In-situ observation of grain growth and phase transformation in weld zone of Ti-6Al-4V titanium alloy by laser welding with filler wire

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Abstract
Through in situ observation at high temperature confocal laser scanning microscope, the microstructures and phase transformation of Ti-6Al-4V titanium alloy welded joint by laser welding with filler wire during heating, heat preservation and cooling process in simulating welding thermal cycle were studied. When the weld microstructure was heated to 380 °C, obvious grain growth was observed. After heating to 1 050 °C, weld microstructures were mostly composed of coarse β-columnar grains. During the heat preservation process of the weld metal, it could be found that the β phase boundary moved slowly. In the cooling process of weld microstructures, with the increase of the cooling rate, the transition temperature for new phase precipitation decreased gradually, and that for ending the precipitation increased gradually. The microstructures after cooling to room temperature at a rate of 0.5 °C/s and 5 °C s⁻¹ were composed of α phases. At 20 °C s⁻¹, acicular α' martensite appeared in the microstructures. At 80 °C s⁻¹, precipitated phases were all composed of acicular α' martensite. The Ti-6Al-4V titanium alloy weld microstructure by laser welding with filler wire is completely hexagonal close packed (HCP) crystal structure. The hardness of the weld microstructures was improved with the cooling rate getting faster.

1. Introduction
Titanium alloys have been widely used in naval equipment and shipbuilding industries due to their low density, high specific strength, excellent corrosion resistance, good toughness, and better fatigue resistance [1, 2]. The behavior of Ti alloys (Ti-6Al-4V and beta titanium alloys) at high-pressures and high-temperatures is receiving a great amount of attention [3, 4]. Ti-6Al-4V titanium alloy is the most representative one. It contains about 6 wt.% α-stable element Al and about 4 wt.% β-stable element V. It is a typical α-β two-phase titanium alloy. The pressure hulls of China’s deep-sea manned submersibles, such as Jiaolong, Fendouzhe, are made of titanium alloys [5]. Welding has become a necessary method for processing thick titanium alloy plates due to the difficulty in its manufacturing and forming. Compared with traditional welding technologies, the ultra-narrow gap laser welding with filler wire has features such as low energy input, fast cooling speed during thick plate welding, narrow heat affected zone, high welding efficiency, small residual stress and deformation of welded joint, etc., so it has been gradually applied for thick titanium alloy plates [6].

The ultra-narrow gap laser welding with filler wire of thick titanium alloy plate is the accumulation of multi-layer filler metal in a single pass. During the process of multi-layer welding, repeated thermal cycles might inevitably lead to the extreme complexity of weld microstructures [7]. The properties of titanium alloy welded joints mainly depend on the composition, morphology, distribution of phases, and dislocation, texture, and other microstructure characteristics. The properties of titanium alloy welded joints with different microstructures vary greatly [8]. Under high energy density laser beam welding, the weld pool temperature of titanium alloy is higher, which prolongs the cooling time and increases the growth time of high temperature β phases.
phase grains. The cooling rate after welding will cause the differences of precipitated phase in volume fraction, grain size, micro morphology and phase composition, and then directly affect the mechanical properties of the welded joint\[9\]. Therefore, it is of great significance to understand the influence of cooling rate on the microstructures and mechanical properties of $\alpha + \beta$ titanium alloy.

