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Microstructure and mechanical properties of Y_2O_3 reinforced Ti6Al4V composites fabricated by spark plasma sintering

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ABSTRACT

The paper presents the fabrication of (0.6–3.0 wt%) Y_2O_3-Ti6Al4V composites by spark plasma sintering (SPS) with the sintering heating rate of 100 °C/min and the sintering temperature of 900 °C. Ti6Al4V powders and Y_2O_3 powders were admixed by rocking mill for 8 h at 30 Hz. Scanning electron microscopy (SEM) equipped with complete energy dispersive spectrometer (EDS) and X-ray diffraction (XRD) were used to characterize the as-received Ti6Al4V powders, Y_2O_3 powders, the admixed composite powders and the sintered samples. The microhardness, the compressive yield strength and the ultimate strength of as-sintered samples at room temperature were enhanced up to 464.1HV, 1346 MPa and 1583 MPa respectively with a plastic strain of 19.1%, when 2.0 wt% Y_2O_3 was introduced. The mechanical behaviors of 2.0 wt% Y_2O_3-Ti6Al4V composite at 450 °C were also carried out with the yield strength of 832 MPa and the ultimate strength of 1088 MPa. Compared with the sintered Ti6Al4V, the 2.0 wt% Y_2O_3-Ti6Al4V composite has higher yield strength and ultimate strength at elevated temperature with the increment of 54% and 37%, respectively. The mode of fracture was transformed from ductile fracture to a combination of ductile and brittle fractures with the increase of Y_2O_3 content.

1. Introduction

Titanium alloy is an important metallic material used widely in various fields of modern industry especially in aircraft industry and medical industry due to its low density, high specific strength, excellent corrosion and biocompatibility [1–5]. As a typical α/β titanium alloy, Ti6Al4V is the most widely used titanium alloy due to its excellent mechanical properties [6]. However, the low hardness leads to the poor tribological properties, which prevents the further application of titanium alloy in many fields [7]. One of the methods improving the surface hardness is using surface modification techniques, such as laser surface cladding, chemical vapor deposition (CVD) and physical vapor deposition (PVD) [8–10]. Generally, the ceramics such as TiB_2, TiB, TiC, Al_2O_3 and Y_2O_3, as the reinforcement, are coated onto the surface of Ti alloys [11–15]. However, some shortages of these methods limit the application. For instance, the adhesion of films by using CVD or PVD is poor [10].

Another method is producing the composites by adding enhanced phase to exhibit the preferred properties of titanium alloys. In general, metallurgical methods of fabrication of composites are divided into two categories: ingot metallurgy (IM) and powder metallurgy (PM). Shen et al. [16] improved the hardness of the Ti6Al4V by investment casting with adding hBN-Y_2O_3. As a new technology of PM, spark plasma sintering (SPS) can produce plasma among the sintered powders by applying the electric field and pressure, so that the particles are activated and purified [17]. The advantages of SPS are short-time, low-pressure and high-density compared with the traditional sintering processes [18,19]. Cao et al. [6] improved fine microstructure of the Ti6Al4V by using TiB through SPS. As it to know, Y_2O_3 can improve the mechanical properties of alloys as oxide dispersion strengthening (ODS) [20–22]. In addition, Y_2O_3 has the potential to improve the high temperature performance of the alloys as the additive. To date, the titanium alloys with Y_2O_3 were usually synthesized by traditional metallurgical methods [23–25], and there are few researches about synthesizing Y_2O_3-titanium composites by SPS. At the same time, the high temperature mechanical properties of Y_2O_3-Ti6Al4V composites produced by SPS are rarely studied.

In the present work, Y_2O_3 powders were dispersed into Ti6Al4V and consolidated by SPS. Our investigation mainly focused on the

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effects of Y_2O_3 on the mechanical properties of Y_2O_3-Ti6Al4V composites at room temperature and at elevated temperature. In addition, the fracture mode of the Y_2O_3-Ti6Al4V composites was also discussed.

2. Experimental procedure

2.1. Raw materials

As shown in Table 1, the chemical composition of the Ti6Al4V powders (99.7% purity), which were supplied by Baoji Baoye Titanium-nickel Industry Co., Ltd, was in agreement with specification ASTM B348 GR5. The average particle size of the Ti6Al4V powders was about 15 μm. The yttrium (III)-oxide powders (Y_2O_3, 99.99% purity) were purchased from MACKLIN, and the average particle size was about 50 nm.

The raw materials were characterized by electron microscopy using scanning electron microscopes (SEM, SSX550, SU8000, HITACHI, JAPAN and FESEM, Gemini SEM 500, Zeiss, GERMANY) equipped with complete energy dispersive spectrometer (EDS) and X-ray diffractometer (XRD) (XRD-7000, SHIMADZU, JAPAN).

2.2. Rock milling

The Ti6Al4V and yttrium (III)-oxide powder mixtures were prepared in four batches, and the content of Y_2O_3 is 0.6 wt%, 1.0 wt %, 2.0 wt%, 3.0 wt% Y_2O_3, respectively. The rock milling was carried out by using a rocking mill machine (Rocking Mill, type RM-05, Japan) at 30 Hz for 8 h. The ball to powder ratio was 10:1 (200 g of agate balls and 20 g of powders). The powder mixtures were sealed in polypropylene vials (100 mL). The agate balls in two different sizes (diameter = 20 mm and 5 mm) were charged with powders to avoid cold welding of powder particles [18]. After rock milling, the powder mixtures were characterized by SEM and XRD, respectively.

