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Original paper

Argon-shielded hot pressing of titanium alloy (Ti6Al4V) powders

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The paper presents the method of the argon-shielded hot pressing of titanium alloy (Ti6A14V) powder (used in medical industry). The powders produced in the GA (gas atomization) process and in the HDH (hydride–dehydride) process were used in the experiments. A pressing process was conducted at a temperature of 800–850 °C for different lengths of time. An unoxidized sintered material, nearly as dense as a solid material and having a lamellar structure (α+β), was obtained from the titanium alloy powder produced in the HDH process.

Key words: *powders, metallurgy, titanium*

1. Introduction

Titanium and its alloys are highly compatible and corrosion resistant and for this reason they are successfully used in medical industry [1]–[2], especially in implantology and prosthetics. They prevent electrochemical reactions and owing to their high mechanical strength and hardness, the products made of titanium and its alloys are highly durable. However, because of the high costs of such materials and the properties of the latter, the costs of manufacturing the products from titanium and its alloys are quite high [3]–[7]. Titanium alloy Ti6Al4V is commonly used for implants [2], [8]. It has very good strength and plastic properties. The products made of this material are manufactured by conventional methods using metallurgical processes, plastic working and heat treatment [9]. The conventional metallurgy of titanium and its alloys supplies a wide spectrum of materials with different properties. But their further improvement through traditional mechanical processing and heat treatment becomes increasingly less effective [10]. It seems that such techniques as powder metallurgy should be developed for this purpose to produce a material with predetermined service properties and to reduce the high production costs (due to the properties of titanium which at high temperatures shows close affinity to oxygen, nitrogen and carbon [2]). Because of the high production costs, titanium alloy Ti6Al4V is used only to a limited extent. Hence it seems worthwhile to search for new methods of bonding powders of titanium and its alloys, which will make it possible to reduce the costs in comparison with the conventional manufacturing methods and such powder metallurgy methods as hot isostatic pressing (HIP) [11]–[13]. This paper presents a new argon-shielded hot pressing method and preliminary structural examinations of the compacts obtained.

2. Materials and methods

Argon-shielded hot pressing

Titanium alloy Ti6Al4V powder [14] produced by gas atomization (GA) and by the HDH (hydride–

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dehydride) process was used in the argon-shielded hot pressing experiment. After GA the powder grains were spherical in shape (figure 1b), whereas those produced by HDH were irregular in shape (figure 1a). The powder grains were below 45 μm in size. The different methods of manufacturing the powders resulted also in their different chemical composition, which is shown, respectively, in tables 1 and 2.

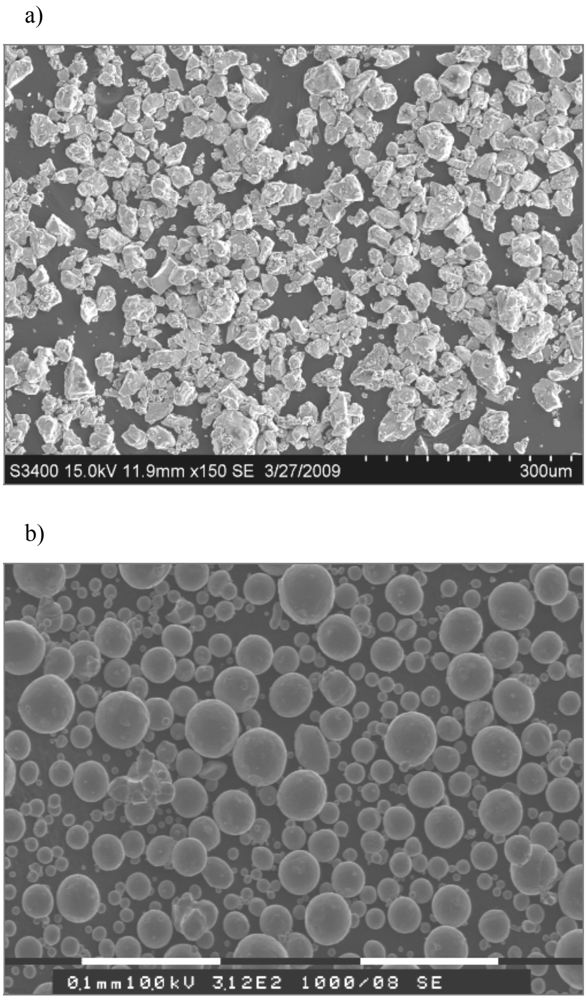

Fig. 1. Titanium alloy Ti6Al4V powder grains under scanning microscope produced by: a) HDH, b) GA

