



Ti-6Al-4V alloy cortical bone screw production by powder injection molding method

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ABSTRACT

In this study, Ti-6Al-4V alloy is targeted to produce cortical bone screw by powder injection molding method. The rheology study was experimentally performed with PEG8000: PP: SA binder system. Thermogravimetric (TGA) analyzes were performed for F1 feedstocks. F1 feedstock showed the ideal properties in terms of viscosity and melt flow index values. Autodesk Moldflow simulation was carried out on a screw part for moldability. In the rheology analysis, F1 feedstock exhibited superior properties with the viscosity of 72–1001 Pa·s, melt flow index of 261–1888 g/10 min and highest volume ratio of 60%. It was decided to perform the injection molding with F1 feedstock at the end of experimental and simulation studies. In this concept, rheology, molding, debinding, sintering and simulation analyses were performed. Experiments with different injections (1000 and 1200 bar) and holding pressures (400 and 600 bar), flow rate (17,260 cm³ and 20, 11 cm³) and temperatures (200 and 237 °C) were made and cortical bone screw were successfully molded. Debinding process was performed at different temperatures until 900 °C with 1 °C/min intervals. Sintering process was performed at 1250 °C and 5 °C/min heat rate. Finally, the powder injection molding parameters of the Ti-6Al-4V cortical screw bone were determined.

Keywords: Ti-6Al-4V, Metal Injection Molding, Cortical Screw Bone, Rheology, Simulation.

1. INTRODUCTION

The purpose of powder injection molding (PIM) method is to produce engineering parts from powders using the plastic injection molding machine and plastic binders.⁽¹⁻³⁾ The powder processing technology is suitable for economical production of small, complex shaped, full sized, precise tolerance, smooth surfaced and high strengthened mechanical parts which require more processing.⁽¹⁻⁵⁾ Examples of PIM applications include complex shaped machine parts, small watch parts, robot arms, medical device parts, dental prostheses, office equipment, laboratory equipment, motor parts, weapon parts, automotive parts, hand tools, jeweler and high temperature parts.⁽¹⁻⁵⁾ Metal injection molding technology included in powder metallurgy is accepted as

one of the important methods to produce specific parts for industry and other fields of technology.⁽⁶⁾ High density, excellent dimensional stability is achieved in complex parts.⁽⁷⁾ Medical implants developed from stainless steel and titanium alloys and cobalt based alloys are the most widely used materials in today's biomedical applications.^(8,9) Production of biocompatible metals for implant devices is limited because of high raw material cost and difficulty in processing complex geometry. These restrictions are stated in the literature as follows. The higher shear force is required to process titanium alloys than that required by steels. Despite having equal hardness, the metallurgical properties of these alloys make them difficult to process. For processing, beta alloys are the most difficult one among titanium alloys. Especially during grinding, it is necessary to perform some works to prevent irregularities in surface integrity for the titanium

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alloy. Otherwise, dramatic losses can occur in mechanical behaviors such as fatigue. Additionally, standard mechanical properties of titanium alloys might be lost at high processing speeds.^(10–12) Because of these undesired effects, new production methods have been investigated for titanium alloys. The difficulties in machining the Ti-6Al-4V alloy require serial production of the cortical screw bone piece. If 316L stainless steel is used as an alternative to Ti-6Al-4V cortical screw bone, Ti-6Al-4V cortical implants are preferred because of their corrosion resistance and superior biocompatibility. Since the mechanical properties of Ti alloys are close to the mechanical properties of the bone, they have widespread use.^(10–12) Metal injection molding (MIM) is an alternative way of overcoming these problems. The MIM process can reduce cost with the advantages of net shape fabrication and the ability to produce small, high-complexity parts with high throughput.^(13, 14)

