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ORIGINAL REPORT



High speed synchrotron X-ray diffraction experiments resolve microstructure and phase transformation in laser processed Ti-6Al-4V

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ABSTRACT

The microstructures of Ti-6Al-4V following laser processing depend primarily on the phase transformation of β to α , but their development is constrained by the rapidly changing temperature in the small fusion zone. *In-situ* synchrotron X-ray diffraction was utilized to probe the rapid phase evolution in single melt tracks with high angular and temporal resolution. Both fully martensitic and mixed $\alpha + \alpha' + \beta$ microstructures were confirmed by microscopy. Cooling rates were inferred from the lattice parameter history and complementary thermal simulation. It was found that the threshold cooling rate for fully martensitic transformation is in the range between 2900 and 6500°C/s.

IMPACT STATEMENT

High-speed synchrotron X-ray diffraction during operando laser processing suggests a new threshold between martensitic and diffusional phase transformation in Ti-6Al-4V occurring at higher cooling rates than previously reported.

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Ti-6Al-4V; martensite; laser melting; X-ray diffraction; *in-situ* /operando characterization

Introduction

Ti-6Al-4V, aka Ti64, has been extensively employed in many industries [1,2] because of its attractive properties in strength, density, corrosion resistance, and biocompatibility. Moreover, based on its weldability, various applications have been developed with laser light as the heat source, such as laser welding, additive manufacturing (AM) as in laser powder bed fusion (LPBF), and laser shock peening [2–4]. Just as in many steels, the solid-state transformation in Ti64 enables considerable flexibility in microstructural design [5]. However, rapid cooling through the β -transus restricts Ti64 microstructures to either an acicular martensite α' with a distorted HCP structure or a tempered $\alpha + \beta$ microstructure. The microstructure affects mechanical properties: it is known that the martensitic structure profoundly affects the ductility and strength of Ti64 [6–8]. Therefore, a comprehensive understanding of microstructure development is crucial for optimizing properties of laser processed Ti64 parts.

A highly focused laser is suitable to selectively melt metals because of its high power density, processing speed, and dimensional accuracy possible with melt pools of the order 200 μm in width. However, much remains to be learned about laser processing because of the rapid evolution of temperature, phase, and microstructure within the small fusion zone (FZ) [9–11]. The rapid heating and cooling during laser processing induce non-uniform temperature fields, gradients, and cooling rates. Additionally, the high β -transus temperature requires specialized tools to examine the evolution involved with solid-state transformation in Ti64 [12–14].

In-situ X-ray diffraction is widely used to examine microstructure development with optimized temporal, spatial, and 2θ -angular resolution. Fast detectors (up to 20 kHz) have been employed to examine phase evolution under rapid processing [10,15–22], such as welding and AM processing, and combination with ultrafast imaging provided new insights into the AM process [9,18,22]. Studies with high angular resolution and

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a relatively large area detector have revealed details of lattice changes for external mechanical and temperature factors [11,23–28]. Our recent report presented an *in-situ* study of Ni-alloy 718 with a multi-panel area detector with an unprecedented combination of high 2θ -angular resolution and a 250 Hz frame rate [29]. Here, we conducted an *in-situ* experiment with the same apparatus to study the rapid microstructural evolution of resolidified Ti64 after laser melting. Recent works on Ti64 employed high-speed detectors (> 250 Hz) to study the microstructural changes in Ti64 under AM (AM-like) processing [16–20]. By placing the faster (but smaller) detectors at off-center positions, the detected scattering ranges were maximized, but full diffraction rings could not be detected, which makes it challenging to separate different effects on the lattice.

In this article, with a focus on β to α/α' transformation, the laser scan speed was controlled to result in microstructures in the FZ that varied from martensitic α' to $\alpha+\alpha'+\beta$. Post-processing electron microscopy validated the features of the microstructural evolution that the x-ray diffraction detected in real-time. Changes in the lattice parameter of each phase were found from the diffraction results and compared with the simulated temperature evolution.