Researchers at home and abroad applied high temperature confocal laser scanning microscope to simulate different thermal cycles and observe the grain growth and microstructure transformation of metal materials under each condition. Fu Kuijun et al\[10\] probed the grain growth and phase transformation in the welded joint heat affected zone (HAZ) of TiNbV micro alloyed steel for large heat input application and found that selecting appropriate duration of high temperature in the welding process can improve the IAF content in the microstructures, thus improving the mechanical properties of the welded joint. Li Yaqiang et al\[11\] carried out in situ dynamic observation on the peritectic solidification characteristics and mechanism of 15CrMoG steel. It was concluded that the $\delta$-phase precipitated in a cellular manner with the cooling rates getting lower, whereas it was in a dendrite manner when the cooling rate were higher. In addition, as the increase of the cooling rate, the peritectic reaction temperature decreased while the reaction rate raised. Guo Junli et al\[12\], based on specimen surface roughness variations, conducted research on the contraction of peritectic transformation of Fe-Si-Mn peritectic steel at different cooling rates. They uncovered that with the increase of cooling rate, the peritectic transformation contraction of peritectic steel first increased and then decreased. At cooling rate of $20 \degree C \cdot s^{-1}$, the surface roughness reached the maximum, which was 2.8 times of that at $2.5 \degree C \cdot s^{-1}$. Ma Yiming et al\[13\] conducted high temperature metallographic in situ observation on ER410NiMo deposited metal obtained by hybrid laser arc welding, and found that it started melting at $1 483.3 \degree C$, and the metal surface was completely melted at $1493.0 \degree C$; crystallization began to occur when it was slowly cooled to $1 490.7 \degree C$, and ended at $1 493.8 \degree C$; the $\delta$-solid solution $\rightarrow \gamma$-solid solution phase transformation took place when the temperature dropped to $1 372.1 \degree C$, and was over at $1 180.1 \degree C$; phase transformation of $\gamma$-solid solution $\rightarrow M$ phase occurred as the metal was rapidly cooled to $325.9 \degree C$, and the transformation terminated at $240.7 \degree C$.

Most of the studies mentioned above focus on the phase transformation of metallic materials with equiaxed crystal structure such as alloy steel and stainless steel, by using high temperature confocal laser scanning microscope. While few on the columnar crystal structure of titanium alloy weld metal, especially on the composition and morphology evolution process of the new phase obtained from high temperature $\beta$ phase zone at different cooling rates. This paper mainly focuses the phase composition and evolution of $\beta$ phase zone in titanium alloy weld microstructure under different cooling rates, and analyzes the room temperature microstructures finally obtained, to provide technical support for subsequent heat treatment process design, and microstructure and properties control.

Table 1. Chemical compositions of base metal and filler metal (mass fraction, %).

| Plate number | Al       | V       | Fe      | C       | N       | H       | O       | Ti     |
|--------------|----------|---------|---------|---------|---------|---------|---------|--------|
| Ti-6Al-4V base metal | 6.30     | 4.11    | 0.018   | 0.024   | 0.007   | 0.001   | 0.14    | Allowance |
| TC3 welding wire     | 4.75     | 3.82    | 0.044   | 0.012   | 0.006   | 0.001   | 0.081   |         |

Figure 1. Sampling location.
2. Experiment

The size of Ti-6Al-4V titanium alloy used in the experiment is 400 mm × 200 mm × 40 mm. The filler metal adopted is TC3 solid wire with a diameter of 1.2 mm. The chemical composition is shown in table 1. The welding test plate was processed into Y-shape groove, with 4 mm root face. The groove gap was set as 3.2 mm, and the single groove angle was 1.5°. Before welding, the test plate was polished and pickled. The pickling solution was 5%HF + 30%HNO₃ + H₂O. First removed surface oil stain and oxide film, and then rinsed the plate with clean water and blow dry. The weld metal was filled by laser welding with filler wire, and the test plate was not preheated before welding. YLS-6000 fiber laser by IPG was adopted. Welding parameters in table 2 were used for one pass of back welding and sixteen passes of filling welding. The temperature difference for each layer was controlled within 100 °C. Ar was used as the shielding gas in the welding process.

4 discs of Φ6 × 3 mm were machined in the sampling position shown in figure 1 after completing laser welding with filler wire. Then they are ground, polished, rinsed with alcohol, and blew dry for later use. In-situ observation of grain growth and phase transformation of Ti-6Al-4V titanium alloy weld was carried out by using ultra high temperature confocal laser scanning microscope produced by Lasertec and Yonekura in Japan. Its temperature control system adopts infrared laser heating. The maximum heating rate can reach 300 °C s⁻¹, and the cooling rate can reach 100 °C s⁻¹. Ar was used as the shielding gas in the experiment, and He was applied in the rapid cooling stage. After the in-situ observation, the microstructures of the specimens were observed by FEI Quanta-200 scanning electron microscope. Its accelerating voltage is up to 30 kV, the maximum amplification is 200000 times, and the resolution is 3.5 nm; the hardness was tested by HVS-1000Z hardness tester; finally, the phase composition of the weld was confirmed by D/MAX-rB X-ray diffractometer, the goniometer can be adjusted flexibly in the diameter of 400–520 mm, the rotation range of 2θ is −8° to 160° and the readable minimum step size is 0.0001° and the maximum positioning speed is 1500°/min.