Injection molding stage is consisting of different steps such as heating and melting of feedstock, inject the melted feedstock to mold cavity, forming the melted feedstock into the desired shape, packing, cooling-solidification and the ejection of the molded product.⁽¹⁵⁾ Some defects can be determined in the molding stages because of the feedstock properties, injection pressure, temperature and holding pressure.⁽¹⁶⁾ To overcome these defects, some software's such as Moldflow, Polyflow and Moldex 3D can be purchased and used. These software's are used to estimate injection parameters during the injection molding.⁽¹⁷⁾ The binder types used in Metal Injection Molding and binder removal parameters are summarized below. In 2013, Chen et al.⁽¹⁸⁾ used PEG as binder in feedstocks prepared with Ti-6Al-4V alloy powder and they debinded PEG in the rate of 28%, in 60 °C water for 20 min. In another study, palmy stearin and polyethylene were used in feedstocks and the debinding was performed in heptane at 60 °C instead of water. The thermal debinding process was performed at 550 °C for 4 hours.⁽¹⁹⁾ Wei et al.⁽²⁰⁾ studied the effect of mold temperature on the flow behavior of

feedstock and they found that heating the mold prevented rapid viscosity changes. In the paper of German,⁽²¹⁾ it has been studied the effect of theoretical density, alloy type and the changing of micro-structure on Ti-alloys and he found that the density increase of Ti-pieces can be achieved by hot isostatic pressing. There have been also some studies showing the comparison of mechanical and geometrical properties of pedicle screws implants.^(22–24)

In this study, it was aimed to mold the cortical bone screw by metal injection molding method which is an advanced manufacturing method. By using rheological properties, Moldflow simulation analysis was performed for getting injection parameters (injection pressure, injection temperature and flow rate of feedstock). Rheology, simulation and injection molding processes were performed to produce screwed implant.

2. EXPERIMENTAL DETAILS

2.1. Materials

Powder Injection Molding (metal and ceramic) is an important parameter for particle size and form. Spherical powders which are smaller than 20 micron for metals is offered for high packing density. Average particle size, the distribution of particle size and the powder form were analyzed at Dumlupinar University Advanced Technologies Design Center. It was determined that the average particle size is 13.4 microns and the powder form is to be spherical. XRD analysis was performed for Ti-6Al-4V alloys to determine the eligibility for Metal Injection Molding. Figure 1 shows XRD analysis results.

In Table I, it was shown the values obtained by EDX elemental analysis and atomic weights.

Figure 2 shows the geometry of the Ti-6Al-4V alloy powder. The distribution of particle size shows the similar results in comparison to SEM analysis. In the SEM pictures, little powder dusts which smaller than 20 micron are seen.

When the XRD, EDS, SEM and size analyzes are examined, the powder size must be below 20 microns, which

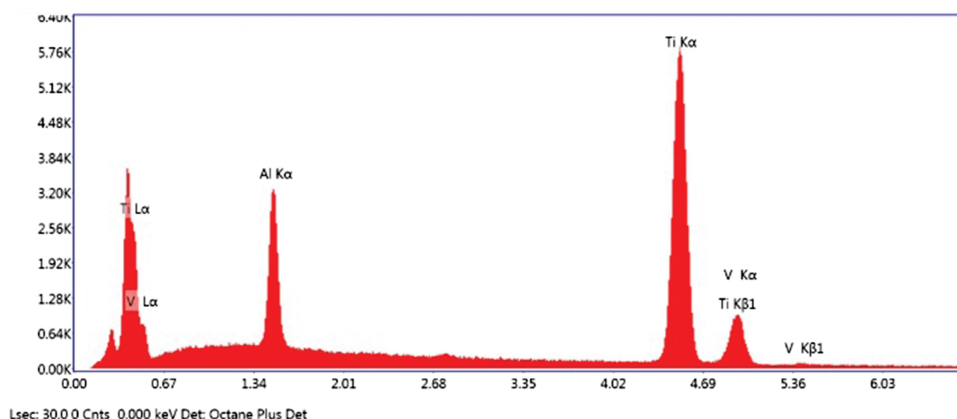


Fig. 1. XRD analysis results.

Table I. EDX analysis.

Element	Weight %	Atomic %
AlK	5.67	9.67
TiK	89.61	86.07
VK	4.72	4.26

is a criterion that helps to achieve a high packing density during injection molding of the spherical shape of the powder. Problems arise during injection molding of powder sizes greater than 20 microns due to high viscous and powder binder disintegration.^(1-5,21)

2.2. Rheology

Rheology plays an important role in the process of Metal Injection Molding. In the rheology, the feedstock granules were obtained by mixing metal powder and binder. The binder is the mixture of polyethyleneglycol (PEG8000), polypropylene (PP) and stearic acid (SA) with a weight ratio of PEG8000:PP:SA = 65:30:5. The obtained feedstocks were passed through the capillary rheometer extruder. The rheology's of the feedstocks were studied using the ASTM D1238⁽²⁵⁾ standard. The rheology experiments were performed to determine flow properties of the feedstocks. The flow type of the feedstocks were determined as pseudo-plastic. It was determined that the viscosity of the feedstocks is below 1000 Pa·s. The injection molding temperature was measured by rheology experiments. Capillary rheometer tests were performed to study F1 feedstocks. The binder used in the feedstocks were given in Table II.

