustrates

the difference in the microstructures of Ti44, Ti64 and Ti84.

3.3. Tensile properties

Figure 4 shows the tensile stress–strain curves of the three as-built materials. From the curves, the yield strength (YS), ultimate tensile strength (UTS), uniform elongation (ε_u), fracture elongation (ε_f), and work hardening exponent (n) values are summarized in Table 2. n is defined by the Hollomon's equation: $\sigma = K \cdot \varepsilon^n$ [15], where σ and ε are true stress and true strain values.

The three alloys' YS increases while their ε_f decreases with the Al content. Notably, both Ti44 and Ti84 exhibit higher ε_u and n than Ti64. The higher work hardening in Ti44 and Ti84 delays the necking of these specimens,

Table 2. UTS, YS, ε_u , ε_f , and n values for Ti44, Ti64, and Ti84.

	UTS (MPa)	YS (MPa)	ε_u (%)	ε_f (%)	n
Ti44	915 ± 4	802 ± 1	4.5 ± 0.16	14.7 ± 2.5	0.078 ± 0.002
Ti64	1068 ± 2	971 ± 9	3.7 ± 0.03	10.5 ± 0.5	0.066 ± 0.004
Ti84	1240 ± 6	1103 ± 17	4.1 ± 0.05	7.4 ± 1.6	0.085 ± 0.001

causing higher ε_u . Figure 4(d) compares the YS and uniform elongation values of Ti44, Ti64, and Ti84 with other as-built LPBF Ti64 in the literature. With a higher Al%, Ti84 exhibits a simultaneous increase in strength and uniform elongation compared to regular LPBF Ti64.

4. Discussion

From the limited number of tensile specimens in Figure 4, both Ti44 and Ti84 exhibit higher work hardening and uniform elongation values than Ti64. In our previous study, we have shown that LPBF Ti44 has a smaller $\text{CRSS}_{\text{prism}}/\text{CRSS}_{\text{basal}}$ ratio (~ 1.19) than that in LPBF Ti64 (~ 1.33) [10]. Therefore, Ti44 is more likely to activate both basal and prismatic slips during plastic deformation, which enhances its work hardening and uniform elongation. Ti44 and Ti64 show a typical trade-off between strength and ductility.

A more interesting result is that Ti84 exhibits a simultaneous increase in strength and uniform elongation compared to Ti64. Indeed, the average ε_u of Ti84 ($4.1 \pm 0.05\%$) is only slightly higher than that of Ti64 ($3.7 \pm 0.03\%$), but this difference is still important, given the small standard deviation values. As a substitutional solute, the addition of Al increases the CRSS of both basal and prismatic slip, but the strengthening effect on prismatic slip is much more significant than on basal slip [23]. Therefore, we presume that the $\text{CRSS}_{\text{prism}}/\text{CRSS}_{\text{basal}}$ ratio in Ti84 will be larger than that in Ti64, making basal slip even more dominant in Ti84. So, instead of the activation of multiple slip modes, the higher work hardening and uniform elongation in Ti84 than those in Ti64 must be attributed to other reasons. Both Ti64 and Ti84 exhibit heterogeneous microstructure, featured by a mixture of secondary and ternary α' laths. During the deformation of such materials, plastic deformation occurs first in the soft zones (i.e. secondary α') while the hard zones (i.e. fine grains) are less deformed. To accommodate the strain gradient, in front of the interface between soft and hard zones, geometrically necessary dislocations (GNDs) are accumulated. Those GNDs will exert a long-range back stress in soft zones and a forward stress in hard zones [24–27]. The former allows

