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Research Progress on Laser Additive Manufacturing and Heat Treatment Processes of Metastable βTitanium Alloys

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Abstract
Metastable βtitanium alloys have become a research focus in aerospace and biomedical fields owing to their high specific strength, excellent corrosion resistance and remarkable microstructural tunability. Traditional processes for fabricating such alloys exhibit numerous limitations, and the synergetic system of laser additive manufacturing and heat treatment has emerged as the core approach for their advanced preparation. This paper elaborates on the characteristics and application scenarios of two mainstream additive processes, and summarizes multi-dimensional control strategies for solidification defects including columnar grains and β flecks. It also analyzes the regulation rules of heat treatment on β phase stability, as well as its regulatory mechanisms and strengthening effects on the precipitation behavior of α and ω phases. The core laws of process regulation summarized herein provide important theoretical reference and technical guidance for the optimization of preparation processes and engineering application of metastable βtitanium alloys.

Keywords:*Metastable β titanium alloys, Laser Additive Manufacturing (LAM), Heat treatment.*

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I. Introduction

Titanium alloys serve as key structural materials for high-end equipment manufacturing and biomedical applications. Featuring high specific strength, excellent corrosion resistance and biocompatibility, they act as the core support for achieving lightweight design and high-performance upgrading in aerospace, medical device, marine engineering and other fields [1, 2]. Among titanium alloy systems, metastable βtitanium alloys possess an extremely high degree of microstructural tunability due to the metastable structure of their β phase, and thus combine the comprehensive advantages of high strength, high toughness and low elastic modulus. In comparison with traditional α+β titanium alloys, metastable βtitanium alloys can meet the stringent industrial requirements for the synergy of multiple material properties through precise phase transformation regulation, making them a research focus in the materials field at home and abroad [3-5]. With the development of high-end equipment toward complexity, customization and high performance, higher demands have been placed on the forming precision, microstructural uniformity and performance matching of metastable βtitanium alloy components. The innovation of preparation processes and the breakthrough in performance regulation technologies have therefore become the core keys to promoting the engineering application of such alloys.

Traditional preparation processes exhibit numerous limitations in the production and application of metastable βtitanium alloys. Restricted by their forming principles and thermomechanical processing characteristics, these processes not only make it difficult to realize the integrated near-net shaping of complex special-shaped components, but also easily cause problems such as compositional inhomogeneity and insufficient precision in microstructural regulation of the alloys, which fail to give full play to the performance potential of metastable βtitanium alloys [6]. Meanwhile, the multi-process processing mode of traditional technologies is associated with disadvantages like low material utilization and long production cycles, which necessitates the development of advanced manufacturing technologies for a breakthrough. As a core technology in the advanced manufacturing of metallic materials, laser additive manufacturing has broken the preparation boundaries of traditional processes by virtue of its unique advantages including rapid solidification, in-situ alloying and complex structure forming, thus providing a novel approach for the high-performance and customized preparation of metastable βtitanium alloys [7].

However, the non-equilibrium thermal cycle characteristic of laser additive manufacturing inevitably leads to challenges in regulating the microstructure and mechanical properties of the prepared metastable βtitanium alloys, which has become a major factor restricting the engineering application of this technology. As a classic method for material performance regulation, heat treatment enables the optimization of alloy microstructure and the improvement of mechanical properties through precise control of phase transformation processes, and it serves as a key supporting technology to solve the performance problems of metastable βtitanium alloys fabricated by laser additive manufacturing[8-10]. Laser additive manufacturing overcomes the limitation of compositional uniformity in traditional casting processes by virtue of its rapid solidification and complex structure forming capabilities, while heat treatment achieves the synergistic optimization of high strength and ductility of alloys through accurate regulation of phase transformation and microstructure. Together, they constitute an advanced preparation technology system for metastable βtitanium alloys. This paper presents a systematic review of the laser additive manufacturing and heat treatment processes of metastable βtitanium alloys, summarizes the research status and technological progress in this field, and concludes the core laws of process regulation, aiming to provide theoretical and technical references for the optimization and upgrading of preparation processes and the engineering application of metastable βtitanium alloys.