Firstly, binders were mixed in a dry environment for 45 min. And then, these binders were mixed with Ti-6Al-4V powders in the same environment for 45 min. Thus, 100 cm³ feedstock mixtures were prepared. Feedstocks were pelleted using the extruder. To prevent the formation of air bubbles in the area close to the part of the mold during the continuous granulation, all pelleting

Table II. Physical properties of binder system constituents.

Material	Manufacturer	Density [g/cm ³]	Melting point [°C]
PEG8000	Alfa Asear	1.204	62
PP	Petkim A.S.	0.85	189
SA	Merck Co.	0.94	69



Fig. 3. Capillary Rheometer.

processing was performed under vacuum. Feedstocks were mixed in dry environment by a mixer (Turbula 3-Dimensional Shaker/Mixer) for 45 minutes. After the mixing process, feedstocks were extruded by a conical twin screw extruder.^(26,27) The rotational speed of the screw was set to 50 rpm, while the temperature of the barrel was maintained at 170 °C. The obtained typical pellet size was about 4 mm. Figure 3 shows the test device for the capillary rheometer. Melt flow index-temperature, viscosity-temperature changes for flow characteristics of the feedstock were determined.

2.3. Mold Manufacturing and Simulation

In this study, a cortical bone screw was produced by MIM technology. Reverse engineering technique was used for all

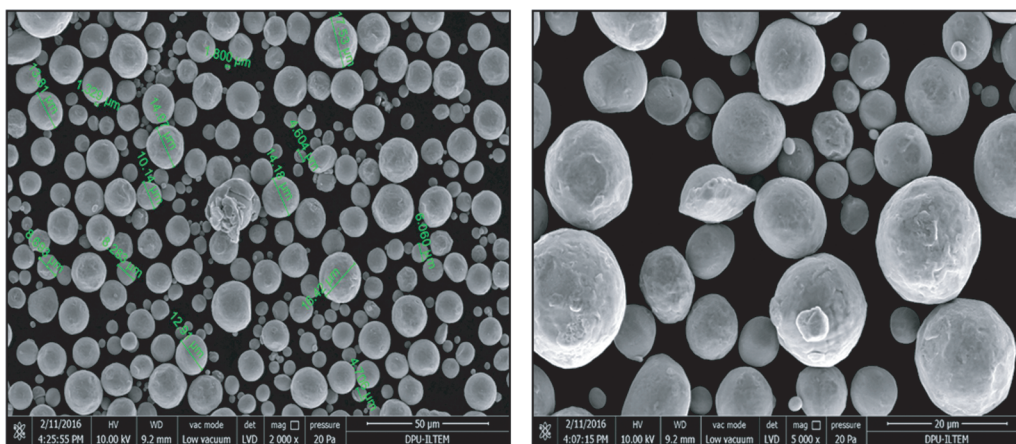


Fig. 2. SEM image of as-grown Ti-6Al-4V alloy powder.

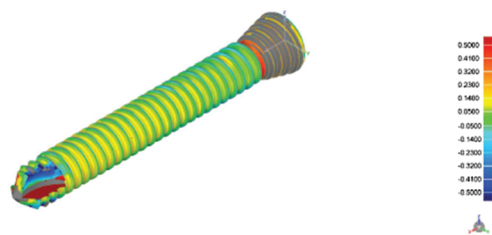


Fig. 4. Cortical bone screw design.

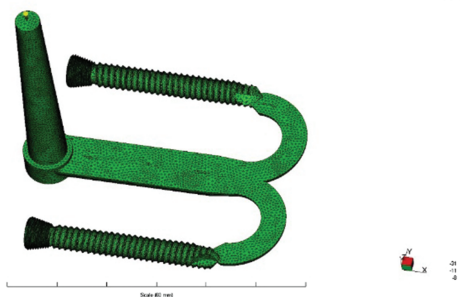


Fig. 5. 3D meshing.

details of the cortical bone screw design. Solid model of the part was obtained using Solid Works program. Implant design is given in Figure 4.

