THE EFFECT OF SPARK PLASMA SINTERING ON THE POROSITY AND MECHANICAL PROPERTIES OF Ti-15Mo ALLOY

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Abstract

Metastable β-titanium alloys are receiving much interest for various applications such as aircraft industry and medicine thanks to their excellent mechanical properties and biocompatibility. The common way of preparing the titanium alloys is hindered by its production costs. Powder metallurgy (PM) approach is a promising route for cost-effective fabrication of titanium alloys due to possibility of near net shaping.

In this study, binary biomedical Ti-15Mo alloy was prepared by PM. Gas atomized powder was sintered by spark plasma sintering (SPS) above the β-transus temperature of the studied alloy. The compaction of the powders was accomplished by short-time sintering. The effect of the time of sintering on the porosity and the microhardness in centre part as well as in periphery part of the sample was investigated. The samples revealed significant inhomogeneity - the porosity increases with the distance from the centre of the specimen. With increasing sintering times the porosity decreases and simultaneously the microhardness increases.

Keywords: Powder metallurgy, spark plasma sintering, metastable β-titanium alloys, porosity

1. INTRODUCTION

Powder metallurgy (PM) is an alternative manufacturing method which overcomes poor machinability of materials and allows production of complex shapes with minimum waste (near net shape processing) and reduced production costs [1]. PM has its advantage to design alloys with desired properties by mechanical alloying of elements [2] and controlling the porosity of the material [3]. This could be beneficial for materials used in biomedicine where low Young's modulus and high strength is required to prevent stress-shielding [4].

A number of sintering methods using high pressure at elevated temperatures have been developed such as hot pressing (HP), hot isostatic pressing (HIP), quasi-isostatic forging (QIF) or spark plasma sintering (SPS) [5, 6]. SPS, a sintering method used in this study, uses very high pulsing direct electric current to heat the powder by Joule heat. The compact microstructure can be reached at shorter times and lower temperatures than with any other commonly used methods [6-8]. However, the distribution of the Joule heat is not homogeneous during SPS. Therefore, temperature distribution in specimen during sintering is also inhomogeneous - it reaches its the highest values near the axis of the cylindrical specimen and lowest near the shell of the cylinder [9].

Titanium-based materials, especially metastable β-titanium alloys are one of the most studied alloys nowadays. Metastable β-titanium alloys have extraordinary properties such as good mechanical properties (high strength, low modulus of elasticity), biocompatibility or good corrosion resistance in many environments what makes them a perspective material to be used for example in aircraft industry or biomedicine [10]. According to basic requirements, metallic biomaterials should not contain toxic elements and should exhibit elastic moduli comparable to the one of bones [11].

The production of metastable β-Ti alloys is expensive, therefore, PM is considered as promising method to produce and design such alloys. However, during compaction at elevated temperatures, these alloys can undergo several phase transformations [12]. In this study, a metastable β Ti-15Mo powder was compacted by SPS method at temperature 800 °C for relatively short times (0,1,3 and 6 minutes). The microstructural changes during sintering were studied by scanning electron microscope in the centre and the periphery part of the cylindrical specimen as well.

2. EXPERIMENTAL MATERIALS AND METHODS

An initial powder of Ti-15Mo alloy was manufactured by gas atomization on demand by company TLS Technik GmbH & Co. Spezialpulver KG, Germany. The size of the initial powder particles was below 20 μm.

The powder was compacted in vacuum in SPS furnace manufactured by FCT Systeme GmbH. The samples of shape of cylinders about 1 cm high and 2 cm in diameter were heated up to 750 °C in one minute and then in 30 s (with the heating rate of 100 °C/min) to the desired temperature of 800 °C. Isothermal sintering was then performed for 0, 1, 3 or 6 minutes. Sintering for time 0 minute corresponds to the heating up to 800 °C and subsequent cooling down without holding. The cooling of the sample cannot be actively controlled. The example scheme of the sintering for time 3 minutes is graphically illustrated in **Figure 1**.

