Materials and Design 132 (2017) 275–282

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Microstructural characteristics and densification behavior of high-Nb TiAl powder produced by plasma rotating electrode process

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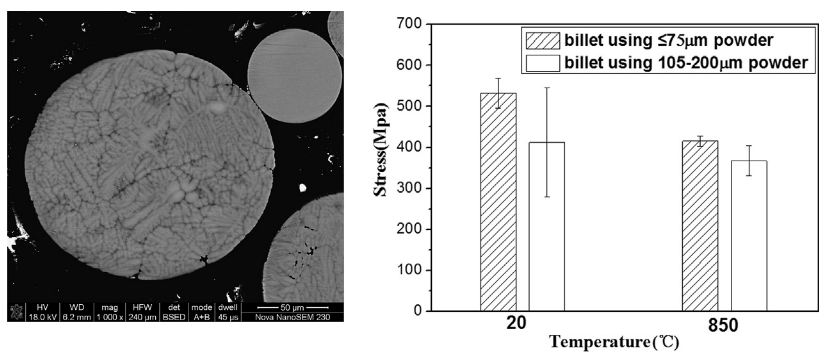
HIGHLIGHTS

- PREPed High-Nb TiAl powder feature two kinds of rapid solidification microstructures.
- Molten droplets will form dendritic or smooth martensitic structure for different cooling rates.
- Characteristic of powder has intrinsic effect on property of HIPed billets.

GRAPHICAL ABSTRACT



In order to produce high-quality high-Nb TiAl billet, the different rapid solidification microstructures and their forming reasons of high-Nb TiAl powder prepared by PREP were investigated, and the microstructure's hereditary effects on the HIPed billet were also studied.

ARTICLE INFO

Article history:
Received 13 April 2017
Received in revised form 21 June 2017
Accepted 30 June 2017
Available online 1 July 2017

Keywords:
Microstructure
High–Nb TiAl
Powder
Densification
Mechanical properties

ABSTRACT

In the present research, the microstructure and phase composition of high-Nb TiAl powder, produced by plasma rotating electrode process (PREP) have been investigated in details. PREPed powder with a mean particle size larger than 150 μm demonstrates a dendrite structure, and that smaller than 75 μm indicates a martensitic structure. PREPed powder is mainly composed of a hexagonal a_2 phase, while in fine powder and coarse powders minor β phase and γ phase are detected, respectively, which is related to solidification path and cooling rate of molten droplets in PREP process. Phase composition and microstructure of the high-Nb TiAl powder have intrinsic effects on the resultant microstructure of hot isostatic pressed (HIPed) billets. After HIP processing, most martensitic and dendritic particles transformed into near γ structure, while few coarse dendritic powder was prone to introduce defects, such as residual primary particle boundaries (PPBs) or coarse lamellae structure, which were harmful to the mechanical properties of HIPed billets. The residual primary particle boundaries (PPBs) or coarse lamellae structure obviously decreased the ultimate tensile strength at room and elevated temperature.

1. Introduction

Nb has been proved to be able to significantly improve the service temperature and strength of TiAl alloys. Therefore, high-Nb TiAl alloys are more suitable for higher temperature application than conventional ones, and have attracted increasing attention [1–5]. However, chemical

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http://dx.doi.org/10.1016/j.matdes.2017.06.072

composition segregation and structure heterogeneity are the major concerns when preparing bulk high-Nb TiAl alloys by traditional casting, often leading to frequent occurrence of cracks during cooling. Powder metallurgy (P/M) is an important method to manufacture high-Nb TiAl alloys, which is effective in preventing compositional and microstructural inhomogeneity and thermal cracks even in a large block. Two typical P/M approaches have been reported in manufacturing TiAl alloy, elemental powder metallurgy and alloyed powder metallurgy, where by using alloyed powder, the properties of TiAl based alloy are generally far more superior compared to those from the elemental-powder technique due to the more homogeneous chemical composition and less impurities [6–8].

There are various powder manufacturing techniques for Ti alloy including plasma melting induction guiding gas atomization (PIGA), electrode induction melting gas atomization (EIGA), and plasma rotation electrode process (PREP) [9–11]. For the gas atomization technique, large amount of inert gas such as argon has to be used, where the entrapment of inner gas cannot be avoided during atomization, and the resultant TiAl powder particles usually show inner porosities. After hot isostatic pressing (HIP), fine pores can be detected in the compacts even after subsequent heat-treatment or hot-rolling. These pores are caused by the atomization gas trapped in the particles, which are believed to deteriorate the creep and fatigue properties and to reduce the plasticity of TiAl products [12]. In contrast, the PREPed powders have nearly no inner pores, since the alloy melts are atomized centrifugally in a very low pressure of inert gas. For these reasons, the sephericity and fluidity of PREPed TiAl powder are better than that of the GA powder, resulting in a higher tap density, and favor of filling into the metallic can for subsequent HIP process [12,13].

There are two kinds of PREPed TiAl alloy powders classified by the microstructure: smooth martensitic structure and dendritic structure. Minoru et al. proved that Ti-rich TiAl alloy powder produced by PREP demonstrated similar microstructure and the powders with different structures gave rise to remarkably different compatibility at elevated temperatures [11]. Fuchs et al. examined the Ti-48Al and Ti-48Al-2Nb-2Cr powders prepared by PREP and GA technique, and found that the microstructure is closely related to atomization technique, alloy content, powder particles size and thermal history [13]. Wang et al. studied the high-Nb TiAl powder prepared by GA process, and proposed that the as-prepared TiAl powder had only dendritic structure. The phase of the TiAl powder was related to its granularity, the powder with particles size <74 μm contained only α_2 phase; when particle size increased, more γ phases could be detected [14]. However, as for the high-Nb TiAl, the microstructural characteristics and the corresponding formation mechanism of the PREPed powder, as well as the effects of high-Nb TiAl powder microstructure on the resultant microstructures and properties of HIPed billet have not been systemically investigated.

