ADVANCED METALLIC-CERAMIC COMPOSITE Ti6Al4V/Al_2O_3 FOR BIOMEDICAL APPLICATION

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https://doi.org/10.37904/metal.2019.913

Abstract

Metallic-ceramic composites are considered as materials with functional properties depending on the production methods. In this work, bi-layered composites Ti6Al4V/Al_2O_3 were obtained using the spark plasma sintering (SPS) technique. The powders (Sulzer Metco) of titanium alloy Ti-6Al-4V with a spherical shaped particle and aluminum oxide (Metco 101SF) with an irregular shaped particle were used. The specimens were compressed at 20 MPa and sintered in a shielding gas (argon) medium at the temperature of 1200 °C in an SPS HP 5 for 25 min. The obtained composites were subjected to microstructural analysis using light microscope, scanning microscopy Joel JSM 5400 equipped with EDS. Hydrostatic weighing in deionized water according to the PN EN standard was used to evaluate the relative density.The results showed that hot pressing proces (SPS method) could be used to produce dense bi-layered composites with proper interface properties between Ti6Al4V and Al_2O_3.

Keywords: Bilayer, SPS methods, composites

1. INTRODUCTION

The interest in the interdisciplinary cooperation of medical and technical environments results in development of new materials (for example bilayer composites) for medical applications. Layered composites, depending on the method of their production may be characterized by porosity and therefore a very important feature in the process of osteogenesis. We distinguish several groups of materials that have been successfully used in medicine and, for example, the most commonly used material in medicine is Ti6Al4V titanium alloy, which has an established position among other metallic materials. Titanium alloys have been used for more than 40 years for biomedical applications: artificial hip joints, artificial knee joints, bone plates, screws for fracture fixation, cardiac valve prostheses, pacemakers, and artificial hearts. Ti-6Al-4V compared to other biomaterials, is characterized by good corrosion resistance in the environment of chlorides, the highest biotolerance, beneficial strength-to-yield-point ratio, the lowest Young's modulus among metallic biomaterials and high fatigue resistance. Ti alloys have a number of advantages, but also some disadvantages like low wear resistance properties and an elastic modulus which is considered to be high when compared to the bone. Solution to the problems associated with the high elastic modulus has been to use advanced manufacturing processes such as powder metallurgy or SPS method to make porous titanium structures. The development of bilayer materials produced by hot pressing seems to present an advantageous and very interesting combination of materials and process for biomedical application. Two main types of pressure-assisted sintering methods are known (see **Figure 1**).

Ceramic/metal composites are mostly created from powder pressed and heated by pressure-assisted methods. The powder is placed into a closed die and pressured between two or more punches. In the hot pressing die sintering process, pressure and heat are applied together at the same time and the sample is placed into a heated die. SPS methods offers many advantages over traditional sintering techniques like hot press HP, hot isostatic pressing HIP or atmospheric furnaces including ease of operation and accurate control of sintering energy, high sintering speed, high reproducibility, safety and reliability. Spark discharge appears in contact point between the particles of a powder of the material and a local high temperature-state to ten thousands of degrees centigrade is generated momentarily. This causes in the SPS process, evaporation and melting on the surface of powder particles, and necks are formed around the area of contact between particles.

