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To cite this article: Xinyu Zhang, Chuanwei Li, Qing Wang, Mengyao Zheng, Zhenhua Ye & Jianfeng Gu (2020) Unusual ordered phases with long periodic stacking structures in an additively manufactured TiAl alloy, Materials Research Letters, 8:12, 454-461, DOI: 10.1080/21663831.2020.1804473

To link to this article: <https://doi.org/10.1080/21663831.2020.1804473>



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Published online: 10 Aug 2020.



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REPORT



Unusual ordered phases with long periodic stacking structures in an additively manufactured TiAl alloy

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ABSTRACT

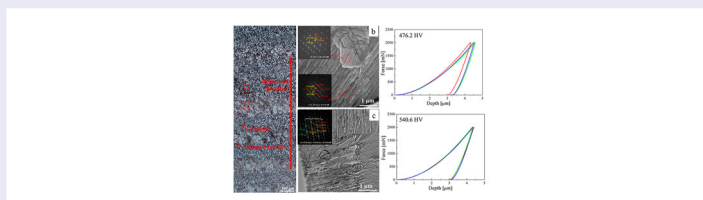
In the present work, we have extensively examined the alternative-band microstructure of a Ti-47Al-2Cr-2Nb alloy fabricated via direct laser deposition by means of high resolution transmission electron microscopy and selected area electron diffraction. The studied samples exhibit two types of long-periodic stacking ordered phase with 6H and 18R structures, which were never reported for those obtained by traditional approaches. Moreover, the ordered phases possess stacking sequences of ABCBACA and ABCABCBACACABCABA, being analogous to the periodically arranged stacking faults in α_2 and γ phases, respectively. With the presence of these unusual structures, the as-deposited sample tends to have superior mechanical properties.

ARTICLE HISTORY

Received 31 May 2020

KEYWORDS

TiAl alloy; direct laser deposition; long-period stacking ordered phase; mechanical property



IMPACT STATEMENT

Unusual ordered phases with long periodic stacking structures, seldom reported in the TiAl alloys obtained by traditional approaches, were comprehensively characterized, and demonstrated to possess excellent mechanical properties.

1. Introduction

Recently, intermetallic γ -TiAl based alloys have gained immense attention for aeroengine applications owing to their light weight and excellent high-temperature properties [1]. However, their high cracking sensitivity and large flow stress at elevated temperatures heavily restrict their production of complex structural components [2]. Additive manufacturing (AM) is an efficient approach for fabricating metallic components with high design freedom [3]. Specimens produced by AM usually exhibit grain-refined microstructures in a deviation from the equilibrium state resulting from the complex thermal cycles of numerous re-heating and re-cooling processes [4].

Consequently, AMed metallic materials tend to achieve outstanding mechanical properties for engineering application [5,6]. Researches on AMed TiAl alloys have been conducted, and their microstructures and mechanical properties appear to vary greatly with the processing parameters [7–10]. In our previous studies, we prepared a Ti-47Al-2Cr-2Nb (at.%) alloy via direct laser deposition (DLD) and discussed the corresponding microstructural formation mechanism [11]. Additionally, an unusual long periodic stacking ordered (LPSO) phase with 18R structure, which was never reported in TiAl alloys, was firstly observed in the alternative-band microstructure [12].

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Supplemental data for this article can be accessed here. <https://doi.org/10.1080/21663831.2020.1804473>

LPSO phases have been widely researched in Mg alloys, and they usually present various types of structures depending on the chemical compositions and processing craft [13–15]. These LPSO phases are demonstrated to not only heavily interact with the deformation twins [16,17], but also effectively increase the critical resolved shear stress of the basal plane [18]. Thus, the existence of LPSO phases would significantly improve comprehensive properties of Mg alloys, especially the strength and creep resistance at high temperatures [19,20]. These enhancements are attractive, especially for TiAl alloys. But due to the different crystal structures between Mg and TiAl alloys, the LPSO phases in TiAl alloys might appear highly different, and very few reports involved LPSO phases in TiAl alloys till now, which contributes to a great information absence for

them. Thus, their detailed structures and the influences on mechanical properties still remain to be a question.

