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European Journal of Advances in Engineering and Technology, 2020, 7(11):1-6

Research Article ISSN: 2394 - 658X

Post Corona Architecture Processing of Metallic Implants using Metal Injection Moulding Process: Mechanical Properties, *In-vitro* and *In-vivo* Evaluations

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ABSTRACT

This paper presents the attempt to manufacture metallic implants particularly fracture fixation plates for orthopedic applications for commercial purposes by MIM process. Furthermore, the in vitro and in-vivo evaluation also has been conducted. The stainless steel powder with the median particle size of 16 μm and a binder consisting of major fraction of palm stearin and a minor fraction of polyethylene were mixed at 160 ℃ using a sigma-blade mixer for one hour to prepare the feedstock of the fracture fixation plates. The medical implant component was injection moulded using 80 ton metal injection moulding machine with the nozzle temperature of 200 ℃. Prior to sintering, the specimens were debound using a combination of solvent extraction and thermal pyrolysis method. The specimens were then sintered under vacuum. The properties of the fracture fixation plates such as physical appearance and densities were presented and discussed. Furthermore, the in vitro biocompatibility and preliminary in-vivo study on the fracture plates produced also been carried out.

Key words: sintering, 316l stainless steel powder, density, microstructure

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INTRODUCTION

Metal injection moulding (MIM) process is drawing much attention as a promising technique, which leads to a large-scale production of metalworking with precision and complex in shape. This industry has established a commercial credibility in the production of many components and it is clear that major growth occur for several types of products ranging from automotive to consumer products. Recent interest, has been directed in particular at MIM components with high added value, including sporting goods, eyeglasses, wristwatches and jewellery [1-2].

The MIM process begins with the selection of the powder and binder. The particles of the powder should be small to aid sintering densification and generally have an average size between 0.1 to 20 μm with ideally, the near spherical shape. The binders are multicomponent system composed of waxes and/or thermoplastic polymers with plastisizing and lubrication additives in appropriate proportions. The feedstock is then given a shape using an injection moulding machine. After shaping, the polymer binder must be removed from the moulded part without significantly disturbing the powder particles. The powder is sintered at high temperature, often to near theoretical density [1-5].

The current manufacturing technique for producing parts of the complex shape includes the machining and also the desired holes and this makes the conventional machining process very costly. The MIM process has recently blossomed into a mature metal shaping process and it was postulated that this process would be ideal for the manufacturing of metal implant particularly fracture fixation plates from stainless steel. Although the conventional processes for producing fracture fixation plates has been accepted for a long time and give a good implants in terms of properties, there are several drawbacks associated with these processes. For example, the chances of corrosion of the implants arising from inhomogeneties induced by casting or mechanical working process. Moreover, this process also exhibit the defects and tolerence limitations of the implants and not suitable for the high melting point materials. Furthermore, the capital equipment costs are relatively high for forging compared to other shaping technologies.

Many implants, particularly fracture fixation plates are produced from difficult-to-machine materials such as stainless steel, cobalt-chromium alloys and titanium alloys. The process is quite complicated and involves an extensive machining operation and time. The economical production of complex shape implants may present a problem. As labour costs for medical manufacturing continue to rise, reductions in component manufacturing costs become ever more important for controlling overall cost.

The main objective of the present study is to investigate the possibility of using MIM process as the manufacturing of the fracture fixation plates for commercial purposes. This will provide an excellent basis for discussing the choice of manufacturing process, material to be used and the adaptability of the process and materials to the mass production.

MATERIALS AND METHODS

Powder Characterization and Mixing Process

The 316L stainless steel powder used in this experiment was a gas-atomised powder having a median particle size of 15 um. The tap density of the powder was determined to be 5.04 g/cm^3. A Coulter laser particle size analyser was used to measure the particle size distribution. The particle density for stainless steel was 7.9 g/cm^3. The powder was mixed with a proprietary organic binder that consists of a major fraction of palm stearin and a minor fraction of polyethylene. The mixing was carried out in a sigma blade mixer for 1 hour at 160°C before it was removed from the bowl, cooled and then granulated into feedstock. The powder loading chosen in this investigation was 65 vol. %. Mixing experiments were conducted in a Brabender Plastogram at 160°C and speed of 50 rpm for 2 hours. When the required mixing temperature was reached, the binder with the composition shown in Table 2 was loaded into the bowl little by little with the powder. The torque value is a measure of the resistance on the rotor blades.

