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An Investigation of Injection Parameters of Ti-6Al-7NbFabricated by Powder Injection Molding

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ABSTRACT

In this study, the optimized injection parameters for a feedstock obtained by mixing the powder of Ti-6Al-7Nb and 31wt% binder system (comprised of high-density polyethylene, Polypropylene, PEG 20000, paraffin wax and stearic acid), were determined. In order to investigate the fabrication of a component, flow rate, injection pressure, holding pressure, injection temperature and mold temperature were studied at three levels to obtain the optimum injection parameters. It was proven that for feedstock comprised of 69wt% Ti-6Al-7Nb powder, the suitable set of injection parameters were as follows: flow rate of 20 cm3/s, injection pressure of 1300 bar, holding pressure of 60%, injection temperature of 140 °C and mold temperature of 60 °C. At the end of the experiments the tensile strength of specimens fabricated with optimum parameters, were as high as 631 MPa.

Keywords: Ti-6Al-7Nb, Powder Injection Molding, Injection, Sintering.

1. INTRODUCTION

Ti and its alloys have high strength and good corrosion resistance as well as excellent biocompatibility. Hence, they are used for various automotive, aerospace and medical applications. Because of high strength and good corrosion resistance, high purity titaniumhas been utilized as a biomaterial for many years. A big difference between elasticity modulus of this alloy and that of bone (102-105 GPa and 4-30 GPa, respectively) causes bone structure to be destroyed easily. In order to solve this problem, different alloys of Ti have been developed. While the elasticity modulus of α-phase Ti alloys is 100-120 GPa, this value for β-phase alloys is as low as 60-80 GPa. Ti-6Al-4V, Ti-15Mo and Ti-6Al-7Nb are good examples of biocompatible Ti alloys. It was determined that although addition of some elements such as V, Ni and Al increases material strength, it causes bone cells breakage and some health problems. As the findings of some studies indicate, β phase or semi β phase Ti alloys do not exhibit such a property. Niobium (Nb) is an important element acting as a β phase stabilizer and it simultaneously allows obtaining high biocompatibility. In addition, the alloy of Ti-6Al-7Nb is preferred because of good strength, high wear and fatigue resistance and good machinability. As a result, Ti-6Al-7Nb alloy is a very attractive material for implant applications such as dental roots, joint endoprosthesis, and surgical implant components for hip replacement[1-7].

However, the high cost of components made of Ti, limits the areas of its application. The time of production is the main reason for such a high cost[8].

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Powder Injection Molding (PIM) could reduce the production costs and improve the structure of components in many areas. PIM is an economical production method for components with small and complex geometry. In addition, PIM application causes unique flexibility in components design which cannot be achieved by other manufacturing techniques[9, 10].These features of PIM result in higher desirability for production of components made of Ti and its alloys.

Recently, Powder Injection Molding has found an important status in production of components of titanium and its alloys. The principles of PIM production for Ti and its alloys are almost the same as other materials like stainless steel and ceramics. However, the process parameters must be more carefully optimized due to high reactivity of the material. There are many titanium powder and feedstock suppliers in the market whose technical knowledge and data of binder systems, and injection molding and sintering parameters are mostly confidential.

In light of the existing literature, it is known that the production of titanium and titanium alloys components by PIM is increasing. Although some researchers have simulated the injection process of stainless steel feedstock to optimize the parameters[11, 12], there are no efficient optimizations regarding powder and binder system and injection parameters (injection temperature, injection pressure, injection flow rate, mold temperature) for titanium based feedstocks [7, 13-19].

The aim of this study was to determine the required optimum injection parameters of PIM in biomedical applications to produce components of Ti-6Al-7Nb instead of Ti-6Al-4V(the latter is harmful for human body due to vanadium toxic effects).

2. MATERIAL AND METHOD

2.1. Powder and Binder

In this study, gas-atomized spherical Ti-6Al-7Nb powder with size of <25 µm and repose angle of 58° was supplied by TLS Technik GmbH company. The chemical properties of the powder used in the experiments are given in Table 1.

In PIM, the characteristics of the binder are almost as important as those of the powder. The required binder should shape powders in desired geometry and exhibit properties such as good packing and adequate strength of green and brown part before sintering; it should also be completely extracted out of component after sintering. Powder and binder are mixed to form a homogenous feedstock.