Simulation work was performed with Autodesk Moldflow. The mold gaps formed during design were checked by surface control. The surface of the screw implant is subjected to surface mesh. 3D meshing method is preferred for mesh analysis since it gives more detailed results (Fig. 5). After 3D meshing, the best path and input analyzes were done.

Two-plate molds are selected and each piece is symmetrically placed for stability. Based on the simulation analysis, injection pressure, injection temperature and flow rate parameters were determined. Machining, electro erosion and heat treatment processes were carried out for the injection mold. The mold design and manufacture

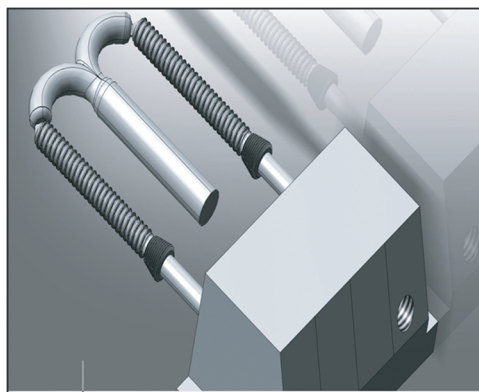


Fig. 6. Mold design and manufacturing.



Fig. 7. Arburg all-rounder 220 s injection molding machine.

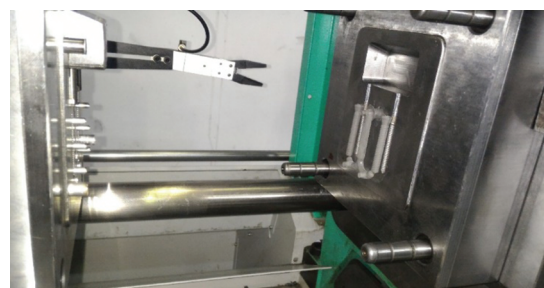
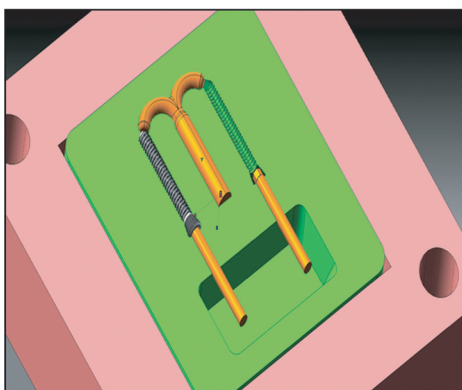


Fig. 8. Molding processes.

used in the injection molding machine has been shown in Figure 6.

2.4. Injection Molding Machine

The molding process was carried out with 60 vol.% Ti-6Al-4V. Cortical Screw bonesamples shown in Figure 4 were produced under the PIM parameters using Arburg All-rounder 220S injection molding machine (Figs. 7 and 8) in the PM Laboratory of Gazi University.