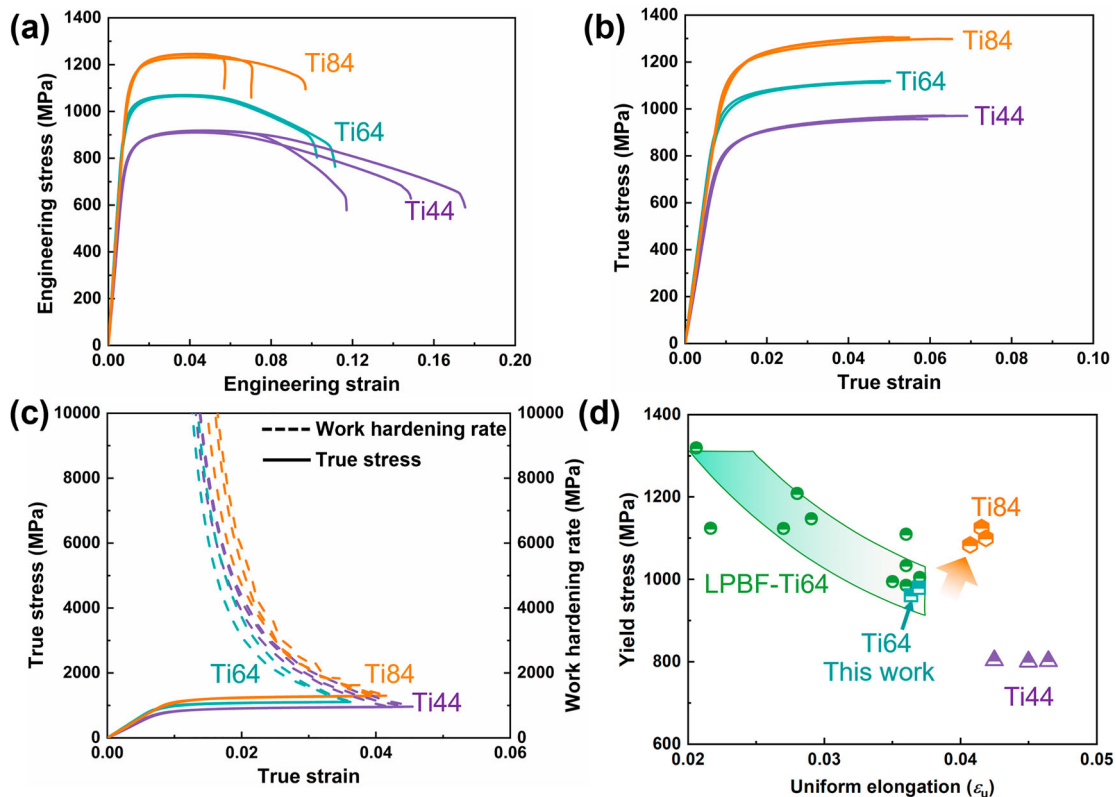


Figure 4. Tensile properties of the three materials. (a, b) Stress-strain curves of Ti44, Ti64, and Ti84 specimens. (c) Work hardening rates in the three alloys. (d) Comparing the YS and uniform elongation values of Ti44, Ti64, and Ti84 with other as-built LPBF Ti64 in the literature [3,10,16–22].

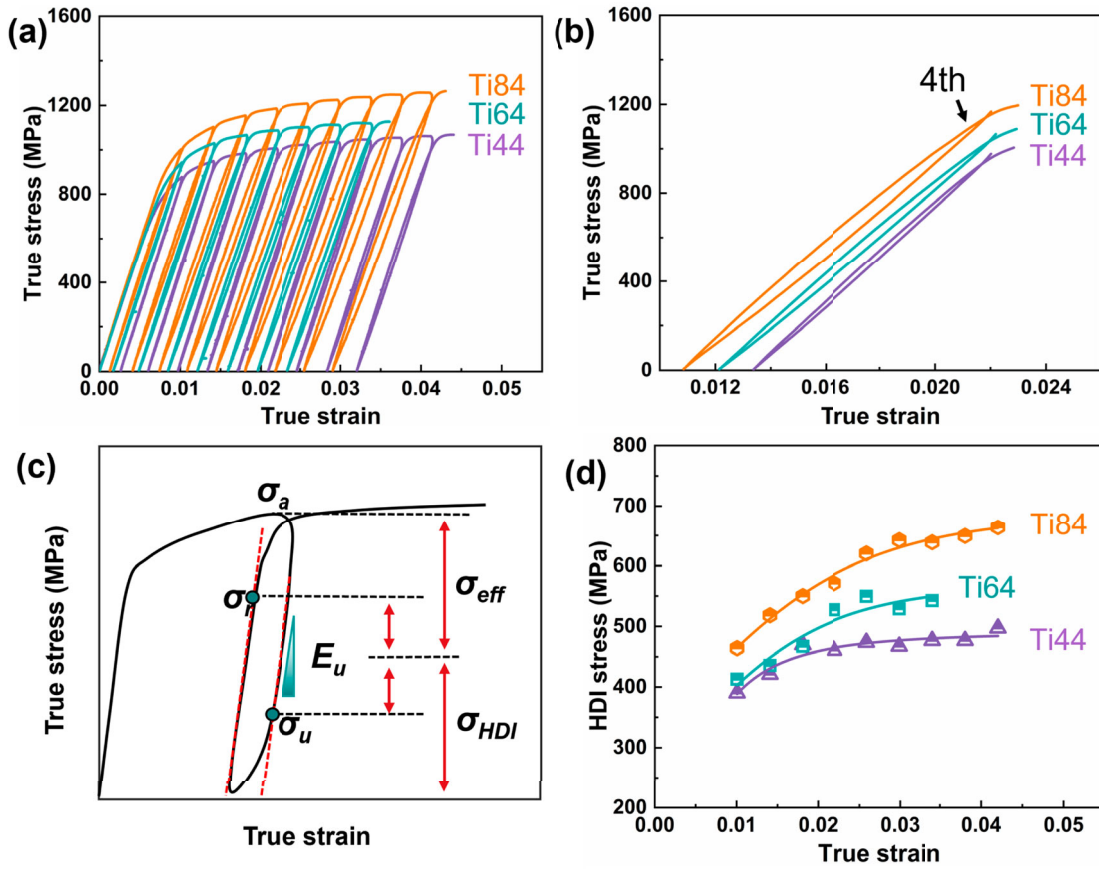


Figure 5. (a) Stress-strain curves from the LUR test of Ti44, Ti64, and Ti84. (b) The fourth hysteresis loops in the three alloys. (c) Schematic of calculating the HDI stress. (d) Evolutions of the HDI stress values in Ti44, Ti64, and Ti84.