Table 1. The chemical properties of Ti-6Al-7Nb powder

Element	Al	Nb	Fe	O	N	H	Ti
%	5.82	6.58	0.050	0.190	0.006	0.001	balance

The feedstock should be homogenous and have low viscosity so that it can be injected easily. These properties are provided by the aid of the binder system. The binder should consist of multiple elements which are needed to be gradually and sequentially extracted from the injected components. The binder system should be cheap and also possess some properties such as long shelf life and low thermal expansion coefficient. It should also be chemically neutral toward the powder [20, 21].Accordingly, high density polyethylene, polypropylene, PEG 20000, paraffin and stearic acid have been used as the binder system in this study.

2.2. Powder Injection Molding

ArburgAllrounder220S injection machine was used to perform the injection process. The standard tensile specimens were injected using a standard mold manufactured based on dimensions presented by Metal Powder Industries Federation [22].

2.3. Debinding Process

Solvent and thermal debinding steps were applied to extract the binder out of injected components. Solvent debinding was carried out by immersing the components inside high purity heptane with temperature of 60°C for almost 20 hours. The specimens were then dried for 12 hours under the atmosphere of 60°C temperature. The thermal debinding process was completed in an atmosphere controlled furnace with high purity (99.999%) argon gas, passing through copper chips to ensure its purity. Figure 1 depicts the sequences of thermal debinding process. The heating ramping of1°C/min was selected to be constant for all sequences of thermal debinding.

2.4. Sintering Process

Sintering process was carried out after thermal debinding (pre-sintering step) in the same furnace and atmosphere. Sintering heating rate, temperature and dwell time were 3°C/min, 1250°C and60 min, respectively. Argon gas flow rate was constant and was selected to be as high as 5 L/min. After sintering, the specimens were cooled slowly with cooling ramping of 5°C/min.

2.5. Design of Experiment

Based on the literature review and some pervious experiments, the important process parameters, exhibited in Table 2, were selected to be studied. The effect of injection parameters on sintered components tensile strength was determined.

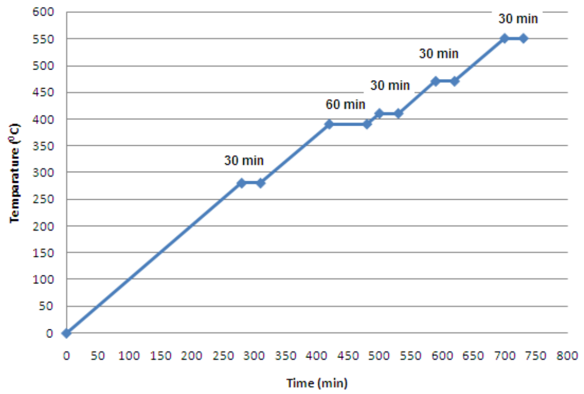

Figure 1. The sequences of thermal debinding

2. 6. Tensile Experiments

The tensile experiments were carried out by 50 kN Instron tensile machine, with cross-head speed of 1 mm/min, according to TS EN ISO 6892-1 standard.

3 . RESULTS AND DISCUSSION

As a result of our experiments, specimens made of Ti-6Al-7Nb feedstocks with powder loading of 69% reached the highest tensile strength as high as 590 MPa. As another result of the experiments, the highest tensile strength of 590 MPa was achieved for components made of Ti-6Al-7Nb with powder loading of 69%. The optimum injection parameters leading to the highest tensile strength were determined as listed in Table 3. Using these optimum injection parameters, components were produced and sintered, giving tensile strength of 631MPa. According to Table 4 and Figure 2, the obtained optimum injection parameters were evaluated.

Table 2. The injection parameters

Injection parameters	Injection flow rate (cm^3/s)	Injection pressure (bar)	Holding pressure (%)	Injection temperature (°C)	Mold temperature (°C)
1	20	1100	50	140	40
2	25	1200	60	150	50
3	30	1300	70	160	60

Table 3. The optimum injection parameters for feedstock Ti-6Al-7Nb

Flow rate (cm^3/s)	Injection pressure (bar)	Holding pressure (%)	Injection temperature (ºC)	Mold temperature (ºC)
20	1300	60	140	60

high structural porosity. They have also mentioned that the furnace atmosphere and its control have a noticeable impact on the amount of porosity inside. This is while Limberget al.[16], who have utilized pressurized atmosphere and shot peening, have reported a tensile strength of 630 MPa. In the present study, the specimens were sintered without shot peening by normal atmosphere controlled furnace. The effect of shot peening on the mechanical properties of specimens is proven[26]. In this study, the tensile strength of 631MPa was developed without shot peening and with lower sintering temperature and dwell time (1250°C, 60 min) than those of other studies.

