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Effect of TiB₂ addition on microstructure and mechanical properties of AA8009 alloy fabricated by laser additive manufacturing

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Effect of TiB₂ addition on microstructure and mechanical properties of AA8009 alloy fabricated by laser additive manufacturing

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Abstract

The research involves the addition of 5 vol.% TiB₂ particles into AA8009 alloy powder to synthesize TiB₂/AA8009 composite parts produced via laser powder bed fusion

(LPBF). The addition of the TiB_2 particles causes the $\text{TiB}_2/\text{AA8009}$ composites with and without annealing have lower compressive strength than AA8009 alloy due to the change of the strengthening mechanism. The results further indicated that solid solution strengthening was the main strengthening mechanism of the LPBF AA8009 alloy at room temperature whereas Orowan strengthening became the primary strengthening factor after annealing at 673 K. In contrast, Orowan strengthening always remained the main strengthening mechanism for the $\text{TiB}_2/\text{AA8009}$ composite, irrespective of the annealing temperature. In addition, after annealing of the LPBF parts at 673 K, the compressive yield strength (CYS) of the unblended AA8009 alloy specimens had a ~2.5 times greater reduction (from 705 ± 16 MPa to 459 ± 30 MPa) compared to that of the composite $\text{TiB}_2/\text{AA8009}$ samples (from 466 ± 23 MPa to 368 ± 3 MPa). Therefore, TiB_2 particles can suppress the drop in yield strength of LPBF AA8009 alloy below 673 K, providing a theoretical and experimental basis for the applications of both LPBF AA8009 and $\text{TiB}_2/\text{AA8009}$ alloys at low and medium temperatures.

Keywords: Laser powder bed fusion; AA8009 alloy; Microstructure; Mechanical properties

1. Introduction

Laser powder bed fusion (LPBF) uses the idea of layer-by-layer processing to create metallic components from three-dimensional models as a popular additive manufacturing technique [1-4]. Compared with conventional manufacturing methods,

LPBF has many advantages in terms of flexible design/design-freedom, high material utilization, and grain refinement stemming from high cooling rate (reaching 4×10^7 K/s) [5-7]. Following comprehensive research involving LPBF over the past 2 decades, several bottlenecks during the LPBF processing have been overcome, e.g., pores, cracks and keyholes [1,7]. Due to the excellent mechanical properties at medium temperatures (573 ~ 673 K), an extensive study on LPBF of heat-treatable Al alloys has been carried out [2,7], for example, defect-free Al-Si and Al-Cu-Mg-Si were successfully prepared by the LPBF process [8,9]. However, research on the LPBF of heat-resistant Al alloys and composites is rather limited. Due to its exceptional mechanical performance, including high specific strength, yield strength, and fracture qualities, the heat-resistant Al-8.3Fe-1.3V-1.8Si alloy (AA8009), is very popular in the automobile sector [10,11]. In the authors' previous work, Yu et al. [12] successfully fabricated fully dense AA8009 alloy parts via LPBF with different laser powers and scanning speeds (200 W, 300 mm/s; 350 W, 1200 mm/s), and reported the formation of different heterogeneous microstructures that led to different mechanical properties. Sun et al. [13] found that the AA8009 alloy prepared by using LPBF had substantial crack formations that results in reducing their hardness, mechanical properties, and ductility. Sun. et al. [14] further found that LPBF AA8009 alloy was composed of α -Al and $Al_{12}(Fe, V)_3Si$ phase through simulation and experiments. At high temperatures, the ultrafine grain second phase ($Al_{12}(Fe, V)_3Si$) had a low coarsening rate, which resulted in excellent yield strength. The formation of $Al_x(Fe, V)_ySi$ phase was very sensitive to the cooling rate, thus a uniform distribution of $Al_x(Fe, V)_ySi$ phase depended on a high cooling rate

[15,16]. Although much of the previous research has examined the process factors, microstructure, and mechanical characteristics of the LPBF AA8009 alloy, a rare endeavor has been made on the influence of temperature, which is important for the application of heat-resistant Al alloys.

Generally, thermal stability is defined as the ability of a material to maintain its properties at required temperatures, large and textured columnar grains in heat-resistant Al alloys are correlated intimately with their thermal stability [17]. During the solidification process of LPBF, columnar grains frequently develop from the inner of melt pool (MP) to the outside along with thermal stress, which causes intergranular thermal tearing and lowers the yield strength of heat-resistant aluminum alloys [18]. In contrast, a microstructure with uniform, fine equiaxed grains is more likely to improve mechanical properties of heat-resistant aluminum alloys [19]. To promote the transformation from columnar grains to equiaxed grains of LPBF alloy, inoculation treatment is one of the common methods adapted [11]. The principle of inoculation treatment is to introduce a nucleating agent to promote heterogeneous nucleation and generate sufficient supercooling at the solid-liquid interface to promote nucleation [20]. Ceramic particles are currently mainly used as nucleating agents in LPBF process. For Al alloys, TiB_2 as a nucleating agent can effectively transform columnar grains into fine equiaxed grains, and the yield strength of composites has been improved [21,22]. Li et al. [23] fabricated nano- $\text{TiB}_2/\text{AlSi10Mg}$ composites by LPBF and found that composites with a texture-free microstructure have excellent tensile strength and hardness. Xi et al [22] found that the TiB_2 particle's addition is beneficial to the

transformation of Al alloy from coarse columnar grains (Al-12Si alloy) to fine equiaxed grains ($\text{TiB}_2/\text{Al-12Si}$ composites). These works present a chance for the development of $\text{TiB}_2/\text{AA8009}$ composites with good **mechanical properties** at medium temperature (673 K).

The current study aims at fabricating parts via LPBF from unblended AA8009 alloy and composite $\text{TiB}_2/\text{AA8009}$ alloy powders. This is followed by a comprehensive investigation of the microstructure and strengthening mechanism of the built parts before and after annealing.

2. Material and methods

AA8009 powder (Fe: 8.28 wt.%, V: 1.34 wt.%, Si: 1.81 wt.%, and balanced Al) was prepared by using gas atomization, with particle size range between 15 to 53 μm , ($D_{10} = 16.2 \mu\text{m}$, $D_{50} = 30.5 \mu\text{m}$, and $D_{90} = 52.3 \mu\text{m}$, shown in Fig. 1(a)). TiB_2 particles (size range of 3-5 μm , shown in Fig. 1(b)) displayed an irregular polygonal morphology and were relatively dispersed with no agglomeration phenomenon. Before mixing the powder, we add TiB_2 particles to the AA8009 powder according to the 7.66 wt.% (equivalent to 5 vol.%) [24]. 5 vol.% TiB_2 particles were added to the AA8009 powder using a 3D vibratory mixer for 2 h at a speed of 72 rpm (Fig. 1(c)) and micron-sized TiB_2 particles were dispersed and inevitably adhered to the AA8009 powder during the mechanical mixing process (inset in Fig. 1(c)).

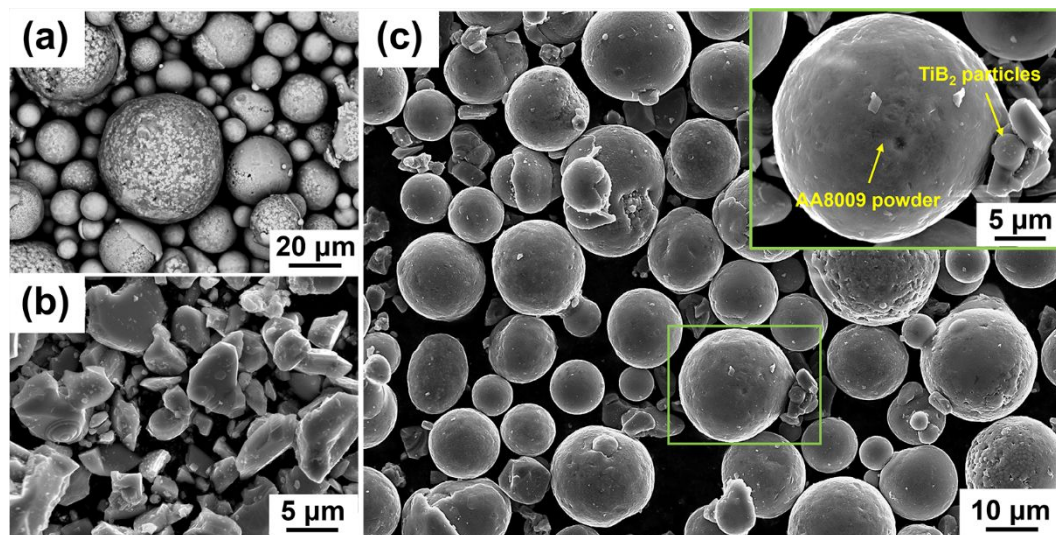


Fig. 1 SEM images: (a) AA8009 powder, (b) Micron-sized TiB₂ particles, (c) TiB₂/AA8009 powder mixture (inset: a high-magnification image).

All LPBF specimens (with a relative density $\geq 99\%$) were fabricated on a DiMetal-100 system in an argon atmosphere (purity ≥ 99.99 wt.%). The processing of the near-fully dense AA8009 alloy and TiB₂/AA8009 composite parts was carried out with the following laser parameters: scanning speed: 1200 mm/s, hatch style rotation: 90°, laser power: 350 W, layer thickness: 0.05 mm, hatch spacing: 0.08 mm. All specimens were fabricated using a single-melt continuous scanning strategy. Rectangular samples (6 mm $\times$ 8 mm $\times$ 12 mm) were made for the microstructure characterization, Columniform rods ($\varnothing$ 3 mm $\times$ 6 mm) were prepared for the compression test. The room temperature (RT) is considered as 298 K. The LPBF AA8009 and TiB₂/AA8009 parts were annealed at 373, 473, 573, 673, and 773 K for 6 h (named as AA8009 373 K, AA8009 473 K, AA8009 573 K, AA8009 673 K, and AA8009 773 K; TiB₂/AA8009 373 K, TiB₂/AA8009 473 K, TiB₂/AA8009 573 K, TiB₂/AA8009 673 K, and TiB₂/AA8009 773 K), followed by furnace cooling.

