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Texture evolution, phase transformation mechanism and nanohardness of selective laser melted Ti-45Al-2Cr-5Nb alloy during multi-step heat treatment process

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ABSTRACT

This paper for the first time systematic study the effect of heat treatment on the grains characteristics, texture evolution, phases transformation mechanism and nanohardness of Ti-45Al-2Cr-5Nb (at.%) alloy processed by selective laser melting (SLM). With increasing the annealing temperature, the grain size generally increases while the crystallographic orientations basically remain unchanged. The grains of all the samples are dominated by high-angle grain boundaries, with its volume fraction increases with increasing the annealing temperature. The content of B_2 phase decreases, while the contents of α_2 and γ phases increase with the increase of annealing temperature. The phases evolution mechanism during the heat treatment is described as: $(200)\beta \rightarrow (11\bar{2}0)\alpha_2 + (111)\gamma$. The structure change from $\beta(B_2)$ phase to $\alpha(\alpha_2)$ phase is most likely achieved by the dispelling of Nb and Cr atoms, namely through transforming from the "… Ti-Nb/Cr-Al-Ti-Nb/Cr-Al …" stacking sequence in (110) plane of the bcc structure to the "… Ti-Al-Ti-Ti-Ti-Al-Ti-Ti …" stacking sequence in (0001) plane of the $D0_{19}$ (hcp) structure. The structure change from $\alpha(\alpha_2)$ to γ is most likely achieved by the rearrangement of Ti and Al atoms through transforming from the "… Ti-Al-Ti-Ti-Ti-Al-Ti-Ti …" stacking sequence in (0001) plane of the $D0_{19}$ (hcp) structure to the "… Ti-Al-Ti-Al …" stacking sequence in (110) plane of the $L1_0$ (bcc) structure. The nanohardness ranging from 8.36 ± 0.42 to 9.75 ± 0.49 GPa with increasing the annealing temperature, which is much higher than the roll bonding fabricated TiB_2 reinforced TiAl alloy.

1. Introduction

Titanium aluminides (TiAl) represent a novel class of light-weight intermetallic alloys targeted for industrial applications especially in aerospace and aircraft fields [1,2]. The exceptional properties of TiAl-based alloys facilitate their applicability are their low specific weight (3.8–4.1 g/cm^3), good oxidation (up to 1073 K) together with a high elastic stiffness and enhanced strength at elevated temperatures [3–5]. Despite these incomparable characteristics of TiAl-based alloys, their inherent low room temperature ductility (circa 1% total fracture strain) and poor hot deformability causes the serious difficulties in the fabrication complex geometries structural components via most of conventional processing routes, which inhibits the practical applications of TiAl-based alloys [6]. Thus, the manufacturing technologies of TiAl-based alloys require further development [7–12].

Selective laser melting (SLM), a burgeoning additive manufacturing (AM) technology, has the capability of fabricating near fully dense metal components with freeform geometries layer-by-layer directly from three-dimensional computer aided design (CAD) models [13,14]. SLM has been considered as one of the most promising manufacturing technologies for net shape industrial scale production of metal parts [15]. Moreover, the extremely high cooling rate (about 10^6 K/s) of the SLM process gives rise to very fine microstructure of as-processed metal parts [16]. It has been reported that microstructure refinement helps to improve the ductility of TiAl-based alloys [17,18]. Therefore, SLM shows great

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potential in manufacturing complex TiAl-based alloy parts with enhanced ductility. Recently, some preliminary researches have been performed focusing on the effect of SLM processing parameters on the microstructure and properties of TiAl-based alloys. Gussone et al. [19] investigated the effects of energy density on chemical composition and phase transformation of a γ-TiAl. Vilaro et al. [20] investigated the relationship between microstructure and mechanical properties of Ti-47Al-2Cr-2Nb alloy specimens produced by SLM and direct metal deposition (DMD), and found that the cooling conditions are fast enough to generate ultra-fine and metastable structures, which resulted in high microhardness. More recently, our previous works studied the effects of laser power [21] and scanning speed [22] on the microstructure evolution and nanohardness of the TiAl-based alloys processed by SLM. However, due to the rapid solidification and high temperature gradient of the SLM process, there exist segregation phenomena and non-equilibrium phases in the SLM-processed TiAl-based alloys [21]. Generally, as an important post-processing technology, heat treatment is considered to play a significant role in adjusting the microstructure features, phases composition and mechanical properties of TiAl-based alloys [23–25]. Yang et al. [26] systematically studied the $\beta \rightarrow \alpha$ phase transformation of Ti-45Al-8.5Nb-(W,B,Y) alloy during heat treatment, and the result suggested that the volume fraction of α phase decreased as the cooling rate reduced, which could be attributed to the competitive growth mechanism of the α and β phases during the $\beta \rightarrow \alpha$ transformation. Huber et al. [27] introduced a sequent three-step heat treatment to process turbine blades with balanced microstructure and mechanical properties of a forged Ti-43.5Al-4Nb-1Mo-0.1B alloy, by this three-step heat treatment process, it is possible to obtain targeted microstructures and strength for both low and high ram speed deformed ciabatta forgings. However, although it has been reported that heat treatment is an effective way to tailor the microstructure and thus improve the properties of TiAl-based alloy parts that produced by the conventional processing technologies, however, as far as the authors' knowledge, there is not yet the research systematically investigating how heat treatment affects the microstructural characteristics and mechanical properties of TiAl-based alloys fabricated by SLM. Thus, the objective of the present work is to reveal the grains characteristics, texture evolution and phases transformation mechanism of SLM-processed Ti-45Al-2Cr-5Nb alloy with a multi-step heat treatment process, these findings would be a valuable reference for the understanding of texture evolution, phase transformation mechanism and nanohardness of Ti-45Al-2Cr-5Nb alloy during multi-step heat treatment.