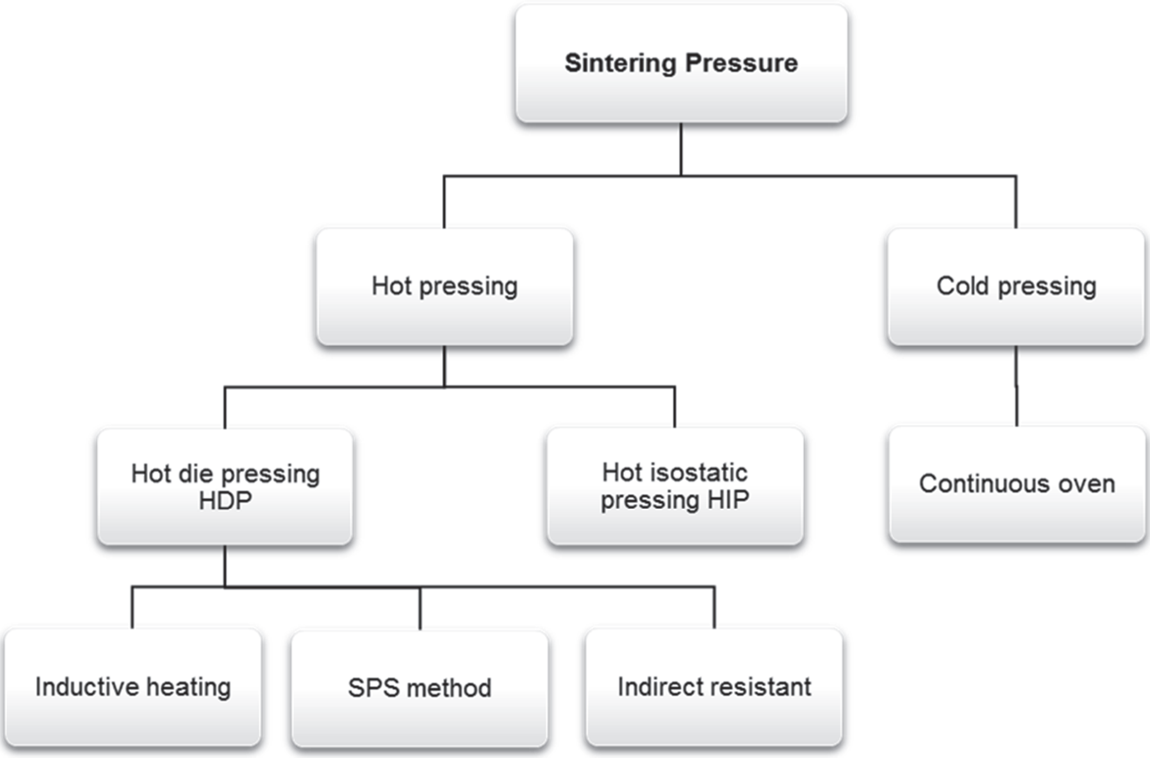


Figure 1 Standard classification of the sintering pressure process

At present, articles of research groups dealing with the subject of Ti6Al4V (or Ti) composites with the addition of Al_2O_3 ceramics can be found. However, these are proposals for the production of composites resulting from the mixing of both powders. Hayun et al. [1] and Miriyev et al. [2] studied the effects of titanium concentration in the initial mixture on phase composition, the static and dynamic mechanical properties of the SPS-processed composites were investigated. There are very few articles about layered composites manufactured using the SPS method as a proposition for medicine [3,4]. The aim of the study was to evaluate the ability of spark plasma sintering to obtain bilayer metallic-ceramic composites titanium alloy (Ti-6Al-4V) with an addition of inert ceramics (Al_2O_3) for medical applications. The possible results may be also useful in similar investigations in materials science [5,6], especially in surface layers technologies [7,8] combined with nanoparticles [9,10] as well as in a full-scale industry [11-16].

2. MATERIALS AND METHODS

The powders (Sulzer Metco) of titanium alloy Ti-6Al-4V with spherically shaped particles (size 45 ± 5 µm), aluminium oxide (Metco 101SF) with irregularly-shaped particles (size 22 ± 5 µm) were used to obtain bilayered (metallic-ceramic) material. Bulk density of individual powders is presented in **Table 1**.

The titanium alloy powder was placed in a die of a diameter of 20 mm with two stamps. Then, the aluminium powder was applied onto titanium powder. Graphite foil was placed between the powders, die and stamps for technological purposes. The sintered material was obtained using the spark plasma sintering method in an SPS HP 5 device in a shielding gas medium at the pressure of 20 kPa. The samples were compressed with the force of 7 kN and a piston moving rate of 1 mm/s. The specimens were compressed in 1200 °C. For comparison, the 100 % Ti6Al4V and 100 % Al_2O_3 sinters was also made by this method.