Phase analysis was performed via X-ray diffraction (XRD, 2 θ range: 30°-90°, scanning speed: 5°/min, Rigaku MiniFlex 600, Japan) with a Cu-K α target. For the corrosion of the sample surface, Keller reagent was used for 15 s. Microstructural characterization was carried out via a field emission scanning electron microscope (FE-SEM, FEI-Quanta 450-FEG, USA) equipped with an electron backscatter diffraction device (EBSD, EDAX-TSL system) and an energy-dispersive X-ray spectroscopy detector (EDS; OXFORD X-Max^N). All specimens were characterized along the building direction. Compression tests were conducted using a versatile material device (E44.304, MTS Co, USA) at a cross-head speed (0.001 mm/s). To guarantee the reproducibility of results, five samples of each category were measured.

3. Results and Discussion

3.1 XRD analysis

The XRD patterns of gas-atomized AA8009 powder, LPBF AA8009 alloy, and LPBF TiB₂/AA8009 composite as shown in Fig. 2. The α -Al phases and the Al_x(Fe, V)_ySi phases are found on the XRD patterns in all materials. These findings align with those obtained from studying rapid solidified AA8009 alloys [14]. The diffraction peaks of TiB₂ phase can be found in the TiB₂/AA8009 composite. The Al_x(Fe, V)_ySi phase peak has higher intensity in the TiB₂/AA8009 composite than in the AA8009 alloy, indicating that solute atoms (Fe, V, and Si) are less solid-dissolved in TiB₂/AA8009 composite than in the AA8009 alloy. The (111) peaks of α -Al phase shift to the higher diffraction angle from 37.94° (LPBF AA8009 alloy) alloy to 38.32° (LPBF TiB₂/AA8009 composite), which indicates the interplanar distance of the

TiB₂/AA8009 composite is reduced. Crystallography further proves that lattice parameter of α -Al in TiB₂/AA8009 composite (0.407 nm) is lower than that of AA8009 alloy (0.410 nm). The lattice parameter reduction of α -Al is caused by precipitation of Fe, V, Si elements. These findings imply more precipitation of Al_x(Fe, V)_ySi phase in LPBF TiB₂/AA8009 composite [25].

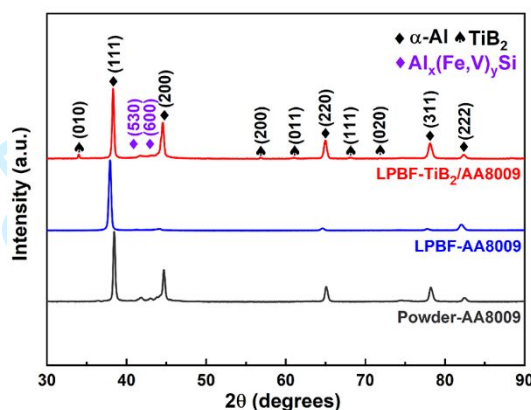


Fig. 2 XRD patterns of AA8009 powder, LPBF AA8009 alloy, and LPBF TiB₂/AA8009 composite.

3.2 Microstructure

The melt pools of the TiB₂/AA8009 composite and the AA8009 alloy appear to have similar microstructures in specific zones (MP and melt pool boundary (MPB)) as shown in Figs. 3(a) and (b). Owing to the higher cooling rate in MP than that of MPB, there is a lower solid solubility at the MPB [12,26], making for the formation of coarse precipitates at the MPB (the red boxes in Figs. 3(a), (b) insets). The similar scenario was also reported in literature [27,28].

However, there are no obvious nano-precipitates in the melt pool of AA8009 (Fig. 3(a)), which is more uniform than that of the TiB₂/AA8009 composite (Fig. 3(b)).

However, there are coarser $\text{Al}_{12}(\text{Fe,V})_3\text{Si}$ phases ($\sim 170 \text{ nm}$) at the MPB of $\text{TiB}_2/\text{AA8009}$ composite than that of AA8009 alloy ($\sim 5 \text{ nm}$), which has been indicated by our previous study [12]. According to EDS analysis, the contents of Ti and Al elements are $91 \pm 1 \text{ at.}\%$ and $9 \pm 1 \text{ at.}\%$ respectively, which can also be inferred that TiB_2 particles did not diffuse in $\alpha\text{-Al}$ during solidification and form a new phase, which is same to the phenomena in the previous references [21]. Meanwhile, The TiB_2 particles are usually added to aluminum alloys as nucleating agents, which makes the precipitation of second phase easier and accelerates the precipitation [22]. Therefore, comparing the melt pool center of LPBF $\text{TiB}_2/\text{AA8009}$ composite and LPBF AA8009 alloy (the green box in Figs. 3(a) and (b)), the precipitates of LPBF $\text{TiB}_2/\text{AA8009}$ composite is increased, and further demonstrates that the solid solution is reduced in LPBF $\text{TiB}_2/\text{AA8009}$ composite. More $\text{Al}_{12}(\text{Fe,V})_3\text{Si}$ phase precipitated in $\text{TiB}_2/\text{AA8009}$ composite, which is compatible with the XRD analysis.

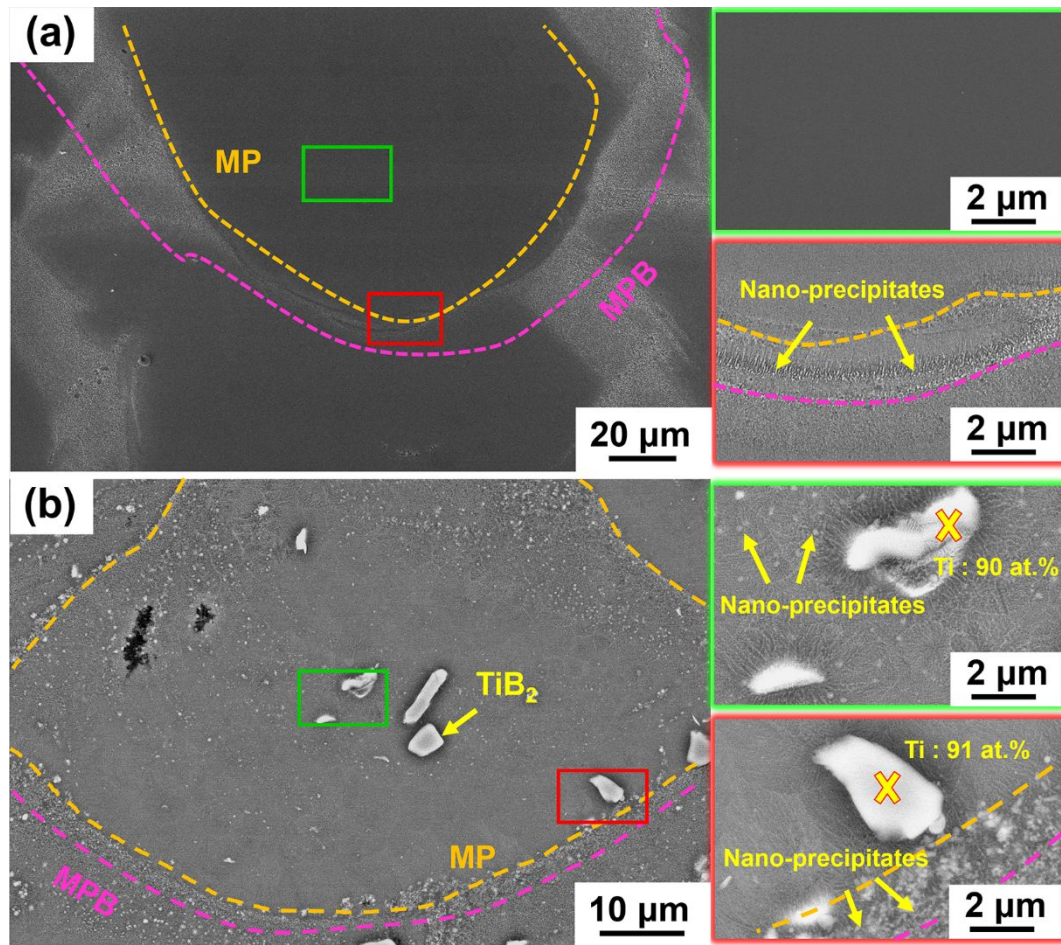


Fig. 3 SEM images: (a) LPBF AA8009 alloy, and (b) LPBF TiB₂/AA8009 composite. Insets are high-magnification images of the melt pool boundary (red box) and center (green box)).

Fig. 4 shows EBSD maps of the LPBF AA8009 alloy and the LPBF TiB₂/AA8009 composite. The main microstructure of LPBF AA8009 alloy (Fig. 4(a)) is a large columnar grain with an average grain size (Avg. GS) of $16 \pm 4 \mu\text{m}$. However, the LPBF TiB₂/AA8009 composite (Fig. 4(b)) is made of refined equiaxed grains with an Avg. GS of about $2.5 \pm 1 \mu\text{m}$. Micron TiB₂ particles can effectively promote grain refinement of the AA8009 alloy. Similar phenomena are also observed in LPBF TiB₂/Al-12Si composite [22] and TiB₂/CP-Ti composite [28]. The IPF plot confirms that the

orientation of these grains is more random in LPBF TiB₂/AA8009 composite, indicating its fiber texture-free characteristics. The presence of <111> fibrous texturing is observed in the LPBF AA8009 alloy, and the <111> fiber texture weakening of LPBF TiB₂/AA8009 composites is correspond with the above XRD analysis results (Fig. 2). In addition, the geometrically necessary dislocations (GND) of LPBF AA8009 alloy is $1.5 \times 10^{14} \text{ m}^{-2}$, the GND of LPBF TiB₂/AA8009 composite is $1.4 \times 10^{14} \text{ m}^{-2}$.

