Mechanical Properties of SS316L and Natural Hydroxyapatite composite in Metal Injection Molding

M.H.I.Ibrahim[1], N.Mustafa[2], A.M.Amin[3] and R.Asmawi[4]
[1]Advanced Manufacturing & Material Center (AMMC) Universiti Tun Hussein Onn Malaysia, Batu Pahat, Malaysia
mdhalim@uthm.edu.my
[2] Advanced Manufacturing & Material Center (AMMC) Universiti Tun Hussein Onn Malaysia,Batu Pahat, Malaysia.
nj.wawa@gmail.com
[3] Advanced Manufacturing & Material Center (AMMC) Universiti Tun Hussein Onn Malaysia,Batu Pahat, Malaysia,
azriszul@uthm.edu.my
[4] Advanced Manufacturing & Material Center (AMMC) Universiti Tun Hussein Onn Malaysia,Batu Pahat, Malaysia,
roslias@uthm.edu.my

ABSTRACT

Metal injection molding (MIM) is a net shapping process in order to achieve the desired molded part via mass production using metal or ceramic powder. MIM is drawing much attention as a promising technique which is leads to a large scale production of metalworking with precision and complex in geometry. It is an elegant blend of metal injection molding, based on the use of fine metal powder particles mixed with binder to form a feedstock that can be molded. The granulated feedstock is then given a shape using an injection molding machine. Stainless Steel 316L (SS316L) and Natural hydroxyapatite (NHAP) powder derived from tilapia fish bones has been chosen as a model materials mixed with an established binders. Polyethylene glycol (PEG), Polymethylmethacrylate (PMMA) and Stearic Acid (SA) were acted as binder systems. Moreover, the optimum powder loading that have been used are 63 vol. % and 64 vol. %. The feedstock was mixed by using Platograph brabinder at 70°C within 95 minutes duration. The feedstock completely filled the injection mold cavity at 155°C of molding temperature in order to produce a green part. The density and strength of the green part was determined. Green Part with 64 vol. % powder loading has higher density which is equal to 4.7159 g/cm^3 while 63 vol. % powders loading is 4.6462 g/cm^3. In terms of green strength, feedstock with 64 vol. % powder loading has higher maximum stress with the value 14.8415 Mpa compared to 63 vol. % powders loading has 12.1714 Mpa maximum stress.

Keywords: Natural Hydroxyapatite, SS316L, Metal Injection Molding, Tilapia Fish Bones.

INTRODUCTION

Metal injection Molding (MIM) is referred to the manufacturing technology that explored due to mass production of green part with intricate size, complex shapes and geometry. This technology is based on injection molding but respect to focus on metal powder [1]. MIM is a net shaping process to achieve the desired molded part via mass production using metal or ceramic powder. Processes such as compression and base coating of metal mixed with ceramic have been reported, but exclusively a few used the injection process [2]. Currently the areas of application can be applied in the biochemical, aerospace, computer peripherals and automobile industries. In MIM, binder systems play an important role as it acts as a temporary vehicle as homogeneously factor in order to packing the powder into the desired shape [3].The development of new powders has always been as the most interest of researcher for future improvements such as cost reduction and less environmental issues. To date, extensive study has been done by using hydroxyapatite powder (HAP) but none of them focused in waste material and most of them used synthetic HAP. The researcher believes such waste (Tilapia Fish Bones) can be converted and explored into more beneficial medical products. Earlier investigation on hydroxyapatite (HAP) as a powder in MIM employed by using the HA /SS316L with powder loading of 58%. [2].The other researcher use the combination of HA/ Ti6Al4V with 78.21 vol % [4]. Both of researchers used the synthetic HAP. This is by the fact that the hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) consist in structure of bone where as similar to their natural mineral composites [5]. Regarding to their similarity, its make it suitable to apply as an implant materials in dental and orthopaedic field. In this paper, the researcher is highlighted to study the mechanical properties of SS316L and Natural Hydroxyapatite composite in Metal Injection Molding with binder system (PMMA, PEG and SA). Natural Hydroxyapatite (NHAP) was derived from Tilapia Fish bones by calcination process.