Table 1. Chemical composition of titanium alloy Ti6Al4V powder produced in HDH process (%)

Ti	Al	V	Fe	Zr	O	N	H
90±1	6±1	4±1	0.03	0.03	max 0.55	max 0.5	max 0.3

Table 2. Chemical composition of titanium alloy Ti6Al4V powder produced by GA (%)

Ti	Al	V	Fe	Zr	O	N	H	C
90	5.9	3.9	0.19	0.03	0.12	0.01	0.004	0.01

Because of the close affinity of titanium to oxygen, nitrogen and carbon [2] at elevated temperatures (above 237.5 °C), hot pressing was conducted in argon shield 5.0. Taking into consideration the high process temperature, the powder was pressed in a die made of high-temperature creep-resisting nickel alloy INCONEL.625. The die was a split device consisting of an external part and an internal part (figure 2). In order to decrease the forces needed to push the internal die out of the external die after the process, the former was designed as based on a cone with an angle ensuring no self-locking. In addition, high-temperature lubricant Molykote P37 was used to provide lubrication between the dies. Since the die was induction-heated, it was separated by corundum brick from the top plate to enhance the action of the eddy currents produced by the alternating magnetic field generated by an inductor and to reduce their action on the top plate (made of a different material with different ferromagnetic properties). The inductor had the shape of a four-coil winding with a diameter and a coil spacing ensuring uniform heating of the die over time. The induction heating system included a transformer (to which the inductor was connected), a GRC20 generator with a rated power of 20 kW, a control cubicle and a closed feedwater system (responsible for cooling the inductor and the generator during heating) (figure 3).

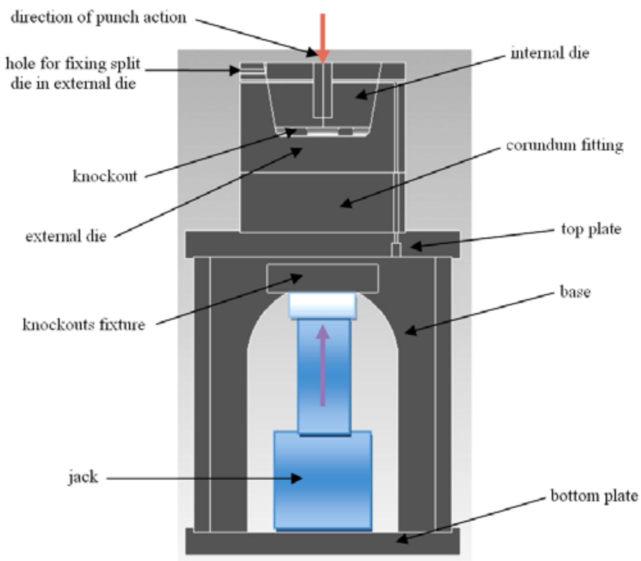

Fig. 2. Schematic of device for hot compaction of titanium powders

Argon was supplied to the top plate, flowing into the die through the hole marked in figure 4. Being denser than air (argon 1.784 kg/m^3; air 1.2 kg/m^3), argon would push out air.

Fig. 3. Device for argon-shielded hot pressing of titanium and titanium alloy powders

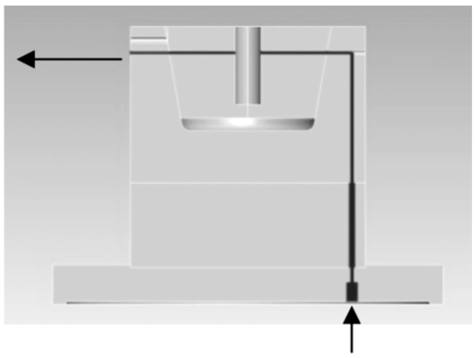

Fig. 4. Flow of argon in hot pressing device

Titanium alloy powder pressing by GA was conducted at a temperature of 800–850 °C (i.e. above the temperature of titanium recrystallization) and under a pressure of 160 MPa. The powder was pressed under the maximum pressure for 15 min, 35 min and 60 min. Twenty grams of the powder would be poured into the unheated die. Then argon was let in to create an argon shield for the powder and the induction heating of the die started. When the process ended the samples, 20 mm in diameter, would be obtained. The powder produced using the HDH process was pressed for only 60 min.

