PARAMETER OPTIMIZATION OF NATURAL HYDROXYAPATITE/SS316L VIA METAL INJECTION MOLDING (MIM)

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Abstract. Metal injection molding (MIM) are well known as a worldwide application of powder injection molding (PIM) where as applied the shaping concept and the beneficial of plastic injection molding but develops the applications to various high performance metals and alloys, plus metal matrix composites and ceramics. This study investigates the strength of green part by using stainless steel 316L/ Natural hydroxyapatite composite as a feedstock. Stainless steel 316L (SS316L) was mixed with Natural hydroxyapatite (NHAP) by adding 40 wt. % Low Density Polyethylene and 60 %wt. Palm Stearin as a binder system at 63 wt. % powder loading consist of 90 % wt. of SS316 L and 10 wt. % NHAP prepared thru critical powder volume percentage (CPVC). Taguchi method was functional as a tool in determining the optimum green strength for Metal Injection Molding (MIM) parameters. The green strength was optimized with 4 significant injection parameter such as Injection temperature (A), Mold temperature (B), Pressure (C) and Speed (D) were selected throughout screening process. An orthogonal array of L\(_9\) (3\(^4\)) was conducted. The optimum injection parameters for highest green strength were established at A\(_1\), B\(_2\), C\(_0\) and D\(_1\) and where as calculated based on Signal to Noise Ratio.

Introduction

Metal injection molding is a new technologies that offers to produce metal and ceramic in small and intricate part geometry with more economically and large volume of production which is consist of four major process in MIM includes of mixing, molding, debinding and sintering [1,2]. In metal injection molding, stainless steel has become as a most popular materials and at the mean time broadly used as metallic implant material in medical implant application. In term of medical application, SS316L have an excessive mechanical property such as fracture toughness, fatigue strength and cost effectiveness [1, 3, 4]. SS316 L water atomized was selected as a metal powder since owing to their superior characterization in MIM such as, high corrosion resistance, high packing densities and non spherical particle shape plus resulted to the higher interparticle friction [5,6, 7].

Lately, hydroxyapatite (HAP) was revealed to be an attractive material for biomedical applications since it offerings a chemical composition close to the bone mineral [8]. Hydroxyapatites have resembled properties to bone structure and teeth based on the chemical and structural observation. HA with the stoichiometric formula \(\text{Ca}_{10}(\text{PO}_4)_{6}(\text{OH}_2)\) and a Ca/P molar ratio=1.67 respectively. HA is a promising biomaterials for bone substitutes and have an excellent properties such as biocompatible, osteoconductive, non-toxic, non inflammatory, non immunogenic agent, and bioactive. Bioactive denotes to the ability to form a direct chemical bond with living tissues [9, 10].
The green part of SS316L/NHAP feedstock were fabricate by using the high class features of the MIM process that can be practical to manufacture the metal matrix composite (MMC) or ceramic matrix composite (CMC) parts [11] and established by earlier researcher such as [4,12,13,14] but they laboring the synthetic HA. Sample that manufactured from MIM process are recognized as a green part. Usually, the properties of green part have to be evaluating in terms of density, strength and defect. Even though the MIM process compromises many advantages, it requires the proper molding condition to produce better quality of green part. Regarding to this concern, an optimization of injection molding parameter could be increased or decreased the quality of green part hooked on a tolerable assessment [15,6].

Taguchi’s parameter design is an imperative tool for robust design. It promises a simple and systematic approach to optimize design for performance, quality and cost. Robust design applied two major tools which is Signal to noise ratio and orthogonal array. Signal to noise ratio (S/N), concentrate on measures quality with stress on variation, and orthogonal arrays, which accommodates various design factors simultaneously [16].

Taguchi method applied the Design Experiment (DOE) techniques owing a systematic and efficient methodology and outcomes the optimum combination input parameter of process [17, 18, 19]. The beneficial of Taguchi Method includes simplification of experimental plan and feasibility study of interaction between different parameters. Previous researchers that work with Taguchi technique are [20, 6]. Taguchi Methods also popular as the best method in order to optimize the design, performance and costing issue [17].

Taguchi method is supreme technique in order to optimize the green part in metal injection molding [21]. There are various injection parameters were optimized in determination the highest green strength which is injection pressure (A), injection parameter (B), mold temperature (C), injection time (D) and holding time (E) and the outcome proves that Taguchi Method could be the best methods in order to solve the problem with a minimum number of trials [6,22]. Other researcher that used Taguchi method as a tool in injection molding optimization parameter is [23, 24].

The main objective of this paper is to optimize the injection parameter for highest green strength by using Taguchi (L₉³⁴) Orthogonal Array. As addition information, NHAP was derived from Tilapia fish bone used to apply as ceramic powder in metal injection molding with the combination of metal powder SS316 L and Low density Polyethylene (LDPE) and Palm Stearin as binder system.