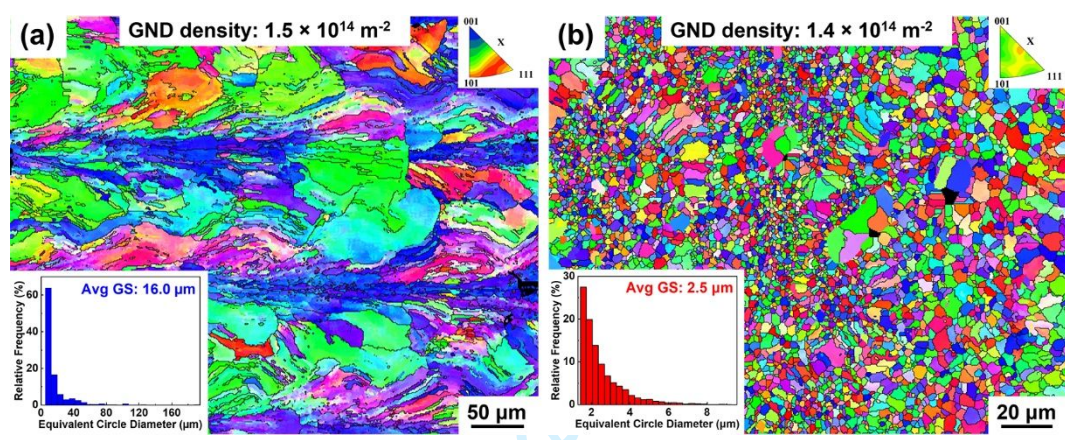


Fig. 4 EBSD maps of the materials on as-built condition: (a) LPBF AA8009 alloy and (b) LPBF TiB₂/AA8009 composite. Inset are the equivalent grain size diameters in EBSD maps.

3.3 XRD analysis associated with different heat treatment

The XRD patterns of LPBF AA8009 alloy and LPBF TiB₂/AA8009 composite without heat treatment and treated under different annealing temperatures as shown in Fig. 5. The Al_x(Fe, V)_ySi phase's diffraction peak angle steadily lowers as the annealing temperature increases. This is because there are fewer solid dissolved elements in the α-Al matrix. The (530) and (600) peaks of Al_x(Fe, V)_ySi phase gradually become more evident. When the annealing temperature is 673 K, more elements are precipitated from

the α -Al matrix [29], and the (200) peak of TiB_2 phase becomes evident in the LPBF $\text{TiB}_2/\text{AA8009}$ composite.

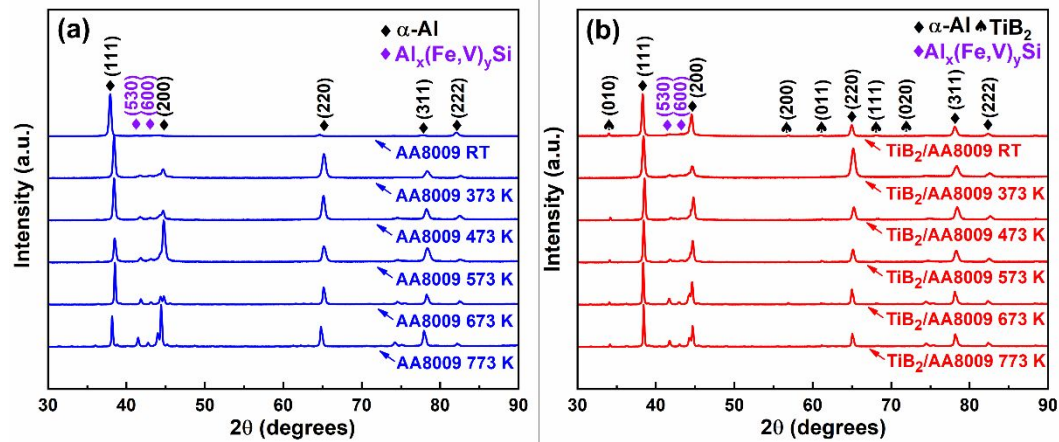


Fig. 5 XRD patterns of (a) LPBF AA8009 alloy and (b) LPBF $\text{TiB}_2/\text{AA8009}$ composite processed in different heat treatment conditions.

3.4 Microstructure associated with different heat treatment

The phase and microstructure of LPBF AA8009 alloy (Figs. 6(a1-a5)) and LPBF $\text{TiB}_2/\text{AA8009}$ composite (Figs. 6(b1-b5)) were further characterized to understand the annealing effect. As the annealing temperature increases from 373 K to 773 K, the melt pool area in LPBF AA8009 alloy increases 38.3 %. At the temperature of 573-673 K, the morphology of the melt pool becomes irregular, and phase particles are precipitated inside the melt pool. The solid solution elements are precipitated to form particles at the MPB. The same phenomenon was also found in the LPBF AA8009 alloy [14]. Sun et al. [30] found that the impact of the Fe volume diffusion causes the $\text{Al}_{12}(\text{Fe,V})_3\text{Si}$ phase to coarsen after annealing the AA8009 alloy at 673 K. During the annealing process of LPBF $\text{TiB}_2/\text{AA8009}$ composites (Figs. 6(b1-b5)), the content of $\text{Al}_{12}(\text{Fe,V})_3\text{Si}$ phases gradually increases in melt pool, its morphology becomes more

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4 irregular compared with LPBF AA8009 alloys. Meanwhile, at the annealing
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6 temperature of 673 K, a lot of fine $\text{Al}_{12}(\text{Fe,V})_3\text{Si}$ phases are noted throughout the melt
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8 pool. A more interesting phenomenon is that the melt pool structure of the two materials
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10 disappears after annealing at 673 K (Figs. 6(a4) and (b4)). Therefore, to further
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12 investigate the influence of temperature, the parts after 673 K annealing were selected
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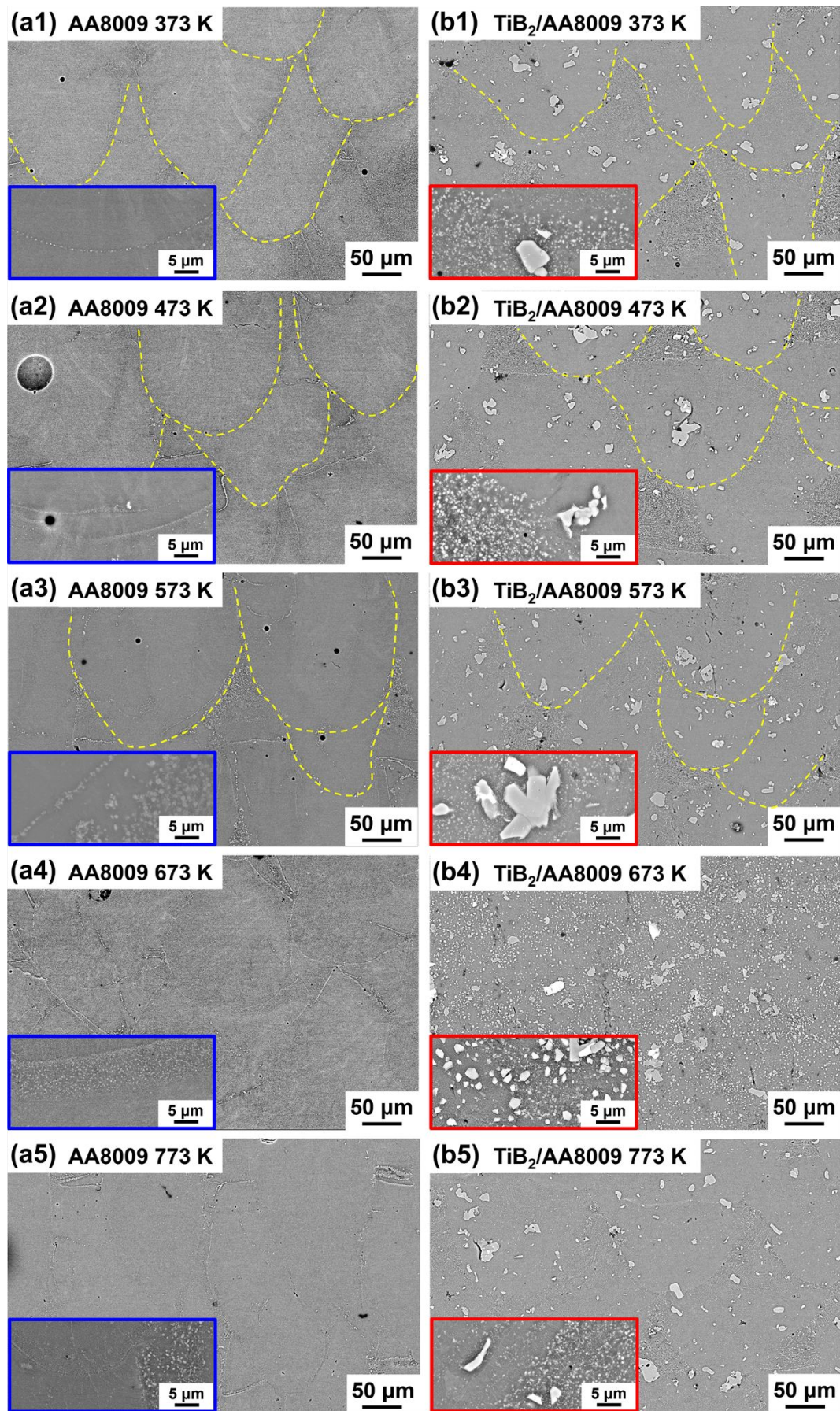


Fig. 6 SEM images of the (a1-a5) LPBF AA8009 alloy and (b1-b5) LPBF

TiB₂/AA8009 composite annealed for 6 h at different temperatures (373-773 K). Insets are the SEM micrographs for the melt pool boundary.