Table 1 Bulk density of powder

Powder	**Bulk density** (g/cm^3)
Ti-6Al-4V	2.95
Al_2O_3	1.75

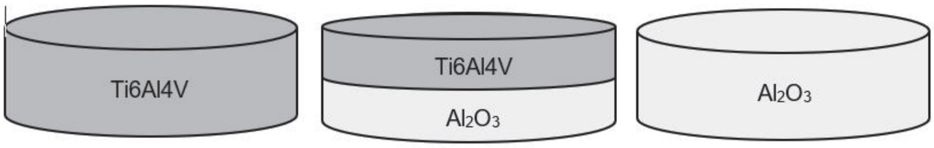


Figure 2 The sample scheme

The sample scheme is shown in **Figure 2**. After sintering process, the specimens were cut and polished in surface and cross-section. The samples were subjected to metallographic examination after polishing and chemical etching. The Kroll reagent, composed of 4 cm^3 of nitric acid, 2 cm^3 of 40 % hydrofluoric acid (HF) and 100 cm^3 of water, was used to reveal the structure of the metal material. The volumetric density of individual sinters was evaluated using the geometric method, expressing the apparent density that would be reached by the sample, calculated from the volume it fills and its mass. The measurements were performed with accuracy of 0.01 g for 3 specimens of each composite. Analysis of microstructure of metallic-ceramic composites was conducted using the optical microscope Axiovert 25. Selected samples (bilayered specimens) were sectioned and polished for microstructural characterization by means on Scanning Electron Microscopy (SEM) Jeol JSM 5400 equipped with EDS. In further investigation, a more complex analytical techniques will be employed as e.g. advanced computer image analysis [17,18], complex statistical techniques [19-21] with simulation [22-24].

3. RESULTS AND DISCUSSION

The microstructure of the 100 % Ti6Al4V sinters is presented in **Figure 3** and of the 100 % Al_2O_3 sinters in **Figure 4**, obtained by the optical microscope Axiovert 25.

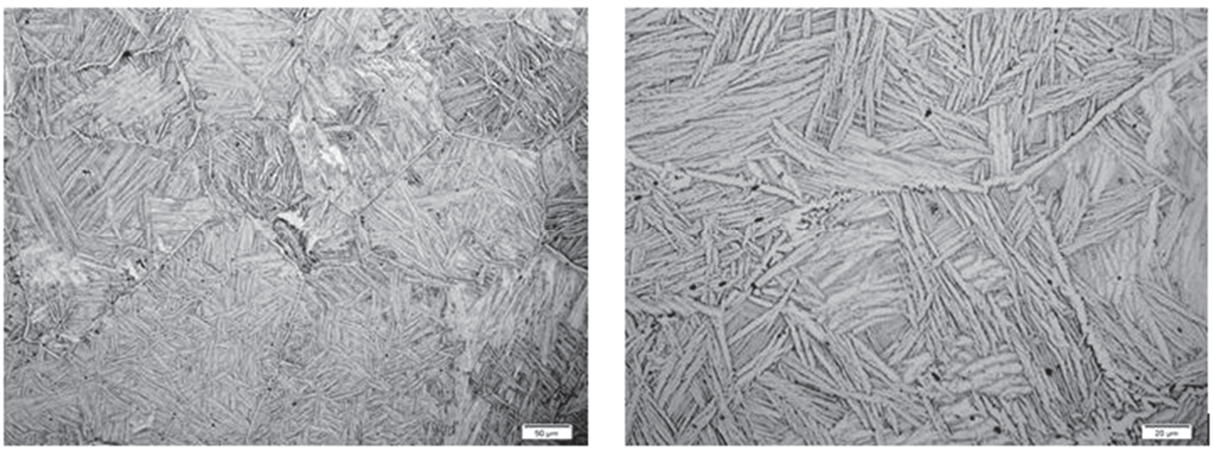


Figure 3 Microstructure of the 100 % Ti6Al4V sinters

The structure of the sintered titanium alloy was composed of a lamellar α/β structure with α' separation formed during rapid cooling. Al_2O_3 ceramics, as indicated by the structure, are characterized by a lower degree of sintering of ceramic powder. The pores are also represented by dark fields.