**Materials and Methods**

SS316L water atomized (Epson Atmix Corp) with irregular shapes and high corrosion resistance was selected as a metal powder and NHAP powder was categorized as a ceramic powder. The Tilapia fish bone was subjected to calcination process in order to produce NHAP powder. Table 1 presented Combination characteristic of SS316L water atomized powder/NHAP characteristic. Low Density Polyethylene (LDPE) and Palm stearin (PS) were used as binder component in this research. LDPE and PS are well known as an established binder system in MIM and the detailed properties described in Table 2. The importance of binder system are plays as transportation agent for feedstock homogeneity and packing powder into desired shaped before sintering phases [25].
Table 1. Combination characteristic of SS316L water atomized powder/NHAP characteristic Details

| Identification | SS31L PF-10F / Ca₁₀ | PO₄₆(OH)₂ |
|----------------|---------------------|-----------|
| Powder source  | Epson Atomix Corp / Tilapia Fish bones |
| Tap density g/m³ | 4.06 |
| True pycnometer density g/m³ | 8.0471/ 3.156 |
| Powder size    | D10 = 2.06 μm |
|                | D50 = 6.553 μm |
|                | D90 = 17.712 μm |

Table 2. Binder Properties

| Binder         | Type    | Designation | Composition % | Melting Temperature °C | Density g/cm³ |
|----------------|---------|-------------|---------------|------------------------|---------------|
| Palm Stearin   | Primary | PS          | 60            | 70                     | 0.891         |
| Low Density    | Secondary| LDPE        | 40            | 111.5                  | 0.92          |

Feedstock Mixture

Theoretically, the combination of powder and binder should be homogeneous and injectable [25, 26]. Concerning to this facts, feedstock was produced by mixing the combination of NHAP/SS316L powder and binder system with suitable composition thru plastograph brabinder. Palm Stearin (PS) was acted as a primary binder where as LDPE was secondary binder system. The mixing temperature used is 150°C by referring the melting point of LDPE which is occurs at 111.5°C. Mixing temperature chooses based on melting point of the secondary binder system temperature. The mixing temperature were set up higher compare to highest melting point and lower than the lowest degradation temperature [27]. The composition ratio between NHAP powder and SS316L is 90 % SS316L and 10% NHAP at 63 Vol % powder loading. Moreover, the composition for binder systems is 60 Vol% Palm Stearin and 40 Vol% LDPE. The optimum powder loading usually in range of 2% -5% lower than critical powder volume concentration (CPVC) [1, 28.] The value for CPVC for NHAP/SS316L feedstock was found to be 68.189 Vol %. In consequence, this paper are concentrate on investigate the feedstock with 63 Vol % powder loading consider within optimal powder loading range.

Taguchi Method

Parameter optimization of Natural Hydroxyapatite/SS316L via Injection molding process is an important process in order to produce the higher strength and great quality green part. The injection parameters are nominated based on the most significant parameter via screening trial by using classical analysis of variance (ANOVA). From ANOVA results the whole control factors are orthogonal, hence interactions effects are neglected [29, 30] the preferred injection parameters are injection temperature, mold temperature, pressure and speed.

The optimization process are conducted by using L₉ (3⁴) Orthogonal Array (OA) which is proposed of three level designs of experiment with 4 selected parameter in 9 trial. Orthogonal arrays can be develops as plans of multiple factor of experiments. The columns denote to the factors, though,
the entries in the represents to the test levels of the factors and the rows resemble to the test runs [31]. Table 3 demonstrates the three level of injection parameter design

| Indicator | Parameter                        | 0   | 1   | 2   |
|-----------|----------------------------------|-----|-----|-----|
| A         | Injection Temperature (˚C)       | 165 | 170 | 175 |
| B         | Mold Temperature (˚C)            | 40  | 45  | 50  |
| C         | Injection Pressure (%)           | 55  | 60  | 65  |
| D         | Speed (%)                        | 55  | 60  | 65  |

**Green Strength Determination (MPIF 15)**

This testing are beneficial in determining the green strength of unsintered compacted powder metallurgy specimens by subjecting them to a uniformly increasing transverse loading under controlled conditions. Green strength is representing 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 centres of the supports [32].

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. [33] Figure 3 exhibit the Universal Tensile Machine

![Universal Tensile Machine](image-url)

**Figure 1. Universal Tensile Machine**
The calculation for green strength is referred the following equation [6]:

\[ S = \frac{3PL}{2T^2W} \]  

(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)

**Results and Discussion**

Green strength optimization in MIM is significance process and it focused on investigate the performance and achieves the superiority green part. Signal refer to desirables value (mean) for the output characteristic whereas Noise can be observed as the undesirables value (S.D) of output characteristic [6, 34]. S/N ratios approach are applied to measure the quality characteristic diverge from the desired value [32].

