Sintered Strength of Ti-6Al-4V by Metal Injection Molding (MIM) using Palm Stearin Binder

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ABSTRACT

Metal injection molding (MIM) is a metal working process used to compact Titanium, Ti-6Al-4V and the binder through injection moulding process. In this work, Titanium Ti-6Al-4V was mixed with palm stearin (PS) and polyethylene (PE) which act as a binder. Palm stearin (PS) is a potential binder system that can be used because it is inexpensive and highly available in Malaysia. Besides that, the application of MIM can reduce time and energy compared to the conventional process which high cost in production and difficulty of producing complex shape of materials. The green parts were then gone through of debinding process and sintering process. This project is to study the effect of the sintered Ti-Al-4V using palm stearin as a binder system. The properties of sintered specimen were investigated by physical properties such as its shrinkage and density. The average shrinkage of the sintered specimen was about 10.35% in length and 10.96% of width body of the specimen. Density of the sintered specimen was about 4.0343 g/cm3 and the tensile strength of the specimen was about 805.7 MPa.

Keywords: *Metal Injection Molding (MIM), palm stearin, Ti-6Al-4V, sintering and strength*

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Introduction

Powder metallurgy is the art and science of producing metal powders and utilizing them to make serviceable objects. In other words, it also can be defined as material processing technique which used to consolidate particulate matter for example metal or non-metals powder [1]. The combination between plastic injection molding and conventional powder metallurgy technologies will create the metal injection molding (MIM) process [2]. MIM process is an alternative for manufacturing process for small and medium shaped precision component that were beforehand produced by conventional techniques at a higher cost. It fits high efficiency and high complex shaped [3]. MIM can reduced a cost to manufactured depending on the requirements of complexity and the quantity of specimen that to be produced, especially when compared to other conventional method such as machining, die casting, and investment casting as shown in Figure 1[4].

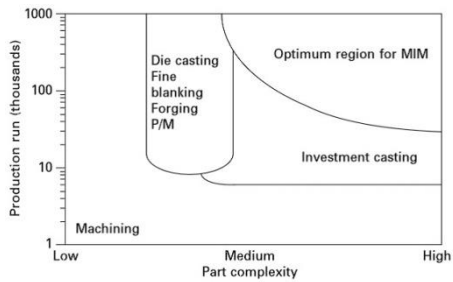

Figure 1: Comparison of manufacturing technique as influenced by quantity of specimen and complexity [4]

After commercialization of MIM process in industry, the technology reached their mature and has been accepted in many industries, including automotive application, medical and dental instruments, orthodontic, firearms, hardware and computer and electrical systems [5]. Typically, the MIM product are 95% to 98% dense, approaching wrought material properties. Besides that, the MIM products get a higher strength, good corrosion resistance, and improved magnetic properties compared to the conventional process [5-6]. Basically, metal injection molding (MIM) had four processing steps:

The first step is mixing which involve compounding the metal powder and binder into feedstock. The binder usually based on a common thermoplastic such as polyethylene or wax. However, food-grade polymers, gels, water, and various inorganic substances are also in use. Normally, the binder system consists of two or three component [7-8]. Feedstock is a term for the mixture of powder and binder. The feedstock is then granulated. The feedstock will be heated into the machine injection molding before injected

into a mold cavity under pressure to produce green specimen. Second step in metal injection molding is molding which shape the part from feedstock as in plastic injection molding. Next step is debinding which aims to remove the binder in the molded part by pyrolysis or solvent soaking. It will then produce brown specimen which is very brittle and needs to be handled with extra care. Lastly, sintering process will carry out which this step aims to identifying the debound parts to a high final density. Sintering can be incorporated directly into a thermal debinding cycle. Sintering bond the particles together and lead to densification. Usually sintering shrinkage is uniform and isotropic, so the molded component is oversized to deliver the desired final dimensions [9-12].

