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European Journal of Advances in Engineering and Technology, 2019, 6(7):15-19

Research Article ISSN: 2394 - 658X

The Potential of Cassava Starch as Binder in Injection Molding of 316L Stainless Steel Powder for Biomedical Applications

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ABSTRACT

Stainless steel is one of the most frequently used biomaterials for internal fixation devices owing to their favorable combination of mechanical properties, corrosion resistance and cost effectiveness when compared to other metallic implant materials. Human implantation for decades has successfully proven the biocompatibility of implant quality stainless steel. Despite the great progress that has been achieved in orthopaedic biomaterials, fixation of implants to the bone host remains a problem. Mismatch of Young's moduli of the biomaterials and the surrounding bone has been identified as a major reason for implant loosening following stress shielding of bone. Appropriate balance between strength and stiffness has to be found to best match the behaviour of bone. In this research work, application of cassava starch as a major constituent of a binder for metal injection moulding of medical devises 316L stainless steel has been investigated. Injection moulded parts that exhibit substantial porosities possesed properties tailored to match the modulus of bone, thus reducing the problems associated with stress shielding.

Key words: stainless steel, metal injection moulding, debinding, sintering

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INTRODUCTION

Powder metallurgy which is based on the partial densification during sintering of metal powder is the simplest fabrication technique for producing both porous coated and fully porous metallic implants. This technology of producing metallic implants involved compacting, binding, and sintering of metal powders. However, the shape limitation of traditional powder compaction technique is an essence which has led to utililization of metal injection molding (MIM) technique which provides advantages for manufacturing complex, precision, net-shape metallic implant. Further, the ability of the technology to hold close tolerances, offers excellent dimensional and productivity limits, defect free and good mechanical strength has making the MIM process highly cost competitive as compared to machining and investment casting [1].

The MIM process starts with the preparation of a homogeneous powder feedstock by mixing metal powders with a suitable binder system. The homogeneity of a powder feedstock is crucial in MIM processing to avoid any in homogeneity, including bubbles, left during the mixing stage which will not be carried over to the subsequent processing stages. An inhomogeneous feedstock usually leads to a poor flow behavior during injection molding stage, which consequently resulted in low densification and poor dimensional stability in the final injection molded products. The selection of binders in MIM is usually based on consideration of producing homogenous feedstock which is free of binder separation or particle segregation in order to offer high quality injection molded product. Typically, the binder present in the feedstock acts as a temporary vehicle for shaping the feedstock into the required geometry and holding the particles in that shape until the start of sintering stage [1].

Generally, the binder formulation in MIM comprised of at least two components. The major component such as wax functions to wet the metallic powder particles and provide necessary flowability [2-3]. Meanwhile, the secondary component that is usually a high molecular-weight polymer will ensure that the green injection molded component has sufficient green strength. In addition, the polymeric component which is also known as backbone component provides sufficient brown strength to the injection molded component after the major component is removed during the debinding stage. In most cases, the binder system also contains a third component, such as a surfactant, which serves to enhance

compatibility between the metallic powders and the polymer. Despite the great progress that has been achieved in binder formulation, it is crucial to ensure that all the binder components are compatible with each other [4-6].
Natural polymers which have gained widespread use in different industries have raised interesting issue related to environmental concerns. Conventional wax-based binders have proven their suitability as a binder sytem in production of intricate shape metallic component by using MIM route. However, the injection molded components which usually suffer long debinding time make them susceptible to defects formation [1, 4-5]. In addition, extracting the wax from the green components may contribute to toxicologic and environmental concerns. The focus in recent years thereby has mainly been on the development of new environmentally friendly and safer binders. Thus, in this study, the feasibility of cassava starch (CS) as a major constituent of a binder system for metal injection moulding of stainless steel 316L has been investigated.

MATERIAL AND METHODS

Materials

The gas atomised 316L Stainless Steel powder used in this study was obtained from Sandvik Osprey Powder with the average particle size of 14.2 μm. As shown in Figure 1, all the particles were approximately spherical in shape. The sizes of some particles are large, while some are very small. These demonstrated that the powder had a relatively wide particle size distribution which is desirable for efficient particle packing in MIM powder. The analysis result showed that the 316L Stainless Steel powder has a mean particle size distribution of around d_{50}= 14.22μm.

