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Mechanical Properties and Biocompatibility Evaluation of Medical Implant using Metal Injection Moulding Process

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***Abstract*- Metal Injection moulding (MIM) is an advanced near net shape forming process for high quality of complex shapes combined with high properties of materials. For economic reasons, it is necessary to have demand for a large quantity of parts. 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°C 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°C. 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**

***Keywords* – metal injection moulding, fracture plates, debinding, sintering**

I. 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 □ m with ideally, the near spherical shape. The binders are multi component 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] [2] [3] [4]. 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 in homogeneities 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

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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. It is hope that this project will create a positive spin-off for Malaysia especially in the area of R & D in orthopaedic technology.

II. MATERIALS

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. %. Five feedstock with the binder system consisted of a locally available binder known as palm stearin and polyethylene was prepared varied between 50 and 80 wt.% as shown in Table 1. The increasing in palm stearin was primarily intended to reduce the viscosity of the binder so that the injection temperature might be lowered. The volume fraction of the powder in these mixtures was kept constant at 65 vol%. Mixing experiments were conducted in a Brabender Plastogram at 160^{o}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. By observing the mixing torque values, the homogeneity of the feedstock prepared can be predicted: the lower the value, the better is the mix [7]. Uniform mixing was assumed to have occurred when the torque reached a steady state value. After each mixing experiment, the feedstock viscosity was measured using a Shimadzu CFT-500D Capillary Rheometer. During the capillary rheometer test, the feedstock was forcibly extruded through a small cylindrical orifice with a 1.0 mm diameter and 10 mm length (L/D=10). The palletized feedstocks were placed in the rheometer barrel and allowed to preheat for 120s under 1, 2, 3, 4 and 5 MPa test load before initiating testing. 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 flow line. The densities of as-moulded parts were also determined by gas pycnometer. 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 heptanes 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^{o}C with holding time of 1 hours.

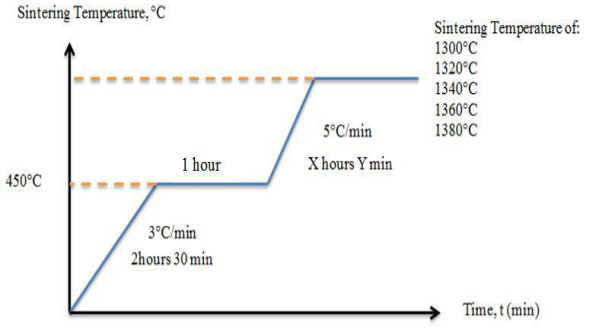

Fig. 1 Sintering profile for fracture plates

The sintered samples were carefully weight, dimensioned and their densities were determined by water displacement technique. [5]. The biocompatibility test were conducted according to the ISO standard [6].

III. RESULTS AND DISCUSSION

1. Injection Moulding of the Fracture Fixation Plates

After several trials, the feedstock was easily moulded at a nozzle temperature of 200^{o}C with the maximum injection pressure around 1000 bar. 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. 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 2 shows the green body of the fracture fixation plates.

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1(a)

2(b)

Fig. 2 a and b.Injection moulded 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/cm3 or 65 % of theoretical density.

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

2 Debinding Process

Debinding is is the process where 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 followed by sintering. Figure 3 shows a network of polyethylene ligaments remained, binding and holding the powder particles together to provide the brown part with sufficient brown strength to be handled.

This phenomenon is explained by the fact that the remaining binder in the contact region serve to retain the shape of the brown part. Figure 4 clearly shown the presence of various size spherical shape metal powder particles in the brown part after thermal pyrolysis at 450°C. The pores between the interstitial powder particles indicate that the binders have been completely removed from the brown injection moulded 316L SS. The strength of the brown parts decreases markedly and great care is necessary in handling the parts

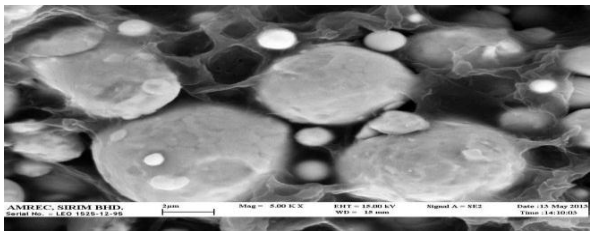

Fig. 3 A network of polyethylene ligaments remained, binding and holding the powder particles together