2. Experimental methods

2.1. Powdered materials

A Ti-45Al-2Cr-5Nb powder, supplied by Beijing Institute of Aeronautical Materials (Beijing, China), which is argon atomized from a Ti-45Al-2Cr-5Nb ingot. The starting powder is spherical with an average particle diameter of 27.6 μm [21,22].

2.2. SLM and heat treatment processes

A HRPM-II-type SLM machine developed by Huake 3D Technology Co. Ltd. (Wuhan, China) is used for the sample preparation. The SLM machine is equipped with a continuous single-mode ytterbium fiber laser (its maximum output is 400 W, focal laser beam diameter is 100 μm and wavelength is 1069 ± 10 nm) and a HURRY SCAN-30 type scanner. Based on a series of preliminary experiments [21,22], the processing parameters have been optimized as follows: the laser power of 250 W, scan speed of 800 mm/s, hatch spacing of 100 μm, and layer thickness of 30 μm. The scanning strategy used for the cubic samples (8 mm × 8 mm × 8 mm) fabrication is shown in Fig. 1(a). The arrows indicate the scanning direction of the laser and 90° rotation angle of the scanning direction between two consecutive layers N and N + 1.

A multi-step heat treatment process is then performed on the SLM-fabricated samples in vacuum condition as shown in Fig. 1(b). Temperatures are selected based on the Ti-Al binary phase diagram and previous literature [21,28–30]. At the first heat treatment step, the samples are annealed at the different temperatures: 298 K (room temperature, RT), 1373 K, 1423 K or 1473 K for 2 h, and cooled down to room temperature in calm air (AC) afterwards. At the second step, all the samples are annealed at 1173 K for 6 h, then followed by furnace cooling (FC). For convenience, the samples are annealed at 298 K, 1373 K, 1423 K and 1473K during the first heat treatment step are denoted as T_1, T_2, T_3 and T_4, respectively.

2.3. Microstructure and nanohardness characterization

X-ray diffraction (XRD) measurements are conducted on a XRD-7000S type instrument (Shimadzu, Japan) with a Cu tube at 40 kV and 30 mA, and the diffraction angle of 2θ varied from 20° to 110° with a scan rate and step size of 10°/min and 0.02°, respectively. Electron backscattered diffraction (EBSD) measurements are carried out on a HKL Nordlys orientation imaging microscope system (Oxford, Oxford Instruments, United Kingdom) with a step size and accelerating voltage of 0.5 μm and 20 Kv, respectively, and the HKL Nordlys orientation imaging microscope system is mounted on a JSM-7600F field emission scanning electron microscope (FESEM, JEOL, Japan), the EBSD data is interpreted by HKL Channel 5 software packages. Prior to the EBSD measurements, all the samples are electrolytic polished on a LectroPol-5 type machine (Struers, Denmark) with A3 reagent (vol. 10% perchlorate and vol. 90% ethanol) at 20 V for 20 s. The presence of α_2, γ, B_2 phases in the samples are detected using a JEM-2100 transmission electron microscope (TEM, JEOL, Japan), the TEM machine is equipped with a LaB6 source and the operating voltage is set at 300 kV. Instrumented nanoindentation experiments were carried out on a commercially available Hysitron Nano-scratch tester (Berkovich Indenter TI750, Hysitron, American) equipped with a diamond Berkovich tip. The indentation load and hold time were set at 3500 mN and 2 s, respectively. Prior to the indentation tests, all the samples were mechanically grinded and polished. Twenty indents were conducted on each sample and the average value was taken.

3. Results

3.1. Study of grains orientation and crystallographic texture

An analysis of the microstructure characteristics of sample T_1 is given in this section. The EBSD orientation map, grain misorientation angle distribution and pole figure (PF) from the top view of sample T_1 are presented in Fig. 1(d), (e) and (f), respectively. The stereographic triangle inverse pole figure (IPF) map shown in Fig. 1(c) is colored with respect to the grain crystal orientation in this paper. It can be found from Fig. 1(d) that most of the presented grains possess (0001), $(10\bar{1}1)$ and $(11\bar{2}1)$ crystal orientations, and the average grain size is calculated to be 5.81 μm. In order to facilitate the analysis of grain misorientation angle distribution, low-angle grain boundaries (LAGBs, 2–15°) and high-angle grain boundaries (HAGBs, >15°) are introduced to separate the grain boundary angles. As illustrated in Fig. 1(e), the grain misorientation angle distribution is dominated by HAGBs, and the statistics show