In the present study, powder with a nominal composition of Ti-45Al-7Nb-0.3W (at%) is produced by PREP. The physical properties, microstructure and phases of PREPed powders are characterized by scanning electron microscope (SEM), X–ray diffraction (XRD), electron probe X-ray microanalysis (EPMA) and transmission electron microscopy (TEM). The relationship between microstructure and particle size of powder, as well as the resultant HIPed microstructure and tensile properties were also investigated.

2. Experimental materials and procedures

High purity sponge Ti (purity>99.9 wt%,size from 3 to 15 mm), Al buttons (purity >99.7 wt%, size from 6 to 13 mm), Al-Nb (Nb content ≥60 wt%,size from 1 to 5 mm) and Al-W (W content ≥50 wt%,size from 1 to 5 mm) master alloy were used in this study. Ingots with a nominal composition of Ti-45Al-7Nb-0.3W (at%) was prepared by vacuum arc remelting (VAR). The ingots were melted for three times in order to ensure chemical homogenization. The ingots were then machined to round bars with dimension of $\Phi 75 \times 400$ mm^3, which were used in the PREP as electrode material. The TiAl powders were obtained through rotating the alloy electrode with 15,000–16,000 rpm and with plasma electric current up to 1100 Å. The heat treatment of the powder was carried out through putting classified powder into evacuated silica tube, heating to 1250 °C for 2 h, and then cooled in air. Two kinds of PREPed powders with particle size <75 μm and 105–200 μm through sieving were filled into cylindrical stainless steel cans individually. Subsequently, the steel cans were degassed under 500–600 °C for nearly 12 h, and sealed gas-tight. HIP process was conducted at 1250 °C holding for 4 h at a pressure of 150 MPa, followed by furnace cooling.

The PREPed high-Nb TiAl powder was sieved into 6 size fraction given by the sieve sizes: <46 μm, 46–75 μm, 75–105 μm, 105–150 μm, 150–200 μm, and >200 μm. The phase of powder with various particle sizes is analyzed by Bruker D8 Advance XRD using Cu Kα radiation. The microstructure of the HIPed TiAl billet and powders were observed by a Leica EC3 optical microscope and a Sirion200 scanning electron microscope (SEM) in back scattered electron (BSE) mode. The element distribution of the powder cross section and HIPed TiAl billet were analyzed by a Jeol Jxa-8530F electron probe mapping analyzer (EPMA). A Tecnai G^{2}20 Transmission electron microscope (TEM) was used to further investigate the powder's microstructure. To prepare TEM TiAl powders specimens, the powders were first mixed with epoxy resin, and then filled the mixture into a copper tuber with diameter of 3 mm. After epoxy resin was fully solidified, cutting the copper tube to attain discs, the discs were thinned through mechanical polishing and ion milling to obtain the TEM specimens. For tensile tests at room temperature, the specimens were cut from the center of HIPed high-Nb TiAl billet, with a gauge section of 3×1.2 mm^2 and 8 mm gauge length. The tensile tests were conducted on an Instron 3369 material testing machine in air at ambient temperature 20 °C and 850 °C with nominal tensile tests speed of 0.5 mm/min and 0.1 mm/min, respectively.

3. Experimental results

Fig. 1 shows the morphology, particle size distribution and element content of the PREPed high-Nb TiAl powder. The particles demonstrate perfect spherical shape. The irregular particles or particles with satellites, which often appear in GA process products, are scarcely observed (Fig. 1(a)). Moreover, no porosity in the inner particles was observed as shown in Fig. 1(b). From the particle size distribution (Fig. 1(c)), it can be seen that the particle size is mostly from 46 to 150 μm, >60% particle is <105 μm. In addition, the particle size is a bimodal distribution, and it is within a small range. The weight percentages of interstitial element in TiAl powder with different particle sizes are presented in Fig. 1(d). With the decrease of particle size, the O content increased while the N and H contents remained constant. When the particle size was larger than 150 μm, O content was 600–700 ppm, O content increased to 1200 ppm when the particle size was <46 μm.

Fig. 2 shows the microstructure of high-Nb TiAl powders and the alloying element line distribution along the particle's cross section. Similar to the other previous research [11,13–16], two kinds of microstructures (surface morphologies) in the PREPed high-Nb TiAl powders were observed: a dendritic structure (Fig. 2(a)) and the featureless smooth structure (Fig. 2 (b)).The smooth structure is similar to a martensitic surface relief, and very faint laths can be distinguished upon closer observation. Moreover, the difference in powder microstructure was found to be related to the particle size: coarse particles generally exhibit dendritic structures, while finer particles show smooth martensitic microstructure. Statistic calculations by SEM (Fig. 2(c)) on percentages of dendritic and smooth surface particles in different size ranges indicates that particles larger than 105 μm generally demonstrate a dendritic structure surface, while particles <75 μm are mostly martensitic structures. Similarly, the cross sections of these particles in BSE mode (Fig. 2(d)) show the large dendritic networks in the cross sections of coarse particle. However, featureless smooth structure was observed