II. Laser Additive Manufacturing Process

With the extensive application of metal additive manufacturing in the field of α/β titanium alloys, the additive manufacturing of metastable βtitanium alloys has attracted widespread attention and undergone numerous exploratory studies [11, 12]. Its core advantages lie in the rapid solidification effect, flexibility of in-situ alloy design and capability of complex structure forming. At the same time, it is still imperative to address the key problems of solidification defects and microstructural anisotropy.

2.1Laser Additive Manufacturing Process

Laser additive manufacturing (LAM) has been an effective technology for fabricating titanium alloy components since the early 21st century. It occupies an important position in the advanced manufacturing of titanium alloys due to its advantages such as near-net shaping of complex geometric structures, high production efficiency and costeffectiveness. Over the past decades, research has mostly focused on the LAM technology of the α+β Ti-6Al-4V alloy [13], while relevant studies on metastable βtitanium alloys were relatively limited previously due to the scarcity of suitable powders. In recent years, the discovery and research of metastable βtitanium alloys such as Ti-7Mo-3Nb-3Cr-3Al and Ti-5Al-5V-5Mo-3Cr [14, 15] have made their LAM research a growing hotspot. These alloys not only have higher tensile strength than Ti-6Al-4V, but also combine economic efficiency with excellent strength and toughness. The main LAM technologies for metastable βtitanium alloys include Laser Powder Bed Fusion (LPBF) and Laser Directed Energy Deposition (LDED). LPBF realizes component forming by selectively melting powder layers, while LDED constructs deposition layers through coaxial powder feeding. The two processes show significant differences in laser power, molten pool diameter and scanning speed, resulting in drastically different cooling rates: the typical cooling rate of LPBF reaches 10^5~10^6 K/s, whereas that of LDED is only 10^2-10^4 K/s [16]. This difference in cooling rate directly leads to the divergence in microstructure and mechanical properties of the fabricated alloys. For example, the TA15 alloy prepared by LPBF has a finer microstructure and higher strength than the alloy of the same composition fabricated by LDED[17].

In terms of process characteristics and application scenarios, LPBF can effectively inhibit the decomposition of β phase and retain the metastable structure by virtue of its ultra-high cooling rate, thus demonstrating prominent advantages in the preparation of high-precision and complex metastable βtitanium alloy components. For instance, H. Schaal et al. [18]successfully prepared the metastable βtitanium alloy Ti-22Zr-9Nb-2Sn (atomic percent) by optimizing the parameter matching of laser power and scanning speed in the LPBF process. Relying on the synergistic effect of the fine-grained β matrix and dispersed precipitates, this alloy achieved a strain hardening rate as high as 15 GPa, which is significantly superior to that of the alloy of the same composition prepared by the traditional forging process. Recent studies have further expanded the application scenarios of LPBF: the LPBF process with an infrared top-hat laser configuration enables the precise regulation of the texture of the metastable βtitanium alloy Ti-42Nb. By constructing a strong <001> texture along the building direction, the elastic modulus of the alloy is reduced to 44 GPa while a yield strength of 674 MPa is maintained, opening up a new path for the preparation of low-modulus metastable βtitanium alloys for biomedical applications [19].