The samples were subjected to metallographic examinations. 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 material [15], [16].

3. Results

Figure 5 shows the change in porosity, depending on sintering time. It appears that the porosity of titanium alloy Ti6Al4V powder hot pressed in an argon shield increases during the first 30 minutes. In the hot pressing process, nearly 100% material density (4.43 g/cm^3 – the density of titanium Ti6Al4V [15], [16]) at a material porosity of just under 0.5% can be achieved.

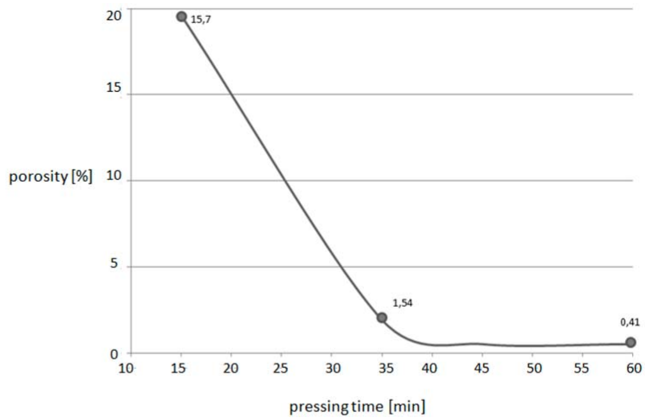

Fig. 5. Porosity versus argon-shielded hot pressing time for Ti6A14V (GA) sample

Figure 6 shows the change in porosity, the distance covered by the ram and the evolution of the structure in the course of hot pressing. In the first stage (from the start to the point *A*), the powder is pressed mainly as a result of plastic deformation, mutual adjustment and displacement of powder grains. It can be assumed that the main stage occurs between the points *A* and *B* where the powder is simultaneously sintered and pressed. The percentage of sintering increases with time and so it can be assumed that for the time longer than *B* = 30 min only sintering takes place.

The structure of the titanium alloy hot pressed in an argon shield for 15 minutes shows still distinct shapes of powder grains. The grain structure is composed of phase α in a phase β matrix (figure 7).

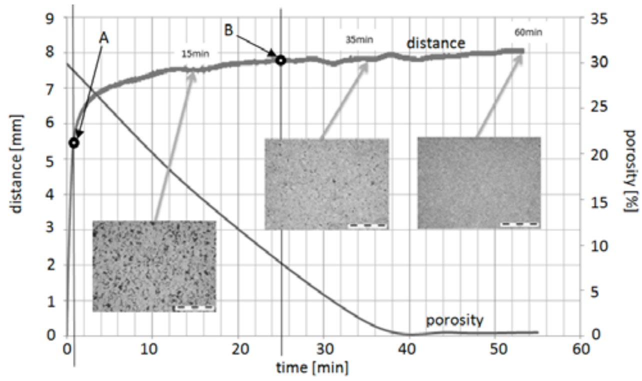

Fig. 6. Ram advance and porosity change during argon-shielded hot pressing of titanium alloy powder Ti6Al4V (GA)

When the process time is extended to 35 min, the volume and the amount of porosities (so substantial in the previous structure) decrease and as a result of recrystallization and pressing a more compact structure than that of the material pressed and sintered for 15 min is obtained. The material obtained has a lamellar structure (average grain size of 30 μm) with indistinct grains of original phase β. The lamellar structure is characterized by an irregular distribution of the phase α lamellae in the phase β matrix (figure 8), which makes it different from the chain structure where most of the phase α lamellae are oriented in one direction [7].

The structure of the material pressed for 60 min is also lamellar with the average grain size the same as that of the material subjected to pressing for 35 min, but no pores are visible (figure 9).