Experimental

Ti64 bar (89.33Ti, 6.20Al, 4.06V, 0.20Fe, 0.021C, and 0.18O by wt%), obtained from McMaster-Carr, was cut by electrical discharge machining into thin plates with dimensions, $40 \times 3 \times 0.9$ mm³. The experiments were performed at the 1-ID-E beamline at the Advanced Photon Source of Argonne National Laboratory. The prepared Ti64 plates were placed inside a stainless-steel vacuum chamber on an XYZ translation stage. After evacuating and filling the chamber with argon gas, a ytterbium fiber laser scanned the top surface of the plate along the direction perpendicular to the x-ray incidence, as described in Supplementary Materials (Figure S1) and our previous report [29]. Monochromatic x-rays (55.615 keV) passed through Kapton windows of the chamber and penetrated the side of the Ti64 plates. The diffracted x-rays were collected with a Pilatus3X 2M CdTe detector at 250 frames per second, placed 685 mm away from the sample. The area illuminated by the x-rays was $100(h) \times 40(v)$ μm^2 , with the upper edge positioned 100 μm below the sample surface. Laser scan speeds of 50 and 30 mm/s were used (denoted by SS50 and SS30). The laser was set to ~ 120 μm spot size, 560 W power, and 2.0 mm scan length.

To enable interpretation of phase evolution, the collected 2D diffraction patterns were integrated around

Debye-Scherrer rings using the HEXRD package [30] to form 1D profiles (2θ -Intensity). A Pseudo-Voigt function was employed to fit the individual peaks. Time-resolved lattice spacings obtained from the fitting results were used to estimate temperature changes based on the reported coefficient of thermal expansion of Ti64 [17,31] using a method described in our previous research [29]. The estimation was not performed over the temperature range 800–1100°C where lattice changes could be impacted by the solid-state transformation. For comparison, heat flow simulation was performed with QuikCASTTM software. More details of the assumptions and models used in this study are described in the Supplementary Materials.

To examine the post-melting microstructure and composition variation in the laser melted region, microscopic analysis was conducted on polished cross-sections of the Ti64 plates after etching with Kroll's reagent (2 ml HF, 5 ml HNO₃, and 93 ml H₂O) for 30 s. The etched surface was observed with an optical microscope (OM, Leica DM750M) and a scanning electron microscope (SEM, TESCAN MIRA-3 FEG). For transmission electron microscopy (TEM, FEI Themis) analysis, samples with < 100 nm thickness were prepared by focused ion beam milling (FIB, Nova NanoLab 600). To identify compositional variation, elemental mapping was performed with energy-dispersive X-ray spectroscopy (EDS).

Results and discussion

Figure 1 presents the diffraction results of Ti64 during laser processing. The 2D diffraction patterns in Figure 1(a) to (c), taken at room temperature, show the overall phase composition. The sharp and complete Debye-Scherrer rings of the α phase in the base metal (BM), Figure 1(a), indicate numerous randomly oriented α grains. Figure 1(b) and (c) show that the FZs have fewer and coarser grains which are reflected by the discontinuous rings and spotty distribution of the diffraction [32].

Figure 1(e) and (f) show time-resolved intensity maps that summarize the sequentially stacked 1D diffraction profiles as functions of time. As a reference, the time when the laser passed across the x-ray beam was defined as 0 ms. For SS50 (Figure 1(e)), as the laser approaches from -20 ms, the temperature in the measured volume increased, reflected by the peaks shifting to lower 2θ due to the thermal expansion. Then, while the Ti64 is melted completely, no diffraction peaks are observed. After 16 milliseconds of melting, solidification began with the β phase appearing first. Upon cooling, thermal contraction caused the β peaks to shift to higher 2θ . Around 80 ms, the β phase transformed into the hexagonal phase. After the transformation, no peaks from the β phase were

