Effect of substrate preheating on the texture, phase and nanohardness of a Ti–45Al–2Cr–5Nb alloy processed by selective laser melting

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ABSTRACT

The crystallographic texture, phase composition and evolution, and nanohardness of a Ti–45Al–2Cr–5Nb alloy processed by selective laser melting (SLM) at various substrate preheating temperatures were investigated. The α2 phase decreases whereas the γ and B2 phases increase with increasing preheating temperature, and the relationship in orientation between B2, α2 and γ phases is described as follows: (1120) α2// (111) B2// (111) B2. The SLM-processed TiAl alloy shows much higher nanohardness than its traditional casting counterpart, which increases with the increase of preheating temperature. The findings would be a valuable reference for fabricating TiAl components with acceptable texture, phase compositions and nanohardness by SLM.

TiAl-based alloys are considered as extremely promising candidates for aerospace and aircraft applications due to their extremely high specific stiffness, hardness and yield strength and excellent creep resistance at elevated temperature [1–5]. Their inherent low room temperature ductility and poor hot deformability, however, greatly hinder the production of TiAl-based alloy components for the practical applications [6]. Recently, some special manufacturing techniques such as isothermal forging and hot extrusion have been developed to process TiAl-based alloys and improve the ductility by means of microstructure refinement and phase transformation [7]. However, these thermomechanically fabricated components show extremely high costs, heterogeneous microstructure and limited structural complexity [6].

Selective laser melting (SLM) is capable of fabricating near-fully dense and complex metal parts directly from computer-aided design (CAD) models [8–11], therefore showing great potential for fabricating TiAl alloy components with acceptable quality and costs and complex geometries. Recently, some preliminary investigations have been carried out to find the optimal SLM process window for TiAl alloys. Loeb et al. found the existence of cracks and pores in the TiAl samples mainly because the SLM processing parameters were not properly optimized [12]. Löber et al. adopted single track experiments to optimize the SLM processing parameters for a TiAl alloy, and near-full dense TiAl parts could be achieved [13]. Gussone et al. investigated the effect of SLM processing parameters on the chemical composition and tensile strength of TiAl alloys [14]. However, to the best of the authors’ knowledge, few previous research works studied the grain orientation, crystallographic texture and phase composition of TiAl alloys processed by SLM, and their effects on the mechanical properties of SLM parts. It has been demonstrated that the substrate preheating in the SLM process has an important role in controlling the microstructures and texture of SLM-processed metals [14]. Therefore, this work concentrates on investigating the influence of substrate preheating temperature on the crystallographic texture, phase transition and evolution, and mechanical properties (nanohardness) of a TiAl alloy, Ti–45Al–2Cr–5Nb, processed by SLM.

A Ti–45Al–2Cr–5Nb (at.%) alloy powder with an average particle size (D50) of 27.6 μm was supplied by Beijing Institute of Aeronautical Materials (BIAM, China). Prior to SLM, the TiAl powder was dried in air at 50 °C for 24 h and sieved (200 mesh) to reduce the aggregated particles and thus improve its flowability. An HRPM-II type SLM machine with a 400 W single-mode ytterbium fiber laser (SP-400C, SPI Lasers, America) was used, and the wavelength and laser beam diameter (Dbeam) of the laser source were 1064 ± 10 nm and 100 μm, respectively. The SLM process was conducted in a high purity argon atmosphere (99.99%) to avoid the pick-up of reactive oxygen. Based on a series of preliminary experiments, the SLM parameters were optimized as follows: the laser power (P) = 200 W, laser scan speed (V) = 400 mm/s, scan line hatch spacing (h) = 100 μm, and powder layer thickness (d) = 30 μm. Four different levels of substrate preheating temperature, namely 298 K (room temperature), 423 K, 523 K and 623 K were selected in the SLM experiments and the corresponding obtained SLM samples were denoted by T0, T1, T2 and T3, respectively.