Motivated by this, the current work will specially explore the LPSO phases generated in AMed TiAl alloys, and comprehensively characterize these structures by high resolution transmission electron microscopy (HR-TEM) and selected area electron diffraction (SAED). Moreover, the mechanical property of the as-deposited samples containing the LPSO phases are preliminarily tested.

2. Experimental

Ti-47Al-2Cr-2Nb pre-alloyed powders with a diameter of 53–105 μm were dried in air at 50°C. Then, single-track walls were deposited on a Ti-48Al plate using the

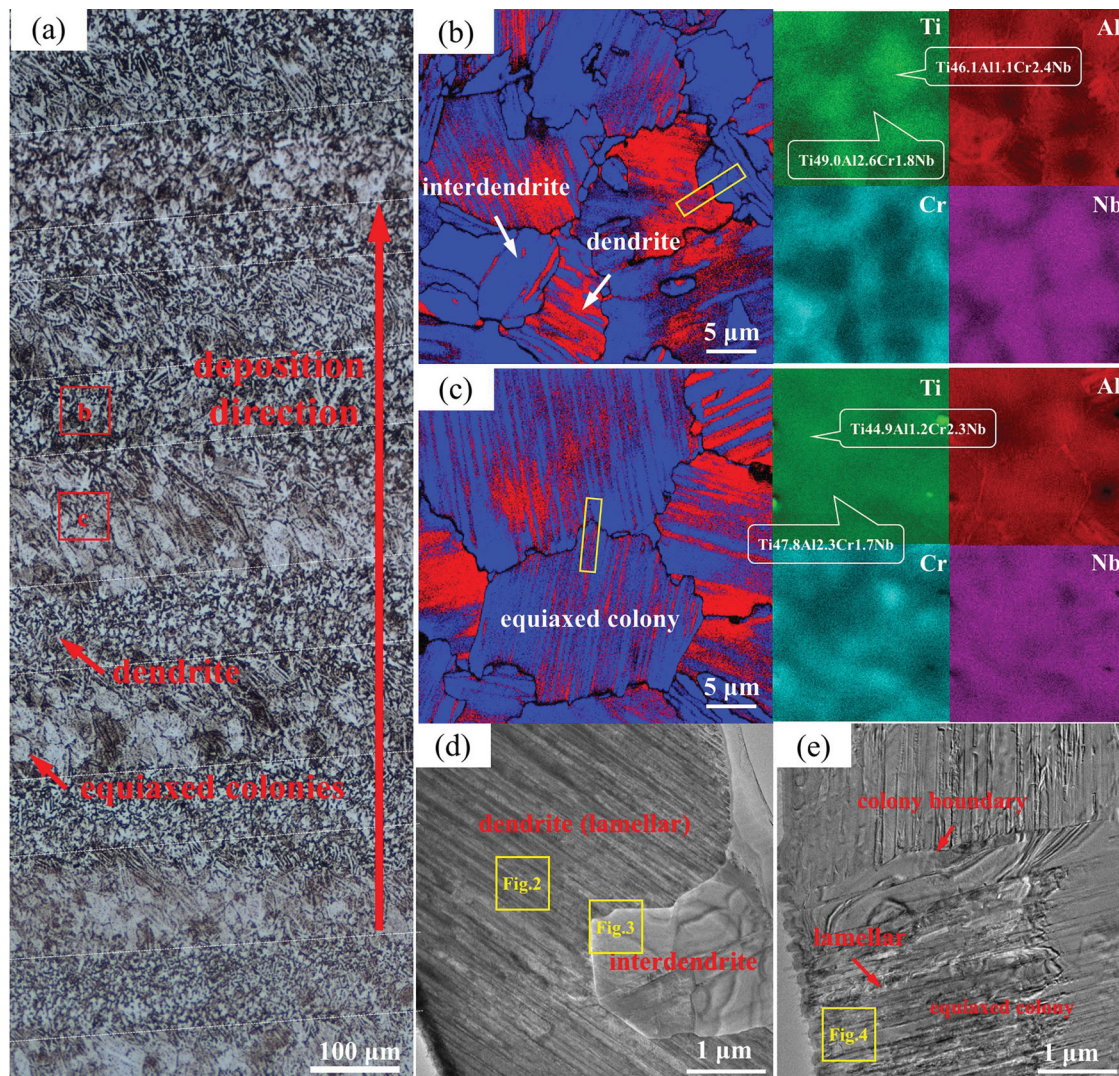


Figure 1. (a) Low-magnification OM image of the as-deposited specimen consisting of alternating dendritic and equiaxed-colony bands. (b, c) Phase maps of the dendritic and equiaxed-colony bands. The red and blue regions represent α_2 and γ , respectively. The right images show the corresponding chemical distribution maps. The yellow rectangles are the locations of the FIB slices. (d, e) Low-magnification TEM images of the dendritic and equiaxed-colony bands.

back-and-forth scanning strategy. The microstructure was characterized by optical microscopy (OM) (Axio Observer A1m, Zeiss, Germany), scanning electron microscopy (SEM) (LYRA3 GMU, TESCAN, Czech), electron backscattered diffraction (EBSD), TEM (TALO SF200X, FEI, America), and scanning transmission electron microscopy (STEM). The resolution of the STEM can achieve 0.16 nm. The chemical compositions were analyzed using super-X energy dispersive X-ray spectroscopy (EDS) system with four silicon drift detectors. The acceleration voltage was 200 kV, the spot size was set as 7, and the acquisition time was 5 min. The focused-ion beam (FIB) technique was used to obtain slices at the selected zones. The hardness of the specimens was determined using nano-indentation (HM2000, FISCHER, Germany).

3. Results and discussion

