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Microstructure Evolution of Gas-Atomized β -Solidifying γ -TiAl Alloy Powder during Subsequent Heat Treatment

Sung-Hyun Park ¹, Ozkan Gokcekaya ^{1,2} , Ryosuke Ozasa ^{1,2} , Ken Cho ^{1,2}, Hiroyuki Y. Yasuda ^{1,2},
Myung-Hoon Oh ³ and Takayoshi Nakano ^{1,2,*} 

- ¹ Division of Materials and Manufacturing Science, Graduate School of Engineering, Osaka University, 2-1 Yamadaoka, Suita 565-0871, Osaka, Japan; sung Hyun.park@mat.eng.osaka-u.ac.jp (S.-H.P.); ozkan@mat.eng.osaka-u.ac.jp (O.G.); ozasa@mat.eng.osaka-u.ac.jp (R.O.); k_cho@mat.eng.osaka-u.ac.jp (K.C.); hyasuda@mat.eng.osaka-u.ac.jp (H.Y.Y.)
- ² Anisotropic Design & Additive Manufacturing Research Center, Osaka University, 2-1 Yamadaoka, Suita 565-0871, Osaka, Japan
- ³ School of Materials Science and Engineering, Kumoh National Institute of Technology (KIT), 61 Daehakro, Gyeongbuk, Gumi 39177, Republic of Korea; ohmyung@kumoh.ac.kr
- * Correspondence: nakano@mat.eng.osaka-u.ac.jp; Tel.: +81-6-6879-7505

Abstract: To promote the use of γ -TiAl alloys in various domains, such as the aerospace industry, it is pivotal to investigate the unusual phase transformation from rapidly solidified and metastable γ -TiAl toward the equilibrium state. In this study, the microstructure characteristics of gas-atomized β -solidifying Ti-44Al-6Nb-1.2Cr alloy powder, in terms of the effect of rapid solidification on microstructure evolution, were explored in comparison with cast materials. The phase constitution, morphology, and crystallographic orientation between phases were noted to be distinct. Furthermore, subsequent heat treatment was conducted at different temperatures using gas-atomized powder. The transition from the metastable to equilibrium state was observed, wherein firstly, the γ phase precipitated from the retained α_2 phase, forming an α_2/γ lamellar microstructure. In intensified heat-treatment conditions adequate for cellular reaction, β/γ cells were formed at the grain boundaries of α_2/γ lamellar colonies. The findings highlight the overall phase transformation during rapid solidification and continuous microstructural evolution from the nonequilibrium to the equilibrium state. This research can bridge the gap in understanding the effect of the solidification rate on microstructural evolution and contribute to enhanced comprehension of the microstructure in other domains involving rapid solidification, such as the additive manufacturing of γ -TiAl alloys.

Keywords: gas atomization; rapid solidification; nonequilibrium state; β -solidifying γ -TiAl alloy; recrystallization



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1. Introduction

Gamma (γ)-titanium aluminide alloys (γ -TiAl) are promising alternative structural materials to the superalloys currently used in the aerospace industry, owing to their low density, high creep resistance, and excellent oxidation resistance [1–3]. Although conventional Ti-48Al-2Nb-2Cr (at.%) alloy has been successfully used in aero-engine low-pressure turbine blades [1,4], it has become necessary to attain superior temperature capabilities and expand the application scope of γ -TiAl alloys. Therefore, considerable research has been focused on exploring different compositions, and various pathways for microstructure optimization and manufacturing have been proposed [4,5]. Notably, additive manufacturing (AM) has emerged as a promising manufacturing strategy owing to the unique microstructure characteristics involved, and its competitiveness relative to traditional manufacturing processes has been demonstrated [6–8].