X-ray diffraction (XRD) measurements were performed on a XRD-7000S instrument (Shimadzu, Japan) with a Cu tube at 40 kV and 30 mA. The scattering angular (2θ) varied from 20° to 110° with a

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scan rate of 10°/min. The specimens for electron backscattered diffraction (EBSD) examinations were electrolytically polished on LectroPol-5 (Struers, Denmark) at 25 V for 20 s. EBSD was performed on the HKL Nordlys orientation imaging microscope system (Oxford, Oxford Instruments, UK) mounted on a JSM-7600F (JEOL, Japan) scanning electron microscope. Transmission electron microscope (TEM) and high resolution transmission electron microscope (HRTEM) measurements were executed on a JEOL-2100 type machine (JEOL, Japan). Nanoindentation tests were performed using a high-precision nanohardness scratch tester (TI750, Hysitron, American) with the test force of 3500 mN and hold time of 2 s.

Fig. 1(a) illustrates the scanning strategy of the SLM process. The arrows indicate the movement of the laser and 90° rotation angle of the scanning direction between two consecutive layers N and N + 1. The inverse pole figure (IPF) is shown in Fig. 1(b), representing the relationship between colors in the EBSD images and crystal orientations of the SLM-processed samples. The EBSD orientation image map and grain boundary misorientation angles of the samples T0, T1, T2 and T3 from the top view are illustrated in Fig. 1(c), (d), (e) and (f), respectively. The applied substrate preheating significantly influences the grain size and orientation of the SLM-processed Ti–45Al–2Cr–5Nb alloy. As the SLM process was carried out at room temperature (298 K) without substrate preheating, the average grain size of T0 was calculated to be 8.3 μm, and the majority of the grains have a green-blue color as shown in Fig. 1(c). The grains at (10T1) and (1121) orientations are present along the laser scanning direction [11]. When the SLM process was conducted at a relatively low substrate preheating temperature of 423 K, the grains generally grew up with an average grain size of 10.2 μm. Also, it can be easily found that the red areas increase while the green regions decrease in the EBSD map in Fig. 1(d) compared with that in Fig. 1(c), indicating that the (0001) orientation is strengthened whereas the (10T1) orientation is weakened on the top view of sample T1. When increasing preheating temperature to 523 K, the grains size further increases and the average size reaches 12.8 μm. Moreover, the EBSD orientation map is dominated by reddish color and the green-blue regions are further reduced, as shown in Fig. 1(e). This illustrates that the gains of T2 reveal a strong (0001) orientation. At an even higher preheating temperature of 623 K, the average grain size attains the maximum of 15.2 μm. But, the EBSD map in Fig. 1(f) shows decreased reddish areas but increased green-blue regions compared with that in Fig. 1(e). It is worthy noted that the areas of the three colors basically remain the same with each other, showing that the grains at (0001), (10T1) and (1121) orientations are equally existing in T3. The fineness of the grains is determined by thermal gradient (G), the solidification rate (R) and cooling rate (T = G × R) [15]. The higher the cooling rate of the material, the finer the microstructure. The SLM process is able to generate an extremely high cooling rates (10⁶ K/s), therefore resulting in a very fine microstructure of the SLM-processed Ti–45Al–2Cr–5Nb. But, the substrate preheating has a negative effect on the cooling rate. With increasing preheating

Fig. 1. Scanning strategy of the SLM process (a), the crystal orientation-color relation map referred to as the inverse pole figure (IPF) (b). EBSD orientation maps obtained from the top view of the SLM-processed Ti–45Al–2Cr–5Nb samples of T0, T1, T2 and T3, which are represented in (c), (d), (e) and (f), respectively.
temperature, the cooling rate decreases, thus leading to growth of the grain size. Consequently, varying substrate preheating temperature can tailor the grains size and orientation of the SLM-processed Ti–45Al–2Cr–5Nb alloy.

