In-situ observation of temperature effect on the tensile deformation behavior in laser melting deposited TA15 alloy

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Abstract. In this study, laser melting deposited TA15 alloy was examined by in-situ SEM tensile deformation at a range of temperatures; RT, 300°C and 500°C. The as-fabricated microstructure consists of prior β grains boundaries and fine basketweave microstructure. Due to LMD fabricated alloys’ anisotropic behavior, the tested samples were collected from the same region and direction. The XRD and SEM results showed that the temperature up to 500°C has no impact on the phase constituent and structure morphology, revealing a stable microstructure. The tensile deformation at room temperature (RT) revealed that slip lines were the primary deformation mechanism generated in the parallel and larger alpha grain. An increase in temperature triggered grain boundary sliding during deformation, which enhanced the elongation in the material at the cost of tensile strength reduction. The grain boundary resistance to dislocation slip was affected by the sliding phenomenon due to increased temperature. Overall, the material showed stable tensile characteristics related linearly to the temperature variation.

1. Introduction

Ti-6Al-2Zr-1Mo-1V (TA15) a kind of near-α titanium alloy commonly used in aerospace large-scale complex geometrical components such as modern jet engine front fans, nacelle center beam frame, bulkheads, and compressor blades due to distinctive characteristics of low density, good heat durability, high corrosion resistance, and excellent room and moderate temperature mechanical properties [1-3].

Due to some metallurgical flaws, the traditional machining fabrication faces challenges of low thermal conductivity of titanium alloys, high cost of the final component and excessive material waste [4, 5]. In contrast, additive manufacturing (AM) has gained much attention, particularly for structural parts [6]. Laser melting deposition (LMD) is a kind of AM techniques, working on the principle of layer-by-layer metal deposition by laser heat source operated from a digital software [7, 8]. The technique has the prestigious merits of producing near-net-shape in short fabrication time, full dense and low production cost, high buy-to-fly ratio, and flexible design and geometry of the components [9, 10].

The microstructure and texture of titanium alloys are highly reliant on process parameters and thermo-mechanical-processing, which in turn influence the mechanical behavior of the final component [5, 11]. During laser deposition, the layer-over-layer mechanism and thermal cycles cause a complicated phase transformation behavior [12]. Laser deposited material composed of columnar β-grains growing in deposition direction due to steep thermal gradient towards the substrate [13-15]. Depending on the process parameters and cooling rate, the columnar β-grains transform to lamellar α'-martensitic, fine basketweave α, and widmanstätten microstructure [16, 17].

Due to the critical structural applications, many researchers investigated the LMD process parameters and post-heat-treatments’ impact on the microstructure and mechanical properties of TA15
alloy. Li and Wang [18] investigated the influence of aging temperature and holding time on the microstructure of LMD-TA15 alloy. Li et al. [9] studied the thermal-expansion during α + β zone annealing of the LMD TA15. Xie et al.[19] processed the annealing temperature to transform the as-deposited β-transformed typical lamellar microstructure to special bimodal structure in the LMD-TA15 alloy, enhancing both strength and elongation of the material. Chen et al. [20] investigated the effect of morphologies and textures of α-phase obtained by various annealing temperatures on the high strain deformation of LMD TA15. However, much attention has been paid to laser deposited TA15 alloy microstructure and mechanical properties. In most of these studies, the effect of microstructure evolution on mechanical properties is studied at RT and based on the post-fracture analysis.

In order to get a deep knowledge of LMD TA15 alloy, the current work is focused on the sensitivity of microstructure and mechanical properties of TA15 to the temperature variation. The tensile testing of as-fabricated samples was carried out at RT, 300°C and 500°C. The entire testing has been carried out by in-situ observation during the tensile deformation in SEM. The fractured surface of the deformed samples is also discussed in the current work.

2. Material and method

The bulk material of the LMD TA15 alloy shown in Fig. 1a was prepared based on a coaxial power supply system, where 10 KW IPJ laser power was used as a heat source. TA15 powder (size ≈ 75-250 μm) was deposited on the as-cast same alloy substrate. The process parameters set after build and inspection method are: Laser power 2200-2600W, powder supply rate 0.8-1 kg/h, laser scan spacing 2-2 mm, layer thickness 0.7-0.9 mm, and scanning speed 1250 mm/min.