The EBSD maps of LPBF AA8009 alloy and TiB₂/AA8009 composites annealed at 673 K are shown in Fig. 7. Apparent grain coarsening is observed for the LPBF AA8009 annealed at 673 K, and the Avg. GS of the specimen increases significantly from $16.0 \pm 4 \mu\text{m}$ (Fig. 4(a)) to $28.6 \pm 2 \mu\text{m}$ (Fig. 7(a)). According to the IPF, the strong $\langle 111 \rangle$ texture of the LPBF AA8009 alloy remains, this is corresponded with the results of XRD patterns (Fig. 5(a)). The GND density of the LPBF AA8009 alloy specimen also decreases from $1.5 \times 10^{14} \text{ m}^{-2}$ to $1.4 \times 10^{14} \text{ m}^{-2}$ after annealing. Fig. 7(b) shows the EBSD IPF map of the LPBF TiB₂/AA8009 composite annealed at 673 K. Small grain size is kept in the composite even though the annealing procedure causes a minor rise in the Avg. GS of the specimen from $2.5 \pm 1 \mu\text{m}$ (Fig. 4(b)) to $5.2 \pm 1 \mu\text{m}$ (Fig. 7(b)). The coarsened α -Al grains of the composite show strong $\langle 111 \rangle$ and $\langle 001 \rangle$ textures as presented in Fig. 7 (b), and the GND density of the specimen decreases from $1.4 \times 10^{14} \text{ m}^{-2}$ to $0.6 \times 10^{14} \text{ m}^{-2}$.

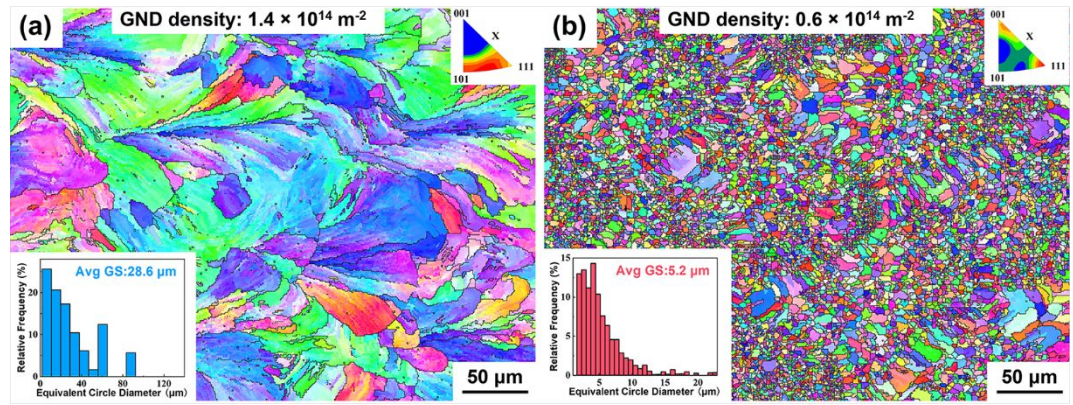


Fig. 7 EBSD maps for (a) LPBF AA8009 alloy 673 K and (b) LPBF TiB₂/AA8009

composites 673 K. Insets show the equivalent grain size diameter in EBSD maps.

3.5 Mechanical properties and strengthening mechanisms

3.5.1 Mechanical properties

Fig. 8 shows the compressive yield strength (CYS) and stress-strain curves of LPBF AA8009 alloy and LPBF TiB₂/AA8009 composite before and after annealing. It is noteworthy that none of the specimens fractured up to 35 % strain and the LPBF TiB₂/AA8009 composite has compressive plastic deformability similar to that of the LPBF AA8009 alloy, indicating that the addition of TiB₂ has less effect on the compressive plasticity of AA8009 matrix. During annealing, the CYS value keeps decreasing. And the CYS of LPBF AA8009 alloys is always higher than that of LPBF TiB₂/AA8009 composites. The CYS slightly decreased from 705 ± 16 MPa to 646 ± 3 MPa for LPBF AA8009 alloy and from 466 ± 23 MPa to 458 ± 7 MPa for LPBF TiB₂/AA8009 composite from 298 K to annealing temperature 573 K. During this annealing temperature range, only insignificant changes take place in the microstructure of LPBF AA8009 alloy and LPBF TiB₂/AA8009 composite (Figs. 6(a1-a3) and (b1-b3)). After annealing at 673 K, the compressive yield strength declines sharply to 459 ± 30 MPa (about 71.1 % of compressive yield strength of the counterpart annealed at 573 K) for LPBF AA8009 alloy and 368 ± 3 MPa (about 80.3 % of compressive yield strength of the counterpart annealed at 573 K) for LPBF TiB₂/AA8009 composite. Compared with LPBF TiB₂/AA8009 composite, the compressive yield strength of LPBF AA8009 alloy drops more, this indicates that the

TiB₂ particles can curb the drop in yield strength [30]. After annealing at 773 K, the CYS values of LPBF AA8009 alloys and LPBF TiB₂/AA8009 composites decrease to 399 ± 14 MPa and 349 ± 15 MPa, respectively. So as to clarify the evolution of mechanical properties clearly, the strengthening mechanisms are discussed as following.

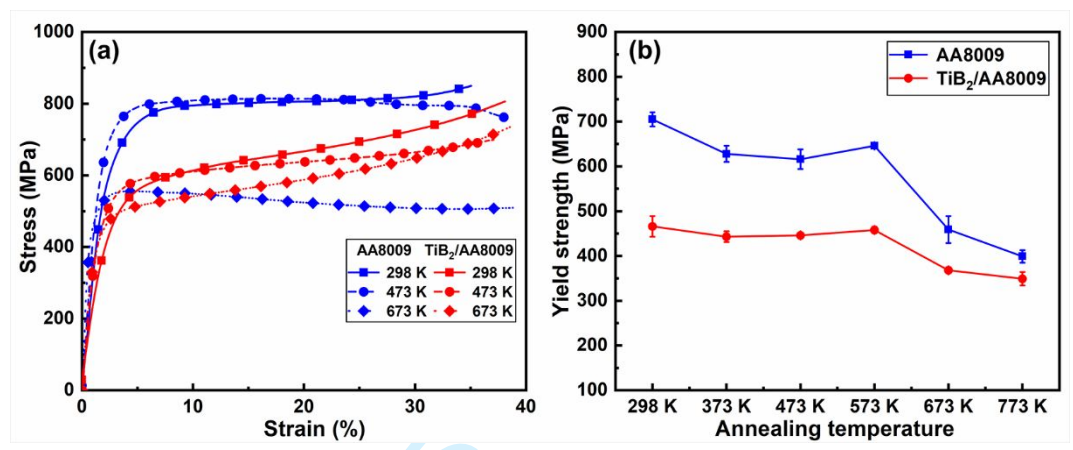


Fig. 8 The compressive stress-strain curves (a) and CYS curves (b) of LPBF AA8009 alloys and LPBF TiB₂/AA8009 composites before and after different temperatures annealing.

3.5.2 Strengthening mechanism

According to our previous work [12], this work combines the microstructure of the material and analyzes the four strengthening mechanisms of LPBF AA8009 alloy: grain refinement strengthening (σ_{hp}), Orowan strengthening (σ_{or}), solid solution strengthening (σ_{ss}), and dislocation strengthening (σ_{dis}) [7,23]. Therefore, the "calculated yield stress" ($\sigma_{A,cal,RT}$) of AA8009 alloy is defined as:

$$\sigma_{A,cal,RT} = \sigma_0 + \sigma_{A,hp,RT} + \sigma_{A,or,RT} + \sigma_{A,ss,RT} + \sigma_{A,dis,RT} \quad (1)$$

According to EDS analysis of LPBF AA8009 373 K and TiB₂/AA8009 373 K, the contents of Al and Fe elements are 95 ± 1 at.% and 5 ± 1 at.%, respectively, but V and

Si elements are not found in the α -Al matrix, which can be inferred that the solid solution of alloying elements reduces dramatically with increasing annealing temperature. Meanwhile, Khatri et al confirm that the matrix consists primarily of aluminum with low levels of other alloying elements, and the low solubility and diffusivity of Fe and V in solid aluminum result in low coarsening rates in the study of Al-Fe-V-Si alloy [31]. Due to the precipitation of more secondary phases during the annealing process, it can be seen in Fig. 6 (a4) that the melt pool and the MPB have developed rounded granular phases. The obvious grow-up of the $\text{Al}_{12}(\text{Fe,V})_3\text{Si}$ phases seems stable after increasing annealing temperature, which indicates that the solid solubility of alloying elements is too less to support the further grow-up. Therefore, in the theoretical analysis of the AA8009 673 K, all the solid solution strengthening can be ignored, and the calculation of the yield stress ($\sigma_{A,cal,673K}$) can be simplified do:

$$\sigma_{A,cal,673K} = \sigma_0 + \sigma_{A,sp,673K} + \sigma_{A,gr,673K} + \sigma_{A,dis,673K} \quad (2)$$

where σ_0 is the friction stress, available in the literature ($\sigma_0 = 72$ MPa) [18]. Refined alloys and composites change in grain size can lead to changes in their strength. This change in grain boundary bulk density by changing the grain size leads to a change in material strength that can be evaluated by the Hall-Petch equation (σ_{hp}), which is defined as [32]:

$$\sigma_{hp} = kd^{-1/2} \quad (3)$$

where d is the Avg. GS ($d_{RT} = 16.0$ μm measured in Fig. 4 (a), and $d_{673K} = 28.6$ μm measured in Fig. 7 (a)), and $k = 0.04$ MPa $\text{m}^{1/2}$, which is the Hall-Petch coefficient [33].