The microstructure characteristics in AM depend on the composition of TiAl alloys and the cooling rates which can be controlled with AM process parameters [6,7,9]. In addition,

the cooling rates during the AM process have been estimated to be 10^5 – 10^7 K/s [10–12]. Moreover, this process involves multiple reheating and cooling cycles, resulting in rapid solidification and complex thermal variations, leading to the unique microstructures observed during AM. However, most experimental data have been derived under low cooling rates, such as those typically used during casting and subsequent heat treatment to control the microstructure [13]. Although certain studies have furnished data pertaining to the quenching [14–16], the effect of rapid solidification on the microstructure remains to be clarified.

In the domain of powder metallurgy, gas atomization is a powder manufacturing method that yields spherical particles [17]. The cooling rate during gas atomization is similar to that in the AM process [18]. Thus, equilibrium phase transformation is likely suppressed during solidification, resulting in the dominance of the α/α_2 phase in the final microstructure [19–22]. Despite controversial results concerning the ordering in the α/α_2 phase, it is clear that the microstructure of γ -TiAl alloys depends on the cooling rate. Several researchers have attempted to illustrate the evolution from the metastable α/α_2 phase toward equilibrium [21,23]. These studies highlighted that the γ phase precipitated in a nano α_2/γ lamellar structure during subsequent heat treatment, then the α_2/γ disappeared and fully recrystallized. There was no plastic deformation during the subsequent heat treatment that could have triggered the recrystallization. Thus, it is necessary to investigate the unusual phase transformation of the rapidly solidified, metastable γ -TiAl toward the equilibrium state.

Therefore, this study was aimed at exploring the microstructure development of metastable alloys, especially during the transition from the metastable to equilibrium state. We observed the microstructure characteristics of gas-atomized Ti-44Al-6Nb-1.2Cr (at.%) powder subjected to heat treatment, in comparison with those of cast materials. The findings are expected to fill the gap between the different microstructural developments depending on the cooling rate. In addition, the direct observation after rapid solidification with subsequent heat treatment provides further understanding of the enhancement in the microstructure after rapid solidification and complex thermal cycling, such as those typically involved in the AM of γ -TiAl alloys, and useful guidance for microstructure optimization.

2. Materials and Methods

Ti-44Al-6Nb-1.2Cr (at.%) alloy powder was fabricated using the gas-atomization process (Osaka Titanium Technologies, Amagasaki, Hyogo, Japan). The mean particle diameter was measured to be 36.0 μm using a laser-diffraction-type particle size distribution measuring device (Mastersize 3000E, Malvern Panalytical, Malvern, UK). To investigate the evolution of the microstructure during the subsequent heat treatment, two temperatures were selected: The raw powders were isothermally exposed at 800 °C and 900 °C for 3 h followed by quenching in water to room temperature. All samples were sealed in an Ar atmosphere to prevent oxidation. For comparison, the same alloy composition of the ingot (as-cast) was attained using arc melting, and identical heat treatment procedures were conducted.

The phase constitution of the powder and as-cast samples was identified through X-ray diffractometry (XRD; X'pert PRO, Philips, Amsterdam, The Netherlands) using Cu K-alpha radiation. Other microstructural characteristics, such as the phase morphology, composition distributions, and crystallographic orientations, were investigated from backscattered electron (BSE) images, energy dispersive X-ray spectroscopy (EDS; Aztec 4.4, X-Max^N, Oxford Instruments, Oxfordshire, UK), and electron backscatter diffraction (EBSD; Aztec 4.4, NordlysMax3, Oxford Instruments, Oxfordshire, UK) images obtained with through field-emission scanning electron microscopy (FE-SEM; JEM-6500F, Tokyo, JEOL, Japan). The powder samples for BSE, EDS, and EBSD characterization were embedded in acrylic resin (KM-CO, PRESI, Eybens, France) and polished to a mirror finish.