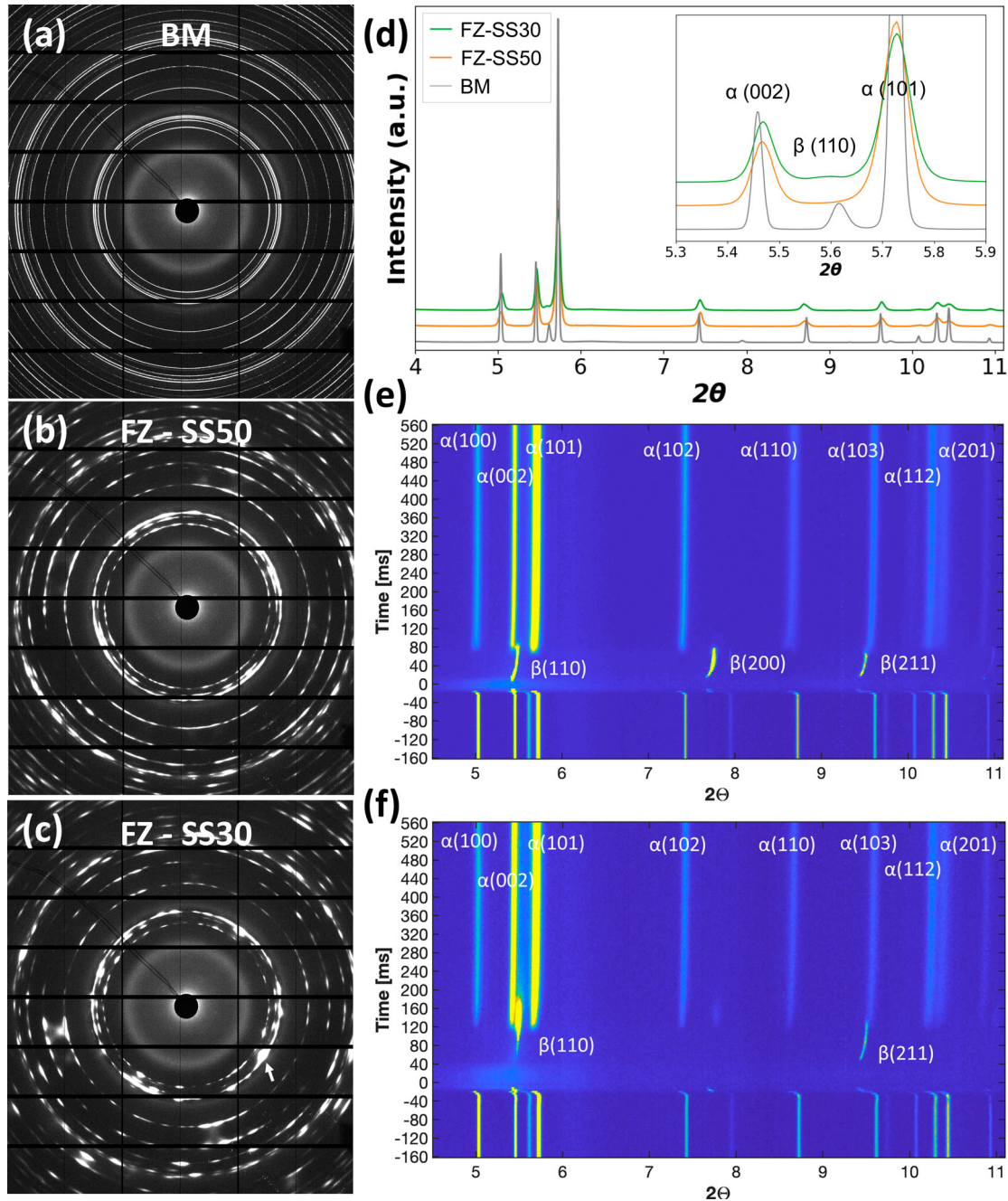


Figure 1. (a)–(c) 2D diffraction patterns of the BM and the SS50 and SS30 FZs at room temperature, (d) 1D diffraction profiles integrated from (a) – (c), (e) and (f) time-resolved intensity maps of Ti64 for SS50 and SS30.

observed: the β completely transformed into the martensitic phase (α') due to the high cooling rate of SS50 [5]. The broadened α/α' peaks imply the micro-strains from the defected lattices or the presence of fine domains in the grains [24,33–35]. Also, the formation of the distorted HCP phase can be inferred from the difference in the peak positions of α between BM and FZs in Figure 1(d).