The values of grain refinement strengthening of AA8009 alloy at 298 K and 673 K can

be directly calculated as 10 MPa and 7 MPa, respectively.

The rate of dislocation migration during rapid solidification is limited by dense dislocations [18]. To calculate the value of dislocation strengthening, geometrically necessary dislocations (GND) are a crucial component. Using the Bailey-Hirsch equation, it is possible to calculate the connection between GND and dislocation strengthening [12]:

$$\sigma_{dis} = M_{Taylor} \alpha G b \sqrt{\rho_d} \quad (4)$$

where, b is Burgers' constant (0.286 nm) [12], according to the analysis of EBSD data, G is the shear modulus of pure aluminum ($G = 26.5$ GPa), ρ_d is the GND value, α is the material correlation constant ($\alpha = 0.16$), the M_{Taylor} of AA8009 alloy is 3.33 [34]. The GND value of LPBF AA8009 alloy is $1.5 \times 10^{14}/\text{m}^2$, and the GND value of LPBF AA8009 alloy annealed 673 K is $1.4 \times 10^{14}/\text{m}^2$. Via calculation, the contributions of dislocation strengthening for AA8009 alloy 298 K and 673 K are 54 MPa and 44 MPa, respectively.

One way to describe σ_{or} contribution is as follows:

$$\sigma_{or} = \frac{0.4 M G b \ln \frac{r\pi}{2b}}{\pi L \sqrt{1-\nu}} \quad (5)$$

where $L = r \sqrt{\left(\frac{2\pi}{3f} - \frac{\pi}{2}\right)}$, r is the average particle radius, f is the volume fraction of the second phase. The average particle size of the $\text{Al}_{12}(\text{Fe,V})_3\text{Si}$ phase was measured in LPBF AA8009 alloy at 298 K and 673 K is 5 nm and 75 nm, respectively, as shown in Figs. 3(a) and 6(a4). Orowan strengthening of LPBF AA8009 alloy at 298 K and 673 K contributes 243 MPa and 450 MPa, respectively.

The Fe, Si, and V atoms in LPBF AA8009 alloy diffuse more easily into the

aluminum matrix under the high cooling rate's help, where differences in the number, size, and shear modulus of the solute atoms lead to local strain fields that impede dislocations and atomic motion in the lattice. For this solid solution strengthening enhanced by supersaturated atoms can be described as [35]:

$$\sigma_{SS} = M_{Taylor} \left(\frac{3}{8} \right)^{\frac{2}{3}} \left(\frac{1+v}{1-v} \right)^{\frac{4}{3}} \left(\frac{w}{b} \right)^{\frac{1}{3}} G |\varepsilon|^{\frac{4}{3}} c^{\frac{2}{3}} \quad (6)$$

where v is the Poisson's ratio of aluminum elements (0.345), M_{Taylor} is set to 3.33, $w = 5b$, and ε is the lattice mismatch strain (Fe: 4.6, Si: 0.87, V: 3.11) [35]. c is the atomic fraction of solute elements in the Al solid solution [from ref. 12]. The solid solution strengthening's estimated contribution to yield strength is 375 MPa.

In summary, the calculated CYS of LPBF AA8009 alloy at RT and after 673 K annealing are 754 MPa and 573 MPa, respectively.

The TiB_2 particles are added to AA8009 alloy as nucleating agent can reduce the solid solubility of Fe, V, and Si elements, and form a large number of precipitated phases.[22] Therefore, solid solution strengthening can be ignored in the CYS calculation of LPBF TiB_2 /AA8009 composite.

To more fully describe how TiB_2 particles affect the mechanical characteristics, the strengthening mechanism based on microstructural characteristics needs to be more clearly defined. Therefore, this work combines the microstructure of the material and analyzes the four strengthening mechanisms of LPBF TiB_2 /AA8009 composite: grain refinement strengthening (σ_{hp}), Orowan strengthening (σ_{or}), load boundary strengthening (σ_{lbs}), and dislocation strengthening (σ_{dis}) [7,23]. The strengthening mechanism ($\sigma_{B,cal}$) of LPBF TiB_2 /AA8009 composite at 298 K versus 673 K can be

described as:

$$\sigma_{B,cal,RT} = \sigma_0 + \sigma_{B,hp,RT} + \sigma_{B,or,RT} + \sigma_{lbs} + \sigma_{B,dis,RT} \quad (7)$$

$$\sigma_{B,cal,673K} = \sigma_0 + \sigma_{B,hp,673K} + \sigma_{B,or,673K} + \sigma_{lbs} + \sigma_{B,dis,673K} \quad (8)$$

where σ_{hp} is the grain refinement strengthening, whose specific strengthening value can be calculated from Eq. (3); the strengthening data of σ_{dis} calculated in the same way as Eq. (4). The grain size and GND density data of LPBF TiB₂/AA8009 composite at 298 K and 673 K, respectively, can determine that grain refinement strengthening contributes 25 MPa and 17 MPa, respectively, and dislocation strengthening contributes 46 MPa and 29 MPa, respectively.

In LPBF AA8009 alloy, the Al₁₂(Fe,V)₃Si phase exists only in MPB (Fig. 3(a)), and Eq. (5) is more suitable for partition calculation of Orowan strengthening. However, in LPBF TiB₂/AA8009 composite, the Al₁₂(Fe,V)₃Si phase exists in the entire melt pool, and Eq. (9) is more suitable for computing non-partitioned Orowan strengthening. Therefore, using different calculation formulas for two different alloys can make the calculation more accurate. Unlike the LPBF AA8009 alloy, the expression of σ_{or} for the LPBF TiB₂/AA8009 composite is defined as:

$$\sigma_{or} = \frac{4\alpha Gb}{d_p} \left(\frac{6v_p}{\pi} \right)^{\frac{1}{3}} \quad (9)$$

where d_p and v_p are the average particle size and volume fraction of the Al₁₂(Fe,V)₃Si phase [12], and the volume fraction annealed at 298 K and 673 K is 1.86 vol.% and 4.01 vol.%, and the d_p of LPBF TiB₂/AA8009 composites annealed at 298 K and 673 K are 4 nm and 12 nm, respectively. The Orowan strengthening values of 298 K and 673 K annealed LPBF TiB₂/AA8009 composites are 249 MPa and 166 MPa,

respectively.

A good bonding interface is a key factor for load transfer from the softer α -Al matrix to the harder TiB_2 particles, and this strengthening mechanism is defined as load boundary strengthening (σ_{lbs}) [23]. Due to the consistent crystal orientation between TiB_2 particles and α -Al, the strongly bonded interface can increase σ_{lbs} [36,37]. The contribution of σ_{lbs} can be defined as:

$$\sigma_{lbs} = 1.5V_{\text{TiB}_2}\sigma_{\text{TiB}_2/\text{Al}} \quad (10)$$

where V_{TiB_2} is the volume fraction of TiB_2 particles (5 vol.%), and $\sigma_{\text{TiB}_2/\text{Al}}$ is the bond strength of TiB_2 with α -Al (676 MPa) [23]. The strength contribution of all σ_{lbs} calculations is kept at 51 MPa because the effect of TiB_2 particles at high-temperature conditions is negligible. In summary, the calculated CYS of LPBF $\text{TiB}_2/\text{AA8009}$ composites at RT and after 673 K annealing are 443 MPa and 355 MPa respectively.

According to the quantitative analysis presented above, the contributions of each strengthening mechanism are depicted in Fig. 9, which includes the calculated and measured compressive yield strengths of LPBF AA8009 alloy and LPBF $\text{TiB}_2/\text{AA8009}$ composite at 298 K and annealed 673 K. The theoretical CYS value of LPBF AA8009 alloy treated at RT is 754 MPa, and solid solution strengthening and Orowan strengthening contributed 375 MPa and 243 MPa, respectively. The deviation from the measured value (705 ± 16 MPa) is 7.0 %. The deviation between the theoretical CYS value of 573 MPa and the measured yield strength (459 ± 30 MPa) of LPBF AA8009 alloy annealed at 673 K is 24.8 %, and Orowan strengthening contributed 450 MPa. The σ_{ss} is the main reason for the better CYS of LPBF AA8009 alloy, but the σ_{ss}

becomes σ_{or} (decrease by 168 MPa) leads to a sharp drop in CYS value of LPBF AA8009 alloy after 673 K annealing. The theoretical CYS value of 298 K LPBF TiB₂/AA8009 composite is 443 MPa, and the deviation from the actual compressive yield strength value (466 ± 23 MPa) is 4.9 %. The estimated compressive yield strength value of the LPBF TiB₂/AA8009 composite annealed at 673 K is 355 MPa, and the deviation from the actual compressive yield strength value (368 ± 3 MPa) is 3.5 %. The computed outcomes demonstrate that the theoretical results and the experimental results are in strong accord. Due to the reduction of solid solution elements in the α - Al matrix, the second phase precipitates more in LPBF TiB₂/AA8009 composite at 298 K and after 673 K annealing treatments. Therefore, the σ_{or} is the major strengthening mechanism. And the CYS decrease of LPBF/AA8009 composite is mainly due to the weak decrease (~ 83 MPa) of Orowan strengthening after 673 K annealing. Therefore, TiB₂ particles can suppress the drop in yield strength of LPBF AA8009 alloy below 673 K.