3. Results and Discussion

3.1. Differences in Microstructures of As-Cast Alloys and Gas-Atomized Powder

Figure 1 shows the XRD profiles and SEM-BSE images of the as-cast alloy and gas-atomized powder, respectively. The XRD analysis (Figure 1a) revealed three different phases, β , α_2 , and γ , in the as-cast sample. In contrast, the powder consisted of only the β and α_2 phases, and no γ phase was detected. Furthermore, the SEM-BSE image of the as-cast sample showed an intermediate contrast of coarse α_2/γ colonies surrounded by the brightest β phase (clumps indicated by white ridges) and small amounts of γ phase precipitated with dark contrast inside the β phase (Figure 1b). From the EDS results listed in Table 1, the β phase indicated a lower Al concentration and much higher Nb and Cr concentration compared to the α_2/γ colonies and γ phase. Therefore, the different contrasts are determined from their compositional differences, which are commonly observed microstructural characteristics in β -solidifying γ -TiAl alloys manufactured via casting [13,24]. In contrast, the cross-section of the powder revealed a dendritic and interdendritic morphology with gray and dark contrasts, respectively (Figure 1c). The chemical distribution represented that the dendrite region is rich in Ti and Nb, while the interdendrite is rich in Al and Cr (Table 2). This segregation phenomenon occurs during the solidification because the Al and Cr repelled to the interdendritic regions when the dendrite solidified from the liquid [22]. Furthermore, irregular morphology of grain boundaries across the dendritic and interdendritic regions can be observed (red arrows).

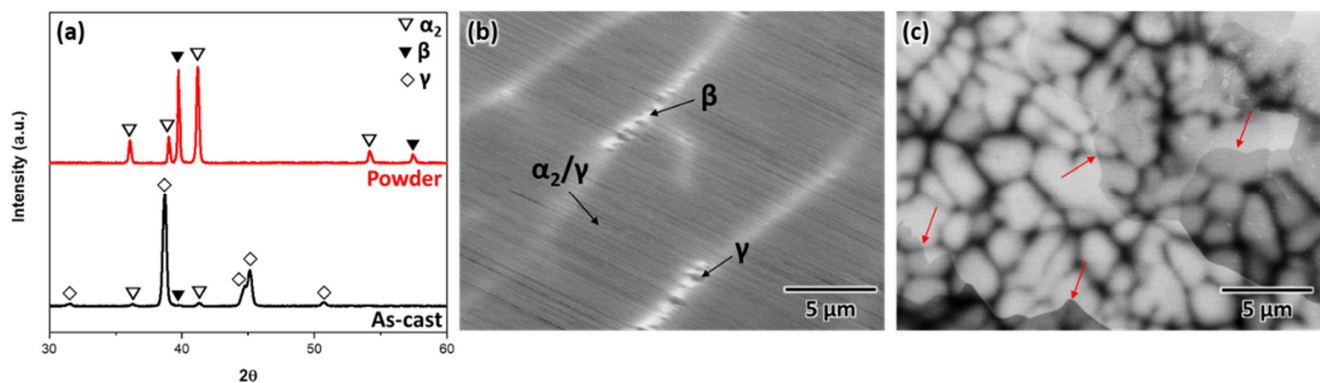


Figure 1. (a) Typical XRD patterns of as-cast samples and gas-atomized powder, (b) SEM-BSE microstructure images of the as-cast sample, and (c) powder in the cross-sections. The red arrows indicate the grain boundaries across the dendritic and interdendritic regions.

Table 1. The EDS results of different phases in cast materials.

Constituent	Composition (at.%)			
	Ti	Al	Nb	Cr
β	52.37 $\pm$ 0.57	37.83 $\pm$ 0.73	6.87 $\pm$ 0.15	2.93 $\pm$ 0.27
α_2/γ lamellae	50.26 $\pm$ 0.29	43.02 $\pm$ 0.29	5.52 $\pm$ 0.04	1.20 $\pm$ 0.10
γ	49.68 $\pm$ 0.24	43.68 $\pm$ 0.23	5.53 $\pm$ 0.08	1.11 $\pm$ 0.04

Table 2. The EDS results of different regions in the cross-section of gas-atomized powder.