The as-deposited specimen exhibited an alternative-band microstructure composed of alternately arranged dendritic bands and equiaxed-colony bands along deposition direction (Figure 1(a)). The dendritic grains in the dendritic bands were only several micrometers, while the equiaxed colonies showed a much larger size, about 20–40 μm . Moreover, the dendritic zones, which mainly consisted of lamellar α_2 phase, were rich in Ti and Nb, the interdendritic zones were identified to be irregular γ phase, and Al and Cr preferentially accumulated in those zones (Figure 1(b)). The equiaxed-colony bands showed regular polygonal colonies filled with α_2/γ laths. In addition, the chemical distribution of the equiaxed colony bands was much more homogeneous than that of the dendritic bands (Figure 1(c)). The TEM slices extracted from the two bands shows that the dendritic band was constituted by the lamellar structure and irregular grains, while the equiaxed-colony bands merely consisted of lamellar structure, which is consistent with the EBSD observations. The comprehensive characterizations were conducted to present the detailed microstructures.

Figure 2(a) shows the bright field (BF) image of the lamellar colony in the dendritic band. These colony is filled with straight nanolaths, and the laths width is less than 50 nm. The corresponding SAED pattern taken along $\langle 11\bar{2}0 \rangle_{\alpha_2}$ shows a composite diffraction pattern (Figure 2(b)). The diffraction pattern of the bright spots corresponds to α_2 phase. Moreover, five weak diffraction spots were observed at $1/6(0002)_{\alpha_2}$, $2/6(0002)_{\alpha_2}$, $3/6(0002)_{\alpha_2}$, $4/6(0002)_{\alpha_2}$, and $5/6(0002)_{\alpha_2}$. This diffraction pattern is significantly different from that of the α_2/γ lamellar structure observed in TiAl alloys, and it seems to be attributed from the formation of LPSO phase. Earlier before, the periodic diffraction spots were sometimes