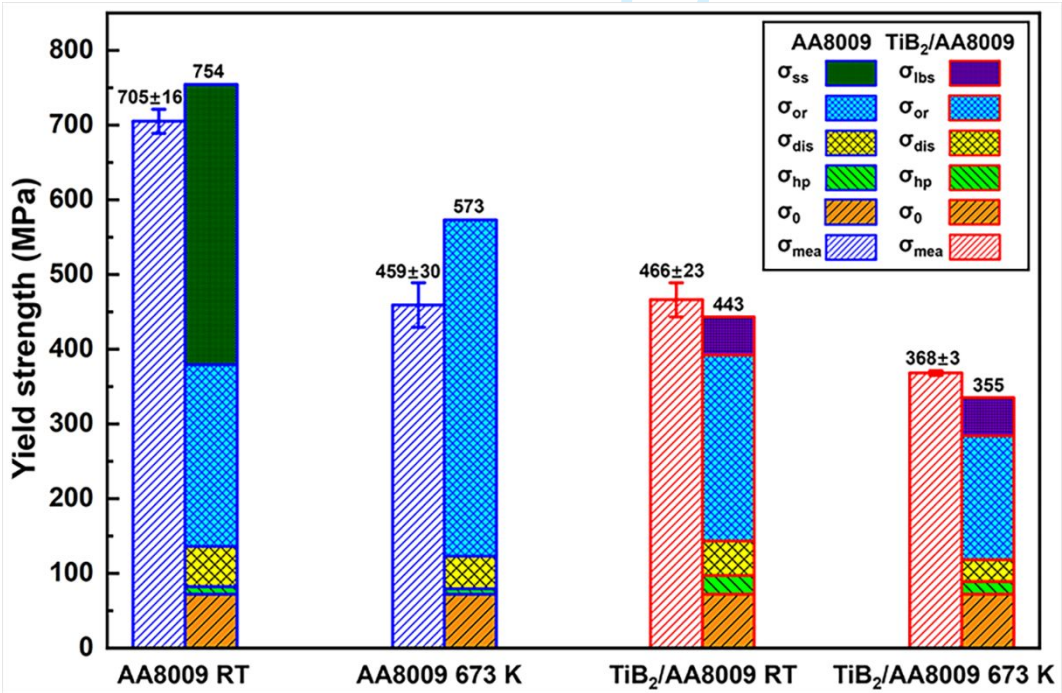


Fig. 9 Compressive yield strength of measured and theoretically calculated values

(AA8009 RT, AA8009 673 K, TiB₂/AA8009 RT, and TiB₂/AA8009 673 K).

4. Conclusions

In this work, TiB₂/AA8009 composite was fabricated by using LPBF. The mechanical properties of LPBF AA8009 alloy and LPBF TiB₂/AA8009 composite (298 K and after 373-773 K annealing treatment) were systematically investigated through microstructure characterization and strengthening mechanism. The conclusions are summarized below:

1. The Al₁₂(Fe,V)₃Si phase is precipitated at center and boundary of melt pool in the LPBF TiB₂/AA8009 composite but only at the MPB in the LPBF AA8009 alloy. However, after annealing 673 K, the precipitation of Al₁₂(Fe,V)₃Si phase in MP is also found in the LPBF AA8009 alloy.

2. The 5 vol.% TiB₂ particles promoted the grain refinement (reduced Avg. GS from 16.0 μm to 2.5 μm) and transformation of columnar grains (LPBF AA8009 alloy) to equiaxed grains (LPBF TiB₂/AA8009 composite). After annealing at 673 K, the grains of LPBF AA8009 alloy and LPBF TiB₂/AA8009 composite are coarsened to 28.6 μm and 5.2 μm, respectively. The weakening of fiber texture along <111> direction in the TiB₂/AA8009 composite.

3. The LPBF AA8009 alloy and LPBF TiB₂/AA8009 composite exhibit CYS of 705 ± 16 MPa and 466 ± 23 MPa, respectively. The TiB₂/AA8009 composite shows lower CYS than the LPBF AA8009 alloy owing to the weakening of σ_{ss} caused by reduction of Fe, V, and Si elements in composite.

4. The compressive yield strength of both LPBF AA8009 alloy and LPBF TiB₂/AA8009 composite after annealing 673 K drops sharply by 246 MPa and 98 MPa, respectively. The sharp CYS decrease of LPBF AA8009 alloy is caused by the disappearance of σ_{ss} . But the CYS decrease of LPBF TiB₂/AA8009 composite results from the drop of the Orowan strengthening caused by coarsen of precipitates in the composite.

5. The addition of TiB₂ particles causes that the CYS of TiB₂/AA8009 composite before and after annealing are lower than that of AA8009 alloy due to the change in the strengthening mechanism of two kinds of materials. In addition, TiB₂ particles can suppress the drop in yield strength of AA8009 alloy due to the 2.5 times CYS drop of AA8009 alloy than that of the composite after 673 K annealing.

Acknowledgments

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Declarations

The authors state that there are no conflicts of interest to disclose.

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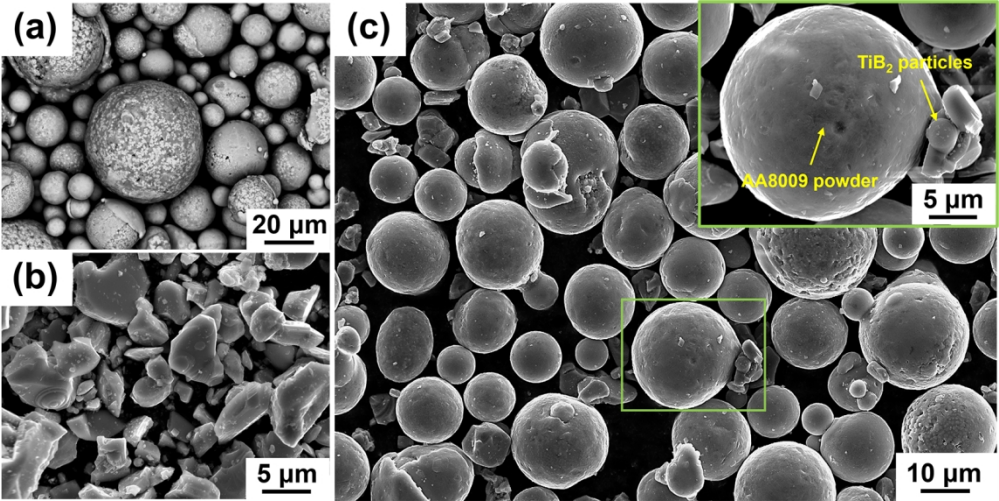


Fig. 1 SEM images: (a) AA8009 powder, (b) Micron-sized TiB₂ particles, (c) TiB₂/AA8009 powder mixture (inset: a high-magnification image).

140x70mm (300 x 300 DPI)

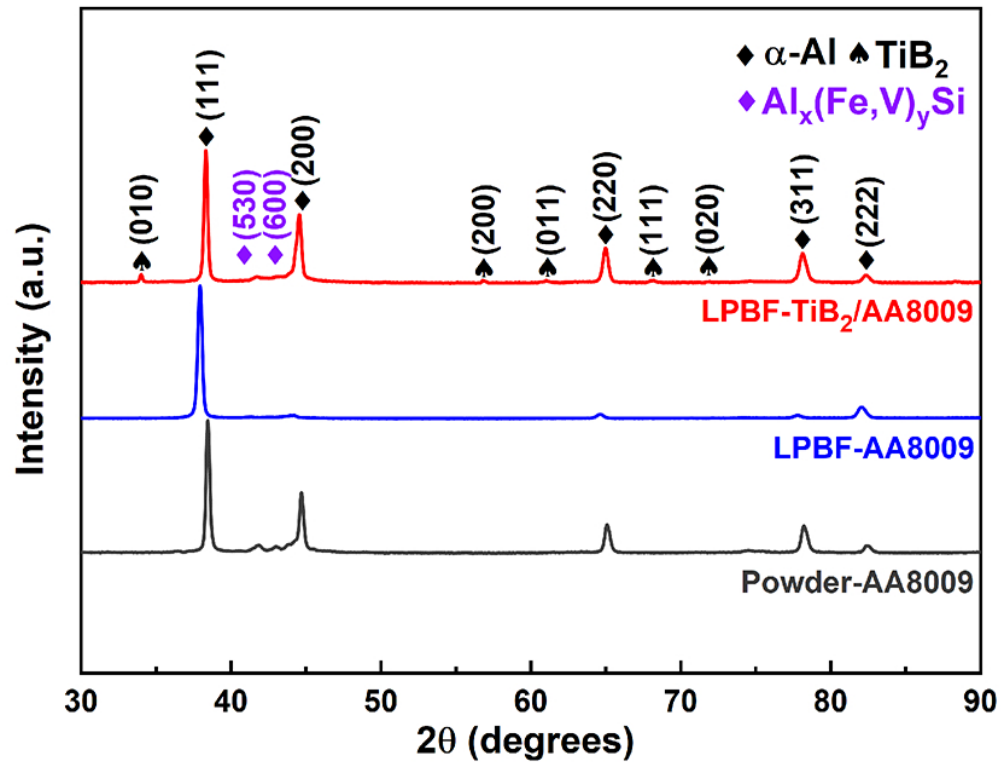


Fig. 2 XRD patterns of AA8009 powder, LPBF AA8009 alloy, and LPBF TiB₂/AA8009 composite.