3. Experimental results and analysis

Ti-6Al-4V alloy is composed of hexagonal close packed hcp-α phase and body-centered cubic bcc-β phase. Figure 2 presents the metallographic photograph of the weld microstructure, which is composed of a small amount of basket like and massive α phase and acicular α′ martensite. The coarse original β columnar grain boundary is faintly visible.

![Figure 2. Microstructure of weld metal.](image)

Table 2. Welding parameters.

| No.of layers | Laser Power PW | Welding speed V₁/cm s⁻¹ | Wire feed speed V₂/cm s⁻¹ | Focal ength f/mm | Defocus quantity Δf/mm |
|--------------|----------------|--------------------------|---------------------------|------------------|------------------------|
| Backing by single laser | 3500 | 1.01 | — | 425 | 20 |
| 2–16 | 3000 | 1.06 | 5.8 |
| 17 | 3500 | 1.06 | 6.0 |

Figure 2. Microstructure of weld metal.
The thermodynamic equilibrium phase diagram is calculated by using JMatPro software and the TC3 titanium alloy welding wire composition database. The results are shown in figure 3. The filler metal of TC3 titanium alloy welding wire is transformed into \( \beta \)-phase when heated to 977 °C. The only existing small amount...
of Ti3Al mesophase disappears when heated to 610 °C. Based on the above basic research data, the thermal cycle process adopted in this experiment is as follows: first heated up to 900 °C at a heating rate of 10 °C/s, then raised the temperature to 1 100 °C at the heating rate of 2 °C s⁻¹, and preserved the heat for 500 s to ensure that the β phase transformation process was complete, and then cooled to room temperature at a rate of 0.5 °C s⁻¹, 5 °C s⁻¹, 20 °C s⁻¹ and 80 °C s⁻¹ respectively.

3.1. Observation of microstructure evolution during heating

Figure 4 exhibits the in situ shooting morphology of the grain microstructure at the characteristic temperature point of Ti-6Al-4V titanium alloy plate narrow-gap weld microstructure by laser welding with filler wire during the heating cycle. When the temperature rises to 380 °C, the grains begin to grow obviously. The weld microstructure consisted of acicular flat α' that stops growing at the grain boundary, basket like α phase, a small amount of massive αₘ and original β grain boundary; when heated to 915 °C, the grains start to grow rapidly, and the weld microstructure is transformed into basket like α phase and massive αₘ phase as temperature reaches 975 °C; at 1 050 °C, the width of grain lath tended to be the same. The weld microstructures are mostly composed of coarse β columnar grains, and the original β grain boundary has been blurred. When the titanium alloy is heated, the volume change during α→β transformation is small, the transformation stress gets low, and the self-diffusion coefficient of the βbcc-β phase becomes high, so the transformation process becomes relatively short. From the whole heating process, it can be observed that the weld microstructure has experienced the process of acicular α' martensite+basket like α phase+massive αₘ→basket like α phase+massive αₘ→β, and the β phase gradually grows up in the way of grain boundary migration. The β columnar grain has grown into a larger size when the temperature exceeds 1 000 °C. It is difficult for the driving force of grain growth to merge small grains into a large one, but the slow movement of grain boundaries is observed. It can be concluded that β phase continues to grow from the original β phase grain boundaries rather than nucleate to form a new β grain structure. Figure 4(a) shows that the appearance of basket like α phase and acicular α' martensite is similar. The main distinguishing feature is that the latter stops growing at the grain boundary of β phase, while the former often begins to nucleate at the position. Figure 5 is the statistical curve of β phase volume with temperature. It can be seen from the figure that with the increase of heating temperature, β phase gradually engulfs the remaining microstructures on both sides through grain boundary migration, and finally a single β phase microstructure is formed. Under the experimental conditions, it can be observed that the transformation temperature of β phase is between 1 000 °C and 1 050 °C.