observed in heavily deformed TiAl alloys [21,22], but the controversy on the formation of LPSO phase structures in TiAl alloys exists, because the overlapping of α_2/γ laths may generate similar features. In our experiment, the specimen was tilted to ensure the incident beam along $\langle 11\bar{2}0 \rangle_{\alpha_2}$ direction, and the lamellar interfaces were exactly edge-on, which impossibly causes large areas of overlapping of α_2 and γ phase. In the dark field (DF) images, the laths attributed to the yellow circle are 80 nm, and both of them extend through the whole specimen, which further means the additional spots is not caused by the overlapping of α_2 and γ phase. Thus, the closely packed diffraction spots are considered to result from the LPSO phases [13–17,19,20]. In addition, the six equal divisions formed by the extra diffraction spots and the vertical relationship between (100) and (001) indicate that the LPSO phase corresponds to a 6H structure. Actually, this LPSO phase with 6H structure was never reported in the AMed TiAl alloys before, and they can coexist with the α_2 phase in form of $\alpha_2/6\text{H}$ laths. The α_2 and 6H laths showed the orientation relationships of $\langle 11\bar{2}0 \rangle_{\alpha_2} \parallel \langle 010 \rangle_{6\text{H}}$ and $(0002)_{\alpha_2} \parallel (006)_{6\text{H}}$, respectively. The magnified high-angle annular dark-field (HAADF) image and its chemical mapping analysis are presented (Figure 2(e,f)). It shows that the 6H laths are rich in Al and Nb, and the average chemical compositions of the 6H and α_2 laths were Ti-52.5Al-0.7Cr-2.5Nb and Ti-42.6Al-1.5Cr-2.2Nb, respectively. Moreover, alternating bright and dark stripes were observed in 6H laths. As the HAADF image usually reveals the atomic-number (Z) contrast [23,24], these stripes indicate that chemical clustering might occur in the 6H laths. These chemical clustering cannot be observed in the EDS mapping because its low spatial resolution, and the low content of Cr and Nb also restrict it distinguish this chemical clustering. To further identify this LPSO phase and its stacking sequence, HR-TEM was conducted, and broad strips were also observed in the 6H laths, and the spacing between the neighboring stripes was 1.40 nm, about six layers of the $(0002)_{\alpha_2}$ planes. Moreover, faint spot lines marked by hollow arrows were observed. These lines were parallel to $\mathbf{g}(001)_{6\text{H}}$ and passed through $\pm(100)_{6\text{H}}$ in the fast Fourier transform (FFT) images of the 6H laths (Figure 2(b)). This implies the atomic configurations in the closely packed planes are ordered [25,26]. Figure 2(h,i) show the stacking sequences of the 6H structure and α_2 phase, respectively. The stacking sequence of the 6H structure was ABCBACA, while that of the α_2 phase was ABA. It should be noted that the stacking sequence of the 6H structure could be obtained from the α_2 phase by introducing stacking faults every three layers, which is also different from the simulated images caused by the overlap of α_2 and γ laths [22].