Constituent	Composition (at.%)			
	Ti	Al	Nb	Cr
Dendrite	50.57 $\pm$ 0.57	41.93 $\pm$ 0.63	6.38 $\pm$ 0.16	1.12 $\pm$ 0.10
Interdendrite	48.65 $\pm$ 0.20	44.86 $\pm$ 0.40	4.93 $\pm$ 0.32	1.56 $\pm$ 0.15

To better understand the microstructural characteristics, additional observations were performed using SEM-EBSD. Consistent with the results of the SEM-BSE analysis, coarse

α_2/γ colonies and an elongated β phase were observed in the phase map (Figure 2a), with each α_2/γ colony exhibiting a different crystallographic orientation in the inverse pole figure (IPF) map (Figure 2b). These different orientations resulted due to the solid phase transformation from the β to α phase during solidification, a key concept in the design of β -solidifying γ -TiAl alloys. In general, solid phase transformation through the complete β phase region without peritectic reactions can suppress chemical inhomogeneities and grain growth in the casting process for different γ -TiAl alloys [25]. Pole figures (PFs) for the β , α_2 , and γ phases obtained from within the black rectangular box in Figure 2a,b show the orientation relationship between each phase (Figure 2c). The PFs demonstrate that the (β and α_2) and (α_2 and γ) phases are crystallographically aligned according to the Burgers orientation relationship (Burgers OR) and Blackburn orientation relationship (Blackburn OR), respectively, as previously reported [16,26].

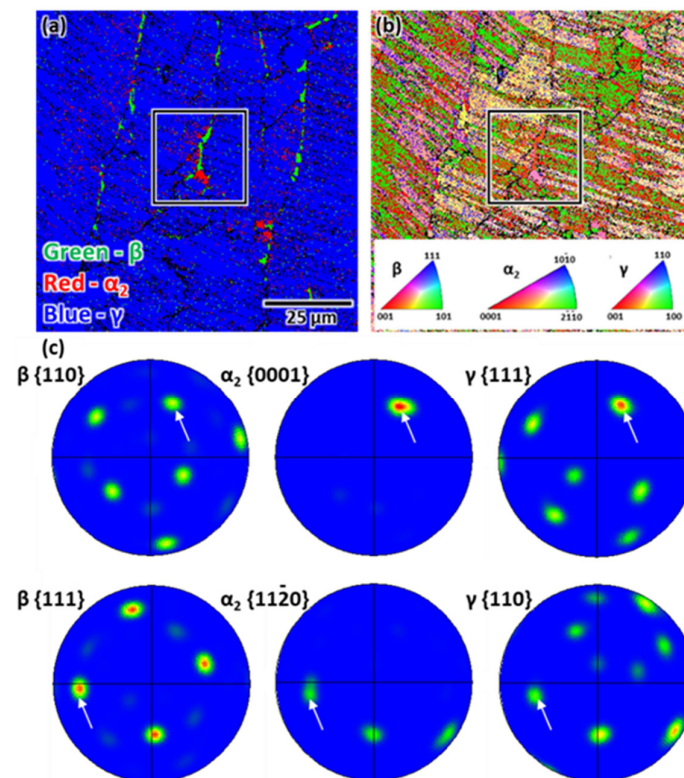


Figure 2. SEM-EBSD analysis of the as-cast sample: (a) phase map, (b) IPF map, and (c) PFs. The PFs pertaining to the phases corresponding to the black rectangular area and white arrows indicate that the (β and α_2) and (α_2 and γ) phases are aligned according to the Burgers OR and Blackburn OR, respectively.

In contrast, the phase map showed a small amount of the β phase and a predominant α_2 phase in the powder cross-sections (Figure 3a). The volume fraction of the β phase increased as the powder size decreased, owing to the higher cooling rate of smaller particles, consistent with observations pertaining to powders of different sizes [27]. In addition, the irregular morphology of the α_2 phases was randomly distributed (Figure 3b). Detailed observation in a specific region (black rectangular box) including the β and α_2 phases revealed no specific orientation relationship between the two phases (Figure 3c), conflicting with the result of the as-cast sample. This difference was attributable to the massive α grain formation owing to rapid solidification [28].