3.2. Observation of microstructure evolution during heat preservation

Figure 6 shows the in situ shooting morphology of the weld microstructure at characteristic time points, namely 100 s, 200 s and 300 s, during the heat preservation at 1 100 °C. It can be observed from the figure that the β phase boundary is moving. With the increase of the preservation time, the grain boundary becomes gradually flattened to reduce the surface energy. Meanwhile, new grain boundaries are formed, and gradually get clear with the extension of the preservation time. Finally, the shape and size of the β phase tend to be stable.

3.3. Observation of microstructure evolution during the cooling process

Figure 7 is the in situ shooting morphology of the weld microstructure at the characteristic temperature points during the cooling process at a rate of 0.5 °C s⁻¹, 5 °C s⁻¹, 20 °C s⁻¹ and 80 °C s⁻¹ respectively, after the heat preservation process at 1 100 °C was over. Figures 7(a), (d), (g) and (j) are four groups of weld microstructures after the completion of the heat preservation process, and at the initial moment of cooling. The β phase boundary can be clearly and obviously observed. Figure 7(b) shows that when the temperature is cooled down to
899.2 °C at a rate of 0.5 °C s⁻¹, the microstructure of a new phase begins to appear at the β phase grain boundary, and the α phase preferentially nucleates at the β grain boundary to form a continuous or discontinuous layer, that is, the grain boundary α_{gb} (grain boundary). Relevant studies have shown that the transformation process of titanium alloys is highly dependent on grain boundaries. In most cases, α_{gb} always presents a certain Burgers orientation relationship with β grains [14] (BOR, Burgers orientation relationship). Figure 7(e) illustrates that when the temperature is dropped to 861.7 °C at a rate of 5 °C s⁻¹, the microstructure of new phase begins to appear at the boundary of β phase, and Widmanstatten structure begins to grow from the boundary of β phase in laminar, i.e. α clusters, and maintains a Burgers orientation relationship at the same time. Figures 7(h) and (k) are the microstructure morphologies of new phase when the temperature is lowered to 826.6 °C and 806.6 °C at 20 °C s⁻¹ and 80 °C s⁻¹, respectively. The short acicular α′ martensite suddenly presents explosive growth mode. Due to the increase of driving force, the nucleation position not only occurs at the grain boundary, but also on
the $\alpha$ lamella inside the grain. From the above-mentioned time points for the occurrence of the new precipitated phase, it is found that with the increase of the cooling rate, the transformation temperature gradually decreases. This is because with the increase of the cooling rate, the diffusion capacity of the $\beta$ phase stabilizing element V weakens, resulting in the difficulty in forming $\alpha$ phase with low V, and gradually reduces phase transformation temperature. Besides, the morphological characteristics of the precipitated phases are also significantly different. As the cooling rate gets faster, the aspect ratio of the precipitated phases gradually is lowered, showing increasingly finer morphological features.

Figures 7(c), (f), (i) and (l) are the shooting morphologies of the microstructure at the characteristic temperature points when new phase no longer increases or grows during phase transformation. When the temperature is lowered to 702.1 °C at a cooling rate of 0.5 °C s$^{-1}$, the microstructure consists of lamellar $\alpha$ phase clusters with aspect ratio close to 20 and intragranular basket like $\alpha$ phase; when the temperature dropped to 754.7 °C at a rate of 5 °C s$^{-1}$, the microstructure is composed of lamellar $\alpha$ phase cluster with aspect ratio close to 5, intragranular basket like $\alpha$, Widmanstatten structure and grain boundary $\alpha$; when the cooling rate is increased to 20 °C s$^{-1}$, the precipitated phase is made up by $\alpha'$ martensite with a smaller aspect ratio and a small number of basket like $\alpha$, and the temperature for terminating the transformation is 763.8 °C; when the cooling rate is increased to 80 °C s$^{-1}$, the original grain boundary is basically broken due to the increase of the undercooling degree of nucleation and the thermodynamic driving force. The temperature for ending the $\alpha'$ phase transformation is 776.5 °C. During the cooling process, it is found that in addition to the nucleation at the grain boundary, some new phases grow on the primary $\alpha'$ martensite. The initial phase becomes the nucleation point, which increases the heterogeneous nucleation rate and greatly increases the growth rate of the new phase. The martensitic transformation of titanium alloy is non-diffusive transformation, in which there is no atomic diffusion but only lattice reconstruction. It has all the features of martensitic transformation. The dynamic feature is that there is no incubation period, the transformation nucleates and grows up instantaneously, and the transformation speed is very fast, with each martensite growing to the final size instantaneously. The crystallographic feature is that there is a strict orientation relationship between the martensite lattice and the parent $\beta$ phase, and the martensite always forms along a certain crystal face of the $\beta$ phase. The thermodynamic characteristic is that the resistance for martensite transformation is very large, and a greater undercooling degree is required during transformation, so the continuous progress of martensite transformation can only be carried out under lower and lower temperature conditions. Therefore, when the cooling rate is low, it nucleates at the grain boundary of $\beta$ phase and grows into the grain, due to the accumulation of a large amount of distortion energy at the phase boundary, which is conducive to the generation of energy, composition and structure.
fluctuation, and provides conditions for the nucleation of new phase. When the cooling rate is high, it grows not only at the grain boundary of $\beta$ phase, but also on the $\alpha$ lamella inside the grain.