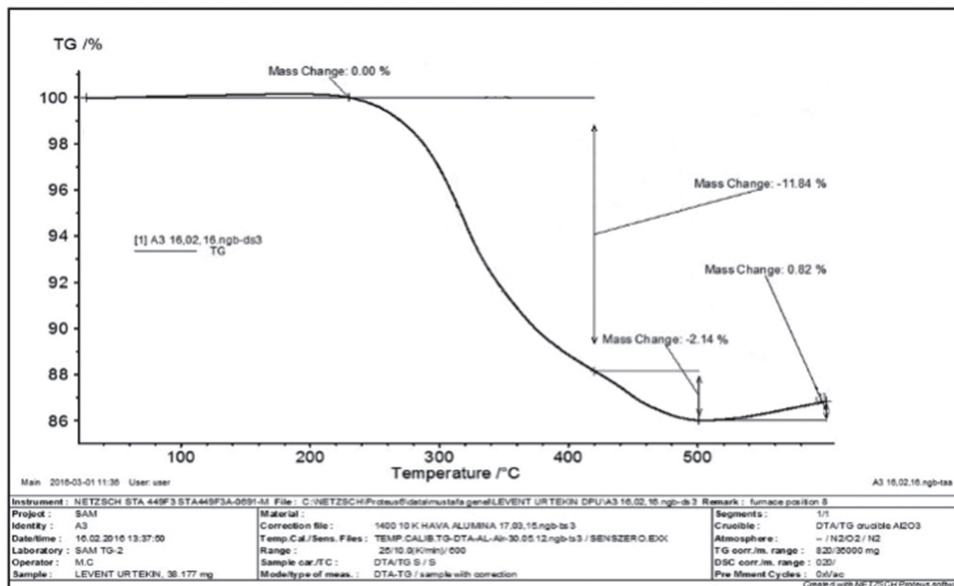


Fig. 9. Thermogravimetric analysis.

2.5. Debinding and Sintering

The binding removal and sintering were carried out in the protective atmosphere furnace. The debinding was performed in two steps depending on the binder, solvent debinding and thermal debinding. PEG8000, a water-based binder, was removed by leaving it in heated water at 60 °C for 24 hours. Thermal debinding was performed at 1 °C/min heating rates under a protective atmosphere (Ar) depending on the TG (Fig. 9) analysis. The heating rate was applied as 5 °C/min and the temperature was increased to 600 °C. In the result of thermogravimetric analysis, it was determined that the starting sample was disrupted at 240 °C. Therefore, during injection molding process, the molding temperature can be reached at 240 °C. This temperature application is expected to provide homogenous filling to the mold. Finally, the feedstock began to deteriorate after 240 °C and the mass decline of the feedstock was experienced at uniform rates up to 420 °C.

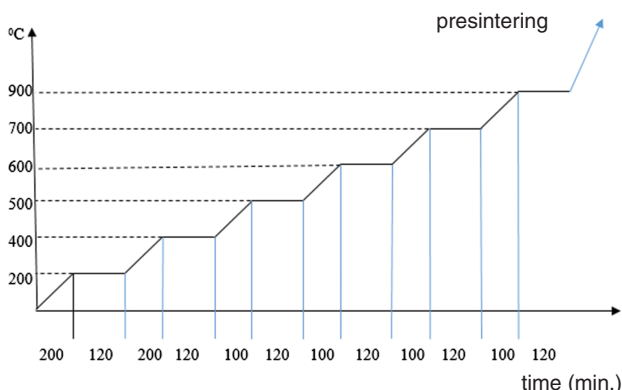


Fig. 10. The debinding process.

Figure 10 shows the debinding and pre-sintering curves. Sintering process was carried out at 1250 °C under a protective atmosphere for 1 hours. Heating rate to sintering temperature and cooling rate to room temperature were same as 5 °C/min.

The ratios of density and porosity were determined. The densities of the sintered test specimens were measured according to the Archimedes principle. The dry weights of the samples are weighed. The surface of the sample was covered with 5% w paraffin and 95% chloroform solution was used to prevent water entering to pores. After the coating, it was air dried and weighed again. Finally, the pure water is immersed and weighed.

3. RESULTS AND DISCUSSION

3.1. Rheological Analysis

The F1 feedstock analyzes given in Figures 11 and 12 showed the most ideal rheological properties. Rheological experiments of powder loading were performed in the volumes of 50, 55, 58 and 60%.

The highest powder loading obtained from the feedstock was %60 by volume. The melt flow index change is ranged from 261 to 1888 g/10 min. and this was found to be the highest feedstock. The viscosity change is ranged from 72 to 1001 Pa · s. When the viscosity-temperature graph is examined, it has been seen that the viscosity values were below 1000 Pa · s.