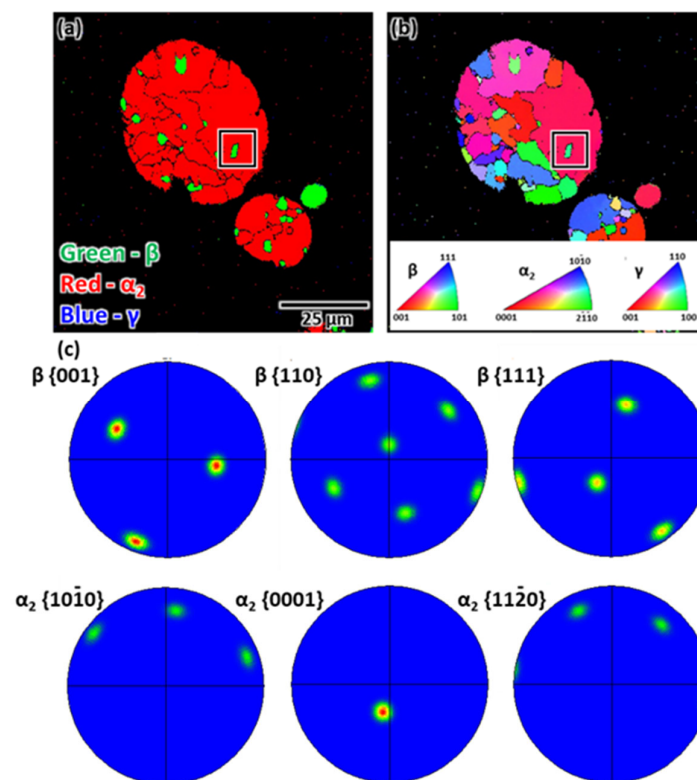


Figure 3. SEM-EBSD analysis of the gas-atomized powder: (a) phase map, (b) IPF map, and (c) PFs. The PFs correspond to the black rectangular area. The alignment of the β and α₂ phases does not comply with the Burgers OR.

As widely recognized, the phase transformation sequence of β-solidifying γ-TiAl alloys can be expressed as $L \rightarrow L + \beta \rightarrow \beta \rightarrow \alpha + \beta \rightarrow \alpha$, where L represents the liquid state [24]. During solidification, the primary β phase is completely solidified from the liquid, followed by the separation of the α phase in different orientations within a single β grain. Subsequent cooling induces the precipitation of the γ phase in the α phase, resulting in the formation of α₂/γ colonies. However, in the case of rapidly solidified gas-atomized powder, the cooling rate is considerably higher than the reaction rate, which impedes phase separation in the α + β phase region. In other words, the high cooling rate results in the massive transformation of the α₂ phase, suppressing the formation of the α₂/γ microstructure [20]. These discrepancies in microstructural characteristics imply that the solidification rate associated with β-solidifying γ-TiAl alloys considerably affects the microstructure development.

3.2. Microstructural Evolution after Subsequent Heat Treatment

The microstructure of the as-cast sample consisted of coarse α₂/γ colonies and an elongated β phase, consistent with the near equilibrium microstructure of β-solidifying γ-TiAl alloys, given the low cooling rate typically experienced during casting. Thus, minimal transitions were observed in the microstructure of the as-cast samples after subsequent heat treatments at 800 °C and 900 °C (Figure 4). Only the volume fraction of the γ phase and α₂/γ lamellar spacing exhibited variations. Notably, the selected temperatures were similar to those used to control the α₂/γ lamellar spacing during the subsequent heat treatment [29].