The tensile samples were extracted from the X-direction (Fig. 1b) of the bulk sample by using a wire-cutting electric discharge machine. The size and dimension of the tensile sample is given in Fig. 1c. The sample is designed with a notch part in the middle section to confine and observe the tensile testing’s deformation activities. To analyze the surface structure, the samples were ground and polished with SiC abrasive papers and diamond paste, respectively. The samples were finalized with an electropolishing technique using a solution of (HClO4:CH3COOH=5 ml: 95 ml). To analyze the surface microstructure and defects (pores/lack of fusion), the samples were etched by the Kroll’ reagent of HNO3: 4 ml, HF: 2 ml, H2O: 94ml. XRD technique was used to identify the constituent phase. The in-situ tensile deformation was carried out by the tensile stage fitted in SEM S8000 chamber shown in Fig. 1d. The tensile stage operates on the principle of bi-axial tension along the principle axis carried out by the left and right clamps of the multi-gear drive set-up shown in Fig. 1e. The set-up consists of a heating system touching the sample’s lower surface to provide a high-temperature condition. A K-type thermocouple was used to test the temperature of the testing sample. The testing temperature was gained step-wise and held for ≥ 30 minutes to ensure the region's temperature uniformity. The specimen was stretched with a tensile speed of 1 μm/s. The SEM images were captured during the relaxation of the tensile loading.
Fig. 1. (a) Bulk sample of LMD TA15, (b, c) Tensile sample dimension and size (d) View of the tensile stage inside SEM vacuum chamber, (e) Schematic of the multi-gear tensile stage with heating system.

3. Results and discussion

3.1 XRD phase determination
Fig. 2 shows the XRD pattern of the tested samples. TA15 is a high Al equivalent titanium alloy containing a small amount of β phase at room temperature. The amount of retained β phase identified by XRD pattern was 10.5, 7, and 8.1% in the samples tested at RT, 300°C and 500°C, respectively. The small difference in the detected amount of β phase in all these three samples could be due to the samples’ positioning. The beta phase peaks were identified at 2Theta of 46.2º, 67.4º, and 85.7º, having hkl index of (110)β, (200)β, and (211)β respectively. The difference in the intensity of peaks shown in the dashed line (I, II) is attributed to the grain orientation in the samples [21].

Fig. 2. XRD pattern of as-deposited LDMD AM TA15. Area detector was used.

3.2 Structure morphology
Fig. 3 shows the structural morphology of as-fabricated LMD TA15 alloy examined by Optical (OM) and Secondary electron microscope (SEM). The columnar β grains are seen clearly in the OM image growing in the deposition direction, Fig. 3a. The columnar β grains grow in the direction of <100>β during the solidification process [22, 23]. The growth rate of the columnar β grain is highly influenced
by the temperature and cooling rate during solidification. During the layer by layer deposition in AM, the fast cooling rate ($10^5 \degree C/s$) enables the epitaxial growth over the previously deposited layer [24]. The columnar beta grains extend over several deposited layers in the building direction due to thermal gradient as the heat sinks downward to substrate by conduction process [25]. The high magnification SEM micrographs of the sample surface are shown in Figs. 3 (b, c). The transformation of $\beta$ phase (bcc) into $\alpha$-phase (hcp) occurs under rapid cooling following the Burger relation (BOR) i.e. $(110)_\beta \parallel (0001)_\alpha$ and $<111>_\beta \parallel <1120>_\alpha$ [26]. As shown in Fig. 3 (b, c), a continuous GB-$\alpha$ phase precipitates at the prior beta grain boundary, which further grows and forms $\alpha$-colonies in the grain interior. The transformation process is carried out in four steps $\beta \rightarrow$ GB-$\alpha$→colony $\alpha$, from beta to alpha phase [27]. The LMD comprise a high cooling rate, and the rapid cooling rate provides a high driving force for the nucleation of intergranular $\alpha$-laths [28]. These newly growing $\alpha$-laths stops further growth of prior $\alpha$-colonies, consequently a fine basketweave structure form in the grain interior. The basketweave structure comprise of weak texture with various lengths of $\alpha$-laths shown in IPF Fig. 3(d).

Fig. 3. (a) OM image showing columnar grains growing in building direction (b) SEM microscopic view of the prior $\beta$ GB (c) the magnified image showing $\alpha$-colony growth along GB-$\alpha$, (d) IPF showing the intergranular fine basketweave $\alpha$-laths with various lengths

3.3 Impact of temperature on tensile strength

Fig. 4 shows the force-displacement curve obtained during in-situ tensile testing. The tensile curve (I, II, III) is used to compare tensile behavior at RT, 300$^\circ$C and 500$^\circ$C, respectively. The comparison shows a noticeable difference in the tensile strength and elongation before fracture at different temperatures. Curve-I RT showed higher tensile strength while low elongation before fracture as compared to 500$^\circ$C (Curve-III). The variation in the strength and elongation before fracture failure shows the mechanical properties sensitivity to the deformation temperature. The drop in tensile strength with increasing temperature was also identified in SLM fabricated TA15 [29]. The ultimate strength decreased while the elongation increased when the deformation temperature increased from RT to 300$^\circ$C and 500$^\circ$C. The interruption in the curves is due to the tensile load releasing during the in-situ observation at the image acquisition stages. The load was resumed from the same points where it was paused.
Fig. 4. Stress-displacement curve from the in-situ tensile testing with pauses shown by letters where the images were taken.