Table 4. The comparison of tensile strength values obtained in different conditions

Feedstock	The average tensile strength obtained for design of experiment (MPa)	The highest tensile strength obtained based on optimized injection parameters (MPa)	Wrought material tensile strength (MPa)
Ti-6Al-7Nb	590	631	900 (ASTM F1295-11)

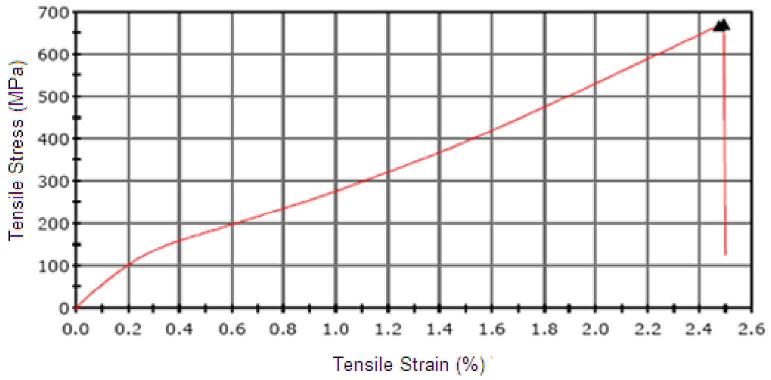

Figure 2. Tensile stress versus strain for the specimen made of Ti-6Al-7Nb feedstock sintered with optimized injection parameters

As it is presented in Figure 2, by applying the tensile strength test, the sintered material behave like cast iron. Components made by powder metallurgy also behave like cast iron[23].

Limberg et al.[16]studied the tensile strength of Ti 45Al 5Nb 0.2B 0.2C made by PIM, and reported the highest value of 630 MPa for specimens sintered and then shot peened under argon gas with pressure of 800kPa.Haoming et al.[24] investigated the mechanical properties of Ti-45Al-8.5Nb-(W, B, Y) specimens which reached the tensile strength of 382MPa. Gerling et al.[25] have reported the tensile strength of Ti-47Al-4 (Nb, Mn, Cr, Si, B) to be as high as 260 MPa for specimens sintered for 2 hours by HIP method.

Haoming[24] and Gerling [25] have pointed out that the main reason for low tensile strength of specimen was

Zhao et al.[7] compared the mechanical properties of high purity titanium and its alloys consisting of Nb with varying percentages (10, 16 and 22%wt). As a result of their experiment, the Nb effect was determined. Ti-10Nb exhibited the tensile strength of 638MPa, whereas the amount for alloys Ti-16Nb and Ti-22Nb are 687 and 754MPa, respectively. It was concluded that Nb element increases the amount of tensile strength. Bidaux et al.[19]investigated the mechanical properties of Ti alloy with 17%wt Nb. The sintering process was carried out under the argon atmosphere with applied sintering temperature and dwell time of 1300°C and four hours, respectively, which resulted in the tensile strength of 749MPa.

Zhao[7] and Bidaux [19] determined the effect of Nb ratio on porosity of micro-structure. In both sintering and HIP processes, the amount of tensile strength increased when Nb ratio increased. In this study, the Ti alloy with 7% Nb was sintered at 1250°C for 60 minutes under argon atmosphere which resulted in a high tensile strength of 631 MPa. The tensile strength is however a little bit lower than the values developed by Zhao and Bidaux. This is due to using lower Nb (7%), and also lower sintering temperature, dwell time and a different atmosphere.

3.1. Evaluation of Molding Parameters

To produce a defect-free component in PIM, it is necessity to prepare a suitable feedstock, and develop optimized molding and sintering conditions. Important molding parameters include injection temperature, injection pressure, flow rate, holding pressure and mold temperature[27].

Injection pressure: In this study, the specimens were injected under the injection pressure of three levels of 1100, 1200 and 1300 bar. In the existing literature, it has been pointed out that utilizing high injection pressures can result in defects such as powder-binder separation, increased mold residual stress, and polymeric chains breakage[28, 29].This condition will result in deteriorated mechanical properties of component. It also should be mentioned that the powder loading affects the injection pressure directly[30, 31].As shown in Figure 3, the weld line was seen in specimens injected at low pressure of 1100 bar. For the feedstock of Ti-6Al-7Nb with powder loading of 69%, the injection pressure of 1300 bar was proven to be the appropriate injection pressure.