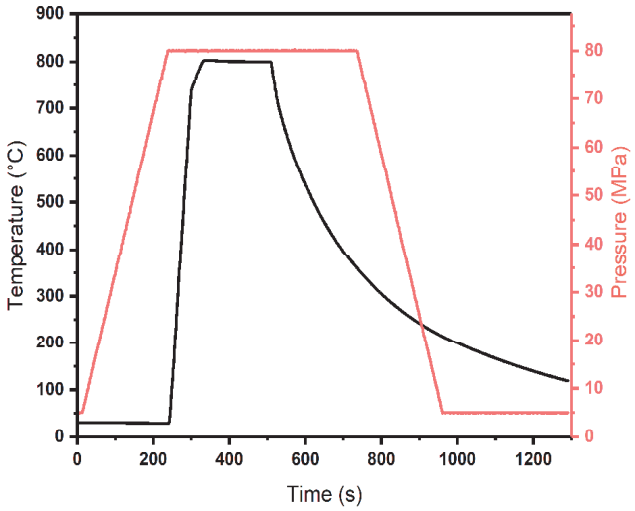

Figure 1 The development of the pressure and temperature during sintering by SPS for 3 minutes

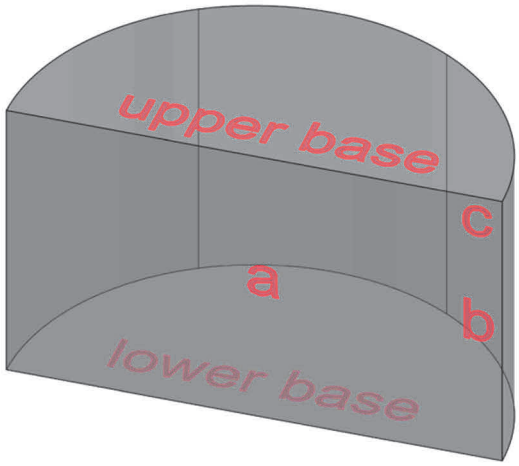

Figure 2 Schematic representation of the areas of interest (a) and b)) for microstructure observation in SEM

For microstructural observation of the compacted samples scanning electron microscopy (SEM) FEI Quanta 200F operated at 10 kV and equipped with EDS detector was used. Samples for SEM observations were prepared by mechanical grinding and polishing followed by a three-step vibratory polishing. The microstructure observation was carried out on two specific positions in each sample - in the centre of the sample and in the periphery part as it is graphically illustrated on **Figure 2**. The porosity was evaluated from secondary electron (SE) images using software ImageJ.

The microhardness of the specimens was measured by Vickers method (0.5 kgf load, 30 indents per sample) using Qness Q10a instrument with automatic evaluation of the measurement. The microhardness measurement was carried out in the centre part of each sample.