Injection Moulding

The granulated feedstock was fed through the hooper of the metal injection moulding machine. The temperature at which the feedstock was injected through the nozzle was varied until satisfactory moulded fracture fixation plates can be produced. All the green parts were carefully weight and some dimensions were measured. All the specimens were checked visually to ensure no defects such as hairline crack, voids and excessive flowline. The density of as-moulded parts was also determined by gas pycnometer.

Debinding and Sintering Process

The green parts were subjected to a solvent extraction step where around two third the volume fraction of the binder was removed. The parts were immersed in heptane for 4 hours at 60°C. The parts which had undergone solvent extraction were subjected to a thermal debinding where all the organic binders were completely removed. Sintering of the fracture fixation plates were carried out in vacuum at the temperature of 1360°C with holding time of 1 hour. The sintered samples were carefully weight, dimensioned and their densities were determined by water displacement technique [6]. The biocompatibility test were conducted according to the ISO standard [7].

RESULTS AND DISCUSSION

Injection moulding of Implants

The detail of the processing parameters during injection is in Table 1. All the injection parts were good and free from normal defects such as short moulding, obvious flashes at the parting surfaces and obvious separation between the powder and binder.

Table -1 Moulding parameters of fracture fixation plates

Moulding parameters	value
Injection temp	200 - 220°C
Injection pressure	1000-1200 bar
Cycle time	10-15 sec
Mould temperature	60°C

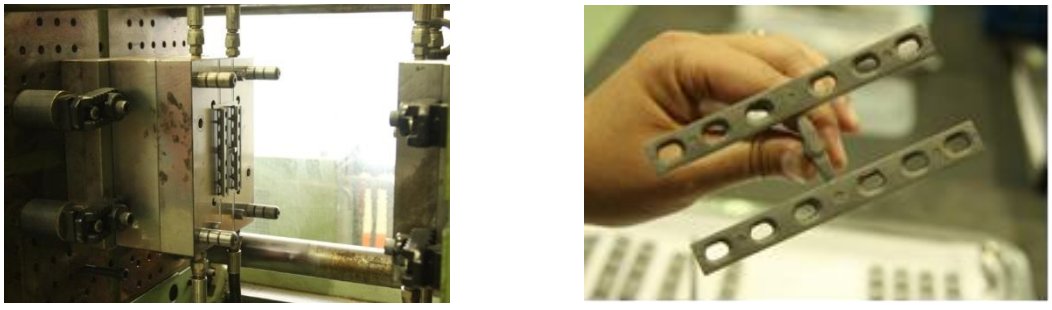


Fig. 1 Injection moulded fracture fixation plates

The die cavity could be filled completely without any porosity or inadequate bonding of flow lines and yet allowing the moulded component to be sufficiently rigid for removal. Figure 1 shows the green body of the fracture fixation plates. The moulded parts hardened sufficiently in the mould, which was at room temperatures, to be removed within 10-15 seconds. The moulded parts had sufficient strengths to be handled. The green density is about 5.2 g/cm^3 or 65 % of theoretical density

Mechanical Properties and Microstructure

A complete sintering process in vacuum at 1360°C for 1 hours resulted in fairly distributed pores around the sample, indicating that the feedstock was well mixed. The sintered density was measured to be 7.8 g/cm3, which is about 99% of theoretical density. Figure 2 showed the microstructure of 316 L stainless steel after etching Furthermore, the pores appear to occupy sites located at grain boundaries and in the grain interiors. It clearly shows a single phase austenitic microstructure, which comply with the ISO 5832-1 for implant quality stainless steel.

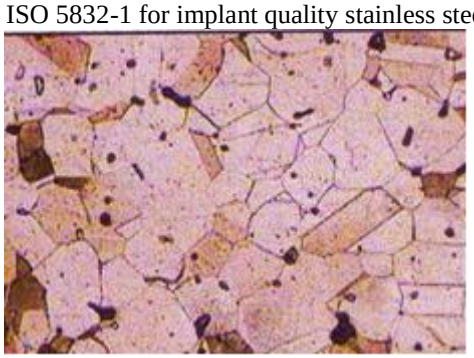


Fig. 2 The microstructure of etched fracture plates sintered at 1360°C

Table 2 shows the physical and mechanical properties of the fracture plates produced by MIM process sintered at 1360°C for 1 hour in vacuum. It is clearly shown that the properties comply to the MPIF Standard 35 for Metal Injection Moulded Parts.