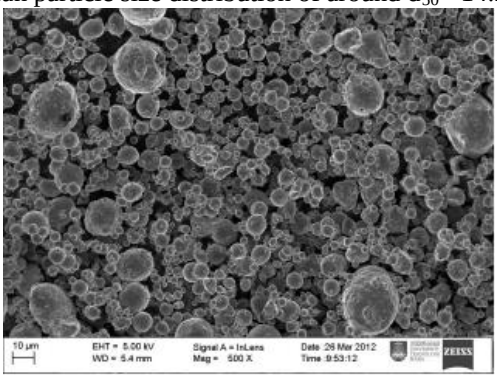


Fig. 1 SEM micrograph of 316L Stainless Steel

Mixing Process and Injection Moulding

The feedstock was prepared by mixing the 316L Stainless Steel powder with the composition binder system as formulated in Table 1. In this study, blends of metal powder and binder containing different solid contents were used to prepare the feedstock samples. The mixing process was carried out using a Z-Blade mixer at a temperature of 160ºC for a duration of 2 hours at a speed of 50 rpm to obtain homogeneous pastes. In order to find suitable solid loding, three types of feedstocks were prepared with 60, 63 and 65% vol. powder loading. Hereafter, these feedstocks are identified as F1, F2 and F3 respectively. The feedstock was then granulated into pallet form so that it could be easily fed into the injection molding machine.

Table -1 The Composition of the Binder System

Binder type	Compositions (wt.%)
Paraffin Wax	40
Polyethylene	10
Cassava (CSV) Starch	40
Stearic Acid	10

The granulated feedstock was molded into tensile bars by using MCP HEK-GMBH vertical injection moulding machine. In order to obtain defect-free molded parts, a suitable set of molding parameters were established. Injection molding temperature and pressure were 155 °C and 500 bar respectively.

Debinding and Sintering Process

The granulated feedstock was molded into tensile bars by using MCP HEK-GMBH vertical injection moulding machine. In order to obtain defect-free molded parts, a suitable set of molding parameters were established. Injection molding temperature and pressure were 155 °C and 725 bar respectively. The green molded parts were subjected to a solvent extraction process where around two thirds the volume fraction of the binder was removed. During the process, the parts were immersed in a bath of n-heptane and held at a temperature of 60°C for 5 hours. A glass container was use to cover the bath to prevent evaporation of the n-heptane. Upon completion of the solvent extraction process, the brown parts were dried in an oven at a temperature of 47°C for 2 hours to remove the remaining n-heptane. The brown parts were

weighed to calculate the amount of binders removed and Scanning Electron Microscopy observation was performed to observe the fracture surface of specimens debound for different elapsed times.

Sintering was performed at different temperatures between a temperature range of 1300°C to 1380°C in vacuum environment. In order to prevent crack formation, the heating rate used was 5°C/min and were soaked for 2 hours and subsequently furnace cooled. The transverse rupture strength (TRS) of sintered specimen was measured by three point bending test by using the Instron Universal Instrument. Specimens for metallographic examination were prepared using standard techniques and etched in 5% Nital solution. Microstructure evaluations were performed by optical microscopy and sintered densities were evaluated by Archimedes' method.

RESULTS AND DISCUSSION

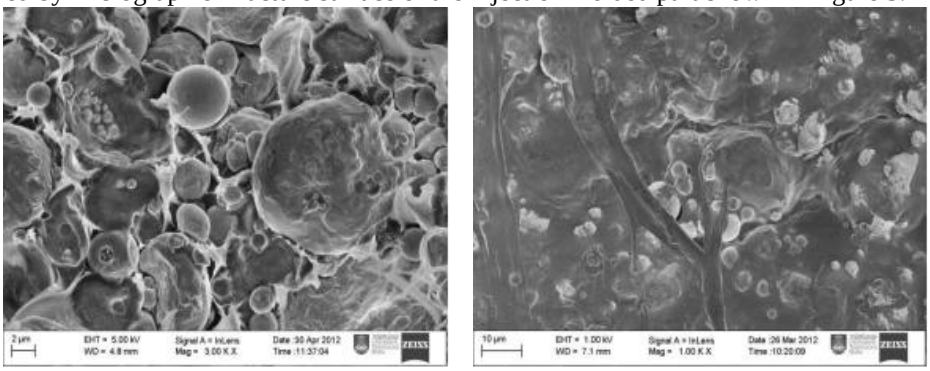
Injection moulding of feedstock

The feedstock was succesfully moulded at injection temperature and pressure of 155°C and 500 bar respectively. Figure 2 presents the tensile specimen of the injection molded parts. The green injection molded parts were fairly good and free from normal defects such as short shot, flashes at the parting surface and binder separation.