The grain boundary misorientation angles of T0, T1, T2 and T3 were also determined through EBSD analysis and divided by low-angle grain boundaries (LAGBs, 2°–15°) and high-angle grain boundaries (HAGBs, >15°). Obviously, the samples T0, T1, T2 and T3 are dominated by HAGBs with the contents of 88.2%, 91.7%, 92.8% and 93.9%, respectively. It is well known that SLM creates parts by layer-wise addition of materials. Due to the high energy of laser beam, when a new layer is created, the previous layer(s) will be partially or completely remelted. This phenomenon leads to recrystallization and then the HAGBs will be aroused. In addition, the content of HAGBs increases with increasing substrate preheating temperature. This is mainly attributed to the fact that the higher preheating temperature brings about a relatively longer duration time in the remelting state and thus prolongs the recrystallization time.

As shown in Fig. 1, there are big differences in the grain orientations between the SLM samples at the different preheating temperatures. Therefore, substrate preheating has a significant influence on the local crystallographic texture of the SLM-processed Ti–45Al–2Cr–5Nb. The most common [0001] pole figures (PF) from the top view for the SLM-processed Ti–45Al–2Cr–5Nb samples, T0, T1, T2 and T3, are represented in Fig. 2(a), (b), (c) and (d), respectively. In the sample fabricated without substrate preheating (T0), a weaker [0001] texture along the scanning direction occurs, as shown in the PF of Fig. 2(a). The low intensities in the PF suggest the presence of a fiber texture [11]. The fiber texture intensity can be described by the texture index, which is calculated from the orientation distribution function (ODF) \( f(g) \) using Eq. (1):

\[
\text{Texture Index} = TI = \int_{\text{eulerspace}} (f(g))^2 \, dg
\]  

where \( f \) is the orientation distribution as a function of the Euler space coordinates \( g \). The texture index of sample T0 was calculated to be 12.54. For isotropic materials, the texture index equals to unity [16]. The texture indexes of T1, T2 and T3 were determined to be 12.96, 20.00 and 13.68, respectively. The texture index results of T0, T1, T2 and T3 are in a good agreement with the EBSD orientation maps shown in Fig. 1(c)–(f). In order to further investigate the effect of substrate preheating on the texture evolution, the normalized texture difference is introduced. The normalized texture difference between samples T0 and Tx with respect to sample T0 can be calculated by Eq. (2):

\[
\text{Normalized Texture Difference} = NTD = \frac{\int_{\text{eulerspace}} (f_{T0}(g) - f_{Tx}(g))^2 \, dg}{\int_{\text{eulerspace}} (f_{T0}(g))^2 \, dg}
\]

where \( f_{T0}(g) \) and \( f_{Tx}(g) \) were the ODF of samples T0 and Tx (\( x = 1, 2, 3 \)) respectively, as a function of the Euler space coordinates \( g \). The normalized texture differences of T1, T2 and T3 with respect to T0 were calculated to be 0.11%, 35.39% and 0.83%, respectively. Therefore, it is evident that the substrate preheating temperatures at 423 K and 623 K have little effect on the crystallographic texture while the substrate preheating
Temperature at 523 K displays a significant influence on the crystallographic texture of the SLM-processed Ti-45Al-2Cr-5Nb alloy.