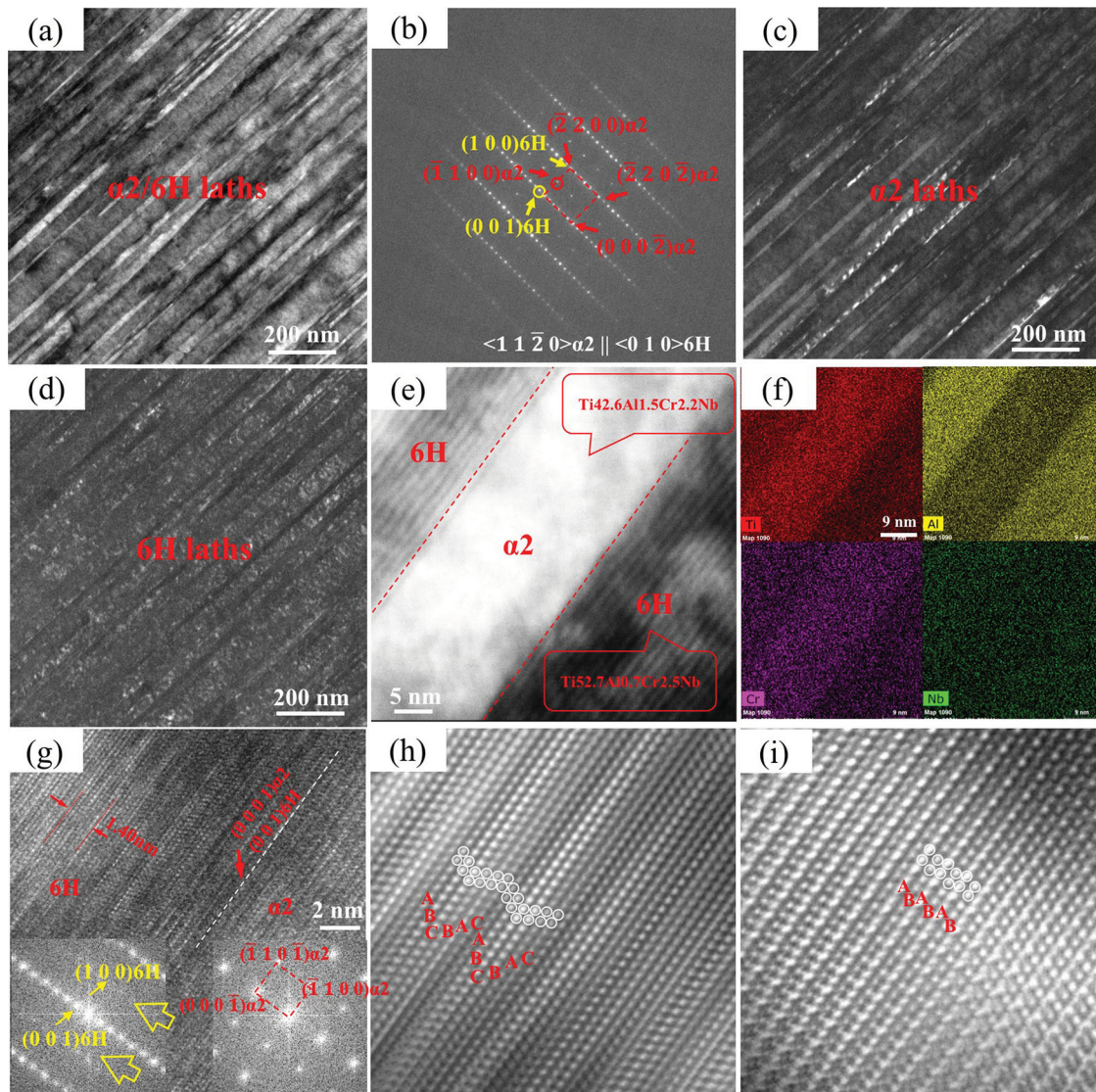


Figure 2. (a, b) BF image of the dendritic zone and the corresponding SAED pattern along the $\langle 11\bar{2}0 \rangle_{\alpha_2}$ direction. (c, d) DF images of the α_2 and 6H laths denoted by the red- and yellow-circled spots, respectively, in (b). (e, f) HAADF-STEM image of the neighboring α_2 and 6H laths and their chemical distribution maps. (g) HR-TEM image of the neighboring α_2 and 6H laths and their FFT images. (h, i) IFFT images revealing the stacking sequences of the 6H structure and α_2 phase.

Additionally, the LPSO phase with 18R structure was observed in the interdendritic zones. A composite diffraction pattern was also observed in the interdendritic grain (Figure 3(b)). The bright spots correspond to the diffraction pattern of the γ phase with the incident beam exactly along the $\langle 11\bar{0} \rangle_{\gamma}$ direction. Meanwhile, the additional diffraction spots observed along the $\mathbf{g}(111)_{\gamma}$ and $\mathbf{g}(11\bar{1})_{\gamma}$ directions indicate the generation of the LPSO phase. The six equal divisions along the $\mathbf{g}(111)_{\gamma}$ and $\mathbf{g}(11\bar{1})_{\gamma}$ directions and the 87.5° angle between (003) and (101) indicate that two 18R structures with different orientations coexisted with the γ phase in the interdendritic grains [27]. The orientation relationships were $\langle 11\bar{0} \rangle_{\gamma} \parallel \langle 010 \rangle_{18R}$ and $(111)_{\gamma} \parallel (00.18)_{18R}$. This was also confirmed by the

HAADF and HR-TEM images (Figure 3(d,f)), which show stripes with two different orientations. The compositions of the γ phase and 18R structure are shown in Figure 3(d,e), respectively. No significant difference was observed in the compositions. The spacing between the neighboring stripes was measured to be about 1.42 nm, identical to the spacing between every six closely packed $\{111\}$ planes in the γ phase. The stacking sequence of the 18R structure was ABCABCBCABCACABCABA, while that of the adjacent γ phase was ABCA, as shown in Figure 3(g,h), respectively. The stacking sequence of the 18R structure was essentially obtained by inserting three intrinsic stacking faults in the γ phase. Thus, three blocks: ABCABC, BCABCA, and CAB CAB were obtained in a complete 18R stacking sequence. Hence,