In contrast, although LDED has a lower cooling rate than LPBF, it is capable of manufacturing large-scale components and designing composition gradients, and can realize in-situ alloying through coaxial feeding of multiple powders, thus playing an irreplaceable role in the low-cost and large-scale preparation of metastable βtitanium alloys. For example, Vrancken et al. [20] adopted the directed energy deposition (DED) technology to in-situ mix and melt 10 weight percent Mo powder with Ti6Al4V-ELI powder, and inhibited the microsegregation of Mo element by utilizing the rapid solidification effect of LAM, finally forming a composite structure consisting of a βtitanium matrix and randomly dispersed pure Mo particles. In comparison with the full α′ martensitic structure of Ti6Al4V formed by LPBF, this new metastable βtitanium alloy achieved an increase in tensile strength by 1100 MPa while maintaining good plasticity. In addition, the layer-by-layer remelting characteristic of LDED can introduce a high density of dislocations inside grains, producing a strengthening effect equivalent to that of cold mechanical processing. For example, the Beta-C metastable βtitanium alloy prepared by LDED by Zhang et al. [12] had a dislocation density of 1×10^{15} m^{-2}, and the nucleation rate of α phase after aging treatment was 40% higher than that of the as-cast alloy, which significantly accelerated the aging response rate of the alloy and provided technical support for the rapid strengthening of metastable βtitanium alloys.

2.1Solidification Defects in Laser Additive Manufacturing

Nevertheless, additive manufacturing processes are accompanied by certain process-induced problems. For example, the formation of columnar grains during printing leads to solidification defects and anisotropy of mechanical properties, and the microsegregation of eutectoid elements is prone to form "β fleck" defects. Both of these defects restrict the performance stability of metastable βtitanium alloys. The thermal conditions during the additive manufacturing process make this phenomenon inevitable, so fabricating a uniform microstructure with fine equiaxed grains has become the ideal goal of researchers.

Aiming at the problem of columnar grains, Bermingham et al. [21] revealed their growth characteristics at different stages through a theoretical model. During solidification, grains undergo a multi-stage evolution process, including epitaxial growth without constitutional supercooling, continuous growth of columnar grains due to insufficient nucleation particles, and the initiation of columnar to equiaxed transition (CET). Based on this, researchers have proposed a multi-dimensional solution involving nucleant regulation, parameter optimization and post-treatment optimization, which enables the fabrication of a uniform microstructure with fine equiaxed grains by adding an appropriate amount of nucleants. When nucleants such as La_2O_3 are present in the molten pool, the grain size can be reduced by lowering the nucleation undercooling, thereby promoting the CET. Similarly, Bermingham et al. [21] added 0.5 weight percent La_2O_3 during the selective laser melting (SLM) of the Beta-C alloy, which increased the volume fraction of equiaxed grains from 15% to 80% and simultaneously inhibited the segregation of Cr element at grain boundaries. Some metastable βtitanium alloys containing eutectoid elements such as Fe are prone to the segregation problem of "β fleck" during additive manufacturing. The only method to eliminate segregation in metastable βtitanium alloys via traditional manufacturing routes is to promote atomic diffusion through long-term thermal homogenization [22]. The multiple thermal cycle characteristic of laser additive manufacturing provides a possibility for the low-cost regulation of this defect. Ng et al. [2] found that during the LAM process, thermal cycles reheat the previously deposited layers, thereby promoting the diffusion and redistribution of solute elements without exerting a significant impact on the uniformity of aging precipitation, which avoids the grain coarsening caused by traditional homogenization treatment.

In terms of process parameter optimization, grain refinement can be achieved by regulating the laser scanning strategy and energy input, which affects the temperature gradient (G), growth rate (R) of the solid-liquid (S/L) interface and cooling rate of the molten pool. For example, the adoption of the island scanning mode reduced the temperature gradient of the Ti-15Mo alloy from 500 K/mm to 200 K/mm and increased the growth rate, which satisfied the critical conditions for CET and significantly raised the proportion of equiaxed grains[23]. In addition, post-treatment technologies such as hot isostatic pressing and ultrasonic vibration assistance provide effective approaches for grain refinement of formed components. Hot isostatic pressing refines the average grain size of columnar grains through dislocation climb and recrystallization under high temperature and pressure, which significantly reduces the yield strength anisotropy index[24]. Molten pool ultrasonic vibration technology promotes dendrite fragmentation and heterogeneous nucleation, as shown in Figure 1. Thus transforming the alloy into fine equiaxed grains completely, and the yield strength and tensile strength of the treated alloy are significantly improved compared with those of traditional SLM samples [25].