Fig. 2 Tensile specimen of the injection molded parts (green)

The green injection molded part exhibits a green density value of 4.42 g/cm^3. The typical density for ferrous based feedstock must be near 5 g/cm^3. This finding (German and Bose, 1997), emphasized that the density of the green injection molded part is considered to be adequate for MIM parts. It should be clear that, it is always important to achieve sufficient value of green density as the green injection molded part is subjected to handling prior to debinding and sintering process. This is due to the fact that the feedstock which possessed pseudoplastic behaviour had allowed the molding pressure to be transmitted uniformly during the injection molding process, and resulted in better packing of the feedstock into the mold cavity.

The binder present in the injection molded part binder provides a mechanical interlocking to the metal powder particles which in turn gives the compact shape and the necessary handling strength required for subsequent processing stages. This is confirmed by micrograph of fracture surface of the injection molded part shown in Figure 3.



a b

Fig. 3 Scanning electron micrograph of (a) fracture surface and (b) outer surface of injection molded part

It can be clearly seen that the binder components consisting of cassava (CS) starch, paraffin wax (PW), stearic acid (SA), and polyethylene (PE) fill the interstitial sites of stainless steel 316L particles and build a binder network which hold the particles in shape. Further, the micrograph also indicates individual stainless steel 316L particles which have been uniformly covered by the binder, thus preventing the stainless steel 316L particles from making a direct surface-to-surface contact.

Debinding Process

In this research work, in order to reduce the total debinding time, solvent debinding was conducted by immersing the green part in a bath of n-heptane where cassava (CS) starch, paraffin wax (PW), and stearic acid (SA) are soluble. It has been observed that the part achieved 90% binder removal after immersion in n-heptane for a duration of 60 minutes

without defects such as cracking, slumping and sagging. M.A. Omar et. al. (2016), has reported the use of n-heptane, a water soluble solvent that has effectively reduce the debinding time and defects formation during thermal debinding of organic binders.

The fractured surface of the as-molded parts were examined by using Scanning Electron Microscopy (SEM) in order to observe the binder distribution and evolution of interconnected porosities resulted from the leaching process. Figure 4 shows the binder distribution at the (a) initial solvent debinding (after 10 minutes) and (b) final stage of debinding (after 300 minutes).The scanning electron micrograph of the as-molded part exhibits that the stainless steel 316L particles were enwrapped by the binders equably and ensuring the density stability of the green parts. It also appeared that there are some voids between the metal particles owing to shrinkage of the binder that occurred during cooling.

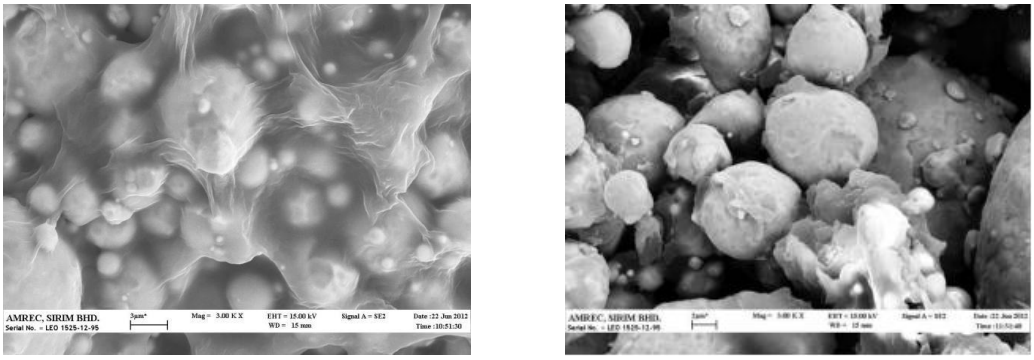


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Fig. 4 Scanning electron micrograph of fractured surface of (a) after solvent extraction in heptane for 10 minutes (b) after solvent extraction in heptane for 300 minutes

When as-molded part was immersed in the solvent bath with preheated heptane, the solvent, heptane, diffused into the PW, CSV and SA. The heptane content decreased with the depth of the as-molded part because the heptane diffused into the as-molded part from the exterior to interior slowly through the pores. Only PW, CSV and SA at the surface began dissolving into the solvent at this stage. As debinding continued, owing to the increase of heptane content in the interior of the part, the solvent insoluble binders-debound depth increased as the debinding time increases. At the final stage of solvent debinding, the interconnected pore channels make leaving of gaseous products by the insoluble binder in subsequent thermal debinding easier and in a shorter period of time. Further, the presence of insoluble binder ligament, which is PE, holding the stainless steel 316L particles together is also noted

Sintering Process and Mechanical Properties

In this stage, temperature increase from 30^{o}C to the sintering temperature at 1360^{o}C with the rate of 5^{o}C/min under vacuum atmosphere and held at the temperature for 1 hour. After sintering, it was not observed any signs of defects such as blister and crack in the specimens.