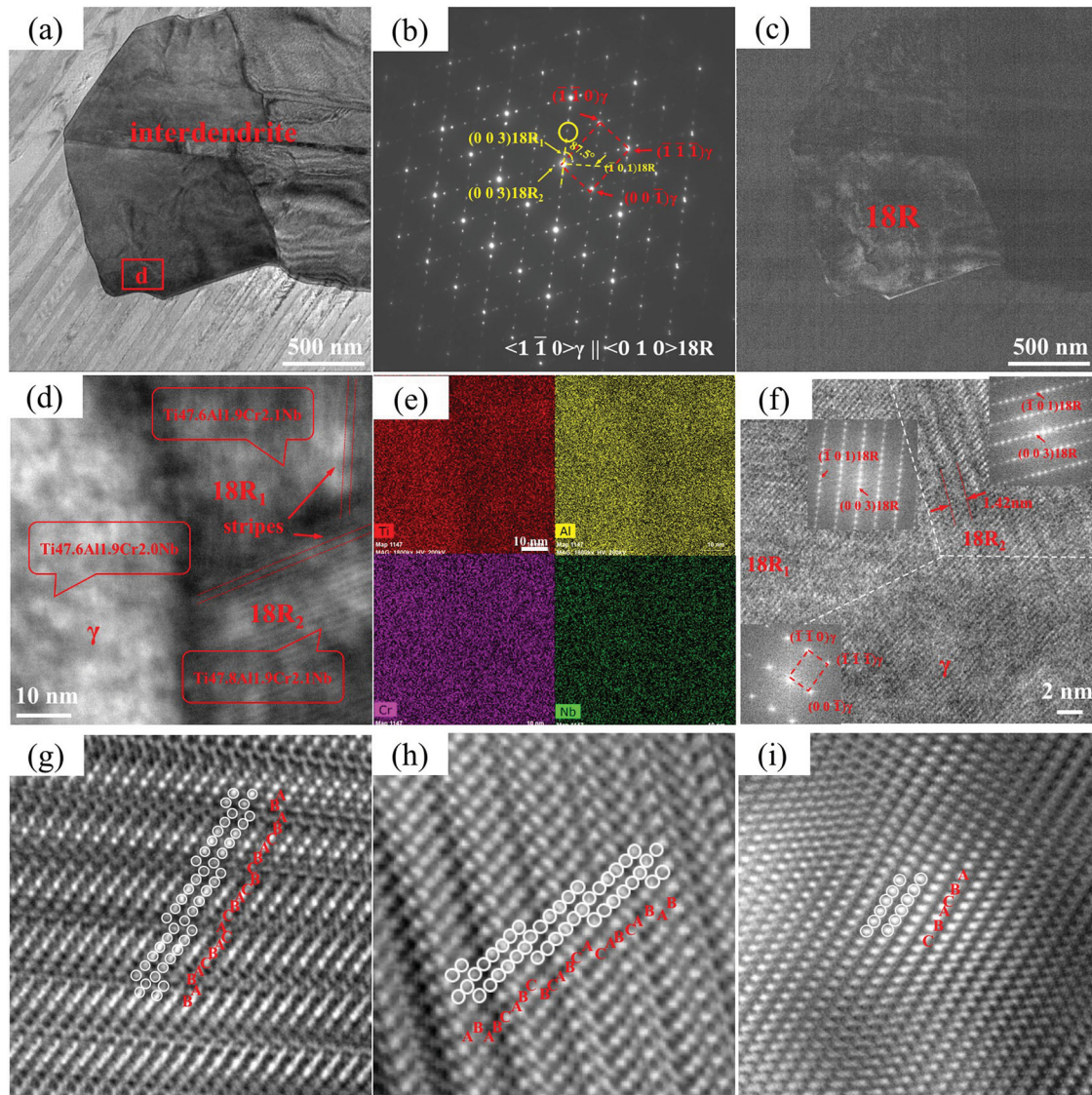


Figure 3. (a, b) BF image of the interdendritic γ phase and the corresponding SAED pattern along the $\langle 1\bar{1}0 \rangle_{\gamma}$ direction. (c) DF image of the 18R structure denoted by the yellow-circled spot in (b). (d, e) HAADF-STEM image of the 18R structure and γ phase and their chemical distribution maps. (f) HR-TEM image of the 18R structure and γ phase. (g, i) IFFT images revealing the stacking sequences of the 18R structures and γ phase.

it can be stated that the periodic occurrence of stacking faults every six closely packed planes resulted in the formation of the 18R structure. In addition, since the stacking faults could be activated on all the four $\{111\}$ planes, 18R structures with different orientations were generated in each γ grain.