Table -2 The properties of fracture fixation plates

properties	**units**
density	7.88 g/cm^3 (98% of theoretical)
hardness	200 Hv
UTS	> 500 MPa
Yield strength	> 300 MPa
elongation	> 40%

Biocompatibility Test

Alamar Blue for direct contact test

The direct contact test accounts for the cytotoxicity test conducted under static condition in which the samples were incubated with the cells for predetermined time. The light microscope observation reveals the increment of the cells proliferation with increasing incubation time. The cells morphology are not much different from that of the control (not shown) and this indicates that there is no morphological abnormalities taking place. Cells growth towards the material under test demonstrates good cytocompatibility, particularly on longer incubation time which also indicates good material-tissue integration.

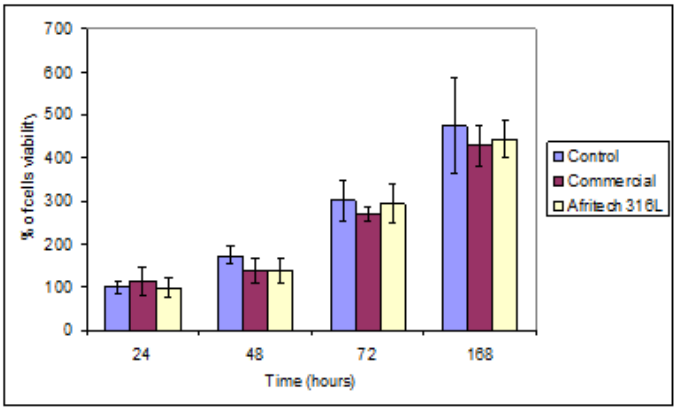


Fig. 3 Percentage of cells viability as a function of incubation time tested using the alamar blue assay. (Afritech=MIM sample)

The percentage of viable cells as a function of time is expressed in Figure 3. The cells viability increased with increasing incubation time in the controls and similar results were demonstrated by both commercial and locally produced samples. The slight difference in the percentage of cell viability between both controls and the test samples shows that the materials tested is not cytotoxic and should be considered for further in vitro testing. In house produced plates showedcomparable results with the commercial ones. This allows the in house produced plates to be considered as a potential material for medical purposes.

Cell Proliferation and Morphology on the Surface of Stainless steel

Scanning electron microscopy (Fig.4) showed the cell were well spread, connected to the stainless steel surface, close and connected to each other by filopodia and also disposed in multilayers. The images showed the surface of stainless steel is very suitable substrate for the cells growth.

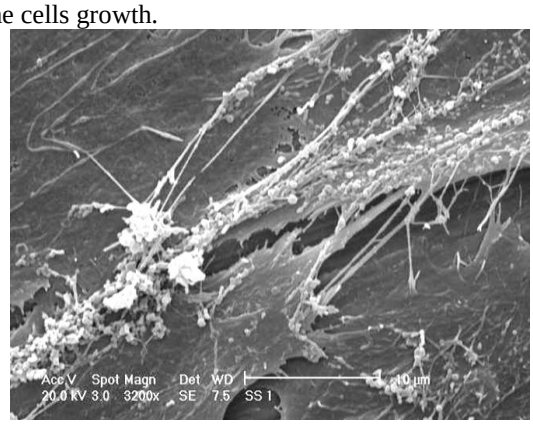


Fig. 4 Scanning electron microscopy showed the cell were well spread, connected to the stainless steel surface, close and connected to each other by filopodia and disposed in multilayers.

Subacute systemic toxicity

No remarkable loss of body weight was found during the 14-day study period. There was no mortality or adverse toxic reaction in the animal groups dosed daily with 50 ml/kg body weight of the test material extract into the peritoneum of mice. The general state and behavior of all animals were normal. Mean body weights of treated mice were not markedly different from those of control animals. Gross necropsy of the brain, spleen, liver, heart, pancreas, lungs, kidneys and stomach did not show any abnormalities

Dermal Sensitization Assay – Modified Buehler Method

In selecting a new material for human contact in medical applications, it is important to ensurethat the material will not stimulate the immune system to produce an allergic reaction. The skinsensitization study also provides the use of test material extracts. The rationale for this is based onthe fact that guinea pig has been shown to be the best animal model to demonstrate contactdermatitis in human.