A similar sintering process carried out for the HDH powder took place over 60 min (which proved to be optimal for the powder produced by gas atomization). The process proceeded at a temperature of 800÷850 °C, yielding a bimodal structure [7] with globular grains of the pure phase α in an acicular matrix (the mixture of lamellae) α + β (figure 10).

The difference in the structure between the two kinds of powder can be due to a slightly different chemical composition and shape of the two powders or due to slight differences in process temperature. According to the literature, a difference of 20 °C in a temperature range of 800–850 °C may lead to a dif-

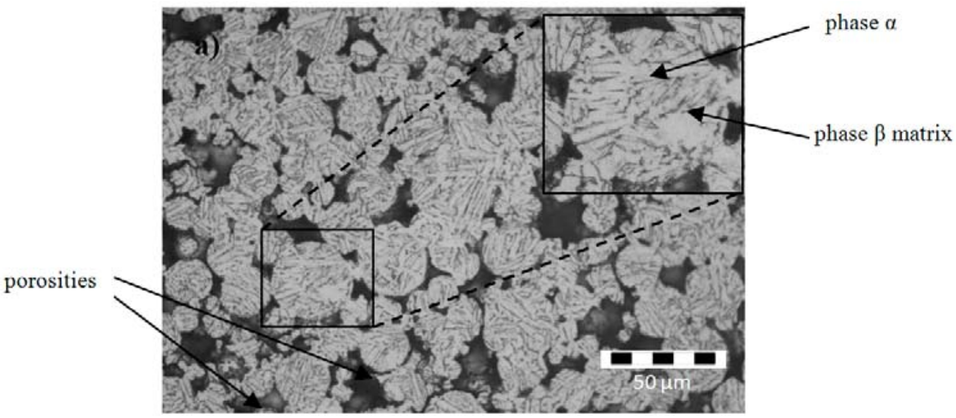

Fig. 7. Structure of titanium alloy Ti6Al4V (GA) pressed in argon shield at temperature of 800÷850 °C for 15 min

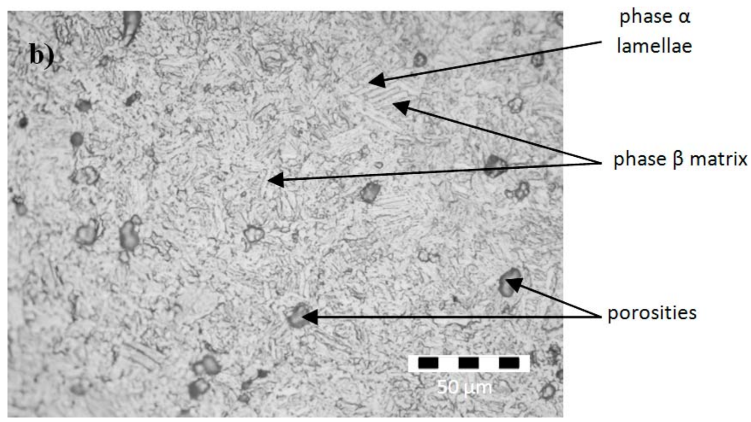

Fig. 8. Structure of titanium alloy Ti6Al4V (GA) pressed in argon shield at temperature of 800÷850 °C for 35 min

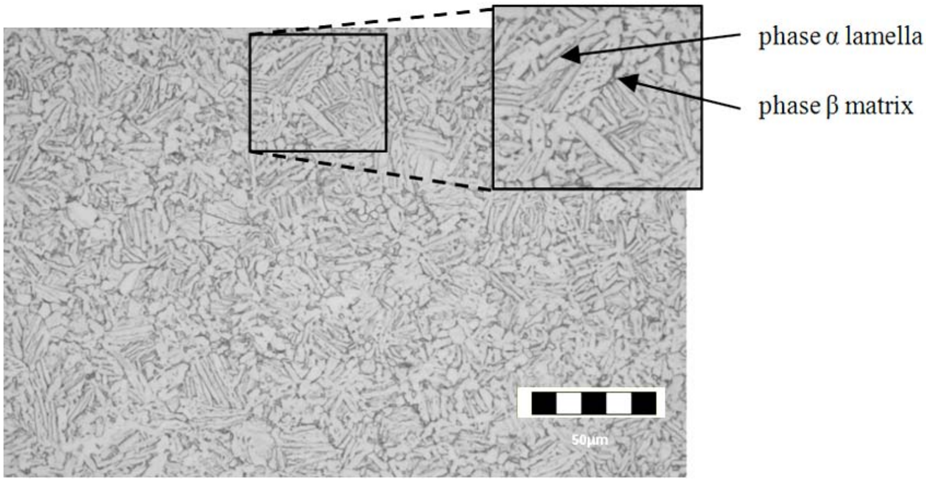

Fig. 9. Structure of titanium alloy Ti6Al4V (GA) pressed in argon shield at temperature of 800÷850 °C for 60 min