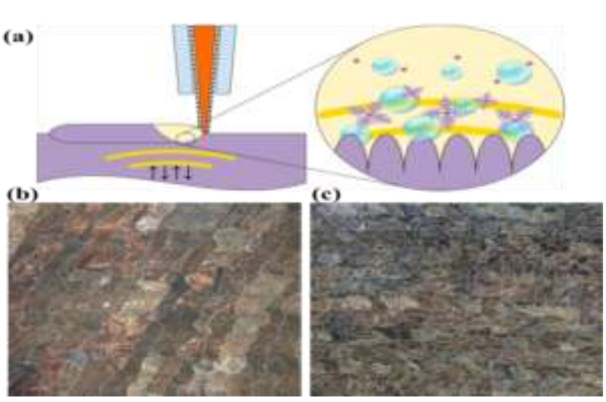

Figure.1 High-strength ultrasonic grain refinement of titanium alloy produced by LAM (a) Schematic illustration of the ultrasonic printing process (b) Columnar grains in the original printed sample (c) Ultrasonically refined grains[25].

III. Research On Heat Treatment Behavior Of Metastable Btitanium Alloys

Heat treatment is a core means for the phase structure regulation and mechanical property optimization of metastable βtitanium alloys. Through the design of various heat treatment regimes, the decomposition path of β phase can be precisely controlled, thereby obtaining multi-phase microstructures with α phase and ω phase as the main strengthening phases to meet the requirements of different service scenarios for strength, ductility and fatigue properties. The key lies in balancing the stability of β phase and the strengthening effect of precipitates, and solving the problem that a single microstructure is difficult to balance both high strength and high ductility.

3.1 Regulation of β Phase Stability by Heat Treatment

The complex thermal cycle process of laser additive manufacturing can induce the formation of unique microstructures such as subgrain boundaries and dislocations in metastable βtitanium alloys, as shown in Figure 2.[26]. The synergistic effect of these microstructures and subsequent heat treatment enables the precise regulation of the strength and ductility of the alloys. Multiple thermal cycles generate and move dislocations, which form dislocation walls and further promote the formation of subgrain boundaries in grains; these subgrain boundaries restrict the movement of dislocations, thus improving the strength of the alloy. For metastable βtitanium alloys with sufficient β phase stability, the heat treatment process is initiated with solution treatment above the β transus temperature. Solution treatment can generate and retain a large amount of metastable β phase in titanium alloys, and subsequent aging treatment decomposes these metastable β phases into fine α phases to realize effective strengthening of the alloy[27]. However, during solution treatment, martensitic transformation usually occurs due to the differences in β phase stability of the alloys, forming a small volume fraction of α″ martensite, which exerts an adverse effect on the deformation behavior and mechanical properties of the alloys. Metastable βtitanium alloys are highly sensitive to the solution temperature: the ductility of the alloys decreases with the increase of solution temperature, and an excessively high temperature above the β transus will lead to the coarsening of β grains. In addition, the stability of β phase decreases with the rise of solution temperature. For example, Zhu et al. [28] treated the Ti-4Mo-4Cr-3Al-2Nb-1.2V-1Zr-1Sn (wt.%) alloy at different solution temperatures to obtain different volume fractions of primary α phase, and found that the alloy containing primary α phase had a higher tensile strength than the alloy composed of only β phase with the decrease of solution temperature. Similarly, Li et al. [29]compared the Ti-6Cr-5Mo-4Al (wt.%) alloy subjected to β solution treatment and α/β solution treatment, and found that although the α/β solution-treated sample contained a small amount of primary α phase, which endowed the alloy with higher strength, both alloys exhibited the same level of ductility after solution treatment. Moreover, solution treatment above the β transus may lead to poor work hardening of the alloy, making the yield strength and ultimate tensile strength almost the same.