The 6H structure was also observed in the equiaxed colony bands, as shown in Figure 4. It should be noted that the lamellar structure was the main feature of the equiaxed colony bands (Figure 1(e)). The laths were measured to be about 93 nm width. The corresponding SAED pattern revealed that the α_2 phase, γ phase, and 6H structure coexisted in the lamellar structure (Figure 4(b)).

The three phases showed the following orientation relationships: $\langle 010 \rangle_{6H} \parallel \langle 11\bar{2}0 \rangle_{\alpha_2} \parallel \langle 110 \rangle_{\gamma}$ and $(006)_{6H} \parallel (0002)_{\alpha_2} \parallel (111)_{\gamma}$. Although the spacing between the neighboring stripes was 1.4 nm (Figure 4(c)), the diffraction pattern was a little different from that in Figure 2(g). The spot lines, denoted by the hollow arrows, became bright and sharp, indicating that the closely packed planes of the 6H structure showed a higher ordering degree compared with that in dendritic band [26]. In addition, the chemical compositions of the α_2 , γ , and 6H laths were measured as Ti-37.9Al-2.1Cr-2.2Nb, Ti-45.4Al-1.2Cr-3.7Nb, and Ti-44.1Al-1.4Cr-2.8Nb, respectively. It is notable that the composition of the 6H laths