The results of SS30 (lower laser scan speed) are presented in Figure 1(f). A longer duration of the liquid stage is evident, and the overall changes in the diffraction

intensity map resemble those of SS50. However, unlike SS50 where the β (110) peak was only present for 60 ms before disappearing, the β (110) peak intensity for SS30 decreased more slowly during the phase transformation and did not disappear completely: see the non-zero intensity between the α (002) and α (101). The features are more clearly demonstrated in Figure 1(c) and (d). Diffuse β reflections are observed between α (002) and α (101) in Figure 1(c), and evident in Figure 1(d), integrated from the 2D patterns of Figure 1(c), as a small peak around

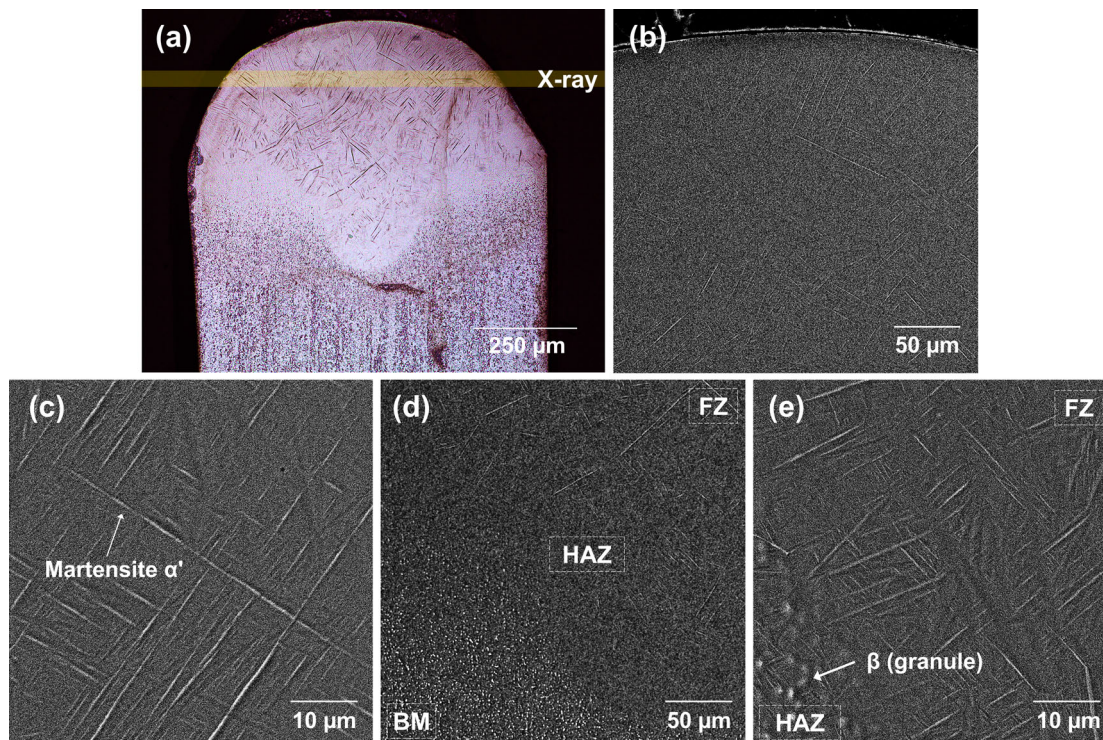


Figure 2. Microstructure of the SS50 FZ: (a) the etched surface (OM), (b) the top and (c) the center and (d), (e) the boundary with the HAZ (SEM-SE).

5.56° between the two α peaks. The results show that a small amount of β phase remained after cooling to room temperature. In addition, as an earlier work suggested [23], increasing the vanadium in the α and β phases would lead to a reduction in their lattice parameters. The peak position of the β (110) of SS30 implies that less V is segregated in the β phase in its FZ than in the BM that was prepared in a more equilibrium-like condition than laser processing.

Figures 2 and 3 present the FZ microstructures. The optical images, Figure 2(a) and Figure 3(a), show the cross-sections of the samples. The approximate positions of x-ray transmission are marked in yellow, showing the x-rays passed through the upper parts of the FZs that contained only resolidified material without including BM or heat-affected zone (HAZ). On the etched surface, the FZ is readily distinguished from HAZ and BM. In SS50, the needle-like microstructures are sparsely dispersed in the FZ, whereas the SS30 FZ presents a darker area with distinct boundaries of the prior β grains. Plus, there are contrast changes from the edge to the interior of the SS30 FZ, indicating a gradual change in microstructure. The maximum depths of the FZ are 630 and 1000 μm for SS50 and SS30, respectively.