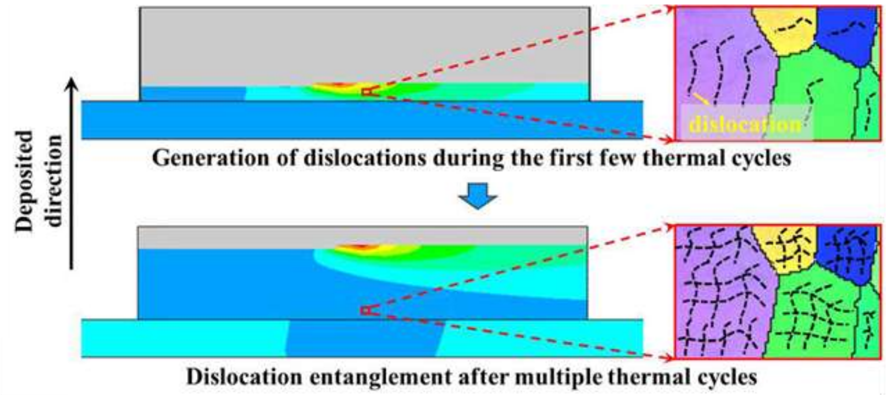

Figure.2 Schematic diagram of subgrain boundary formation during DED deposition[26].

3.2 Effect of Heat Treatment on Precipitates

As the primary strengthening phase of metastable βtitanium alloys, the morphology, size and distribution of α phase are determined by the parameters of heat treatment processes. The customized regulation of the mechanical properties of the alloys can be realized by controlling the precipitation behavior of α phase. The synergistic effect of aging temperature and time dominates the evolution of α phase: low-temperature aging tends to form fine and dispersed spherical α phase, while high-temperature aging promotes the growth of lath-shaped α phase. For example, for the Ti-10V-3Fe-3Al alloy, fine spherical α phase with a volume fraction of 20% can be obtained under low-temperature aging, and the alloy achieves a tensile strength of 1200 MPa and an elongation of 12% at this time; the lath-shaped α phase formed under high-temperature aging increases the tensile strength to 1300 MPa, but the elongation decreases to 8% [30]. Recent studies have shown that the adoption of a step aging process can realize the composite distribution of spherical and lath-shaped α phases, which maintains a tensile strength of 1250 MPa while increasing the elongation to 14%. The underlying mechanism is that the spherical α phase formed in the low-temperature stage can inhibit the excessive growth of lath-shaped α phase in the high-temperature stage and alleviate stress concentration at the phase interface [31]. In addition, the alloy composition exerts a significant influence on the thermal precipitation kinetics of α phase: the presence of αstabilizing elements such as Al and Sn can reduce the nucleation activation energy of α phase and shorten the aging time to accelerate the nucleation of α phase, while βstabilizing elements such as Mo and Nb delay the precipitation process and reduce the growth rate of α phase, which is more conducive to the formation of fine α phase [32]. Alternatively, the rapid heating aging process can realize the synergistic distribution of fine intragranular α phase and discontinuous intergranular α phase in the alloy, avoiding the continuous precipitation of intergranular α phase caused by traditional slow heating and thus improving the fracture toughness of the alloy [33]. Furthermore, the α phase strengthening of as-additive-manufactured metastable β-titanium alloys can be combined with their inherent dislocation structure, and the thermal cycle-assisted aging process can be adopted to significantly enhance the strengthening effect. When Zhang et al. [12] conducted aging treatment on the Beta-C alloy formed by directed energy deposition (DED), the high-density dislocations introduced during the DED process were used as heterogeneous nucleation sites for α phase, which made the nucleation rate of α phase 40% higher than that of the as-cast alloy. After aging treatment, the alloy achieved a tensile strength of 1400 MPa and an elongation of 10%.