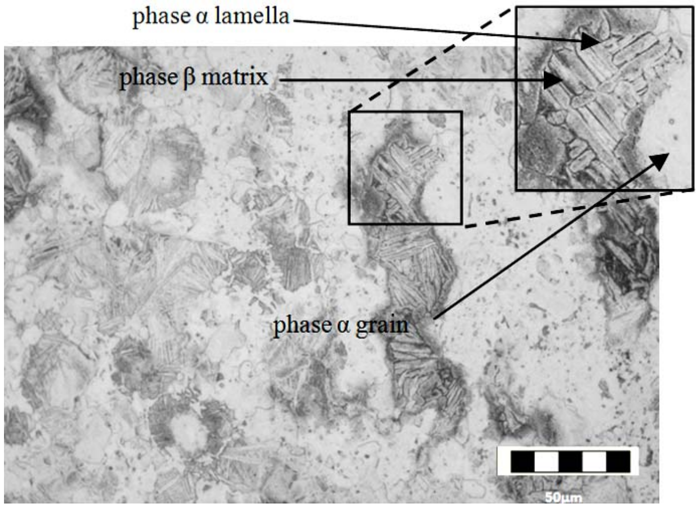

Fig. 10. Structure of titanium alloy Ti6Al4V (HDH) pressed in argon shield at temperature of 800÷850 °C for 60 min

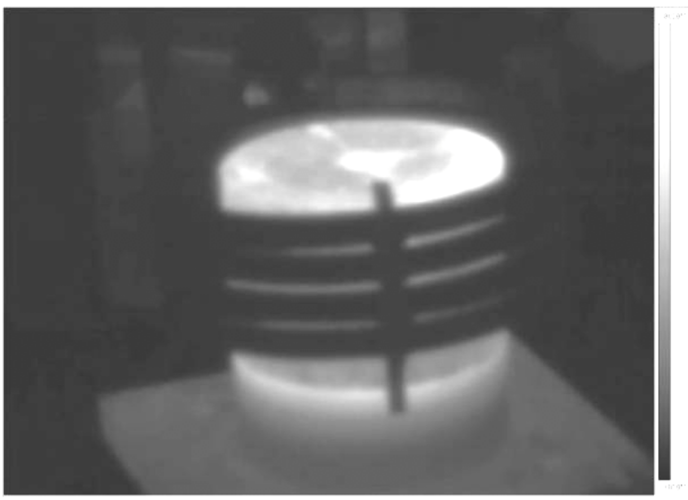

Fig. 11. Thermal photograph of die during argon-shielded hot pressing of titanium alloy Ti6Al4V powder

ferent structure. Since the process temperature was controlled by a thermovision camera (figure 11) recording the temperature of the tools' surface, the temperature differences exceeding 20 °C might have occurred between the surface of the tools and the powder being sintered. Because of induction heating no thermocouple could be used to control the process temperature.

4. Conclusions

1. A special device in which powder can be simultaneously pressed and sintered was built. The device was used to make solid material from GA produced titanium Ti6A14V powder. It appears that since it combines pressing and sintering and does not require expensive equipment, the argon-shielded pressing technology could be used (after some improvements) for more economic industrial production of implants from titanium alloy powders.

2. The optimum conditions for the argon-shielded hot pressing of Ti6Al4V powder produced by GA were determined to be: the time – 60 min, the temperature 800–850 °C and the pressure of 160 MPa. Almost solid material was produced in these conditions.

3. Further research needs to be done for powder produced by HDH in order to find out why a lamellar-globular structure is obtained in this case.

4. Further research is also needed to determine the process parameters (time, temperature and pressure) optimum with regard to the strength and biocompatibility of the material obtained.

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