Methodology

Characterization of powder Ti-6Al-4V

In this project, Ti powder (<20μm) were used. The particle size distribution of the powder was determined using Malvern Instrument Mastersizer 2000 apparatus. The density and particle size distribution of materials titanium (Ti) alloy was determined and the data were recorded using micromeritics AccuPyc II 1340 and Malvern Instrument Mastersizer 2000 apparatus respectively as shown in Table 1.

Table 1: Properties of Titanium alloy

Material Characteristics	**Properties Value**
Particle size (μm)	20.414
Density (g/cm^3)	4.4164
Melting Temperature (^oC)	1660

Characterization of binder

In this study, the binders used were combination between binder PS, and PE. From previous study that have been carried out by researcher before, the characterization of the binder components used was determined by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA).

Mixing Calculation

The feedstocks are consisting a balanced mixture of metal powder and binder components. The mixture between these two components can determine the success or failure of the next step in metal injection moulding process. There are three possible situations that could happen in the mixing process, which are excess of the binder, critical binder concentration, and insufficient of binder. These situations will give different result. For excess binder, it will separate from the metal powder in moulding, lead to flashing, and the most important things are leading to component slumping during debinding. Too little binder will cause high viscosity and trapped air pockets which increase

the difficulties in the moulding. A feedstock is calculated from a weight basis, not a volume basis. The weight fraction of powder Wp can be determine by using Equation below.

$$Wp = \frac{\rho_P \phi}{\rho_P \phi + \rho_B (1 - \phi)}$$

where;

	Wp	weight fraction of the powder
	ρ_P	density powder (g/cm3)
	ϕ	powder loading
	ρ_B	average density of binder system (g/cm3)

For the binder system in MIM process, usually there is more one component that will be used. Because of that, to find the average density of the binder system ρ_B, the equation below can be used.

$$\rho_B = \frac{1}{\frac{fraction\ of\ binder\ 1}{density\ of\ binder\ 1} + \frac{fraction\ of\ binder\ 2}{density\ of\ binder\ 2}}$$

Feedstock Preparation

Feedstock preparation involved of mixing the metal powder and binder system using z-blade mixer. The maximum limit of the z-blade mixer was about 1 litre, but for this feedstock preparation, the powder weight was 250 g.

The temperature for the mixer was heated up to 150°C which above the melting point of the PE binder system. The blade and screw were rotating at 60 rpm for 1 hour. After completing the mixing, the feed-stock could cool with the blade still in motion about 30 minutes. In this pro-cess, there is method during mixing this component. Firstly, PE must be put inside the mixer follow by PS after a few minutes of mixing the PE. Titanium will be the last one to be mix inside the mixer. This method to ensure the entire component will mix together completely and in order.

Injection molding process

The feedstocks were injecting to the mould using a vertical MCP-100KSA injection molding. The temperature on the panel and injection timer was set first. The barrel temperature must be allowed to run about 5 to 10 minutes to stabilize before starting the moulding. The feedstock was put into the barrel and injected through the nozzle into the mold cavity.

In this study, a plunger which driven by pressure from compressor will force the material to be injected into the mold cavity. Pressure must be set correctly because if pressure is too high, it will cause ‘flash’ because the mold is open. Vice versa for that, if pressure is too low, incomplete molding will occur. The barrel temperature must be set higher than the highest melting point

of the binder system. The temperature selection will be based on which temperature will produced satisfactory molding. The molded part known as green part will be removing from the mold. Dimension of the green parts that were free from defects were measured and recorded. It is important for further process to study the changes in the dimension for the next process could be followed. Each specimen was measured along its length, its thick-ness, and its width. The weight of the specimens also was recorded.

For the incomplete specimen produce, it will be crush using crusher machine to make it in form of feedstock again. Then, it will inject again to form a good and desire shape of specimen.