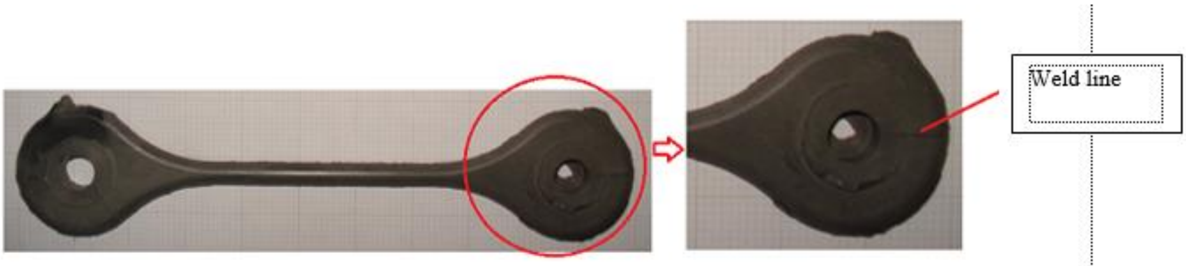

Figure 3. Specimen injected at pressure of 1100 bar and flow rate of 30 cm^3/s

Flow rate: While the high flow rate can result in porosity and jetting specially in thick sections, the low flow rate deteriorates the surface quality[32]. In addition, high flow rate and injection pressure may contribute to powder-binder separation. High flow rate also can increase component surface stresses, which results in component warpage [28, 32]. In this study, no warpage, surface defects, and porosity were seen in specimens injected at three levels of flow rates of 20-25-30 cm^3/s. However, in specimens injected with high flow rates, there were some signs of color changes which were interpreted to be the result of powder-binder separation (Figure 4). Because there was no powder-binder separation in the specimen injected with flow rate of 20 cm^3/s, exhibiting the highest tensile strength, this value was selected as a suitable flow rate.

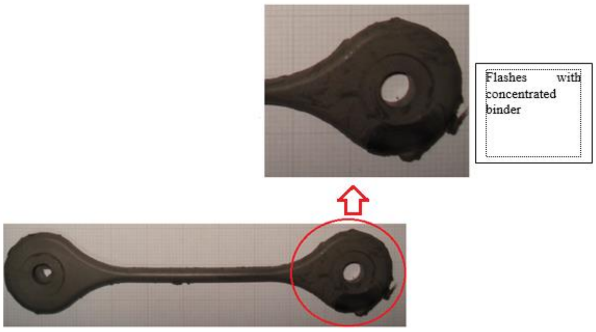

Figure 4. Specimen injected with pressure of 1100 bar, and flow rate of 30 cm^3/s

Injection temperature: The higher the injection temperature is, the better flowability and moldability of feedstock will be. However, high injection temperatures have some disadvantages as the need for higher energy, and increasing the cooling time to eject the injected component from the mold. In addition, high injection temperatures cancause extra thermal stresses which negatively affect the dimensional accuracy of components [29]. In the present study, the dimensional changes of sintered component, known as shrinkage, for specimens injected at temperature of 140°C and 160°C were 11.8 and 13.39%, respectively. Regarding these points, the temperature of 140°C was selected as acceptable injection temperature.

Mold temperature: The other important injection parameter which affects the quality of the component is mold temperature. In PIM, higher mold temperatures result in decreased temperature fluctuations during injection process. It also has a positive effect on feedstock rheological properties[33]. In this study, three levels for mold temperature were selected, as 40, 50 and 60°C, among which the 60°C was the most appropriate value regarding the obtained tensile strength.

Holding pressure: Low holding pressure can cause porosity and slumping. Holding pressure, which guarantees the feedstock flow inside the mold until the end of gate solidification, prevents thermal stresses during the cooling stage[34]. It is also noticeable that holding pressure should not be considered as an independent parameter. For example, low holding pressure and high mold temperature may result in some defects in injected components. Specimens were injected inside a mold with temperatures of 40, 50, 60°C and holding pressures of 50, 60, 70 % of injection pressure. Under such a condition, no defects related to holding pressure (defects such as porosity and slumping) were seen in specimens. For injection process with mold temperature of 60°C, the optimum holding pressure of 60% was selected.

3.2. Evaluation of The Microstructure of Specimens

The optic-microscopic views, SEM and tensile fracture surface views for sintered specimen with the highest tensile strength of Ti-6Al-7Nb are illustrated in Figures 5, 6 and 7, respectively. In this specimen with the tensile strength of 631 MPa, the phases of α+β were present at the structure. As it is graphically shown in the optic, SEM and tensile fracture surface view, the sintering occurred completely in specimen injected at optimum injection conditions.