3.4. SEM observation result

Figure 8 shows the SEM morphology of the room temperature microstructure obtained at different cooling rates. It can be found that with the increase of cooling rate, the lamellar spacing of the new phase gradually decreases and the interfacial area gradually increases. Under the condition of continuous cooling, the higher the cooling rate is, the lower the formation temperature of $\alpha'$ martensite or $\alpha$ phase is. That is, the higher the undercooling degree is, the lower the atom diffusion ability of the stable element V of $\beta$ phase is. It is not easy to migrate in a larger distance, thus forming a new phase with smaller lamellar spacing. In addition, the acicular $\alpha'$ phase has higher dislocation density and twinning, which further hinders the migration of new phase.

It can also be found from the figure that at a cooling rate of $0.5 \degree C s^{-1}$ and $5 \degree C s^{-1}$, the precipitated phase is composed of lamellar $\alpha$ phase clusters, and the grain boundary $\alpha_{gb}$ phase is found in the red circle, which was generated during slow cooling. At a cooling rate of $5 \degree C s^{-1}$, there is also a small amount of massive $\alpha_m$ phase in addition to the $\alpha_{gb}$ phase found in the room temperature structure obtained. When the cooling rate increases to $20 \degree C s^{-1}$, there is a small number of $\alpha$ phase and $\alpha'$ martensite. When the cooling rate increases to $80 \degree C s^{-1}$, the precipitated phases are all composed of bundles of acicular $\alpha'$ phase. The test results are completely consistent with the in situ microstructure observation results. In the observation of the microstructures, it is found that there are two different types of original $\beta$ grain boundaries as shown in figure 8, namely discontinuous grain boundary and continuous grain boundary. Figures 8(a) and (b) exhibit discontinuous original $\beta$ grain boundaries, which are composed of $\alpha_{gb}$ phase intermittently. It is believed that the discontinuous grain boundaries are generated because the microstructures in this region are transformed from the high temperature $\beta$ phase zone at a lower cooling rate. The undercooling degree is so small that the crystal nucleus can only be generated at the grain boundary and grow into the grain boundary $\alpha_{gb}$ phase. The growth is too slow to form a continuous grain boundary. Figures 8(c) and (d) shows microstructures obtained by temperature being cooled down from the high-temperature $\beta$-phase zone at a faster rate. The undercooling degree is larger, so the thermodynamic driving force is sufficient for nucleation to make the primary $\alpha$ phase nucleate and grow into continuous grain boundary.