2.3. Thermo-electrical-mechanical consolidation of powder mixtures

The spark plasma sintering system was from Japan (SPS, LABOXTM-325S, SINTER LAND INC, JAPAN). Y_2O_3-Ti6Al4V powder mixtures of each batch (20 g) and pure Ti6Al4V powders were poured into a Φ 20 mm graphite die respectively. To avoid welding between powders and die and to make sure the current flows uniformly within it, graphite foils (0.2 mm thick, Japan) were placed between the graphite die and the powders. The heating rate was 100 °C/min constantly, and the temperature for SPS was set to 900 °C and held for 5 min. Under high vacuum environment, the applied pressure was 30 MPa. To ensure the good electrical contact among the powders, a pre-compaction pressure of 10 MPa was applied by LAB Mini Press Machine before the sintering. After sintering, the electrical-mechanical load was removed. The sintered compact was taken out when it cooled down to room temperature. Before measuring the relative density by Archimedes' principle, the graphite on the surfaces of the sintered compacts was removed by polishing. The density was tested at least 6 times for each content Y_2O_3-Ti6Al4V composites, and the relative densities of the sintered compacts are shown in Table 2. It was observed that the composites were almost fully densified at the sintering temperature of 900 °C, and there were no significant effects of Y_2O_3 contents on the consolidation. The as-sintered Ti6Al4V and Y_2O_3-Ti6Al4V composite samples were characterized by SEM, EDS and XRD, respectively.

Table 1
Chemical composition of Ti6Al4V.

Elements	Al	V	O	Fe	H	N	C	Ti
Ti6Al4V	6.4	4.1	0.12	0.18	0.0089	0.02	0.01	Balance

2.4. Hardness test and compression test

A microhardness tester (HXD-1000TMC/LCD) was chosen to test the Vickers-hardness. A load of 0.9806 N was applied and held for 15 s. At least 5 different positions were chosen to perform indentations separated by 3 mm to avoid residual stresses for each sintered compact. Before the hardness tests, the surface of the sintered compacts was mirror polished.

Compression tests were performed on an uni-axial 100 kN MTS servo hydraulic testing machine (C45.105) with a load-sensing transducer (LPS.105, 100 KN) with a constant strain speed of 0.005 min^{-1} at room temperature and 450 °C, respectively. The sintered compacts were cut into the samples for compression tests by wire-cut electro-discharge machining (EDM). The diameter and the height of the samples were 4 mm and 6 mm respectively. There were four samples in each group for compression tests. The yield strength (0.2% offset), the ultimate strength and the compressive strain at failure were obtained from the true stress-strain diagrams. The true stress σ_t and true strain ε_t were computed by:

$$\varepsilon_t = -ln(1-\varepsilon_e) \quad (1)$$

$$\sigma_t = \sigma_e(1-\varepsilon_e) \quad (2)$$

where the engineering stress σ_e and the engineering strain ε_e are expressed as:

$$\sigma_e = P/A \quad (3)$$

$$\varepsilon_e = (L_0 - L)/L_0 \quad (4)$$

P and A represent respectively the real-time load of compression machine and the cross-sectional area of the sample. L and L_0 are respectively the real-time height and initial height of the sample.

3. Results and discussion

3.1. Characterization of powders and composites

The morphologies of starting powders and admixed powders are shown in Fig. 1. As shown in Fig. 1 (a) and (b), there was no demonstration of agglomeration in the Ti6Al4V powders and the Y_2O_3 powders. Fig. 1(c–f) show the dispersion of the different weight fractions of Y_2O_3 (0.6 wt%, 1.0 wt%, 2.0 wt%, 3.0 wt%). As shown in Fig. 1(c–f), the Y_2O_3 was relatively dispersed homogeneously in the admixed powders after milling for 8 h with 30 Hz without any process control agents (PCA). Fig. 2 shows the size distribution of particles before and after rock milling with varied Y_2O_3 contents. From Fig. 2 (a), it can be seen that the average size of particles before milling is about 14.78 μm (Ti6Al4V), and the average size of particles after milling with varied Y_2O_3 contents (Fig. 2 (b)-(e)) is 14.85 μm (0.6 wt% Y_2O_3-Ti6Al4V), 15.31 μm (1.0 wt % Y_2O_3-Ti6Al4V), 15.43 μm (2.0 wt% Y_2O_3-Ti6Al4V) and 15.22 μm (3.0 wt% Y_2O_3-Ti6Al4V), respectively. There was no fracturing of the particles and the particles still remained their original sizes and the pristine structure.

Fig. 3 shows the X-ray diffraction patterns of as-received Ti6Al4V powders, Y_2O_3 powders and the admixed Y_2O_3-Ti6Al4V composite powders. There was no evidence of peak broadening, which further corroborates that the sizes of the matrix particles were no obvious reduction. The peaks of Y_2O_3 were observed in the