Conversely, the powders contained only β and α₂ phases because the high cooling rate suppressed the precipitation of the γ phase. Thus, heat treatment at the same temperatures was conducted to induce α₂ → γ phase transformation, aiming to return to the equilibrium microstructure. As shown in Figure 5a, the XRD peaks of the γ phase noticeably intensified, and the fraction of the γ phase sequentially increased with rising heat-treatment

temperature. Figure 5b,c show highly magnified SEM-BSE images of the powders subjected to heat treatment at 800 °C and 900 °C, respectively. The α_2 phase grains were observed in extremely thin lamellae, with varying orientations. Moreover, small β/γ cells started precipitating at the grain boundaries of the α_2/γ colonies (Figure 5b), which is due to a cellular reaction. In addition, coarse β/γ cells were observed when the temperature increased to 900 °C, and the β/γ cells tended to grow away from the grain boundaries and toward the α_2/γ lamellar colonies (Figure 5c). Unlike the cast samples, the microstructure of the powder clearly transitioned during the subsequent heat treatment, owing to rapid solidification.

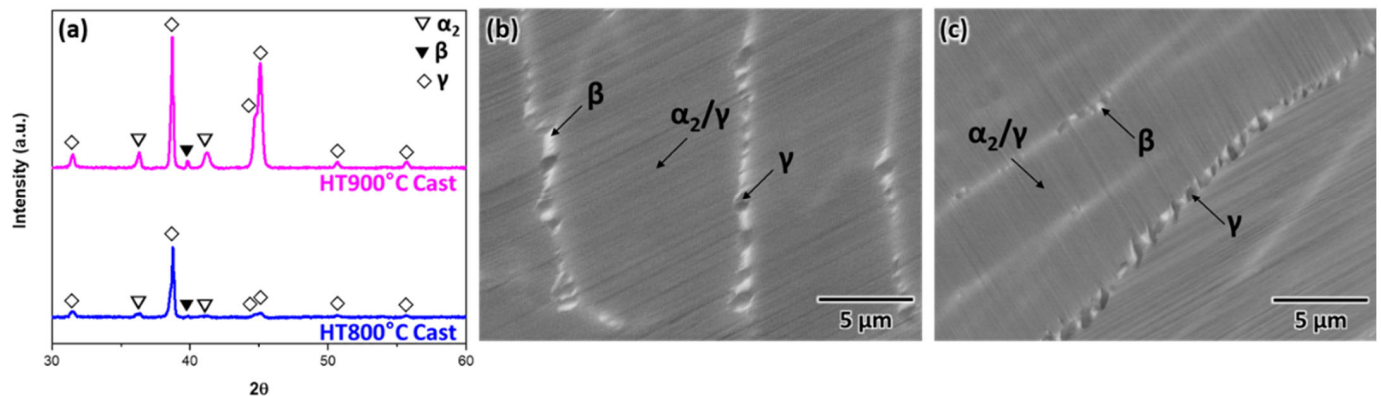


Figure 4. (a) Typical XRD patterns of the as-cast samples subjected to heat treatment and (b,c) SEM-BSE images showing the microstructure after heat treatment at 800 °C and 900 °C, respectively.