In fact, besides α phase, the formation of ω phase in metastable βtitanium alloys is often inevitable during heat treatment, and the response of ω phase to heat treatment is extremely complex. According to the formation mechanism, ω phase can be divided into athermal ω phase (formed by quenching) and isothermal ω phase (formed by aging), which exert significantly different effects on the mechanical properties of alloys. The athermal ω phase is usually formed by diffusionless transformation during water quenching, with a fine and dispersed size. Although it can improve the strength of the alloy, it easily leads to a decrease in plasticity. However, studies have shown that the formation and transformation path of ω phase can be precisely controlled by designing appropriate heat treatment processes: for example, Sun et al. [34] strengthened the Ti-12Mo alloy by two heat treatment regimes including solution treatment and aging treatment. The alloy after solution treatment and quenching was composed of β phase with uniform composition and athermal ω precipitates. Then, short-time low-temperature aging was carried out within the precipitation temperature range of isothermal ω phase to promote the nucleation of isothermal ω phase for precipitation strengthening, without excessive precipitation that would affect the chemical composition of the β matrix. At the same time, the size of ω phase increased by 3 to 5 times, and the elongation of the alloy was significantly improved while the same strength was maintained. The core reason is that the shear modulus of isothermal ω phase (65 GPa) is lower than that of athermal ω phase (75 GPa), which effectively reduces the hindrance to dislocation movement in the matrix. For the above-mentioned micro-mechanism of ω phase transformation, Qian et al. [35] conducted further research on the Ti-12Mo alloy and found that the formation of athermal ω precipitates during water quenching traps Mo atoms in these precipitates, putting the atoms in a metastable "frozen" state; the thermal activation provided by aging within the precipitation temperature range of isothermal ω phase can induce the precipitation of athermal ω precipitates through diffusion transformation, which provides the necessary driving force for the "thawing" of Mo atoms. The depletion of Mo in the precipitates leads to the transformation of ω phase into a chemically stable hexagonal lattice structure and ultimately the growth of isothermal ω grains. This process is characterized by thermally diffusion-controlled kinetics. In addition, the depletion of Mo in the athermal ω phase during aging also significantly increases the shear modulus of the transformed isothermal ω phase, which is the reason for the significant isothermal ω phase strengthening effect observed in βtitanium alloys. This "thawing-diffusion-transformation" process transforms the strengthening effect of ω phase from brittle strengthening in the solution-quenched state to tough strengthening in the aged state.

IV. Conclusion

This review provides a comprehensive synthesis of the research progress regarding the laser additive manufacturing (LAM) and subsequent heat treatment of metastable β titanium alloys. The following core conclusions are drawn:

1.The choice between Laser Powder Bed Fusion (LPBF) and Laser Directed Energy Deposition (LDED) dictates the initial microstructural state due to a vast disparity in cooling rates. LPBF, characterized by cooling rates of 10^5–10^6 K/s, is ideal for suppressing β phase decomposition and producing high-precision components with metastable structures. Conversely, LDED offers cooling rates of 10^2–10^4 K/s and excels in large-scale manufacturing and flexible in-situ alloy design.

2.The inherent challenges of columnar grain growth and β fleck segregation in LAM can be effectively addressed through multi-dimensional control strategies. Promoting the columnar-to-equiaxed transition via nucleant addition, optimized scanning strategies, and high-intensity ultrasonic vibration has proven successful in achieving uniform, fine-grained microstructures.

3.Heat treatment serves as the definitive tool for balancing strength and ductility in these alloys. By leveraging the unique dislocation densities and subgrain boundaries generated during the LAM thermal cycles, post-processing can precisely regulate the morphology and distribution of α and ω phases. Specifically, strategies such as step-aging or the "thawing-diffusion-transformation" of the ω phase allow for a departure from traditional brittle strengthening toward a high-performance, tough-strengthened state.

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