Solvent Debinding

For solvent debinding, the solvent was heat up by put a beaker in a water bath. Heptane was used as agent to remove the PS binder. It was put into the water bath container and completely covers the samples. The temperature was set up at 60 ºC and hold for 6 hours for the leaching process. After soaking debinding process is done, the specimen will be dried in oven for a night to ensure that that specimen has completely dried and have the net weight without excess of binder PS anymore. Then, the weight loss was determined using a balance with a precision of 0.0001 g and the dimensions were measured using the micrometre.

Thermal Debinding

The remaining binders in the specimens were removed by thermal debinding technique after solvent debinding process is finish. This process was one cycle with the next process which is sintering process. The specimens were placed on Yithria plate as shown in Figure 3.4. The calcium hydrate powder (CaH2) was put on top of the specimens before proceed into the furnace for thermal debinding and sintering process.

The heating rate for this process was about 1 ºC/min under gas argon. The specimens was heated up at room temperature to 550 ºC and soaked for 1 hour. For the temperature of thermal binding process was determined based on the TGA analysis of the binder system at which the point that the binder will be decomposed. This process was continuing with the sintering process.

Sintering process

By using the same equipment for the thermal debinding process, the furnace was heated up to 800 ºC with the heating rate was about 5 ºC/min. Then, the specimens were soaked for 1 hour before heated up to 1200 ºC with same heating rate. After that, specimens were soaked for two hours before the specimen cooled into the room temperature for 4 hours. The schematically diagram for the thermal debinding and sintering process were shown in Figure 2.

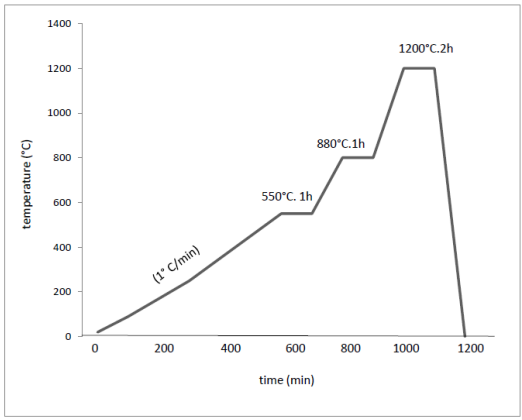

Figure 2: Schematic thermal cycles for debinding and sintering process

Shrinkage

Dimensions of the specimen were measured after sintering process to determine the change in size of green part. The purpose of doing this is to evaluate the shrinkage of the specimen after being sintered. The metal powders have basic characteristics to change the size. The difference between length of the green specimens and the sintered specimens can be expressed in terms of percentage. The percentage of the shrinkage, *L* (%) can be calculated using the Equation below.

$$L(\%) = \frac{L_{gr} - L_s}{L_{gr}} \times 100$$

Where; L_{gr} length of green part (mm)
L_s length of sintered part (mm)

Density

Density of the sintered can be determined by using Archimedes Principle. The density can be calculated using the Equation below.

$$\rho = \frac{A}{(A - B)} \times (\rho_o - d) + d$$

Where; ρ density of the sample
A weight of sample on air
B weight of sample in liquid
ρ_o density of liquid
d density of air

Ultimate Tensile Strength (UTS)
The UTS of the sintered specimens was determined using "Instron Universal Testing Machine 100kN". In this test, speed rate use is at rate 0.5mm/min. The peak load at the maximum load was recorded. This test was done by performing 3 samples in order to obtain average result.

The UTS of the test specimen can be determined using equation below:

$$\text{UTS} = \frac{\sigma}{A}$$

Where; σ stress at ultimate load (MPa)
A cross sectional area of sample (mm^2)

Results and Discussion

Particle Size Distribution
The particle size distribution was done to analyse its range of particle size. Figure 3 showed the result of particle size distribution for *Ti* powder.