LITERATURE REVIEW

Metal Injection Molding is known as a manufacturing process that has looking into producing a large quantities and complex component geometry at a low cost [6]. Metal injection moldings are process that uses the advantages of injection molding but, this method are also applicable to metals and ceramics. The application of MIM can be seen in the industrial sector in the production of automotive components, petroleum refining equipment, appliances and computers, medical, firearms, equipment and dental surgery [1]. The advantages of MIM are savings in the cost of production, complex geometry, good surface finish, flexible material and final properties almost solid. The MIM processes are divided into 4 major steps which are mixing, molding, debinding and sintering [1, 7]. Hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) is one of major mineral that consist in teeth and bones. Besides that, it also has a great biocompatibility with hard tissue, skin and muscle tissues without any effects in terms of cytoxic and can growth directly to the bone [8]. Moreover, HAP has been vigorously investigated as implant materials for orthopedic and dental applications, showing excellent bioactivity, osteoconductivity [9]. However HAP only limited to non load bearing implants due to their poor mechanical properties, such as brittleness and low fracture toughness [10]. Hence, medical grade SS316L is added to increase the mechanical & biological properties so that it can be tailored to meet specific clinical requirements [2, 11].

MATERIALS AND METHODS

MATERIALS

SS316L water atomized was selected as a metal powder (Epson Atmix Corp) with irregular shapes as it is compatible with water leaching and high corrosion resistance. While, NHAP from Tilapia Fish bones was acted as ceramic powder. The NHAP powder was produced from Tilapia Fish bones by calcination process. The both SS316L and NHAP powder were mixed up with multi component binder system which is consisting of PMMA, PEG and SA. PEG is categorized as a water soluble binder in Metal injection molding [13, 14]. The solubility water property of PEG is leading them as an ideally for different applications [15]. PMMA is one of established binder in metal injection molding due to their properties which is faster removed from the moldings in short time [13]. SA is well known as a surfactant factor that useful in improving and increases the particle surface area. Furthermore it reduces the contact angle by reduces the surface energy between binder surfaces [1]. SA plays as important roles as a lubricant agent in terms of improving the powder wetting in feedstock [15]. Table-1 shows the combination of SS316L and NHAP characteristic while Table-2 reported the binder system Properties.

Table-1. The combination of (SS316L) and NHAP characteristic

Characteristic	Details
Identification	SS316L (PF-10F) / $Ca_{10} (PO_4)_6(OH_2)$
Powder Source	Epson Atmix Corp / Tilapia Fish bones
Tap Density g/m^3	4.06
True pynometer density g/m^3	8.0471/ 3.156
Powder Size	D10 = 2.06 µm D50 = 6.553 µm D90 = 17.712 µm

Table-2. Binder system properties [15]

Binder	Designation	Melting Temperature ˚C	Desitiy g/m^3
1 (Primary)	PMMA	257.77	1.19
2 (Secondary)	PEG	63.32	1.23
3 (Surfactant)	SA	70.1	0.94

MIXING

Mixing process is defined as mixed up the metal powder and binder system into a mixer with a correct volume ratio. The feedstock with 63 Vol % and 64 Vol % powder loading mixed with the binder consist of 73 % PEG; 25 % PMMA and 2% SA. These feedstocks were prepared using a sigma type blade mixer with a rotation frequency of 26 rpm. The mixing temperature was set at 70ºC, which is within the highest melting temperature and the lowest degradation temperature of the binder system [16, 3]. The mixture from mixing process should be crush by using Plastic Granulator SLM 50 FY to produce homogenous granules. Figure-1 exhibits the Feedstock in granules form.

Figure-1. Feedstock in granules form

INJECTION MOLDING

The feedstock was injected into the injection molding process. The sample that produced is called as green part. Pressure was applied on the feedstock and it forced it into the mold cavities. The feedstock solidified after the cooling stage and the pressure was released and then continuously with the ejected of green part. The parameter used in injection molding process has been set up to 130°C for nozzle, 125°C for the front, 120°C for the middle, 115°C for the rear 1 and 110°C for rear 2, and lastly 55°C for the feed. Series of trial injection processes have been done for the sample of feedstock with powder loading 63 Vol % and 64 Vol % powder loading. Figure -2 show the Horizontal Screw Type Injection Molding Machine while Figure-3 represents Sample of green part

Figure-2. Horizontal Screw Type Injection Molding Machine

Figure-3. Sample of green part

TRANSVERSE RUPTURE TEST (MPIF 15)

This testing are useful in determining the green strength of unsintered compacted powder metallurgy specimens by subjecting them to a uniformly increasing transverse loading under controlled conditions. The green strength is the stress required to break the powder metallurgy compact as a simple beam. The specimen is supported near the ends, and broken by applying the force midway between the fixed center of the supports [17]. The procedures of strength test begin with position the specimen in the transverse rupture test fixture so that it centrally located and perpendicular to the supporting rods, with the top surface facing upward after that, place the loaded fixture between the platens of the compression testing machine and apply a load to the nearest 0.4N with the cross-head velocity of the compression testing machine has been set at approximately 2.5 mm/ minutes and lastly Record the result of the breaking load [18]. Figure - 4 exhibit the Universal Tensile Machine