70x53mm (300 x 300 DPI)

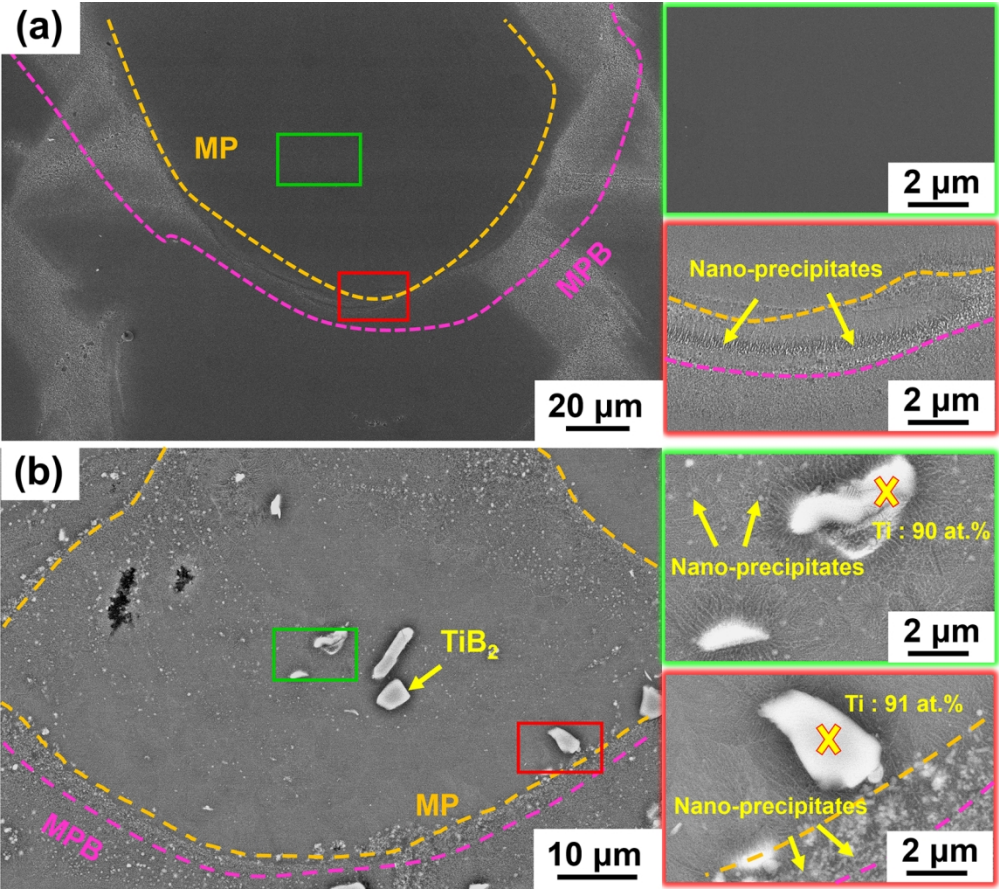


Fig. 3 SEM images: (a) LPBF AA8009 alloy, and (b) LPBF TiB₂/AA8009 composite. Insets are high-magnification images of the melt pool boundary (red box) and center (green box)).

140x124mm (300 x 300 DPI)

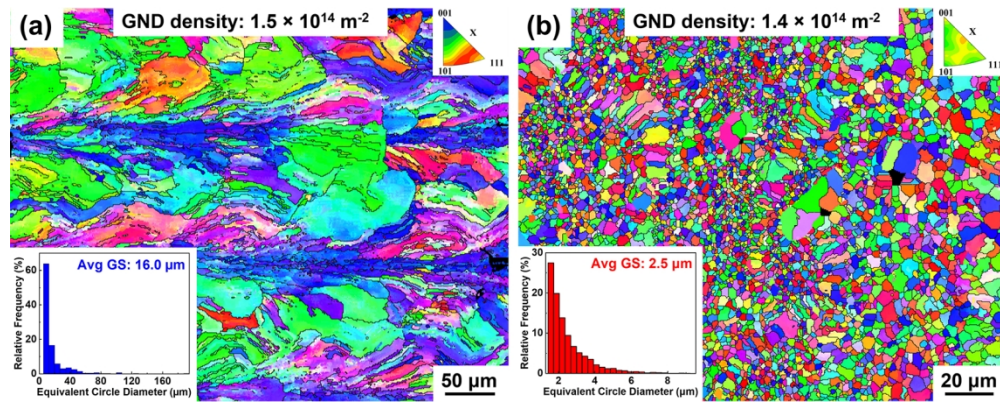


Fig. 4 EBSD maps of the materials on as-built condition: (a) LPBF AA8009 alloy and (b) LPBF TiB2/AA8009 composite. Inset are the equivalent grain size diameters in EBSD maps.

140x55mm (300 x 300 DPI)

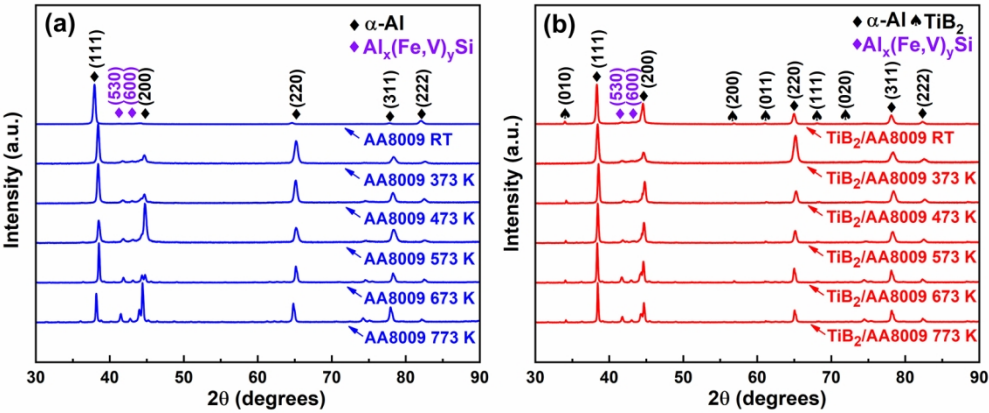


Fig. 5 XRD patterns of (a) LPBF AA8009 alloy and (b) LPBF $\text{TiB}_2/\text{AA8009}$ composite processed in different heat treatment conditions.

140x58mm (300 x 300 DPI)

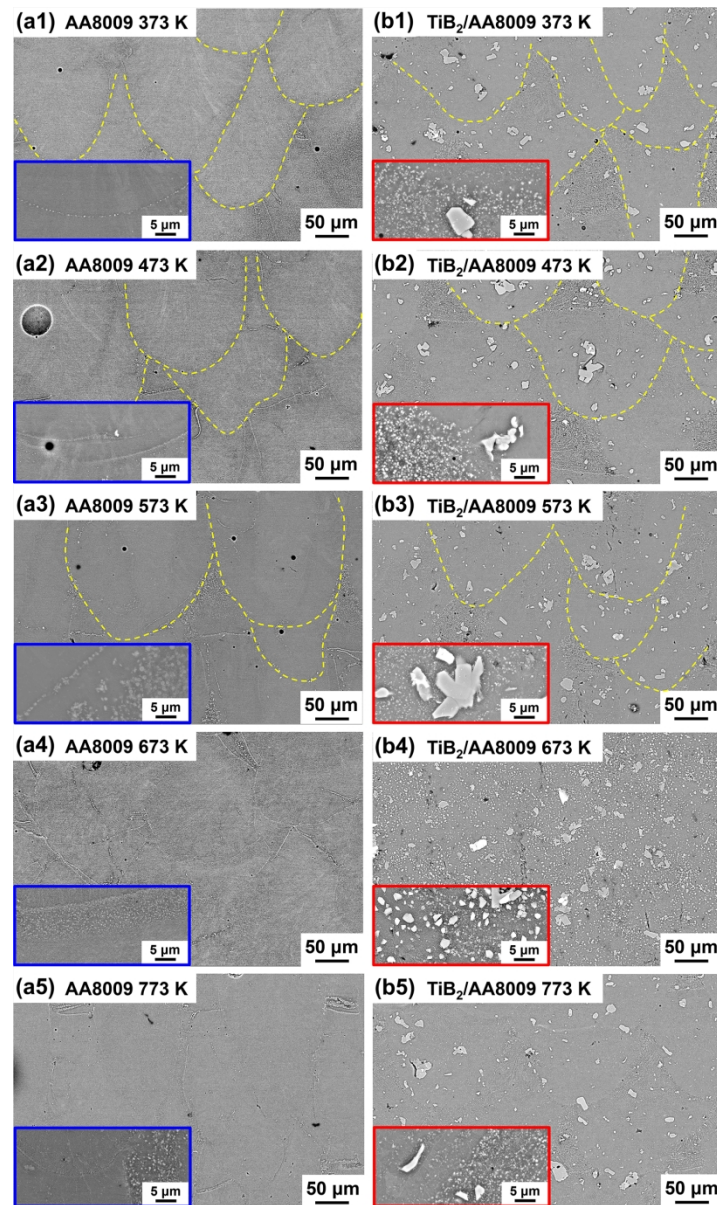


Fig. 6 SEM images of the (a1-a5) LPBF AA8009 alloy and (b1-b5) LPBF TiB₂/AA8009 composite annealed for 6 h at different temperatures (373-773 K). Insets are the SEM micrographs for the melt pool boundary.

