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Fabrication of micro-fine spherical high Nb containing TiAl alloy powder based on reaction synthesis and RF plasma spheroidization

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ABSTRACT

A compact process for fabricating micro-fine spherical TiAl–Nb alloy powders was developed with a combination of reaction synthesis and plasma spheroidization technique. Powders of TiH_2, Al and Nb were firstly mixed and refined by high-energy ball milling, and the mixture was then heated at different temperatures ranging from 600 to 1200 °C for 2 h, allowing synthesis reaction to take place. Ultimately, the synthesized powders melt through radio frequency (RF) argon plasma and rapidly solidified into micro-fine spherical TiAl–Nb alloy powders. The fabrication process and powder characteristics were investigated. With the process described above, the alloy powders with high composition homogeneity and good sphericity can be fabricated. The obtained spherical powders perform a uniform equiaxed-grain microstructure dominated by a supersaturated α_2-Ti_3Al phase. The powders possess a micro-fine average particle size of 9.6 μm, with a distribution uniformity level of 0.622.

1. Introduction

With the rapid development of aerospace industry, highly demanding requirements have been put forward on the aerospace materials. γ-Based TiAl alloys with high Nb addition are deemed as a new generation of high temperature structural materials owing to the superior mechanical properties and oxidation resistance at elevated temperature as well as the intrinsic low density [1–4]. However, poor ductility at room temperature and difficulties in processing are still the major obstacles to the application of TiAl based alloys. Powder metallurgy fabrication technologies can overcome the issues. It is believed to be one of the most effective ways to extending the application of TiAl based alloys.

High-quality raw powders have been in great need to support the fabrications of TiAl–Nb alloys with powder technologies, especially for powder injection molding, plasma spraying, etc. [5–9]. So far TiAl based powders are usually prepared by plasma rotating electrode atomization or inert gas atomization with non-crucible melting. However, the obtained powders exhibit larger particle size and hollow structure, and can hardly meet the requirements for practical applications [10]. In our previous work, TiAl based alloy powders have been prepared by fluidized bed jet milling and subsequent RF plasma spheroidization from alloy ingot materials. The result shows the feasibility of RF plasma spheroidization process, and the particle size of powders can be controlled in the range of 15–60 μm with no pores inside [11]. Based on this work, a short process for producing micro-fine spherical TiAl–Nb alloy powders has been developed with a combination of reaction synthesis and plasma spheroidization from elemental powders. As a traditional process, reaction synthesis is widely used for alloy powder preparation, with simple procedure and cost-effective feature [12–14]. RF plasma spheroidization also can be used in producing spherical powders with high melting point. It is a sufficient method to obtain metal and alloy powders with high degree of sphericity and narrow size distribution [15–17]. By combining these two methods, TiAl based alloy powders are expected to be fabricated. Compared with Ti metal powders, TiH_2 particles are more likely to be crushed during milling process due to lower ductility. The refinement and composition homogenization of the raw material particles can be achieved in a shorter period of milling time, which can contribute to the decrease of impurity absorption. Besides, the highly reactive Ti released from dehydrogenation of TiH_2 may promote the alloying process during heat treatment. Hence, TiH_2 was employed as the raw material in our recent research.

In this paper, homogeneous spherical high Nb containing TiAl alloyed powders in microscale were prepared by reaction synthesis and subsequent RF argon plasma spheroidization from TiH_2, and elemental powders of Al and Nb. The fabrication processing and the characterization of the powders were investigated as well.

2. Experimental

TiH_2 powders and Al, Nb elemental powders with high purity (≥99.7%) were used as the raw materials to give a nominal composition of Ti–45Al–8.5Nb (at. %). Fig. 1 shows SEM images of the as received TiH_2, Al and Nb powders. It can be observed that Al powder is near

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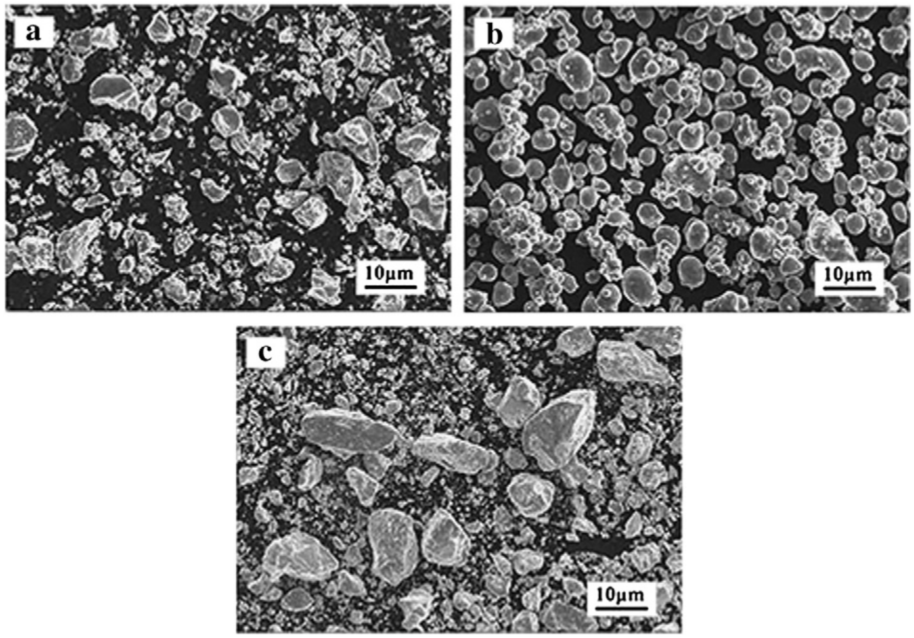