Additionally, S/N ratios compromise as an alternative approach to transform the experimental outcomes into a value for the calculation characteristic in the optimal parameter analysis [34, 35]. S/N ratios can be divided into three characteristics which is larger is better, nominal is better and smaller is better [17, 36]. However, in this paper, larger the better signal type is selected as a green strength measurement characteristics. The data is interprets in S/N ratio emphasizes to obtain the optimal performance of green part [6]. S/N ratios Larger is better could be defined as equation 2

\[ \frac{S}{N} = -10 \log \left( \frac{1}{n} \sum_{i=1}^{n} \frac{1}{Y_{ij}^2} \right) \]  

(2)

Where \( Y_{ij} \) = the amount of score for the green density
- \( N \) = the total number of shots for each trial

As mention before, optimization injection parameter for the strength of the green part were using Taguchi method. Optimization performance processes by Taguchi methods are very popular among researcher especially in engineering analysis and manufacturing process. Fundamentally, Taguchi method optimizes the performance characteristics over the setting of design parameters. Taguchi method is also robust tool for the design of high quality systems. A model based on L9 orthogonal array of Taguchi method was created by employing the S/N ratio optimization process [24]. Table 4 exhibit the Taguchi’s L9(3)4 orthogonal arrays and demonstrates the value of experimental trials (strength) and quality characteristic.
The highest green strength for Natural hydroxyapatite/SS316L feedstock was obtained by using the optimum configuration of injection molding. By referring figure 2, the highest peak for each parameter $A_1$, $B_2$, $C_0$ and $D_1$ presented the optimum configuration. In simplest explanation it reflect to Injection Temperature 170 °C, Mold Temperature 50 °C, Pressure 55 % and Speed 60 %. The main effects plot is developed from Table 4 above by using the mean of S/N ratio. The mean S/N value will be calculated from calculation below:

$$\frac{13.973+13.792+14.058+14.752+14.605+15.538+14.603+14.245+14.229}{9} = 14.422$$

Table 4. Taguchi’s $L_9(3)^4$ orthogonal arrays demonstrate the value of experimental trials (strength) and quality characteristic

| Factors | S/N ratio : Larger is Better |
|---------|------------------------------|
| Trial   | A  | B  | C  | D  | Average S/N ratios |
| 1       | 0  | 0  | 0  | 0  | 4.996 13.973       |
| 2       | 0  | 1  | 1  | 1  | 4.893 13.792       |
| 3       | 0  | 2  | 2  | 2  | 5.045 14.058       |
| 4       | 1  | 0  | 1  | 2  | 5.465 14.752       |
| 5       | 1  | 1  | 2  | 0  | 5.374 14.605       |
| 6       | 1  | 2  | 0  | 1  | 5.983 15.538       |
| 7       | 2  | 0  | 2  | 1  | 5.372 14.603       |
| 8       | 2  | 1  | 0  | 2  | 5.155 14.245       |
| 9       | 2  | 2  | 1  | 0  | 5.146 14.229       |

$\sum_{i=1}^{9} S/N = 129.795$  
$\frac{129.795}{9} = 14.422$

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$$\frac{13.973+13.792+14.058+14.752+14.605+15.538+14.603+14.245+14.229}{9} = 14.422$$

**Figure 2.** Main effects plot (data means) for S/N ratio
Table 5 expressed the detailed response for green strength S/N values and it can further be noted from the rank of the parameters that variation in the value of S/N ratio. The maximum parameter is injection pressure and least for pressure. The optimum of injection parameter was measured by calculates the variances sensitivity between highest and lowest value of S/N ratio for each factor [30]. The most contribute factor to produce strong green part is injection temperature with 1.02 dB delta values. The injection temperature used here is 170 °C where as to be found at moderate level. Conceivably, too higher injection temperature could lead possibilities of injection defect such as powder binder separation and other defects related with injection molding and produce a brittle green part. [6, 34, 37]. The following rank is mold temperature recorded at 0.39 dB for delta value. Based on figure 2, the optimum mold temperature are at 50 °C which is placed at higher level. The higher mold temperature could avoid possibilities of feedstock to stop flow into the die cavity, rapid freezing on the mold walls and caused cracking green part [38]. Besides, at higher mold temperature, the shear rate between the feedstock surface and cavity surface area will be decrease [39]. Consequently, heating the mold is compulsory to prevent the defects [1]. The third rank is speed with 0.38 dB delta values and the fourth rank is pressure with 0.33 dB value. The moderate speed and pressure usually used in order to produce a quality green part [1, 6].

Table 5. Response Table for S/N Values

| Level | Injection Temperature | Mold Temperature | Pressure | Speed |
|-------|-----------------------|------------------|----------|-------|
| 0     | 13.94                 | 14.44            | 14.59    | 14.27 |
| 1     | 14.96                 | 14.21            | 14.26    | 14.64 |
| 2     | 14.36                 | 14.61            | 14.42    | 14.35 |
| Delta | 1.02                  | 0.39             | 0.33     | 0.38  |
| Rank  | 1                     | 2                | 4        | 3     |

Conclusions
This paper summarizes experimental investigations carried out on optimization injection process via L9 (3^4) Taguchi Method for SS316l/NHAP composite as a feedstock in MIM. The results shows the optimum parameter for highest green strength is A1, B2, C0 and D1 represents to Injection Temperature 170 °C, Mold Temperature 50 °C, Pressure 55 % and Speed 60.

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