140x236mm (300 x 300 DPI)

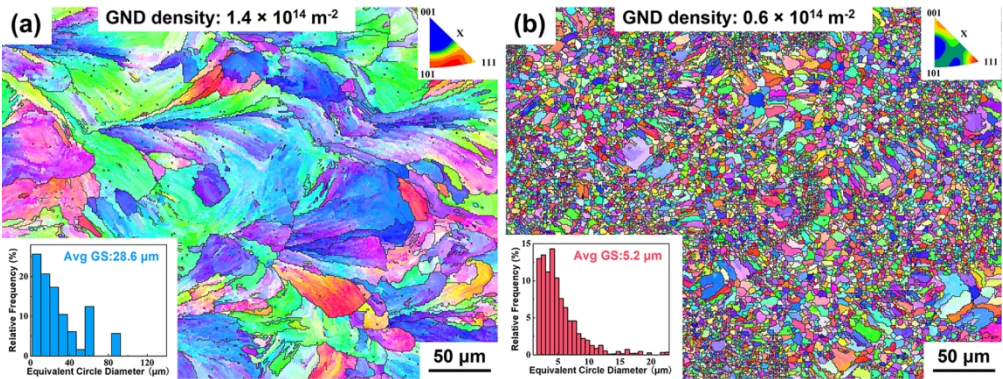


Fig. 7 EBSD maps for (a) LPBF AA8009 alloy 673 K and (b) LPBF TiB2/AA8009 composites 673 K. Insets show the equivalent grain size diameter in EBSD maps.

140x52mm (300 x 300 DPI)

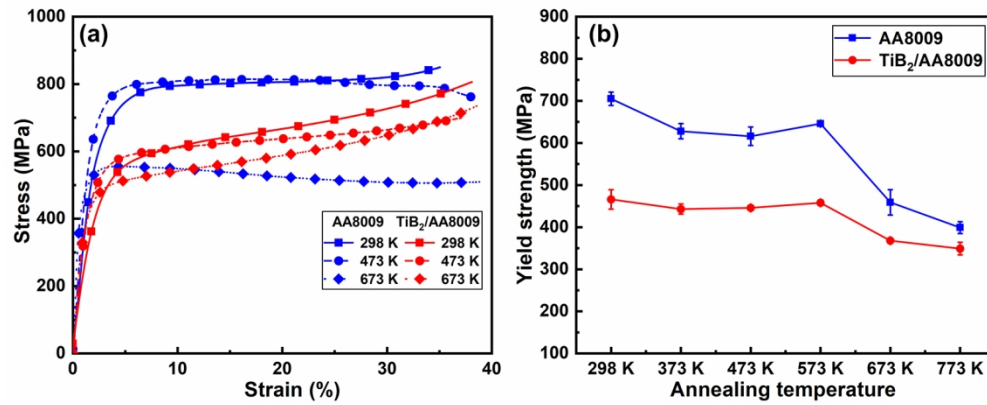


Fig. 8 The compressive stress-strain curves (a) and CYS curves (b) of LPBF AA8009 alloys and LPBF TiB₂/AA8009 composites before and after different annealing temperatures.

140x57mm (300 x 300 DPI)

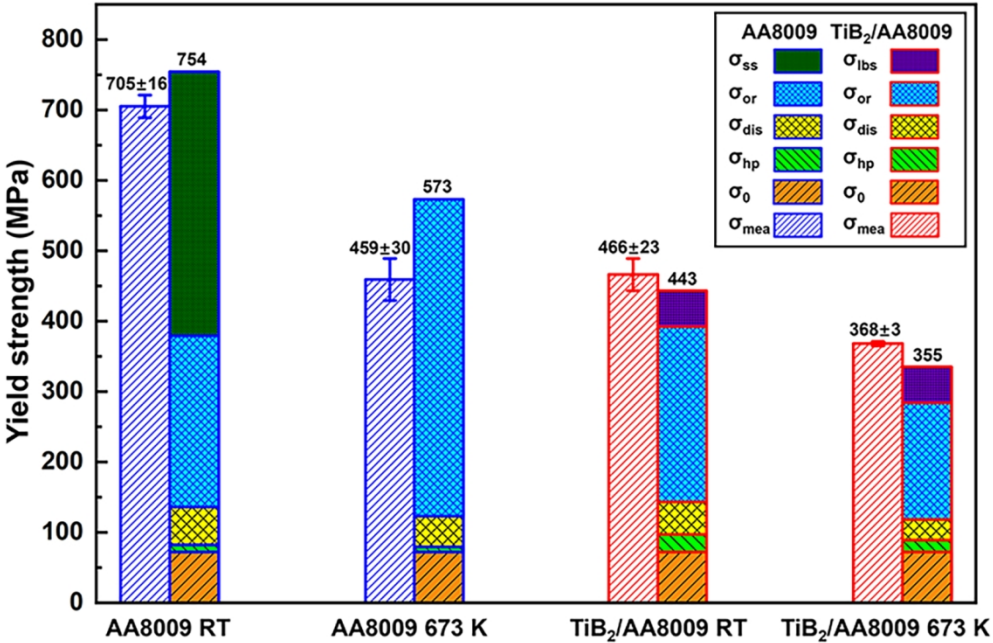


Fig. 9 Compressive yield strength of measured and theoretically calculated values (AA8009 RT, AA8009 673 K, TiB₂/AA8009 RT, and TiB₂/AA8009 673 K).

140x91mm (300 x 300 DPI)

Authors' response (Manuscript: Acta Metallurgica Sinica (English Letters) _ AMS-2023-0281.R1)

Dear editor and reviewers:

We would like to thank all reviewers for carefully reading our manuscript and for providing critical but constructive suggestions. We really appreciated all comments and questions raised, since they helped us to significantly improve our manuscript.

Reviewer 1:

1. There are two factors in thermal stability. One is temperature, and the other is the duration time in that temperature. If there no variation in heat treatment time, the author cannot determine how stable the material is at that temperature. As a result, the reviewer recommends to revise the thermal stability to the influence of temperature in the manuscript. "Thermal stability" in the title, abstract, and main text should be revised.

Answer:

Thank you for your recommendations. As you suggested, the title has been changed to "Effect of TiB₂ addition on microstructure and mechanical properties of AA8009 alloy fabricated by laser additive manufacturing", and "thermal stability" in the abstract and main texts have been revised. The relevant changes are already marked with red in the manuscript.

2. The reviewer has checked reference [31]. There is no "According to previous studies, with increasing annealing temperature, the solid solubility of alloying elements will reduce dramatically, which also leads to a significant decrease in the solid solution strengthening until this strengthening is ignored". The reviewer is curious about the solid solution contribution at high temperature conditions.

Answer:

Thanks for your comments. According to EDS analysis of LPBF AA8009 373 K and TiB₂/AA8009 373 K (Fig. 1), the contents of Al and Fe elements are 95 ± 1 at.% and 5 ± 1 at.%, respectively, but V and Si elements are not found in the α -Al matrix, which can be inferred that the solid solution of alloying elements reduces dramatically with increasing annealing temperature. Meanwhile, Khatri et al confirm that the matrix consists primarily of aluminum with low levels of other alloying elements, and the low

solubility and diffusivity of Fe and V in solid aluminum result in low coarsening rates in the study of Al-Fe-V-Si alloy [Mater. Sci. Eng. A **167**, 11 (1993)]. Therefore, we consider there is no solid solution strengthening for the strengthening mechanism of AA8009 673 K and TiB₂/AA8009 composite according to these analyses. The relevant supplements and changes are already marked with red in the manuscript.

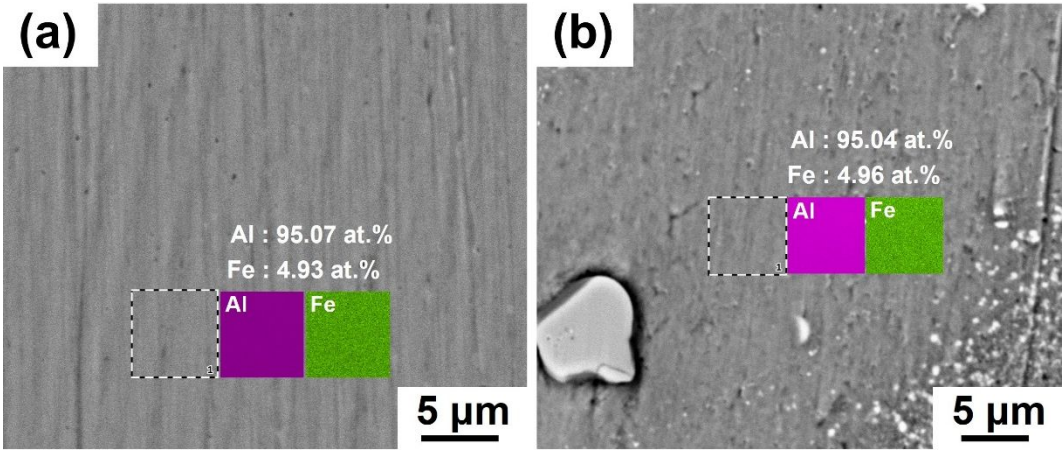


Fig. 1 The SEM images and EDS maps of (a) LPBF AA8009 373 K and (b) LPBF TiB₂/AA8009 373 K

3. There are typos or formatting issues (subscript), e.g., (Al₁₂(Fe, V)₃Si) in section 3.5.2. Please check the manuscript carefully for the next round of revision.

Answer:

Thanks for your careful checks. The manuscript is carefully checked for formatting issues, e.g., “Al₁₂(Fe, V)₃Si”, “at%” and “TiB₂”, and the issues have been revised. The relevant changes are already marked with red in the manuscript.

That’s the reviewers' questions and our response. Relevant revisions will be marked in red in the submitted manuscript, please check it.

At the same time, I hope to get your suggestions and corrections again.

I wish you good health and happy work.

Sincerely,

Pei Wang