As the temperature increases for all mixing ratios, the viscosity drop indicates that the flow type of the feedstock is pseudo-plastic. Accordingly, the viscosity decreased based on temperature (Fig. 11). Low viscosity values in 50% volume feedstocks cause jetting in the mold, which means that the feedstock does not reach the viscoelastic

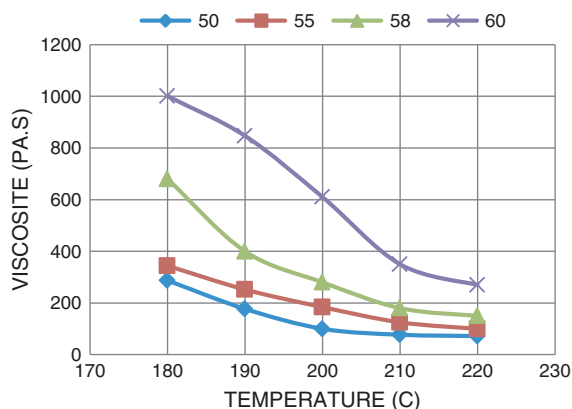


Fig. 11. Viscosity-temperature change.

level. According to German,⁽²¹⁾ In the case of applications under 100 Pa·s, space and jetting occur in the mold. The powder binder mixture in feedstocks of 55%vol. did not reach the desired filling density. In addition, the critical threshold value (1000 Pa·s viscosity value) was not reached for the feedstock of 55% by volume. At 58–60% of the volume, it is seen that the viscosity increase in the feedstocks by dust loading and optimum loading data is reached. As seen in Figure 11, 60% of the volume appears to be based on 1000 Pa·s threshold. When Figure 12 is examined, the change in the melt flow index with temperature is interpreted taking into account the viscosity data. It has been decided that the feedstock at a volume of 60% by volume is suitable in terms of viscosity and melt flow, and that the molding process is carried out with granules prepared with this ratio.

3.2. Simulation Results

The simulation results for the designed mold are given in Figures 13–15. The mold filling was performed at 215 °C and 2–3 °C temperature drops were occurred during this process. The rapid temperature drops can be problem during the mold filling. Considering the temperature

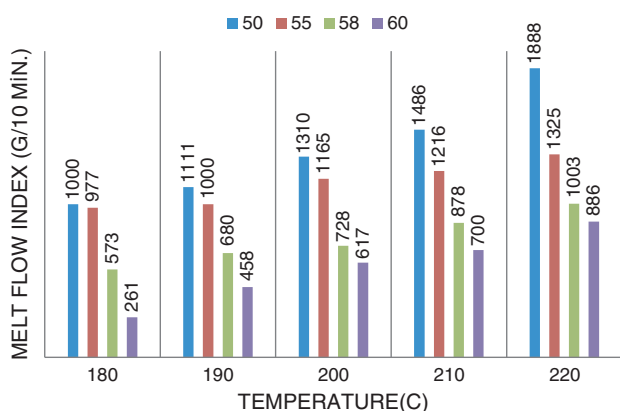


Fig. 12. Melt flow index-temperature change.

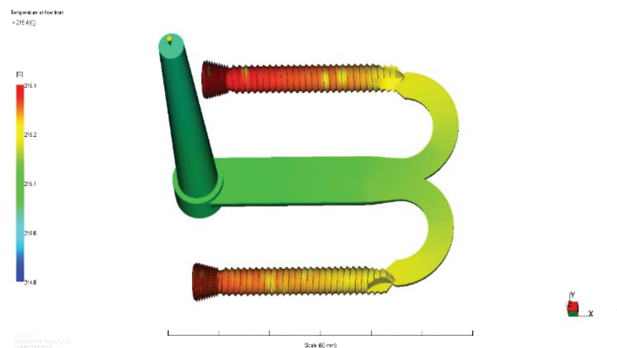


Fig. 13. The injection temperature of screw bone specimens during molding process.

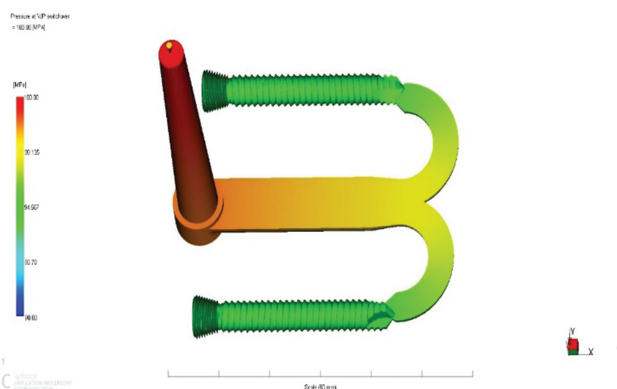


Fig. 14. The injection pressure of screw bone specimens during molding process.

distribution, it can be said that the 2 implant screws are at similar temperatures during simulation analysis (Fig. 13).