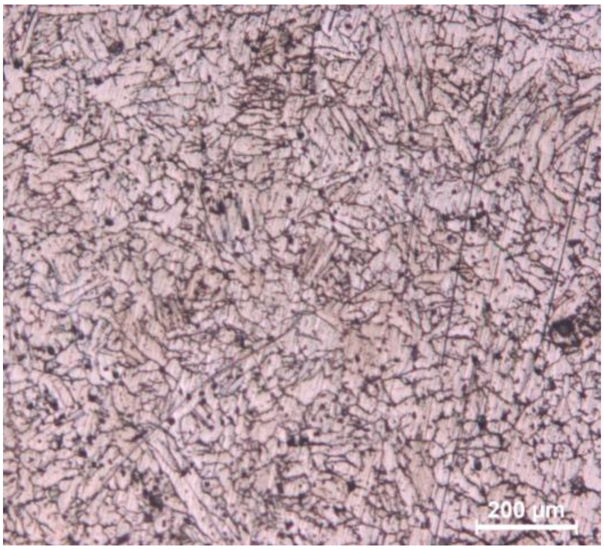

Figure 5. Optic microscopic views of sintered Ti-6Al-7Nb

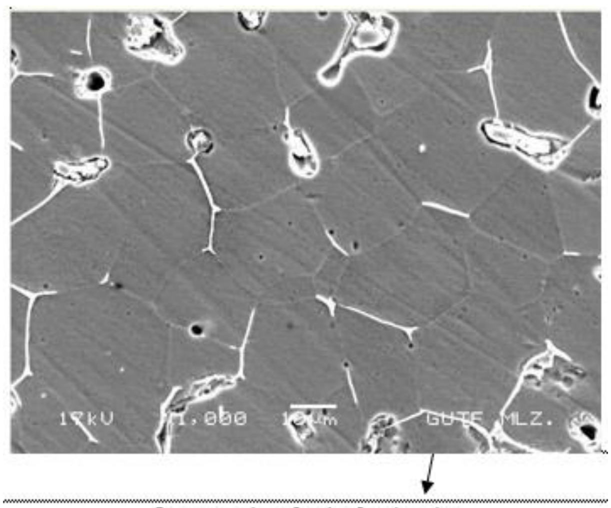

Figure 6. SEM view of specimen sintered at 1250°C and dwell time of 60 min, giving the tensile strength of 631MPa

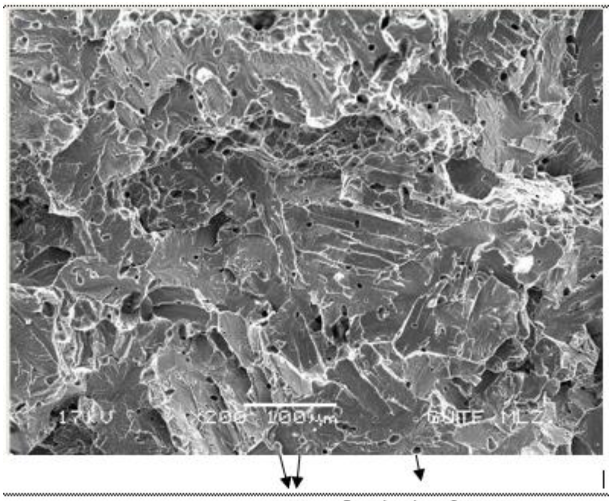

Figure 7. Tensile fracture surface of specimen sintered at 1250°C and dwell time of 60 min, giving the tensile strength of 631MPa

4. CONCLUSION

In this study, a feedstock was prepared using Ti-6Al-7Nb powders with powder loading of 69wt%. Using the prepared feedstock, standard tensile specimens were produced, and then injection parameters were optimized. The results of our experiments can be summarized as:

- By optimizing the injection process of Ti-6Al-7Nb feedstock, tensile strength of sintered specimens reached 631 MPa.
- Argon with high purity as a sintering atmosphere, passing through copper chips leads to sintered Ti specimens with high strength.
- Feedstock powder loading is an essential parameter affecting the injection pressure. For Ti-6Al-7Nb feedstock with powder loading of 69wt%, injection pressure of 1300 bar is a suitable value.
- To have high strength Ti-6Al-7Nb specimens, other injection parameters should also be taken into account. For this study, these parameters are as follow: flow rate of 20 cm^3/s, holding pressure of 60%, and mold temperature of 60°C.

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