The SEM images show details of the FZ microstructures. The SS50 FZ microstructure is uniform everywhere in the FZ, indicating that the resolidified material

completely transformed to the martensitic phase, α' . In Figure 2(b), the acicular α' is present in bundles in different variants, which represents a typical martensitic microstructure [36]. Figure 2(d) shows the FZ-HAZ-BM boundary. Away from the FZ, the HAZ gradually changed to the microstructure of the BM. Figure 2(e) shows that granular β (present in the BM) disappears closer to the FZ.

On the other hand, Figure 3 shows that the SS30 FZ microstructure varied by position. Figure 3(b) to (e) are the microstructures from the top to the middle of the FZ which mainly consists of basket-weave microstructure with grain-boundary α and some acicular α' near the boundary of the HAZ. The dissimilarity of the microstructure of the FZ between SS50 and SS30 resulted from different transformations during laser processing. The element mapping results in Figure 3(f) suggest that the SS30 FZ experienced diffusional (incomplete martensitic) transformation, unlike SS50 [5]. Regions richer in Fe and V (β stabilizers) and depleted of Al indicate that the β phase remained as thin layers, corresponding to the weak and broad diffraction reflections of β in Figure 1. The boundary between the FZ and the HAZ in Figure 3(d) does not show the basket-weave microstructure. Instead, it is similar to the martensitic microstructure shown in Figure 2. In Figure 3(d), the microstructures of the BM, the HAZ, and the FZ are diagonally presented