Figure-4. Universal Tensile Machine

DENSITY TEST (MPIF 42)

Density Test (MPIF 42) was conducted for measuring the density of powder metallurgy products having interconnected porosity by weighing the piece first in the air, second in water and then estimating the density. It is applicable to both green specimens and sintered parts. The density of powder metallurgy products is expressed in grams per cubic centimeter. The procedures of density test starts by fill the beaker with distilled water and suspend the universal holder for solids from the bracket prepared for non-floating samples. After that, record the temperature of distilled water. Then, put the sample in the pan and weighed in air and removed it. Afterwards, put the sample in the basket, ensures that no air bubbles adhere to the sample. Wait until the balance has reached stability and note the displayed weight of sample. Lastly, the density result will be printed by using mini printer provided [19]. Figure-5 shows Mettler Toledo XS 64 equipment for density determination.

Figure-5. Mettler Toledo XS 64

RESULTS AND DISCUSSIONS

SAMPLE DEFECTS

The samples of both 63 Vol % and 64 Vol % powder loading had defect such as ripping and cracked. This problem occurs during injection process which is the runner and gate of the part were stuck and sticky to the mold. So, the part cannot be pushed out to form a finer green part. These problems are having a connection with the properties of PEG that acted as a secondary binder in a feedstock. The melting point and temperature glass, Tg of PEG is about 63.3°C and -22.37 °C [16, 20] but, the mold temperature only could be set around 50°C. The low temperature glass of PEG automatically increased their time to solidify. That is the reason why the part are not cool enough even cooling time were adjusted and produce some parts defects. The mold temperature has a great effect on the surface quality, because temperature keeps the surface constantly heated and maintain the surface structure [21]. Figure -6 presented the defect of green parts

Figure-6. Defect of green parts

GREEN STRENGTH OF INJECTED PART

The terms of green strength is represent to mechanical strength of parts where as a compacted powder must have in order to withstand mechanical operations and it is subjected after pressing and before sintering, without damaging its fine details and sharp edges [15].This test has been conducted for samples of 63 Vol % and 64 Vol % powder loading. Table-3 and Table- 4 presented the green strength results that obtained from transverse rupture test, the calculation of green strength are referred the following Eqn. (1):

$$S = 3PL/2T^2W \quad (1)$$

Where:
S = green strength (MPa)
P = force (N) required to rupture
L = length of specimen span of fixture in inches (mm)
t = thickness of specimen in inches (mm), and
w = width of specimen in inches (mm)

Table-3. Maximum Force and Maximum stress for sample 63 % Vol Powder loading

Sampel.	Maximum Force	Maximum Stress
1	87.8469	11.1566
2	71.4531	9.07455
3	95.8375	12.1714

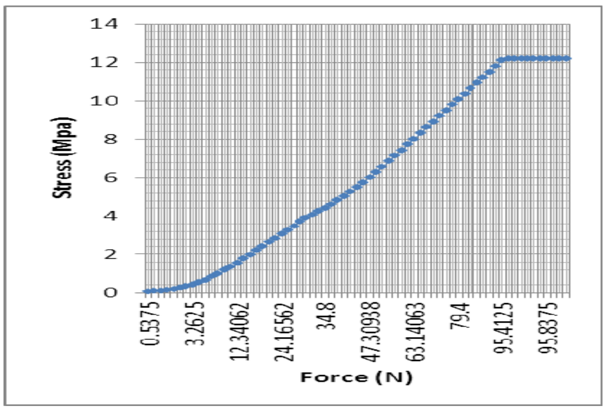

Figure-7. Graph of Maximum Stress against Force for 0.63PL

Table-4. Maximum Force and Maximum stress for sample 64 % Vol Powder loading

Sampel.	Maximum Force	Maximum Stress
1	95.8063	12.1674
2	116.862	14.8415
3	88.15	11.1951