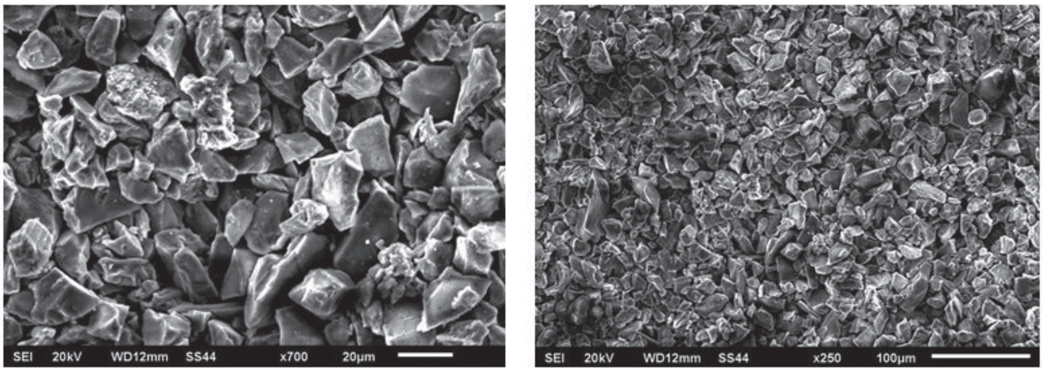


Figure 4 Microstructure of the 100 % Al_2O_3 sinters

Figure 5 shows the image of the produced Ti6Al4V/Al_2O_3 bilayered materials. The microstructure of the bilayered material was obtained by the scanning microscope Jeol JSM 5400.

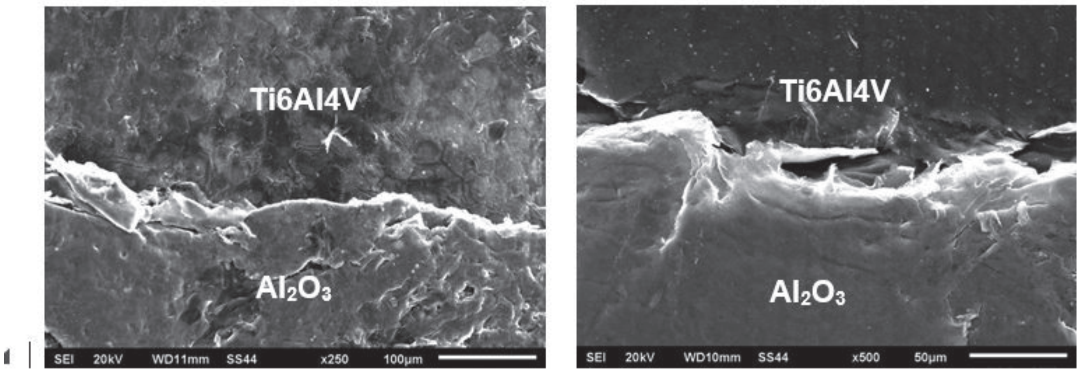
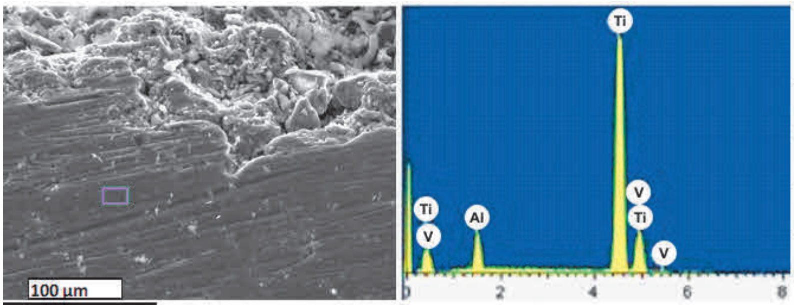
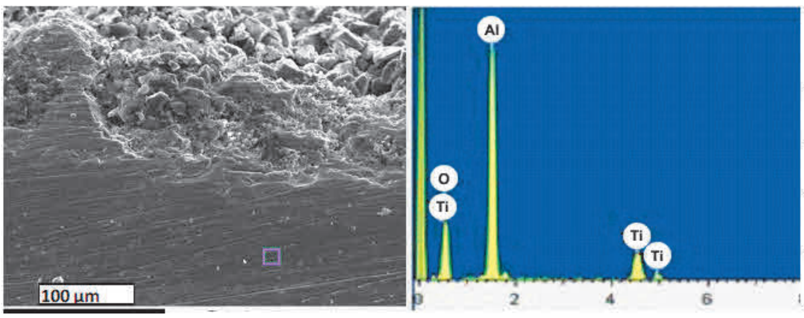