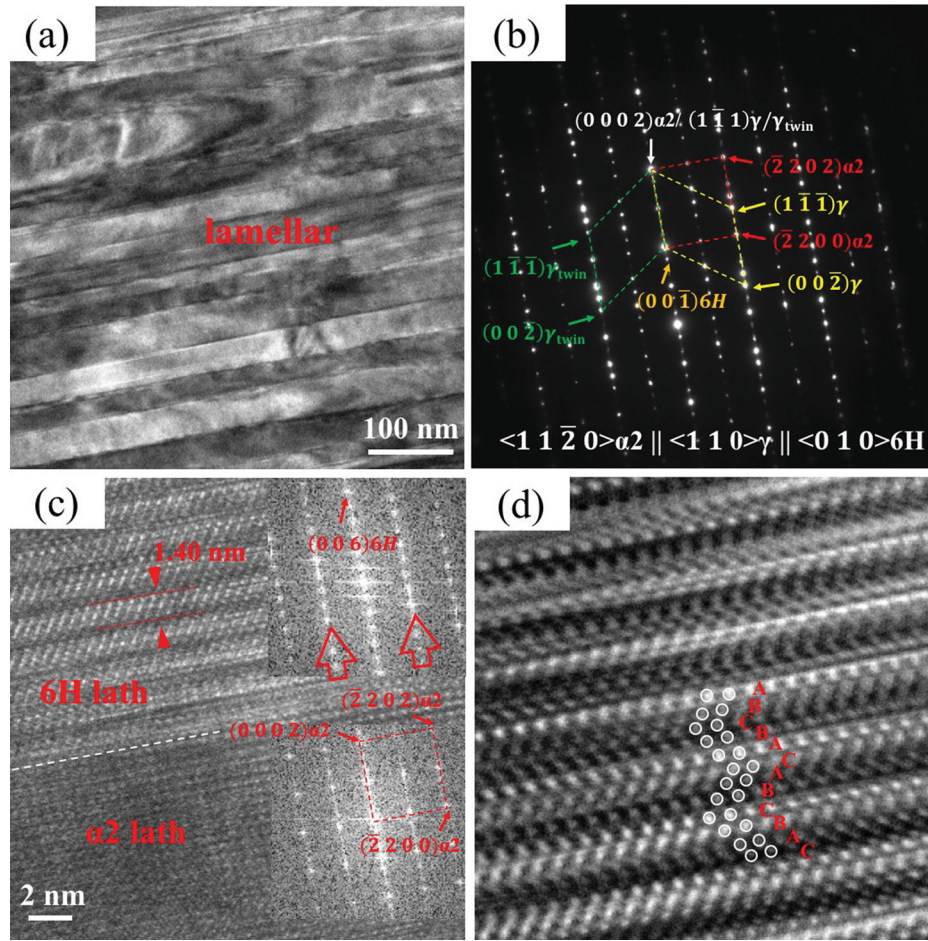


Figure 4. (a, b) BF image of the equiaxed colony and the corresponding SAED pattern. (c) HR-TEM image of the neighboring 6H and α_2 laths. (d) IFFT image revealing the stacking sequence of the 6H structure.

was intermediate between the α_2 and γ laths. Its stacking sequence structure was ABCBACA, identical to the 6H structure observed in the dendritic zones.

Based on the analysis above, the atomic configuration of the 6H and 18R structures were established, and their simulated diffraction patterns are present in Figures S2 and S3. It is observed that both their simulated $<1\bar{1}0>$ diffraction patterns are in accordance with the experimental ones (Figure S1), and the arrangement of the closely stacking spots in the simulated $<010>$ diffraction patterns were observed to be similar to the experimental ones, but it is notable that some extinct spots, especially the $(00l)$ spots, appear in the experimental ones. The occurrence of the extinct spots is attributed to the double diffraction caused by the dynamics of electron diffraction. Thus, both the observations and analysis demonstrate that different types of LPSO phases were generated in the alternative-band microstructure produced by the DLD process, and the two LPSO phases could be obtained from the α_2 and γ phases by introducing periodic stacking faults.

Metzbow [27,28] has proposed an empirical relationship between the stacking-fault possibility (SFP), α , and Al concentration, N_{Al} , for Ti-Al binary systems with hexagonal close-packed structure:

$$\alpha = 0.003 \exp(0.133 N_{Al}) \quad (1)$$

The SFP increases with Al concentration increasing, indicating that stacking faults tend to be activated in alloys with high Al concentrations. We assumed that the equilibrium Al concentration of α_2 phase was only about 32 at.% at room temperature, but as-deposited α_2 phase shows highly supersaturated. This indicates the stacking fault tendency improved significantly and the formation of the 6H structure was favored. Additionally, the accumulation of Cr in the interdendritic zones significantly decreased the stacking fault energy of (SFE) the γ phase [29], contributing to the occurrence of stacking faults in γ grains. Thus, it is possible for these supersaturated α_2 and γ phases to transform into the LPSO phases during the thermal cycling of the DLD process. The 6H structures with different ordering degrees can be attributed

to the difference in the chemical compositions of the two bands induced by their different thermal histories [11]. The coexistence of the α_2 , γ , and 6H laths and the intermediate composition of the 6H structure indicate that the 6H structure might be an intermediate structure obtained during the $\alpha_2 \rightarrow \gamma$ transformation. Moreover, the mechanical properties of the microstructure containing these unusual ordered phases were preliminary tested, which present a considerable improvement compared with the as-casted alloys, as is shown in Figure S4. However, the detailed formation and strengthening mechanism of these LPSO phases deserve to be explored in further studies.

4. Conclusions

In summary, the LPSO phases of the 6H and 18R ordered structures were identified by HR-TEM and SAED in the as-deposited TiAl specimen, showing alternating dendritic and equiaxed colony bands. The 6H ordered structures with a stacking sequence of ABCBACA are present in both the dendritic zones and equiaxed colonies, their ordering degrees are, however, significantly different. For the 18R ordered structure, its stacking sequence of ABCABCBCABCACABCABA is only found in the interdendritic γ phase. Consequently, the formation of the LPSO phases could be rationalized as a result of the SFE reduction and cyclic thermal history.

Acknowledgments

The authors acknowledge financial support from the National Natural Science Foundation of China (grant number 51971145, No.51801126).

Disclosure statement

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

Funding

This work was supported by National Natural Science Foundation of China [grant numbers 51971145, 51801126].

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