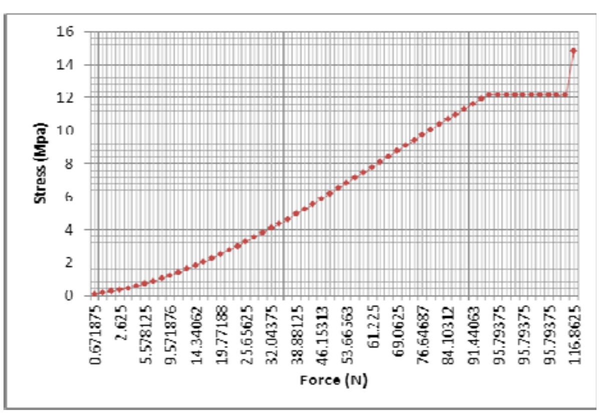

Figure-8. Graph of Maximum Stress against Force for 0.64PL

Figure-7 and Figure-8 demonstrate the highest value of maximum stress for 63 % Vol Powder loading are 12.1714 MPa with the maximum force 95.8375N. While for 64 % Vol Powder loading the highest maximum force equal to 116.862N and the maximum stress is 14.8415MPa. The green part with 64 % Vol Powder loading show the higher green strength compared to 63 % Vol Powder loading. This is due to the amount of metal powder in the green part which is according to [1], adding more metal powder results in greater injected part strength because of the compaction between the metal particles.

DENSITY DETERMINATION OF GREEN PART

Density of green part was measured using the Archimedes water immersion method according to MPIF 42 standard [22] by weighing the piece first in air, second in water and then estimating the density. The density is specified as the mass per unit volume of an unsintered powder metallurgy part and is expresses in g/cm^3 by using Mettler Toledo XS 64. The detailed of the result were exhibit in Table-5 and Figure-9 which is presented the Green part Density of 63 Vol % and 64 Vol % powder loading.

Table-5. Green part's density for 63 Vol % and 64 Vol % powder loading

Sample	Density (g/cm^3)	
	63 Vol %	64 Vol %
1	4.6462	4.7159
2	4.5920	4.6990
3	4.5015	4.7036

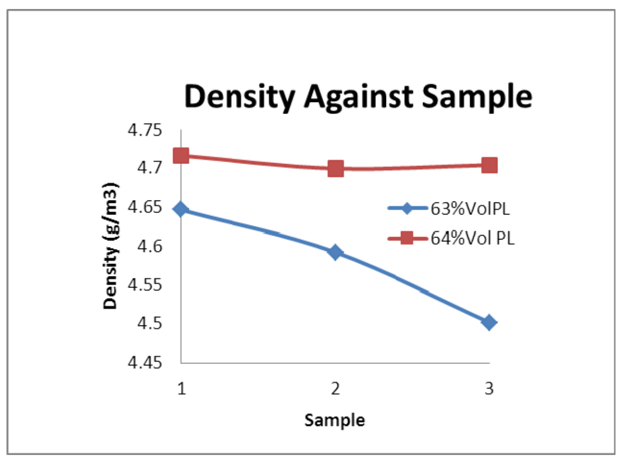

Figure-9. Graph of Density against sample for 63% Vol and 64 % Vol Powder Loading.

Based on Table 5 and Figure 9, the highest density for 63 Vol % powder loading is 4.6462 g/cm^3 while for 64 Vol % powder loading equal to 4.7159 g/cm^3. The green part with 64 Vol % powder loading was resulted in higher density value. Basically, mechanical properties such as density, strength and hardness increase gradually with powder loading. Moreover, the higher density also leads to the overall improvement of mechanical properties [23].

CONCLUSSION

In summary, the density and strength of the green part was determined. Green Part with 64 vol. % powder loading has higher density which is equal to 4.7159 g/cm^3 while 63 vol. % powders loading are 4.6462 g/cm^3. In terms of green strength, feedstock with 64 vol. % powder loading has higher maximum stress with the value 14.8415 Mpa compared to 63 vol. % powders loading has 12.1714 Mpa maximum stress. The mechanical properties of green part such as strength and density are increase gradually with the powder loading. This is proven with the findings that obtained for green part with 64 % Vol powder loading is higher for both strength and density compared to 63% Vol powder loading.

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REFERENCES

[1] German .R and Bose, Injection Molding of Metals and Ceramics. 1997 Powder Industries Federation U.S.A,

[2] Ramli. Mohd Ikram, Sulong. Abu Bakar. Arifin. Amir, Muchtar. Andanastuti, Muhammad. Norhamidi. 2012. Powder Injection Molding of SS316L / HA Composite : Rheological Properties and Mechanical Properties of the Green Part, Journal of Applied Sciences Research, 8(11): 5317-5321,

[3] Z.Y. Liu , N.H. Loh , S.B. Tor , K.A. Khor , Y. Murakoshi , R. Maeda. 2001. Binder system for micro powder injection molding, Materials Letters 48 , 31–38

[4] Arifin, Amir, Sulong, Abu Bakar, Muhamad, Norhamidi, Syarif, Junaidi, Ramli, Mohd Ikram, Powder injection molding of HA/Ti6Al4V. 2015. Composite using palm stearin as based binder for implant material, Materials and Design 65,2015 , 1028–1034 Contents.