The screw implants could be molded under 100 MPa pressure. The filling defects can be seen during the pressure decrease. Because of this, the green part is obtained at low densities. The low green density can cause to produce the low theoretical density after sintering (Fig. 14).

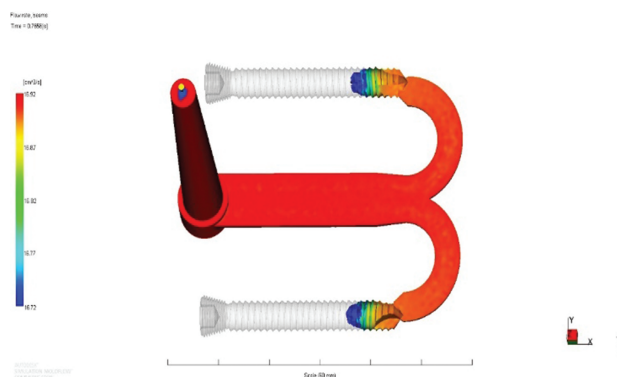


Fig. 15. The flow rate of screw bone specimens during molding process.

Table III. Injection parameter values in simulation and application.

Analyses	F1 mold flow analyses	F1 arburg machine
Flow rate (cm ³ /s)	Max. 16.92	Max. 20.11
Pressure (MPa)	Max. 100	Max. 120
Temperature (°C)	Max. 215	Max. 237
Close force (ton)	Max. 1700	Max. 3200
Molding time (s)	Max. <i>t</i> /2	Max. <i>t</i>
Holding pressure (MPa)	Max. 34.83	Max. 47.1

In Figure 15, the most friction can be seen on the main tureng. This situation can cause the separation of powder-binder.

Injection parameters obtained from injection molding process and pre-operative values obtained from simulation are given in Table III. For feedstocks with a volume of 60%, the values given in Table III were firstly obtained for Mold-flow analysis. Mold-flow analysis was mainly used to obtain preliminary data in this study because it specifies the injection molding parameters of polymer based materials. Finally, metal injection molding is an advanced aspect of plastic injection molding. The obtained data were examined and the lower limit values were determined in real practice. With the help of the simulation program, many trial and error processes have saved time and energy. In real practice, higher data are obtained than simulation results.

The flow rate, pressure, temperature, and hold press pressures obtained by the simulation in Table III are the initial tests. In real practice, there is no need to try a lot and experimental work is facilitated. It was obtained 93% theoretical density in the samples. Sintering is a shrinkage processes. Shrinkage measurements were determined by measuring the dimensional changes of the raw, binder-free, and sintered samples ($\% \Delta L/L_0$).⁽²⁸⁾ The linear shrinkage in the sintered parts was determined as the average of 14.5%. The images of raw were shown on Figures 16 and 17. It can be said that the density of sintering doesn't seem to be at the desired level when density and shrinkage are taken into account.



Fig. 16. Raw (green) cortical screw bone.



Fig. 17. Green part and original cortical screw bone.

4. CONCLUSION

In conclusion, it was found that the viscosity, melt flow index and temperature behaviors of the feedstocks were compatible with PIM. Based on both simulation and rheology studies, PIM can be performed with F1 feedstock. For cortical screw bone, it has been showed that 60 vol.% Ti-6Al-4V and PEG8000/PP/SA can be used. For two-hole cortical screw bone, injection molding parameters were determined as injection pressure (1000–1200 bar), holding pressure (400–600 bar), flow rate (17.26 cm³–20.11 cm³) and temperature (200–237 °C). 93% theoretical density was obtained after the debinding and sintering. The theoretical density and the small amount of linear shrinkage means that the sintering is not at the desired level. Further studies such as improving of sintering parameters are needed to increase the theoretical density. After the necessary condensation has been established, biocompatibility, mechanic and life tests could be performed in terms of using in practice these screw implants. Moreover, it could be investigated the compatibility of different pore rates and pore types with tissue. The final products obtained from this study will be tested according to ASTM F543 and the data will be presented in future.

This study was supported by The Scientific and Technological Research Council of Turkey (TUBITAK) with the grant number of 114M766.

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Received: 16 May 2017. Revised/Accepted: 29 June 2017.