3.4 Influence of temperature on deformation behavior

Fig. 5 elucidate the sample deformation behavior during the tensile test at various temperatures i.e. RT, 300°C and 500°C. Figs. 6 (a, e, g) are the samples’ surface area before tensile deformation at the aforementioned temperatures. To analyze the surface evolution during deformation under various testing temperatures, three stages: sample surface before tensile, close to the fracture, and fracture are chosen for the macroscopic analysis. Fig. 5b shows the plastic deformation (F=915 N, d= 687 µm) in the sample at RT. The surface shows slight necking close to fractured failure point corresponding to the point (c) Fig. 4. A shear deformation was observed in the central notch section of the sample near the prior β grain boundary labeled in Fig. 5b. The deformed surface has no visible major crack at this stage. On further tensile load, the sample fractured at a displacement of 717 µm shown in Fig. 5c. The breaking path shows a high brittle behavior. The microstructure in the vicinity of fracture failure point has no major deformation showing a least elongation during the tensile process. Fig. 5f shows the sample’s deformation behavior at a temperature of 300°C (F=803 N, d= 802 µm). The tensile deformation stage corresponds to point (r) Fig. 5. The sample showed comparatively greater necking behavior than the sample at RT (Fig. 6b). Microcracks were appeared in the sample surface along the prior β GB. By increasing tension to 841 µm, the fracture failure occurred shown in Fig. 6 (g). The fracture path followed the maximum shear stress direction of 45° to the tensile direction. As compared to the sample fractured at RT, the fracture path partially followed the direction of prior β GB at a deformation temperature of 300°C. Fig. 6h shows the plastic deformation (applied force: 580N, displacement: 844 µm) in the sample at a deformation temperature of 500°C. The sample showed larger necking and uniform deformation as compared to the samples tested at RT and 300°C. The strain localization in the sample is along the prior β GB as shown in fig. 5h. A larger crack was observed initiated at an angle of 45° to the tensile direction. The crack tip followed the strain localization and loose microstructure of the sample, shown in Fig. 5h. At high-temperature conditions, the sample showed enhanced ductile characteristics in the deformation zone. Further tension in the sample to 861µm caused fracture failure shown in Fig. 6i. The fracture’s central part is nearly vertical, while the upper and lower ends showed a shear fracture at the angle of 45° to tensile direction describing the cup and cone characteristics. The greater necking characteristic and linear fracture path in the sample tested at 500°C showed a noticeable increase in the ductility at high temperature.
Fig. 5. (a) Before tensile at RT (b) close to fracture, RT (c) fracture path, RT (d) Before tensile 300°C (e) close to fracture 300°C (f) fracture path 300°C (g) Before tensile 500°C (h) close to fracture 500°C (i) fracture 500°C.