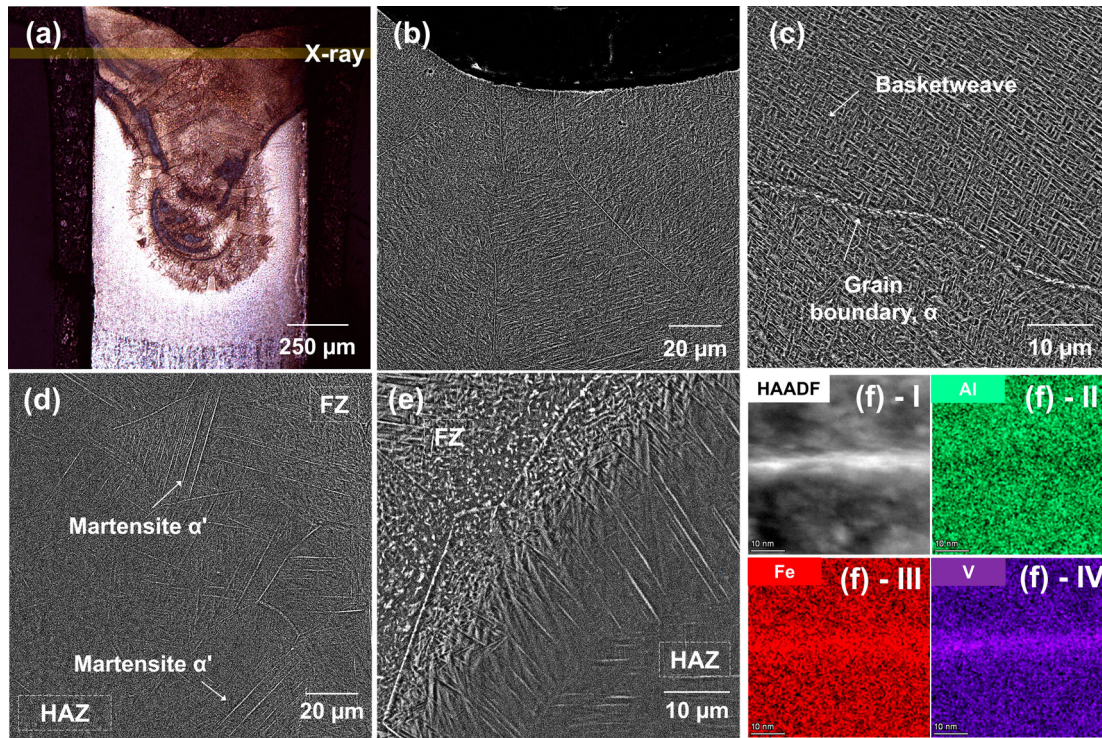


Figure 3. Microstructure of the SS30 FZ: (a) the etched surface (OM), (b) the top and (c) the center and (d), (e) the boundary with the HAZ (SEM-SE). (f) STEM-EDS element mapping results, I – HAADF image, II-IV – Al, Fe, V mapping.

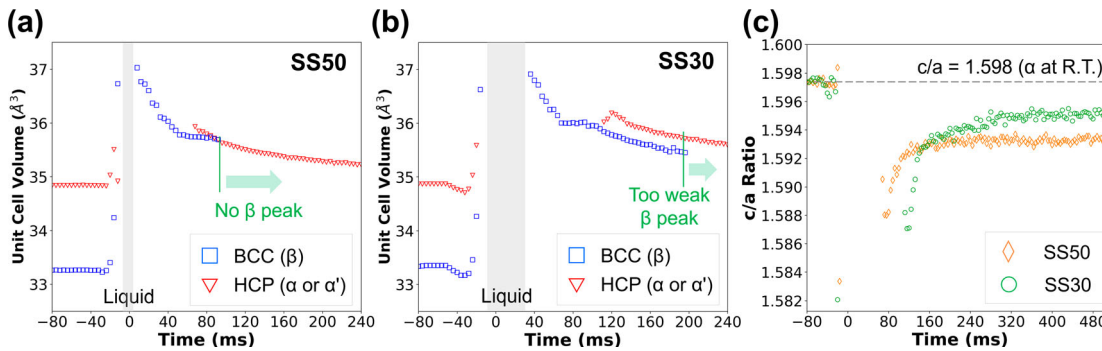


Figure 4. The evolutions in (a-b) the average lattice volume and (c) c/a ratio of α/α' of SS50 and SS30. The β lattice was detectable until 96 ms for SS50 and 156 ms for SS30.

from the bottom left of the image. Regions without the granular phases of the BM show acicular (martensitic) α' . Grain-boundary α and dense basket-weave are observed at the top right of the image.

In Figure 4, the lattice evolution demonstrates various aspects of the phase transformation and the laser process. Each data point was recorded for cases where the corresponding lattices were discernible. Generally, as the laser heating source approached the measured area, the unit cell volume shows a slight contraction. According to the temperature gradient mechanism [37], the neighboring material expands due to laser heating, thus compressing the lattice. During the initial stage of cooling, the solidified β rapidly contracts. Then, as the temperature reaches

the transformation temperature, shrinkage arrests or slows down. The arrest in thermal contraction begins at 20 ms in SS50 and 40 ms in SS30 before the transformation of β starts. However, the lattice reverts to shrinking before the start of the transformation. The behavior is not fully understood but may be caused by two effects combined with the phase transformation. One possibility is heat release during the transformation of β [38] in neighboring regions, i.e. recalescence, that retards cooling in the measured region. Another possibility is a transient imbalance in the cooling rate. As shown in the simulation results in Figure 5, the cooling rate is predicted to increase just after the arrest caused by transformation. Faster contraction in the neighboring region can induce thermal