Figure 5 SEM image of bilayered material - interface zone



Element	Weight (%)	Atomic (%)
Al K	6.35	10.76
Ti K	90.06	86.02
V K	3.59	3.22
Totals	100.00	



Element	Weight (%)	Atomic (%)
O K	43.47	59.19
Al K	42.82	34.58
Ti K	13.71	6.23
Totals	100.00	

Figure 6 SEM and EDS line scan analysis of bilayered material - interface zone

Figure 5 shows that interface reaction between Ti6Al4V alloy and Al_2O_3 is irregular with a large surface roughness. These connections had no cracks or defects and were characterized by high adhesion of both materials. Porosity is visible in the ceramic part of the Al_2O_3. **Figure 6** shows the results obtained by EDS analysis, performed in the interface zone in Ti6Al4V/Al_2O_3.

Figure 6 SEM and EDS line scan analysis of bilayered material - interface zone SEM-EDS line scans showed, that inter-diffusion of some elements was verified between the two materials. Elements constituting of the titanium alloy were found, especially for Ti. Oxygen was the one that diffused the most and with the biggest range. **Figure 7** presents the relative density of the materials.

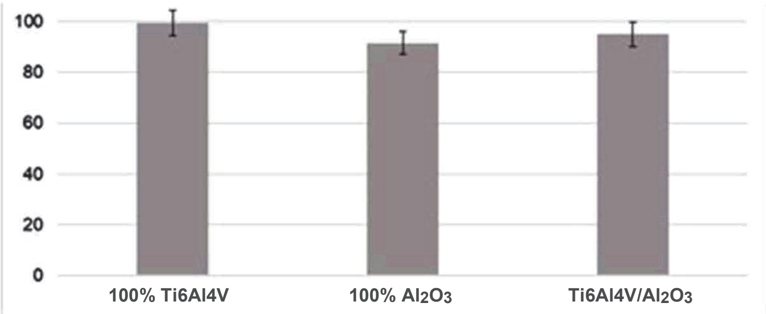


Figure 7 Relative density of the obtained materials

The highest relative density among the obtained materials was found for the sinter with 100 % Ti6Al4V. It was found that the addition of Al_2O_3 ceramics to titanium alloys in bilayered materials is the resultant of the density of both materials. This is connected with bulk density of individual powders (**Table 1**).

4. CONCLUSIONS

The method of spark plasma sintering can be successfully used for obtaining bi-layered composites (Ti6Al4V/Al_2O_3) based on Ti-6Al-4V with addition of inert ceramics Al_2O_3. As a result of the applied method and proper parameters of sintering, multilayer composites were obtained, characterized by good quality connection both materials (without cracks and delamination) with containing of elements Ti, Al, O.

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