Fig. 6 shows the deformation mechanism in the samples tested at RT (a-c), 300°C (d-f) and 500°C (g-i). The temperature variance showed an evident impact on the microstructure and deformation behavior. Fig. 6a shows slip lines appeared along the parallel and larger α-laths, which is the primary plastic deformation mechanism in titanium alloy at room temperature [30]. The appeared slip lines are oriented at an angle of 45º to the tensile direction as the largest force in tensile load. The slip lines are attributed to the dislocation slips in the crystal by an external force. During deformation, the localization of strain occurs in the larger α-laths. The slip line generated in the same orientation combined and formed a larger slips band, shown in Fig. 6b. The slip line grows longer with the increase in external force until resisted by misoriented grains, Fig. 6b. As shown in the EBSD IPF map, the intergranular basketweave region is composed of randomly oriented α-laths. The weak texture of α-laths opposed the growth of slip lines, resulting in low plastic elongation and high tensile strength, as shown in curve (I) Fig. 5. Fig. 6c shows the brittle characteristic evident from the irregular fracture path. The remnant of slips lines shows the cracks and fracture generated sites. The tiniest microstructural damage close to the fracture failure verifies the least elongation. Fig. 6d shows the sample surface tensiled at 300°C. Slip lines were appeared along the prior β GB at a deformation stage (tensile load 770N, displacement 475µm). The GB sliding was observed prompted by the closely oriented slip lines with the GB direction [31]. Due to the thin layer of retained-β-phase, the GB accommodates the incompatible plastic deformation between the neighboring grains instead of cracking [32]. On further tension, the propagation of slip lines and the GB sliding resulted in voids and microcracks shown in Fig. 6e. Void formation during tensile deformation is a characteristic of ductile nature [32]. The enlargement of microcracks due to further tension caused the fracture failure where the remnant of prior β GB can be seen in Fig. 6f. As compared to RT deformation Fig. 6b, the prior β GB showed a critical role in the sample's deformation at 300°C. The microstructure in the vicinity of the fracture point has clear evidence of the slips growth. The uniformity in the fracture surface and distortion in microstructure shows the increase in elongation with increasing deformation temperature. Figs. 6g shows the sample surface undergoing tensile deformation at 500°C.
Initial slip lines were observed when the sample reached a displacement of 560µm. The slip lines were appeared in parallel α-laths along prior β GB and larger α grain in the grain interior shown in Fig. 6g. GB sliding is observed due to low angle incident slip line shown in Fig. 6h. The basketweave region opposes the slip line growth, whereas the slip line comparatively grows easier along the parallel α-laths along prior β GB [28]. The closely oriented slip bands and the higher intensity stirred the grain boundary sliding phenomenon in the sample during deformation shown in Fig. 6h. The GB sliding occurred relatively at low stress at high temperature. The shear crack shown in Fig. 5h, propagated along the deformed GB region, caused fracture failure. The fracture surface shown in Fig. 6i, demonstrates the fracture path along parallel α-laths combined with shear fracture in the maximum shear direction. The slip lines growth along parallel laths and grain boundary sliding showed greater ductility in the sample tested at 500°C.

Fig. 6. Tensile deformation observation at micro scale: (a) initial slip lines appearance in larger α-grains at RT (b) the fusion of slip line at RT point 'c' Fig. 4 (c) slip line remnants along the fracture path (RT) (d) initial slip line generation along GB: 300°C (e) void formation by fusion of slip lines: 300°C (f) fracture path showing breaking at GB (g) initial slip lines appearance along parallel α laths at 500°C (h) GB sliding occurring by incident slip lines (i) the fracture path along GB showing the traces of parallel α laths.

3.5. Fractography comparison
Figure 7 shows the fracture morphology representation of samples subjected to tensile deformation at RT, 300°C and 500°C. The fractured surface at RT mainly consists of river-like cleavage patterns and
stair-cut facets, as shown in Fig. 7a. The surface mainly consists of flat regions where no deep dimples are observed, showing the brittle nature at said temperature. As the deformation temperature was increased to 300°C, the cleavage and stair-cut phenomenon reduced in the fractured surface and equiaxed deep dimples appeared. As shown in Fig. 7b, the fracture surface exhibits fiber characteristics, describing high plastic behavior than sample tensile at RT. According to Li et al. [33], the homogeneous micro-scale deformation characteristics and the ability to withstand a higher deformation determine high plasticity. The fiber region homogenized and the dimples became deeper when the sample was tensiled at 500°C, as shown in Fig. 7c. The depth enhancement of the dimples reveals high necking and better plastic behavior [34]. As compared to the tensiled sample at RT and 300°C, the edges of the sample are perceptibly inclined to the center fiber area of the fracture surface, 7c. The cross-section area reduction of the samples shown in fractured surfaces agreeing with the necking in notch surfaces of the samples shown in Figs. 5 (b,f,i). The characteristics described by the fractured surfaces show a larger impact of temperature increase on the elongation of the samples. This phenomenon is well agreed with the force-displacement curve given in Fig. 5.

Fig. 7. Fracture surface: (a) RT, (b) 300°C, (c) 300°C, along with the magnified images mention by rectangular shape

4. Conclusions
In-situ instigation of LMD TA15 alloy at temperature ranges RT, 300 and 500 during tensile deformation was studied in this study. The main conclusions are drawn as below:
1. LMD-TA15 alloy consists of columnar β grains elongated in the deposition direction that transformed to fine basketweave structure under high cooling rate. In addition, the deformation temperature in the range 500°C has no dynamic impact on phase constituents.
2. Increasing the deformation temperature linearly decreases the tensile strength and increase the ductile characteristics of the material.
3. Prior β grain boundary has a vital role in the deformation mechanism that resists the slips lines to pass and propagate, which in turn causes the strain localization in the region.
4. The temperature increase improves the sample ductility by inducing the grain boundary sliding phenomenon during tensile deformation.

Acknowledgment
The authors wish to thank Basic Science Center Program for Multiphase Evolution in Hypergravity of the National Natural Science Foundation of China (No. 51988101).
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