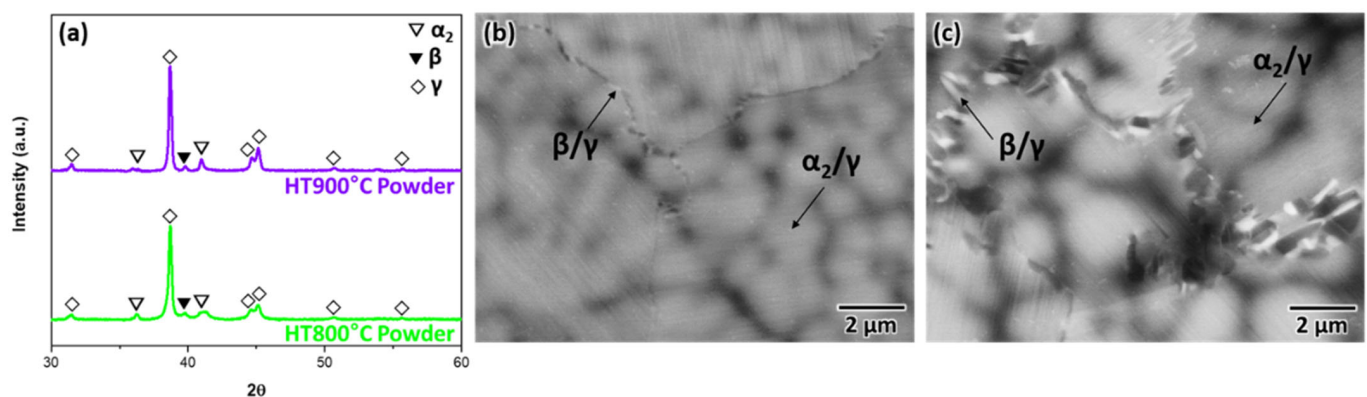


Figure 5. (a) Typical XRD patterns of the gas-atomized powder subjected to heat treatment, and (b,c) SEM-BSE images showing the microstructure post heat treatment at 800 °C and 900 °C, respectively.

TiAl alloys typically exhibit the α_2/γ lamellar microstructure. According to the mechanisms of γ phase transformation reported in a prior study [30], the development of stacking faults in the disordered α or ordered α_2 matrix induces the metastable face-centered cubic phase. Through a change in the chemical composition via atom transfer, the ordering reaction forms the final γ phase. However, the rapidly solidifying gas-atomization process causes solute supersaturation due to the high cooling rate. As shown in Tables 1 and 2, the β phase is a commonly observed phase in the casting and powder materials, but the EDS results showed a significant difference. Although the EDS analysis was carried out in the dendrite and interdendritic regions in this study, the β phase is located in the dendrite regions which is deduced from a previous report [20]. In addition, after rapid solidification or quenching and subsequent aging treatment, the induced α_2/γ lamellar microstructure transforms into an ultrafine nano-lamellar structure [22,31]. The chemical disequilibrium in the α_2 phase and high interfacial energy in the α_2/γ lamellar are the driving forces of

the cellular reaction [32]. In fact, Table 3 shows the EDS results of phases in the powder after heat treatment at 900 °C. It can be found that the chemical distribution is changed toward equilibrium, which is a similar tendency compared to casting (Table 1). Meanwhile, higher temperature promotes atomic diffusion during the subsequent heat treatment, which results in a higher volume fraction of the β/γ cells in the 900 °C than 800 °C heat treatment condition.

Additionally, SEM-EBSD observations were conducted to address the crystallographic orientation of the powder after heat treatment at 900 °C. The phase map indicated a dominant γ phase (Figure 6a), and the IPF map suggested different crystallographic orientation distributions of the grains (Figure 6b). However, the α_2/γ lamellar colony followed the Blackburn OR when considering each grain (Figure 6c). While different types of crystallographic orientation between β/γ cells and α_2/γ lamellar colonies have been reported [32,33], the first type possesses the same crystallographic orientation relationship between the β/γ cells and α_2/γ lamellar, and the lamellar interfaces are the low energy habit plane. Second, the β/γ cells are perpendicular to α_2/γ lamellar, with a high energy faceted lamellar interface. Third, the β/γ cells and α_2/γ lamellar have different crystallographic orientations, but they form a low-energy habit plane as their interface. Thus, the β/γ cells can represent the same crystallographic orientation with the α_2/γ lamellar interfaces or not. The mechanical properties of γ -TiAl alloys are sensitive to changes in phase constitution and/or α_2/γ lamellar microstructure. The appropriate volume fraction of the β/γ cells can enhance the mechanical properties due to the twinning-induced plasticity effect [34], and ultrafine α_2/γ lamellar also contribute to increased hardness [35]. Therefore, the observed β/γ cells and ultrafine α_2/γ lamellar microstructure after rapid solidification in this study may have the potential to improve the mechanical properties of γ -TiAl alloys.