3.5. Hardness and XRD test

Figure 9 shows the HV10 hardness values of the room temperature microstructure of Ti-6Al-4V titanium alloy welds obtained at different cooling rates. It can be seen from the figure that the hardness increases gradually with the increase of cooling rate. This is because the temperature for generating phase transformation decreases and the undercooling degree increases, which makes the lamellar spacing of new phase gradually become narrower (as can be seen from figure 8). The increase of phase contact area can effectively prevent the slip dislocation and increase the hardness. In addition, the order of hardness of each phase of titanium alloy is $\alpha' > \alpha > \beta$ [15]. The weld microstructure obtained at $0.5 \degree C s^{-1}$ and $5 \degree C s^{-1}$ cooling rate is composed of $\alpha$ phase, while that obtained at $20 \degree C s^{-1}$ cooling rate is composed of a small amount of $\alpha$ phase and acicular martensite $\alpha'$ phase. At $80 \degree C s^{-1}$, a
A great amount of acicular martensite \( \alpha' \) phases with high dislocation density and twinning are arranged in clusters in the weld microstructure, resulting in a large number of grain boundaries, which makes the hardness of the weld microstructure significantly higher than that of other regions.

In order to confirm the type of martensite phase in the room temperature microstructure of Ti-6Al-4V titanium alloy weld, XRD phase analysis was carried out on the weld area obtained at different cooling rates, and the results are shown in Figure 10. The Ti-6Al-4V titanium alloy weld microstructure by laser welding with filler wire is completely hexagonal close packed (HCP) crystal structure, and there is no orthorhombic lattice and body-centered cubic (BCC) crystal structure observed. Based on the \( c/a \) constant of each hexagonal lattice, it can be determined that all the microstructures in the weld are close packed hexagonal \( \alpha \) phase and \( \alpha' \) martensite, and no \( \alpha' \) phase, undercooled \( \beta \) phase or other harmful precipitated phases are produced.

4. Conclusions

1. When the temperature rose to 380 °C, the grains began to grow obviously; when heated to 915 °C, the grains started to grow rapidly, and the weld microstructure was transformed into basket like \( \alpha \) phase and massive \( \alpha_m \) phase as temperature reaches 975 °C; when the temperature was raised to 1050 °C, the weld microstructures were mostly composed of coarse \( \beta \) columnar grains, with blurred original grain boundary. The transformation temperature of \( \beta \) phase was then determined between 1 000 °C and 1 050 °C. With the increase of heating temperature, \( \beta \) phase gradually engulfed the remaining microstructures on both sides through grain boundary migration, instead of nucleating to form a new \( \beta \) grain structure.

2. When the temperature was cooled down to 899.2 °C, 861.7 °C, 826.6 °C and 806.6 °C at a rate of 0.5 °C s\(^{-1}\), 5 s\(^{-1}\), 20 °C s\(^{-1}\) and 80 °C s\(^{-1}\) respectively, the microstructure of a new phase began to appear at the \( \beta \) phase grain boundary, and the transformation temperature for new phase precipitation decreased with the gradual increase of the cooling rate. Widmanstätten structure and grain boundary \( \alpha_{gb} \) appeared at the cooling rate of 0.5 °C s\(^{-1}\) and 5 °C s\(^{-1}\). At 20 °C s\(^{-1}\) and 80 °C s\(^{-1}\), short acicular \( \alpha' \) martensite began to show up, with nucleation position not only at the grain boundary. As the cooling rate got faster, the aspect ratio of the precipitated phases gradually was lowered, showing increasingly finer morphological features.

3. When the temperature was cooled down to 702.1 °C and 754.7 °C at 0.5 °C s\(^{-1}\) and 5 °C s\(^{-1}\) respectively, the microstructures were composed of \( \alpha \) phases; when cooled the temperature to 763.8 °C at 20 °C s\(^{-1}\), the microstructure was made up by \( \alpha' \) martensite and a small number of \( \alpha \) phase; when cooled to 776.5 °C at 80 °C s\(^{-1}\), precipitated phases were all composed of acicular \( \alpha' \) martensite. The higher the transformation temperature for new phase, the narrower the temperature range.

4. With the increase of cooling rate, the lamellar spacing of the new phase gradually decreased and the interfacial area gradually increased. Discontinuous original \( \beta \) grain boundaries were composed of \( \alpha_{gb} \) phase intermittently at a low cooling rate. With a relatively high cooling rate, continuous grain boundaries were

![Figure 10. XRD of welded zone for different cooling rates.](image-url)
formed. The hardness of the weld microstructures was improved with the increase of the cooling rate. Weld microstructures were completely hexagonal close packed (HCP) crystal structures.

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**Data availability statement**

All data that support the findings of this study are included within the article (and any supplementary files).

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