3. RESULTS

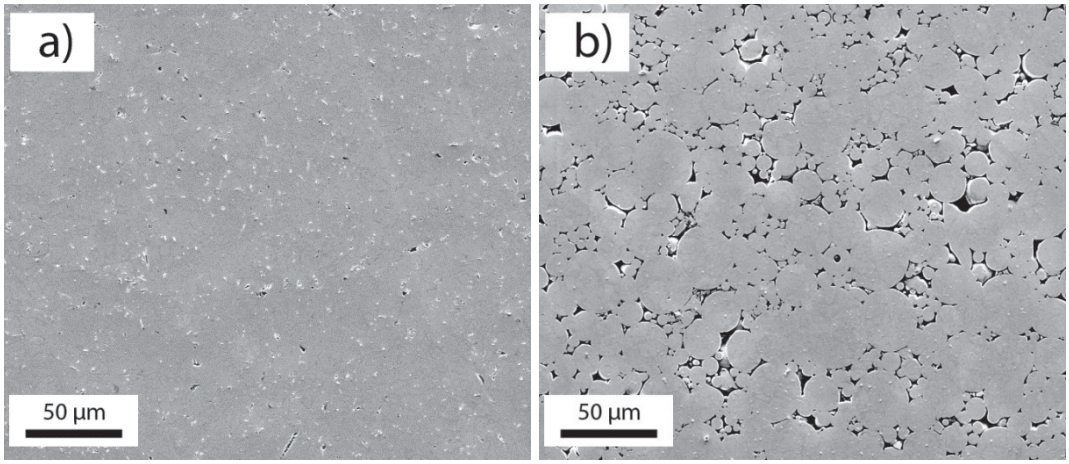

Figure 3 SE micrographs of sample sintered for 0 minutes (a) from the centre part of the sample, (b) from the periphery of the sample

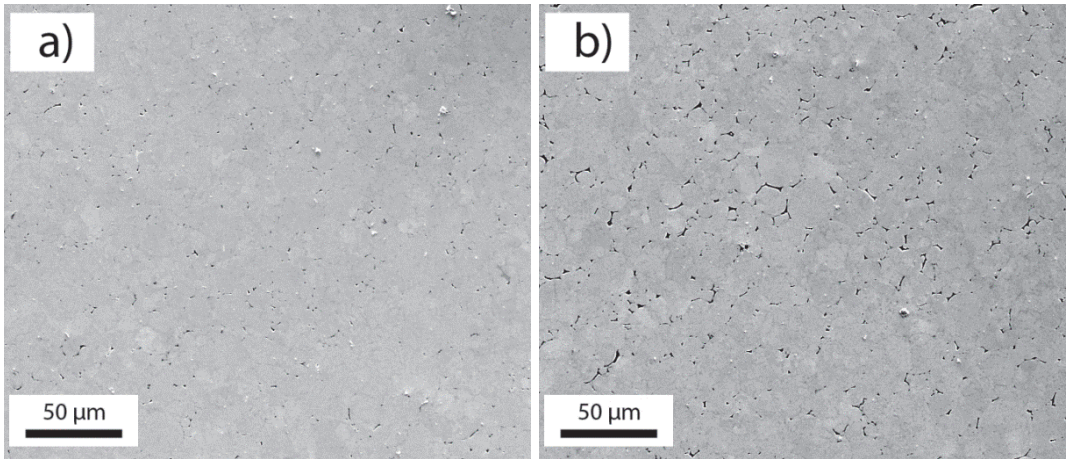

Figure 4 SE micrographs of sample sintered for 1 minute (a) from the centre part of the sample, (b) from the periphery of the sample

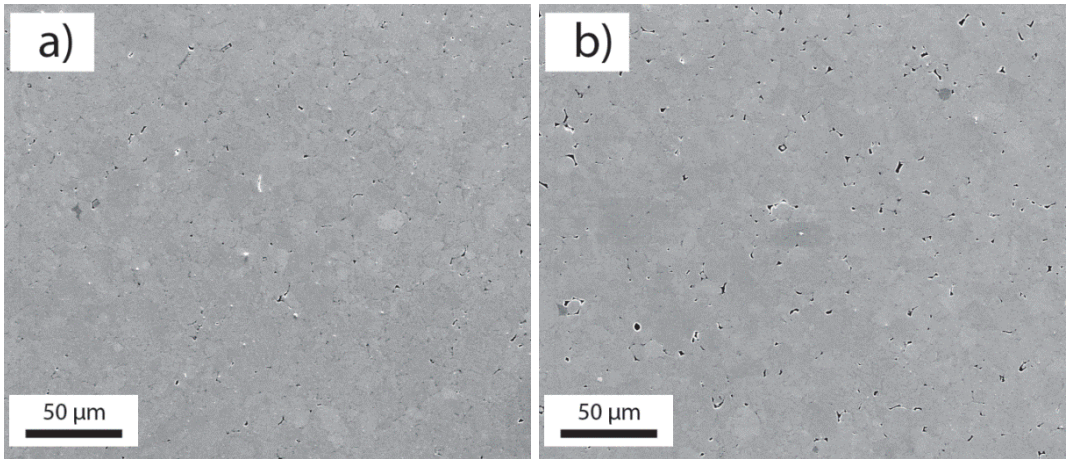

Figure 5 SE micrographs of sample sintered for 3 minutes (a) from the centre part of the sample, (b) from the periphery of the sample

Microstructure of the samples after SPS at temperature 800 °C for 0, 1, 3 and 6 minutes were observed using secondary electrons in SEM.