There was no positive allergic reaction observed during the challenge phase in animals treated with the test material and negative control. No reaction was observed upon removal of the test material during the challenge phase. Similarly, no reaction was observed in the negative control animals. It shows that the MIM implant did not give any contact dermatitis. Figure 5 shows the dermal sensitization assay test on guinea pig.

Fig. 5 Dermal Sensitization Assay Test

Acute Systemic Toxicity

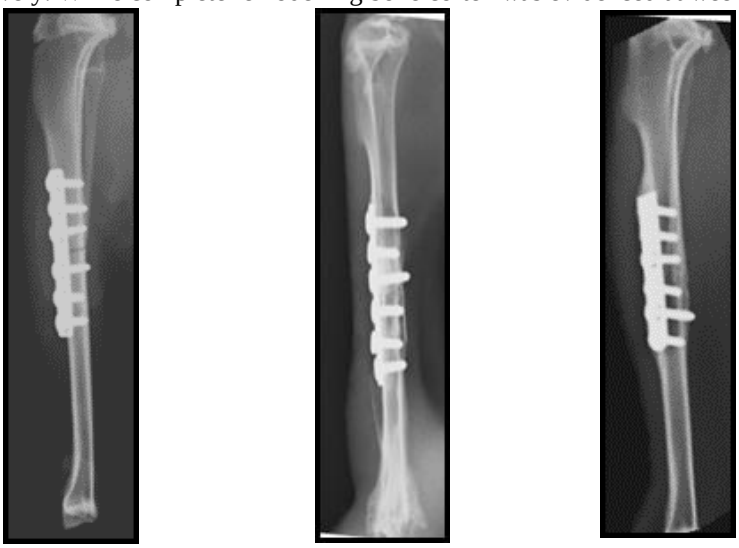
In assessing the toxic potential of a chemical, determination of acute systemic toxicity is usually an initial step. It provides information on the potential health hazards likely to arise from a short-termexposure by the intraperitoneal

route. Data from an acute study serves as a basis for classificationand labeling. It is an initial step in establishing a dosage regimen in subchronic and other studiesand could provide initial information on the mode of toxic action of a substance
Based on the observations and raw data generated, there was no death or remarkable loss of body weight and no adverse reaction in the group of animals treated with 50 ml/kg body weight of test material extract when injected intraperitoneally into mice. Gross necropsy of the brain, spleen, liver, heart, pancreas, lungs, kidneys and stomach showed no abnormal signs.The implants extract did not produce any adverse toxic reaction in mice

***In-vivo* Evaluation**

Radiograph Assessment

In order to evaluate the bone tissue reaction to the 316L stainless steel plate through Metal Injection Moulding technique in bone plate fixation, animal experiment with rabbit was performed by a group of orthopaedic surgeon and researchers. In this study, animal experiment on fracture fixation was investigated by radiograph assessment and gross inspection via hard tissue processing since each sample comprises bone and metal plate.
A total of 40 rabbits were used for this study. Experimental fractures were made in rabbit tibia, and fixed with either MIM plate or conventional mini plate which provided as control. It is interesting to note that there were callus formations in both groups. Bone union was evidenced starting week 6 post-operatively, whilst bone remodelling was completed at week 26. Histological assessment has indicated that both groups possessed mild to moderate callus bridging at week 3 and week 6, respectively. While complete remodelling bone cortex was evidenced at week 26**.**



3 weeks **6 weeks** **26 weeks**

Fig. 6 X-Ray assessment of implantation of MIM plate (lateral view) at different week

CONCLUSION

An advantage of the present invention, with respect to the fracture fixation plates is that the design flexibility and cost effectiveness compared to the conventional processes. The above features lend to versatility for implant fabrication. By deploying the injection moulding, the world has taken the step to rapid manufacturing of said fracture fixation plates, for the first time. Such generative processes overcome the defects and tolerance limitation of casting and the higher cost of machining, opening up a wealth of new possibilities.In-vitro and in-vivo testing is very useful for assessing the cytotoxic potential of new materials and formulations and as part of a quality control program for an established or new medical device and itscomponents. Assessment of cytotoxicity provides useful information in predicting the potential clinical applications in the human

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