Fig. 1. SEM micrographs of raw material powders: (a) TiH_2; (b) Al; (c) Nb.

spherical, while TiH_2 and Nb powders are irregular in shape, and their mean particle sizes were all below 30 μm.

The powder mixture was subjected to high energy ball milling for 5 ~ 20 h in a three dimensional vibration ball milling machine with the frequency of 1400 Hz. The process was conducted in an argon atmosphere with ball-to-powder ratio of 10:1. The as-milled powders were annealed in a tube furnace in an argon atmosphere at different temperatures from 600 to 1200 °C. Finally, the irregular annealed powders were processed by RF plasma spheroidization system. A powder spheroidization system is schematically shown in Fig. 2 [18]. During RF plasma spheroidization, an argon plasma torch with extremely high temperature was formed by the alternating current in the induction coils. When passing through the plasma torch region, the injected powders with irregular shape are melted instantaneously by absorbing great amount of heat, and form spherical droplets under surface tension. And then these droplets are cooled and solidify rapidly to form spherical-shaped particles. The input power was 45 KW, the argon gas flow rate was 85 ml/min and the negative pressure was −1000 Pa.

A Siemens D5000 X-ray diffractometer using Cu radiation was employed to analyze the phase constitution of the powders obtained during the process. The observation of microstructure, morphology and analysis of element distribution, were performed by an LEO1450 scanning electron microscopy (SEM) equipped with a KEVEX Sigma energy dispersive spectrometer (EDS). The particle size distribution was examined by using an LMS-30 laser particle size analyser. The oxygen and carbon contents were measured by the high-frequency combustion-infrared absorption method and the inert gas impulse infrared thermal conductivity method.

The parameters of number-average diameter ($D(1,0)$), and size distribution uniformity (U) were introduced to characterize the powder size and distribution level. Both of them can be derived from the detected particle size distribution data. The value of $D(1,0)$ can be calculated by Eq. (1), where d_i means detected particle diameter and n_i (%) indicates the percentage of the particles with the diameter of d_i (μm).

$$D(1,0) = \frac{\sum\left(n_i \cdot \sqrt{d_{i-1} \cdot d_i}\right)}{\sum n_i} \tag{1}$$

The value of U can be used to evaluate the uniformity level of particle size distribution, calculated by using Eq. (2). A higher value of U indicates a better uniformity of particle size distribution. In general, U value is less than 1, and it is equal to 1 only for the powders consisting of mono-sized particles.

$$U = \frac{D(1,0)}{D(4,3)} = D(1,0) \cdot \frac{\sum\left[n_i\left(\sqrt{d_{i-1} \cdot d_i}\right)^3\right]}{\sum\left[n_i\left(\sqrt{d_{i-1} \cdot d_i}\right)^4\right]} \tag{2}$$

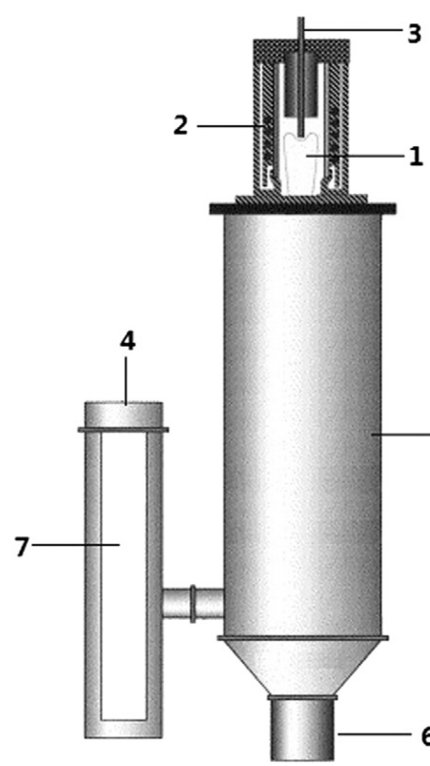

Fig. 2. Schematic of a powder spheroidization system. This system includes: 1. plasma torch, 2. induction coils, 3. powder feeder, 4. vacuum, 5. cooler, 6. collector and 7. filter.