In the **Figures 3 to 6** SE micrographs from the centre (a) and periphery (b) part of the samples sintered for 0, 1, 3 and 6 minutes are displayed, respectively. In each image white and black areas are clearly visible. Black areas correspond to pores and white ones can be attributed to impurities from polishing which was proved by EDS (not shown here). In the periphery part initial round particles can be still recognized for short sintering times (0 and 1 minute). The fractions of pores decrease with the time of sintering, especially for the periphery part of the specimen.

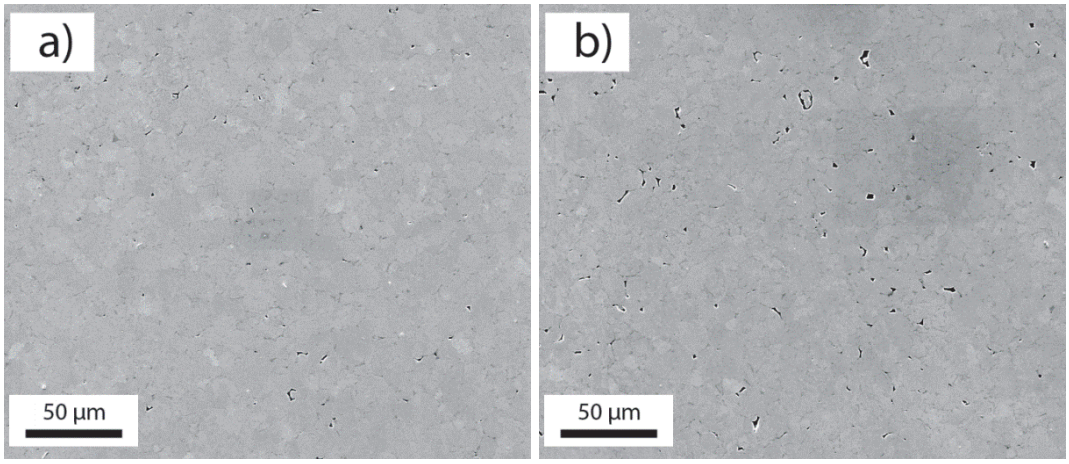

Figure 6 SE micrographs of sample sintered for 6 minutes (a) from the centre part of the sample, (b) from the periphery of the sample

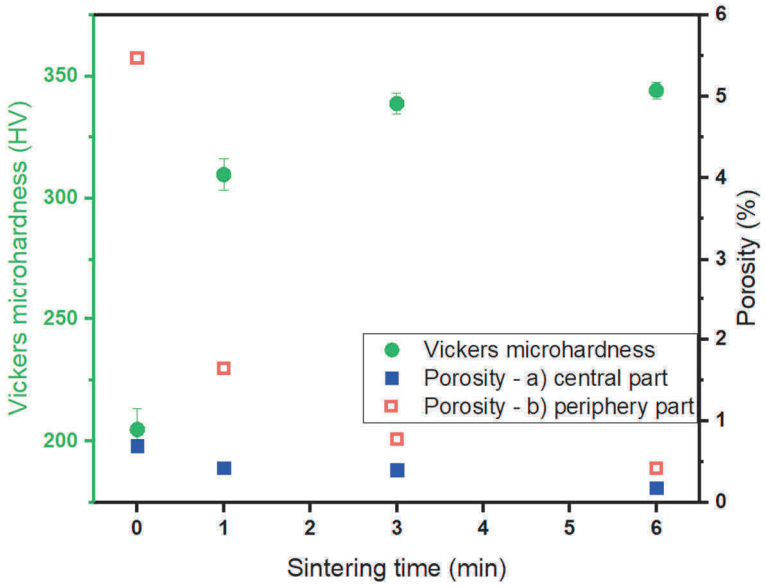

Figure 7 Dependence of the microhardness and porosity of the samples on the sintering time