[5] Horng Yih Juang and Min Hsiung Hon. 1996. Effect of calcination on sintering of Hvdroxvapatite. Elsevier Science Limited, Biomoterial (2059-2064)

[6] K.C. Tam, S.P. Yap, M.L. Foong , N.H. Loh. 2001. Metal Injection Molding: Effects of the Vinyl Acetate Content on Binder Behavior, Journal of Materials Processing Technology 67 (1997) 120-125

[7] Animesh Bose, Isamu.Otsuka. 2007. Metal Injection Molding Of Ultra-Fine 316L Stainless Steel Powders advances in Powder Metallurgy and Particulate Materials, Metal Powder Industries Federation.

[8] Chu Chenglin, Zhu Jingchuan, Yin Zhongda ,Wang Shidon. 1999. Hydroxyapatite–Ti functionally graded biomaterial fabricated by powder metallurgy, Materials Science and Engineering A271. 95–100

[9] M. Boutinguiza a, J. Pou, R. Comesana a, F. Lusquinos , A. de Carlos, B. Leon. 2012. Biological hydroxyapatite obtained from fish bones, Materials Science and Engineering C 32, 478–486

[10] Thian, E S, Loh, N H, Khor. KA, Tor. SB.2002. Ti-6A1-4V / HA composite feedstock for injection moldingMaterials Letters 56, 522–53

[11] Fan Xin, Chen Jian, Zou Jian Peng, Wan Qian, Zhou Cheng, Ruan Jian Ming.2009. Bone Like Apatite Formation on HA/316L Stainless Steel, Composite Surface in simulated body fluid. Trans.Nonferrous Met.Soc.China 19, 347-352

[12] M.A Omar , H.A Davies, P.F Messer, B. Ellis .2001.The influence o PMMA content on the properties of 316L stainless steel MIM compact. Journal of Material Science Technology (477-481)

[13] Chen, Gang, Cao, Peng, Wen, Guian, Edmonds, Neil. 2013. Debinding behaviour of a water soluble PEG/PMMA binder for Ti metal injection moulding, Materials Chemistry and Physics 139, 557- 565

[14] Torsten Henning.2002.Polyethylene glycols (PEGs) and the pharmaceutical industry, Clariant GmbH 65926 Frankfurt, Germany.

[15] M.H.I. Ibrahim, N. Muhamad, A.B. Sulong, K.R. Jamaludin, S. Ahmad and N.H.M. Nor. 2010. Optimization of Micro Metal Injection Molding For Highest Green Strength by Using Taguchi Method International Journal of Mechanical and Materials Engineering (IJMME), Vol. 5 No.2, 282-289

[16] M.H.I Ibrahim, N. Muhamad, A.B. Sulong, Murtadhahadi,K.R. Jamaludin, S. Ahmad, N.H.M Nor.2011. Rheological Charcterization Of Water Atomised Stainless Steel Powder for Micro Metal InjectionMolding , Advanced Materials Research Vols.264-265

[17] N.H.M. Nor, N.Muhammad, M.H.I.Ibrahim, K.R.Jamaludin.2011. Optimization of injection molding parameter of TI-6AL-4V powder mix with Palm Stearin and Polyethylene for the highest green strength by using taguchi method, International Journal of Mechanical and Materials Engineering (IJMME), Vol.6 ,No.1, 126-132

[18] Metal Powder Industries Federation Standard 15, Method for Determination of Green strength of unsintered compacted powder metullugt materials.

[19] Metal Powder Industries Federation Standard 42 Determination of Compacted or Sintered Powder Metallurgy Products

[20] Perkin Elmer, 2007. 940 Winter Street, Waltham, MA 02451 USA,

[21] Muhammad Ilman Hakimi Chua, Abu Bakar Sulong, Mohd Fazuri Abdullah ,Norhamidi Muhamad. 2013. Optimization of Injection Molding and Solvent Debinding Parameters of Stainless Steel Powder (SS316L) Based Feedstock for Metal Injection Molding ,Sains Malaysiana 42(12): 1743–1750

[22] Khairur Rijal Jamaludin, Norhamidi Muhamad, Sufizar Ahmad, Mohd Halim Irwan Ibrahim, Nor Hafiez Mohd Nor And Yusof Daud. 2011. Injection Moulding Temperature And Powder Loading Influence To The Metal Injection Moulding (Mim) Green Compact Scientific Research And Essays Vol. 6(21), Pp. 4532-4538,

[23] Yimin Li, Liujun Li, K.A. Khalil 2007. Effect of powder loading on metal injection molding stainless steels, Journal of Materials Processing Technology 183 432–439