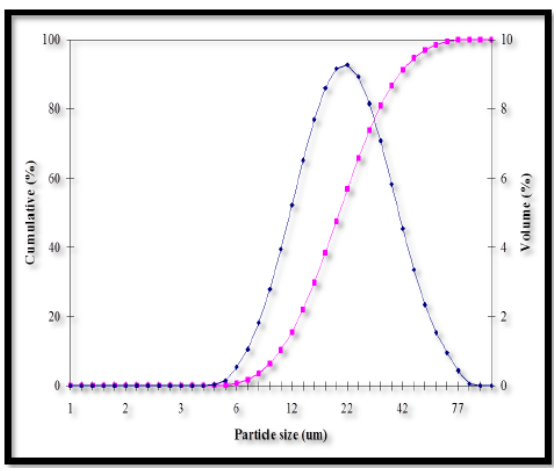

Figure 3: Particle size distribution and the cumulative percentage of the titanium powder

Powder Density
Density of the powder were obtained from the experimental using a gas pycnometer test. Helium gas is used in this test. Table 2 below is the result

obtained from this experiment. Average density of Ti powder is 4.4164 g/cm^3.

Table 2: Pycnometer densities result of Titanium alloy

Run	Density (g/cm3)
1	4.4235
2	4.4208
3	4.4159
4	4.4124
5	4.4091
Average	4.4164

Density of Binder Components

Helium gas is used in gas pycnometer to test the densities of the binder components. The result from the experiment was shown in Table 3.

Table 3: Density of Binder Component

Type of binder	Density (g/m3)
PS	0.9613
PE	0.9528

Densities of these binders are important to determine the weight fraction of the binder that should be used to form a feedstock during mixing process. From the result of density, it found that the difference in term of weight between these two components is not much. For this project, 60% wt of PS and 40% wt of PE were used to mix with 60vol% of Titanium powder.

Injection molding evaluation

For injection molding process, to get the desired shape that is free from any defects the parameter of the nozzle was optimized. The minimum temperature of the nozzle must be above of the highest melting point of the binder component which is above 106.80 ºC (melting point of PE). The ideal temperature for this injection molding process was at 150 ºC. After injection molding process is done, the green parts will be produced, and the next step is to study the green part of the specimen. In this project, small dog bone size mold is used to produce the specimen. Figure 4 show the tensile shape of the specimen with a single gate located at one end of the specimen. From this process, 10 samples of green specimens have been produced and they were free from any defects such as flashes at the surface, short shot, and binder separating.

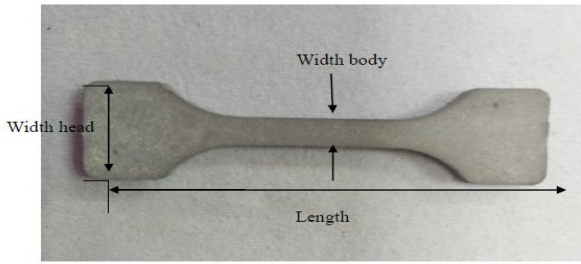

Figure 4: The green part of the specimen

Dimensions of the green part were recorded in the Table 4 below. Dimension of the green part were measured using micrometre in units of mm. The weight of the specimen was being recorded and the result show the weight average of the specimens was about 7.2109g.

Table 4: Dimensions of the green part

Parameter	**Dimension (mm)**
Length	59.62
Width (head)	15.05
Width (body)	4.56
Thickness	4.55

Properties of sintered specimen

Shrinkage

Sintering is the final process in injection molding. In this process, all the binder component was completely remove from the green part and this mean the particle will have a strong bonding when heat is applied. The dimension of sintered specimen will absolutely reduce from the green part specimen as shown in Figure 5.