Fig. 3(a) illustrates the X-ray diffraction (XRD) results of the samples T₀, T₁, T₂ and T₃ from the top view. Apparently, only the diffraction peaks corresponding to α₂, γ and B₂ phases are detected. It is worth noting that α and β are the high temperature disordered α₂ and B₂, respectively. As revealed in the XRD patterns, the strong and dominant diffraction peak belonging to (202) α₂ is identified as the matrix phase. With increasing substrate preheating temperature, the intensities of the (0002) α₂, (2023) α₂ and (2024) α₂ peaks decrease while the intensity of the (2021) α₂ peaks increases. Meanwhile, the intensity of the (202) γ peak increases whereas the (620) γ and (222) γ peaks basically remain unchanged. Similarly, the intensities of the (220) B₂ and (311) B₂ increase, whereas the (420) B₂ peak intensity decreases and (310) B₂, (520) B₂ peaks remain the same. Consequently, the α₂ phase decreases while the γ and B₂ phases increase with increasing substrate preheating temperature. According to previous literature [17–20], the phase transition of TiAl alloys during solidification has experienced a L + β → α peritectic reaction, a α → β + γ eutectic reaction and two α → α₂, β → B₂ ordering transformations. As the β is a high temperature stable phase, substrate preheating may improve the stability of the β phase. Moreover, due to the existence of strong β-stabilizer elements of Cr and Nb [19], the stability of the β phase is further increased. But, the L + β → α transformation will be restricted with the increase in the stability of the β phase. Meanwhile, the eutectic reaction rate of α → β + γ is quite sensitive to the cooling rate [20], and substrate preheating can lower the cooling rate, thus facilitating the eutectic reaction. Therefore, the L + β → α transformation is limited, while the α → β + γ transformation is stimulated with increasing substrate preheating temperature, hence leading to the decrease of α₂ phase and the increase of γ and B₂ phases.

To further confirm the existence of α₂, γ and B₂ phases in the SLM-processed Ti-45Al-2Cr-5Nb, TEM was conducted. In the bright-field image of T₃ in Fig. 3(b), fine grain γ and B₂ grains are found distributing in the α₂ matrix, and the fine B₂ grains are supposed to derive from the uncompleted transformation of primary β → α [21]. Fig. 3(c) shows the selected area diffraction pattern (SADP) corresponding to Fig. 3(b). The different planes of the α₂, γ and B₂ phases showing the diffraction rings can be identified, indicating the polycrystalline structures. The radius ratio of the rings observed from the SADP is 1:1.17 (D₀₁₀):1.44 (L₁₀):1.67 (bcc). Additionally, based on the standard XRD reference codes (12-0085, 65-0428 and 14-0451), the inter-planar spacings were calculated to be d(1011) = 0.331 nm (α₂), d(1120) = 0.282 nm (α₂), d(111) = 0.230 nm (γ), d(200) = 0.198 nm (B₂). Fig. 3(d) illustrates...
the high resolution transmission electron microscopy (HRTEM) of α2, γ and B2 phases in the yellow circle of Fig. 3(b). The lattice-fringe separations of 0.289 nm, 0.234 nm and 0.267 nm are identified as the (1120) planes of α2 (DO3), the (111) planes of (γ L12) and (111) planes of B2 (bcc), respectively. According to the measured lattice parameters and standard reference patterns, it can be concluded that the γ-related and B2-related phases simultaneously precipitate within the α2 matrix. The γ and B2 with the planes (1120) α2/(111) γ/(111) B2 in a range of several hundred nanometers are uniformly distributed within the α2 matrix. As revealed in Fig. 4(b), the dynamic indentation depth decreases gradually with increasing substrate preheating temperature. The indentation depth decreases gradually with increasing substrate preheating temperature. Therefore, the SLM-processed TiAl shows much higher nanohardness than its traditional casting counterpart. In summary, the grain orientations, crystallographic texture, phase composition and nanohardness can be tailored by controlling the substrate preheating temperature during SLM. With the optimal preheating temperature of 623 K, the basically equal existence of the grains with (0001), (1011) and (1121) orientations was successfully achieved. The phase evolution mechanism in the SLM-processed Ti–45Al–2Cr–5Nb alloy is concluded as follows: (102) β transforms to (0002) α2 and (110) γ, and then the residual B2 and incompletely transformed γ phase homogeneously distribute in the α2 matrix. The orientation relationship between B2, α2 and γ phases is expressed as (1120) α2/(111) γ/(111) B2. Compared to the traditional casting TiAl alloy, the SLM-processed TiAl alloy shows much higher nanohardness values ranging from 7.57 ± 0.38 GPa to 8.74 ± 0.42 GPa. The findings would be a valuable reference to the optimization of the substrate preheating temperature for fabricating TiAl components with acceptable grain structure and phase compositions and nanohardness by SLM.

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