The dependence of the microhardness and the porosity on the sintering time is shown in **Figure 7**. It can be clearly observed that the porosity decreases and the microhardness grows with the sintering time. The porosity in the periphery part of sample sintered for 0 minute is more than 5 % but it rapidly decreases after sintering for 1 minute. It also corresponds to the rapid increase of the microhardness for sample after SPS for 1 minute. The difference in porosity between centre and periphery parts is biggest for sample sintered for 0 minute and

decreases with the sintering time which means that material become more homogeneous with the increasing sintering time. For sintering time 3 and 6 minutes the material become denser, thus, the microhardness saturates at around 350 HV.

4. DISCUSSION

In **Figures 3 to 6** the microstructure of the Ti-15Mo powder sintered for 0, 1, 3 and 6 minutes is shown, respectively. The porosity of the samples decreases with the increasing sintering time. Porosity below 1% is achieved after sintering for 3 and 6 minutes disregarding the position in the sample (discussed below). The temperature of 800°C is therefore appropriate for spark plasma sintering of gas atomized powder of Ti-15Mo alloy with the size of 20 μm. Note that the required sintering time and temperature generally depend on the powder particles size and morphology [13]. Significant compaction (porosity < 5%) took place also after sintering at 800°C/0 min. Such sample spent only about 1 minute at temperatures above 700°C.

The residual porosity also changes with the distance from the centre of the cylindrical sample. It grows from the cylindrical axis to the shell of the cylinder, but it does not change along the cylinder axis of the cylinder. It was proved by measurement near the edge of the base - marked as point *c* in **Figure 2** (not shown here). It corresponds to temperature distribution modelling in [9], showing that the temperature dependence on the distance from axis of the cylinder has a Gaussian course with a maximum in the centre of the specimen. The densest condition of sintered Ti-15Mo powder was achieved by sintering for 6 minutes in the centre part with porosity 0.184 %.

The microhardness measurement (**Figure 7**) exhibits a strong dependence of microhardness on the relative density of the material. For sintering time 0 and 1 minute the porosity in the periphery part of the sample is more than 1 % which indicates that the sintering is not complete. This results in a porous and softer material. The microhardness increases with the increasing sintering time and after sintering for 3 and 6 minutes the microhardness saturates at 340 HV. The microhardness is significantly higher than the microhardness of the β solution treated Ti-15Mo alloy prepared by standard casting (278 HV) [14]. However, the microhardness does not achieve as high values as for SPD-deformed Ti-15Mo alloy (458 HV) [15] or after annealing to two-phase α+β condition (491 HV) [14]. The increased hardness when compared to the solution treated material might be caused by precipitation of small particles of other phases (α or ω) or by increased oxygen content. Phase and chemical composition will be characterized in detail during future research.

5. CONCLUSIONS

The porosity of the Ti-15Mo alloy prepared by spark plasma sintering at temperature 800 °C for times 0, 1, 3 and 6 minutes was studied in centre and periphery parts of the samples by scanning electron microscopy. The dependence of the porosity and microhardness on the sintering time was analyzed. The main conclusion from this study can be drawn as:

- The porosity of the material after spark plasma sintering decreases with the sintering time.
- The material is denser in the centre part of the sample than in the periphery part.
- For sintering times of 3 and 6 minutes, the porosity is lower than 1 % throughout the sample.
- Microhardness increases with longer sintering time due to the decreasing porosity of the material. Microhardness exceeds the microhardness of comparable bulk material prepared by standard casting.

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