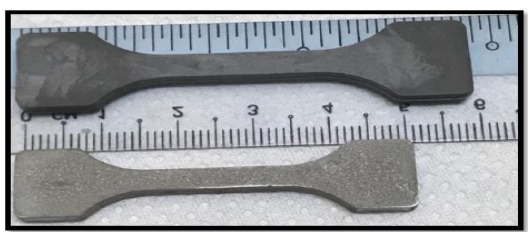

Figure 5: Comparison of dimension between green specimens and sintered specimens

The dimension of all sintered part in average was recorded in Table 5. For the shrinkage, the length of the specimen was show that the specimens reduce about 10.55% and for the width of the body specimen about 10.99%.

Table 5: The dimension of the sintered specimen

Parameter	**Dimensions (mm)**	**Shrinkage (%)**
Length	53.45	10.35
Width (head)	13.40	10.96
Width (body)	4.03	11.62
Thickness	4.39	2.42

Density

Density of the sintered specimen was obtained by using Archimedes principle. All the sintered specimens were doing this test because it does not effect for another test and also to get more accurate result from this experimental. From the average result, the density of the sintered specimens was about 4.0343 g/cm3.

Table 6: Density of the sintered specimens

Specimen	**Density (g/cm3)**
1	4.0390
2	4.0291
3	4.0348
Average	**4.0343**

Ultimate Tensile Strength (UTS)

From the graph that shown in Figure 6, the minimum elastic modulus, (E) of the sample was 44.23 GPa and the maximum was 62.67 GPa. The average of modulus elasticity from this experiment was about 55.90 GPa. These value was lower compare to previous research, that usually got a modulus of elasticity at about 70-116 GPa [13,14].

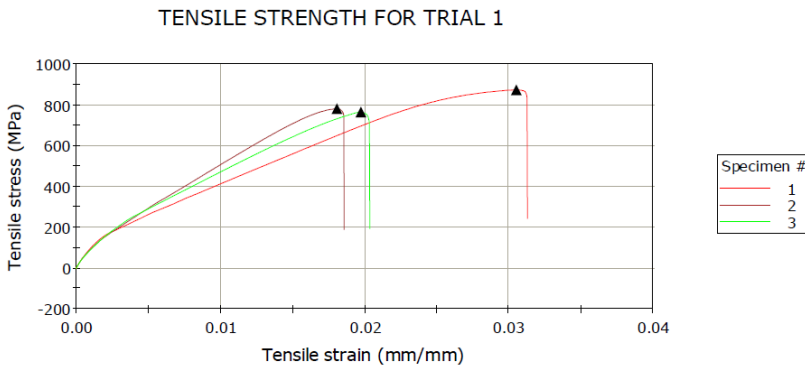

Figure 6: Stress-strain curve from tensile test of MIM Ti specimens

For the tensile stress that can be obtained at the maximum load, it shows that the minimum tensile stress was about 764.4 MPa, and the maximum at 872.3 MPa. The average of the tensile stress for this experiment was about 805.7 MPa. It shows that the tensile stress that have been obtain was much lower compare to the previous research that normally should be around 531-768 MPa. In theory, the sintered body that has a high porosity effect will reduce the density and thereby weaken the strength of the body [13]. This is evident since the sinter body has a low density compared to other researchers [13,14].

Conclusion

In this experiment, by using MIM process, Titanium based alloy powder are successfully developed with a new binder which is PS. This process has a lot of advantages compared to conventional method. The application of MIM can reduce time and energy and it is recommended in industry to reduce their cost of processing. Besides, it was proven that that PS binder can be used as a binder component in this process. This is because the combination of PS and PE worked successfully in this process. They bonded each other completely. The properties of sintered specimen also have been evaluated. From the tests that have been carried out it show that the average shrinkage for the length of sintered specimen was about 10.35%. The average density of the sintered specimen for 5 samples are 4.0343 g/cm3.

However, the mechanical properties of the sintered specimen show a lower in tensile strength compare to previous research. The ultimate tensile strength of the specimen was about 805.7 MPa. These result caused by many factors. This is due to some error occurs during carried out the experiment. In conclusion, the objectives of this project have achieved.

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