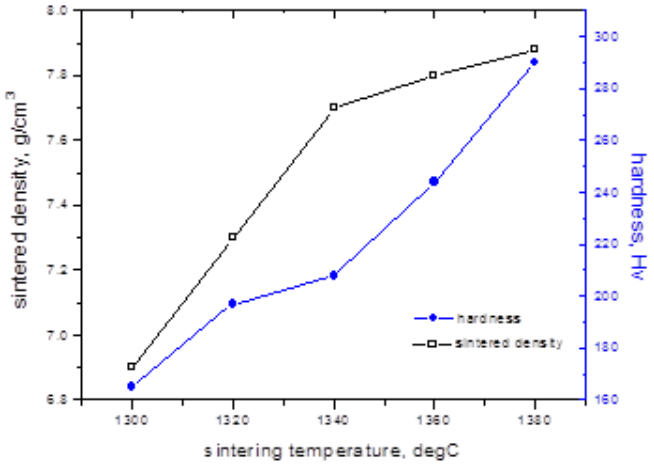

Fig. 5 The relationship between the sintering temperature on the density and hardness at different sintering temperature

Figure 5 show the relationship between the sintering temperature on the density and hardness. As the sintering temperature increased, the sintered density, as expected, improved. For example, a change of sintering temperature from 1300°C to 1380°C causes the density to increase by 10 %, from 6.9 g/cm^3 to 7.88 g/cm^3. The density is also believed to be influenced by volume diffusion, grain boundary diffusion and plastic flow at high temperature, notably at 1360°C [1]. At that temperature, sintering is likely to slow down, as during this final stage of sintering, isolated pores start to form.

Hence, densification of the part will reach a certain level with respect to increasing sintering temperature. At 1380°C, the specimen shows smaller increase of 1 % in density. Vickers microhardness measurements were conducted on the cross sectional of the specimens. From the result obtained, it can be seen that the hardness of the sintered specimens increases when the sintering temperature increases. The hardness increases from 165 Hv to 290 Hv with increasing of sintering temperature from 1300°C to 1380°C respectively.

Tensile testing was performed using Instron Universal Testing machine according to the MPIF Standard 50. The results obtained from the tensile tests are shown in Figure 6. The ultimate tensile strength levels scatter over a large range from 200 to 500 MPa. To meet the MPIF Standard 35, the specimens made with 316L SS must have tensile strength of at least 450 MPa. As shown in the Figure 6, the graph shows a significant linear increment of strength as a result on increasing sintering temperature. High strength greater than 450 MPa was observed in all specimen sintered at 1340ºC,1360ºC and 1380ºC, thus complying with the international standard MPIF 35 MIM specimens

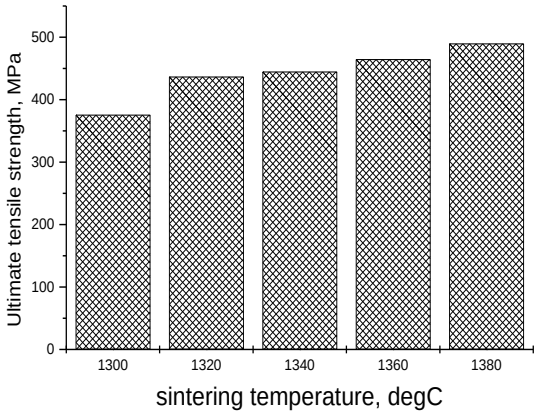

Fig. 6 The Ultimate tensile strength of the sintered specimen at different sintering temperature

CONCLUSIONS

In this study, the feasibility of cassava starch as a major constituent in a binder system for metal injection moulding of 316L SS has been investigated. Results of this study indicate that cassava starch (CV) can be a promising alternative for synthetic polymers in metal injection molding of 316L SS for medical devices applications. The binder comprises of a major fraction of cassava starch (CV), which can be rapidly removed by heptane leaching, and a minor fraction of polyethelene (PE), which retains rigidity of the part. Furthermore, this binder system eliminates the need for seperate debinding and environmental liabilities associated with the debinding process. The results are a cycle time reduction and cost savings compared with polymeric binders.

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