soft zones to sustain the higher applied stress, while the latter increases the stress in hard zones to attenuate the strain incompatibility. Their cooperative action produces hetero-deformation induced (HDI) hardening, which is responsible for the enhanced work hardening and ductility of various heterostructured materials [28,29].

Previous studies indicate that HDI hardening depends on the size, geometry, and distribution of the heterogeneous zones [24]. For instance, Wu et.al [27] developed a pure Ti sheet by asymmetrical rolling and partial recrystallization annealing to obtain a heterogeneous lamella structure: coarse, elongated grains with a volume fraction of $\sim 25\%$ were fully constraint by un-recrystallized ultra fine grains. This microstructure produces strong HDI hardening, so that the Ti sheet exhibits both high ductility and high strength. It was proposed that two structural features are important for HDI hardening: (1) a relatively low volume fraction of the soft zones (about $20 \sim 30\%$), and (2) complete constraint of the soft zones (e.g. coarse grains) by the hard zones (e.g. fine grains).

From Figure 3, volume fractions of secondary α' laths in Ti84 and Ti64 are 52% and 72%, respectively. As shown in Figure 3, secondary α' laths in Ti84 are almost completely constrained by ternary α' laths, while

secondary α' laths in Ti64 are less constrained. In this sense, the microstructure in Ti84 will lead to stronger HDI hardening than Ti64.

To corroborate the above theory, LUR tests were conducted to assess the HDI hardening in Ti44, Ti64, and Ti84. The results are shown in Figure 5. The hysteresis loops (i.e. Bauschinger effect) in the unloading-reloading curves (Figure 5(a)) indicate the existence of HDI hardening. Figure 5(b) compares the shapes of the fourth hysteresis loop in Ti44, Ti64, and Ti84. As Al content increases, the unloading-reloading hysteresis loop becomes wider, suggesting the HDI hardening effect becomes stronger.

We calculated the HDI stress values (σ_{HDI}) in Ti44, Ti64, and Ti84 following the procedure proposed by Yang et al. [30], as schematically shown in Figure 5(c):

$$\sigma_{HDI} = \frac{\sigma_r + \sigma_u}{2} \quad (1)$$

where σ_r is the reload yield stress, σ_u is the unload yield stress. Here, the slope reduction of effective Young's modulus (E_u) by 5% is used as the criterion to determine σ_r and σ_u [30].

Figure 5(d) shows the calculated HDI stress values in Ti44, Ti64, and Ti84. Apparently, Ti84 has higher HDI stress values than Ti64 and Ti44, especially in later deformation. The total increase of σ_{HDI} in Ti84 is about 198 MPa during the plastic deformation, which is larger than that in Ti64 (≈ 146 MPa) and in Ti44 (≈ 95 MPa). This HDI hardening may explain LPBF Ti84 having higher work hardening and uniform elongation than Ti64.

The fracture elongation values in Ti84 are notably lower than Ti64. When both the secondary and ternary α' laths deformed plastically (i.e. the end of HDI hardening), the lack of non-basal slip in Ti84 would make it difficult to accommodate local stress concentration, so cracks could quickly form to cause fracture. To improve the fracture elongation, it will be necessary to employ strategies such as mid-temperature heat treatment to induce non-basal slip while retain the heterostructure. Further studies are needed to explore that possibility.

5. Conclusions

In this work, we study the effect of Al content on the microstructure and mechanical properties of Ti- x Al-4 V alloys ($x = 4, 6, 8$, wt.%). The main conclusions are summarized below:

- (1) All three alloys contain the α' phase only. Each alloy exhibits a hierarchical microstructure characterized by prior- β grains, primary α' laths, and secondary/ternary α' laths. The area fractions of ternary α' laths increase with the Al content. The secondary α' laths in Ti84 are almost fully constrained by ternary α' laths.
- (2) The three alloys' strength increases with the Al content. Both Ti44 and Ti84 exhibit higher work hardening and uniform elongation values than Ti64. This outcome is attributed to the activation of multiple slip modes in Ti44 and a stronger HDI hardening effect in Ti84.

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Disclosure statement

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

Declaration of interest statement

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

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ORCID

Xiaoqin Zeng  <http://orcid.org/0000-0002-1466-070X>

Leyun Wang  <http://orcid.org/0000-0002-1783-6520>

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