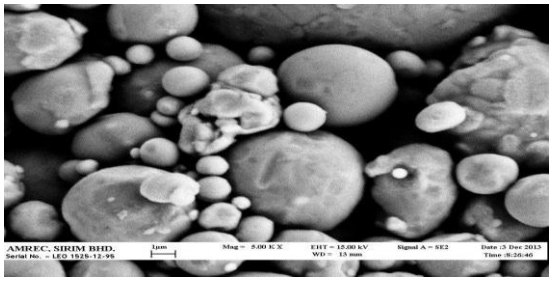

Fig. 4 The presence of various size spherical shape metal powder particles in the brown part after thermal pyrolysis at 450°C

3. Sintering Process

Sintering is a high temperature firing process in a controlled atmosphere to consolidate powdered-metal particles by diffusion. This happens in a sintering furnace at temperatures elevated to just below the melting point of the specific metal. Sintering and densification occurs through multiple processes including volume diffusion, grain boundary diffusion and surface diffusion. In some cases a liquid phase is used to accelerate sintering.

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 with the MPIF Standard 35 for Metal Injection Moulded Parts.

Table 2 The properties of fracture fixation plates

Properties	units
density	7.88 g/cm3 (98% of theoretical)
hardness	200 Hv
UTS	> 500 MPa
Yield strength	> 300 MPa
elongation	> 40%

4. Microstructure Analysis

Microstructure analysis was done to examine porosity and grain boundaries. Figure 5 shows the pore structure of the sintered sample and the grain boundaries of sample after etching with ADLER solution.

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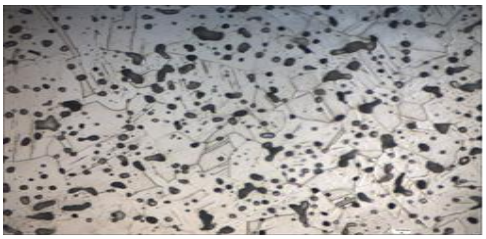

Fig. 5 Microstructure of sintered samples.

5. 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. The percentage of viable cells as a function of time is expressed in Figure 6. The cells viability increased with increasing incubation time in the controls and similar results were demonstrated by both commercial and locally produced samples.

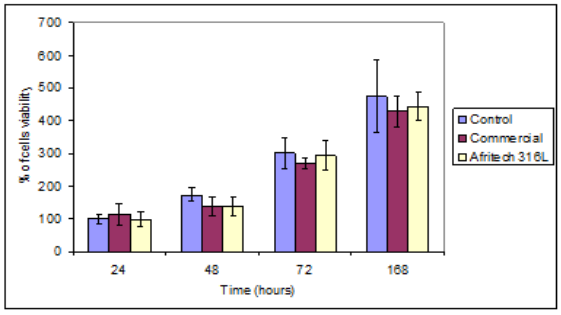

Fig.6 Percentage of cells viability as a function of incubation time tested using the alamar blue assay.(Afritech=MIM sample).

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 showed comparable results with the commercial ones. This allows the in house produced plates to be considered as a potential material for medical purposes.

6. Cell Proliferation and Morphology on the Surface of Stainless steel

Scanning electron microscopy (Fig. 7) showed the cell were well spread, connected to the stainless steel surface, close and connected to each other by filopodia and also disposed in multilayer's. The images showed the surface of stainless steel is very suitable substrate for the cells growth.

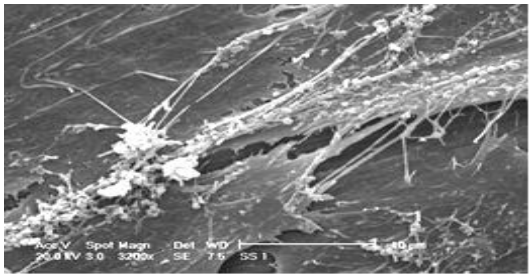

Fig.7 Scanning electron microscopy showed the cell were well spread, connected to the stainless steel surface, close and connected to each other by filopodia and also disposed in multilayer's

IV. CONCLUSIONS

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. A further advantage, this invention would generate more uniform structure, both metallurgical and physically, and further reduce the chances of corrosion arising from in homogeneities' induced by casting or mechanical working process. 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.

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