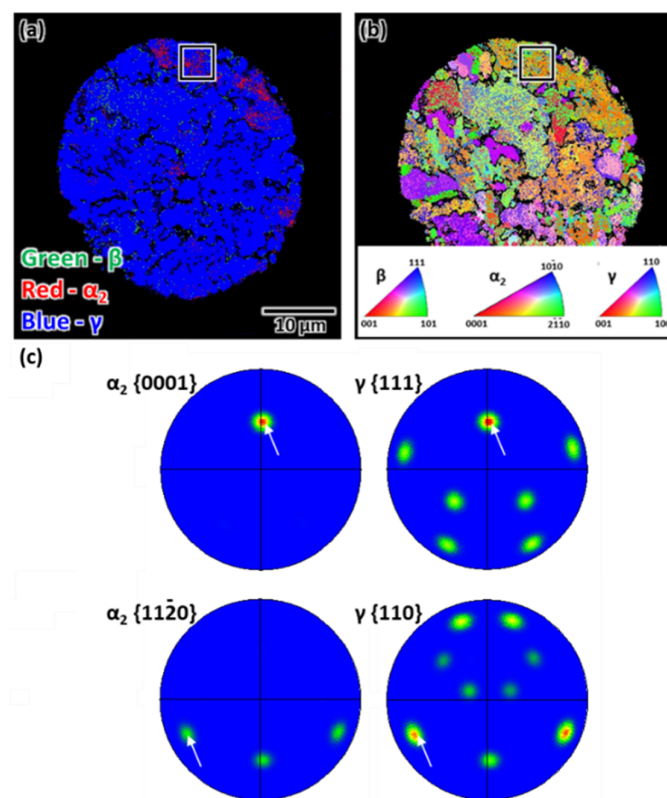


Figure 6. SEM-EBSD analysis of the powder subjected to heat treatment at 900 °C: (a) phase map, (b) IPF map, and (c) PFs. The PFs obtained from the black rectangular area indicate compliance of the Blackburn OR between the α_2 and γ phases as shown with white arrows indicating the (α_2 and γ) phases are aligned according to the Blackburn OR.

Table 3. The EDS results of different phases in gas-atomized powder after the subsequent heat treatment at 900 °C.

Constituent	Composition (at.%)			
	Ti	Al	Nb	Cr
β	51.75 $\pm$ 0.28	39.93 $\pm$ 0.42	6.80 $\pm$ 0.21	1.52 $\pm$ 0.09
α_2/γ lamellae	50.77 $\pm$ 0.37	41.95 $\pm$ 0.37	6.08 $\pm$ 0.08	1.20 $\pm$ 0.07
γ	50.31 $\pm$ 0.21	42.78 $\pm$ 0.20	5.76 $\pm$ 0.09	1.15 $\pm$ 0.10

Lastly, understanding how this microstructure can be achieved on a bulk scale is crucial. The AM process, characterized by rapid solidification and repeated thermal cycling, can enable unique microstructure control. Additionally, this process affords control over the crystallographic orientation [36,37] and can potentially be used to regulate the α_2/γ lamellar orientation [38], which can significantly improve the mechanical properties. Overall, this study suggests that preheating and/or subsequent heat treatment at high temperatures during AM can yield precipitation of fine β/γ cell microstructures. However, appropriate temperature control is crucial to avoid cellular reaction and control the orientation of the α_2/γ lamellar structure through crystallographic texture formation.

4. Conclusions

This study was aimed at providing a comprehensive understanding of the microstructure evolution of metastable gas-atomized Ti-44Al-6Nb-1.2Cr (at.%), especially during the transition from the metastable to equilibrium state, in comparison with cast materials. The following conclusions can be drawn:

1. The cast material with a relatively low solidification rate demonstrated α_2/γ colonies surrounded by the β phase. In contrast, the powder consisted of a small amount of the β phase and predominant massive α_2 phases with high chemical disequilibrium owing to the high cooling rate.
2. The β phase and massive α_2 phases have no specific crystallographic orientation relationship with each other.
3. After subsequent heat treatment, there were no distinguishing differences in the cast materials; only the volume fraction of the γ phase was changed. However, ultrafine α_2/γ lamellar microstructure emerged from the α_2 phase, and β/γ cell microstructures were formed at α_2/γ lamellar grain boundaries via cellular reaction.

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Data Availability Statement: The data supporting the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest: The authors declare no conflict of interest.

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