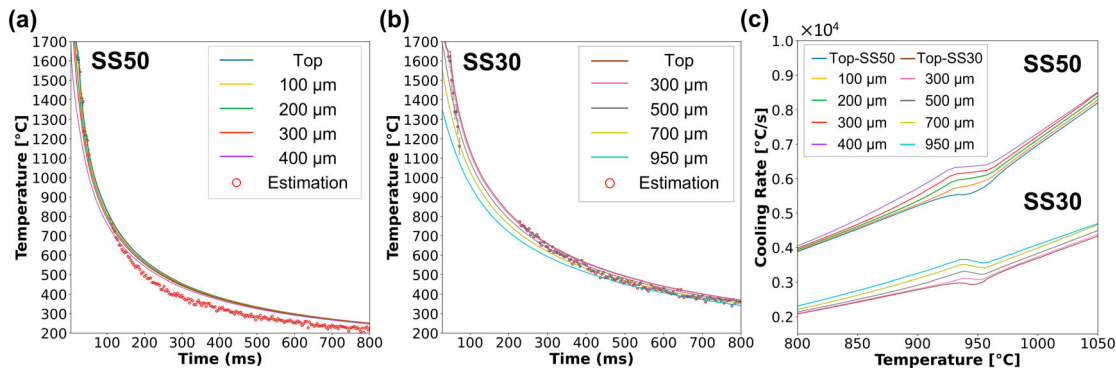


Figure 5. (a) Estimated and simulated temperature evolution in FZ of (a) SS50 and (b) SS30; simulation results are shown at different distances from the FZ top, and (c) cooling rates vs temperature of SS50 and SS30 with the distances from the FZ top. The ‘top’ labels indicate the top surface of each FZ.

strain on the measured volume. Also, Figure 4(a) and (b) demonstrate that the transformed α/α' has a lattice volume that is similar to that of the transforming β (during the transformation). However, the difference between the volumes of β and α in SS30 is larger than SS50. The behavior indicates partial diffusional transformation in SS30, and the occurrence of diffusion of the alloying elements and consequent volume difference between the phases [23,39]. The evolution of the c/a ratio of the α/α' lattice is presented in Figure 4(c). Generally, the ratio increases as cooling proceeds after the transformation is complete. This behavior has not been well studied yet, but it is assumed to originate from anisotropic thermal contraction in the HCP phase [40,41]. At room temperature, the c/a ratios for the two FZs were 1.5942 and 1.5955, owing to the difference in the composition and the residual strain, induced by the different cooling histories of SS50 and SS30.

Figure 5 presents the temperature history and the calculated cooling rates with respect to temperature, at different positions in the FZ. The lines and scatter plots respectively indicate the simulation and experimental estimation results obtained by the methods described in Supplementary Material. The temperatures decreased monotonically, with faster cooling in SS50 than in SS30. At the transformation of β to α/α' , the cooling rate in the SS50 FZ ranged from 5600°C/s to 6500°C/s, which is faster than for SS30 which ranged from 2900°C/s to 3600°C/s, shown in Figure 5(c). The presence of the β phase in the SS30 FZ indicates that the complete martensitic transformation was not attained at 3000°C/s. Although cooling is retarded slightly by the heat of transformation, the effect is small because of the small enthalpy change (48 J/g) compared with the heat of fusion (286 J/g) [42]. Even though the temperature evolution predicted by simulation and estimated from the lattice parameters do not correspond exactly, the general trends agree. The critical cooling rate in this work

is substantially larger than the well-known research of Ahmed & Rack [5] reporting fully martensitic transformation over 410°C/s during continuous cooling from 1050°C. On the other hand, Kenel et al. [18] reported that incomplete martensitic transformation was observed at 4500°C/s at the solidification front of Ti64 during laser processing. The close agreement with our results for incomplete martensitic transformation at a high cooling rate supports the suggestion that the critical cooling rate under laser-processing conditions is higher than previous work.

Conclusion

We have characterized the evolution of FZs in Ti64 during laser processing with high angular-temporal resolution. Two different laser scan speeds, SS50 and SS30, were employed to develop a complete martensitic microstructure, α' , and an incomplete martensitic microstructure, $\alpha+\alpha'+\beta$ in each FZ. The X-ray diffraction and microscopy analysis demonstrate different features in the evolution of each FZ and verify the existence of the β phase in the FZ of SS30. The temperature evolution in the FZ was simulated and compared with the estimation based on the thermal expansion of the lattice. Based on the good agreement found, the results were combined to estimate the cooling rate required for the martensitic transformation in Ti64. SS50 resulted in a fully martensitic microstructure at a cooling rate ~ 5600 – 6500 °C/s, whereas SS30 was only partially martensitic at a cooling rate ~ 2900 – 3600 °C/s. This result suggests the critical cooling rate for fully martensitic transformation during cooling of Ti64 following laser melting.

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Disclosure statement

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

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