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Original Research Article

Effect of compaction pressure and heating rate on microstructure and mechanical properties of spark plasma sintered Ti6Al4V alloy

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ABSTRACT

In the present paper, the use of spark plasma sintering on Ti6Al4V powder was investigated. Sintering experiments were conducted at the temperature of 1000°C for 5 min. The simultaneous effect of compaction pressures of 5, 25 and 50 MPa and heating rates of 200, 300 and 400°C/min on the structure, density, microhardness, elastic modulus and compressive strength were analyzed and ranged between 4.14 and 4.43 g/cm^3, 293 and 373 $HV_{0.05}$, 116 and 142 GPa, 1169 and 1414 MPa respectively. With increasing compaction pressure, the effect of an increase in grain size was observed. The obtained results show that very good mechanical properties can be achieved using spark plasma sintering at a rapid heating rate and already with the 25 MPa compaction pressure. The best results of microhardness (373 $HV_{0.05}$) and compressive strength (1414 MPa) with an elastic modulus of 138 GPa were obtained by the compacts sintered under the compaction pressure of 50 MPa and at the heating rate of 300°C/min.

1. Introduction

The Ti6Al4V alloy was discovered in 1954 through the addition of 6% aluminum to stabilize the α phase and 4% vanadium to stabilize the β phase [1]. Nowadays, this two-phase (α + β) alloy is widely used in different industries such as aerospace, automotive, marine, chemical processing and medicine [2,3]. The Ti6Al4V alloy is a kind of advanced material with low density, high specific strength, excellent corrosion resistance, good weldability and high-temperature performance [4,5]. A serious disadvantage of the Ti6Al4V alloy is its poor performance in sliding, hardness and wear [6,7]. Moreover, sintering by conventional powder metallurgy (PM) methods is difficult due to the high melting point and the extreme chemical affinity to atmospheric gases like oxygen, hydrogen, and nitrogen, especially at elevated temperature [1]. That is why better PM methods are sought. Some of them are field assisted sintering techniques (FAST) with the most popular of them – spark plasma sintering (SPS). This method is sought not only for earlier cryomilling of powder materials in order to protect grains before they increase in size but also for powder

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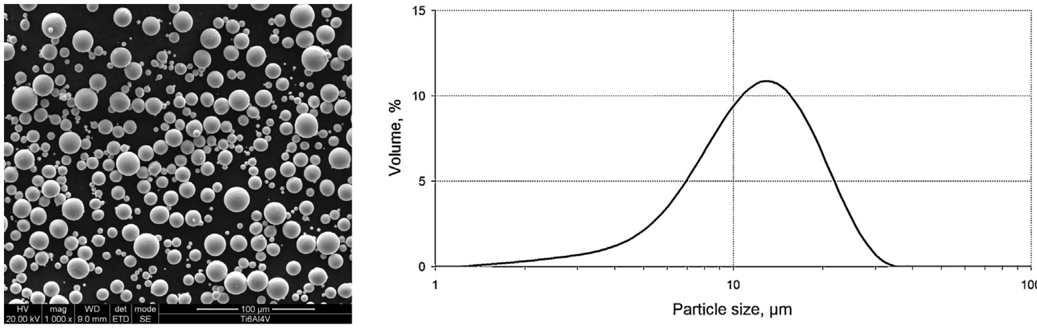

(a) (b)

Fig. 1 – Morphology of powder particles (a) and particle size distribution (b) of Ti6Al4V powder.

materials that are difficult to sinter by conventional PM methods [8,9]. Using SPS, various powders can be consolidated at a temperature usually 100–300°C lower than by other PM techniques, and in a much shortened time. That is why SPS is highly efficient for processing a wide range of powder materials [10]. The Ti6Al4V alloy and composites based on it produced using SPS have been characterized in many research centers worldwide in recent years. In papers [1,2,7,8,11–13], SPS processes were performed mostly on the Ti6Al4V alloy and some composites based on it at temperatures ranging from 700 to 1500°C and compaction pressures ranging from pressureless to 80 MPa. A holding time ranging from 2.5 to 20 min and heating rate of 100°C/min were also applied. Liu et al. [10] reported that a rapid heating rate is highly effective in densifying powder materials by SPS. That is why the authors of the present paper decided to investigate the effect of various heating rates as well as compaction pressures on the microstructural evolution and mechanical properties of the Ti6Al4V alloy spark plasma sintered above the β transus at 1000°C. In some applications, this is the critical temperature for sintering titanium and its alloys e.g. Ti6Al4V-HAp composites [14]. Moreover, the simultaneous effect of various heating rates and compaction pressures on the densification of Ti6Al4V powder using spark plasma sintering has not been studied yet in open literature.

2. Materials and methods

Fig. 1 shows the morphology and particle size distribution of gas-atomized Ti6Al4V powder of 99.9% purity supplied from Kamb Import-Export, Poland. Table 1 presents the chemical composition of this powder.

The powder was densified by spark plasma sintering using a HP D 25-3 furnace (FCT, Germany). The sintering temperature of 1000°C was reached at heating rates of 200, 300 and 400°C/min. The pressure level on the compacts was kept constant at 5, 25 and 50 MPa throughout the sintering process. The vacuum level of the sintering chamber was set at 5 Pa. After hold time for 5 min, the Ti6Al4V bulks were cooled to room temperature for 10 min. From spark plasma sintered Ti6Al4V compacts with dimensions of ∅ 40 mm × 10 mm, samples for tests were cut by the wire electrical discharge machining.

Particle size distribution using the laser diffraction technique was carried out by a Mastersizer 3000 (Malvern, UK) analyzer. The density of the spark plasma sintered compacts was measured by the Archimedes method. The microstructure of the sintered compacts was observed by light microscopy (LM) using an Eclipse L150 (Nikon, Japan) microscope on polished cross-sectioned surfaces and etched using Kroll's reagent. The grain size was measured using NIS Elements software by the line intercept method and mean of the values were calculated. A Kristalloflex 4 (Siemens, Germany) X-ray diffractometer with Mo-K_α radiation was also used for the structural studies. The microhardness measurements were carried out on the polished surface of the samples using a Vickers hardness tester FM-800 (Future-Tech, Germany) and applying a load of 0.4903 N for 15 s. The study of the indentation elastic modulus was conducted using a nanoindentation tester Picodentor HM500 (Fischer, Germany). A Vickers indenter was used under a load of 0.5 N applied for 5 s. The compressive strength was measured using a 4483 Instron mechanical testing machine of a measuring range of up to 150 kN (constant crosshead speed) with an initial strain rate of 0.0033 s^{-1}. For the tests, samples with dimensions of ∅ 10 mm × 10 mm were used. The compressive strength and strain were defined.

3. Results and discussion

The Ti6Al4V powder was densified by spark plasma sintering. The obtained compacts were analyzed and the results listed in Table 2. Table 2 shows the density and selected mechanical properties. The relative density of the spark plasma sintered compacts increased from 93.45% to 100.00% with increasing

Table 1 – Chemical composition of Ti6Al4V powder (manufacturer's data).

Ti, %	C, %	O, %	N, %	H, %	Fe, %	Al, %	V, %
Balance	0.01	0.17	0.02	0.013	0.16	6.19	3.80