CHARACTERIZATION OF INJECTION MOLDED 17-4PH STAINLESS STEEL PREPARED WITH WASTE RUBBER BINDER

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ABSTRACT

This study is to investigate the sintering characteristics and to establish the best heating rate and soaking time used for sintering process, by determining the physical, mechanical, and microstructural properties of the injection molded 17-4PH stainless steel using waste rubber as a new developed binder system. By using the feedstock which having 65 vol.% of metal powder, the molding are injected into the tensile test bar and immediately processed with two stage debinding process that involves of solvent extraction and thermal pyrolisis to remove the binder. The specimens were sintered at 1360°C under vacuum atmosphere and tested for a critical property analysis of tensile test. Later, the observation on tensile tested specimens fracture surface are done to understand the fracture behavior, distribution of grain and porosity and the significant correlation of fracture morphology to the mechanical properties. From this study, it is found that the combination of 5^0C/min heating rate and 60 minutes of soaking period resulted in higher density value, higher tensile strength, less porosity and homogenous grains distribution of the sintered specimens.

KEYWORDS*: Injection molded; sintering; binder system; stainless steel powder; waste rubber.*

1.0 INTRODUCTION

There are many improvisation and development in the field of powder metallurgy. Newly invented and the very recent method of powder metallurgy is by combining the concept of plastic injection molding

with the utilization of metal powder feedstock (Rodriguez-Senin *et.a*l., 2005). However, this process is more complicated than regular plastic injection molding process. This is due to the process based on the use of fine powder particles mixed with the small quantity of wax binders and/or thermoplastic polymer to form the molded feedstock.

As the novel binder system in Metal Injection Molding (MIM) processing, the use of waste rubber to replace natural rubber is receiving great attention due to the advantage of renewability, thermal stability and high shear viscosity (Tan *et.al*., 2008). In addition, a new binder system used in Metal Injection Molding (MIM) processing also exhibit economical and environmental friendly characteristics especially for the application in automotive, tooling, medical and hardware component. In this study, the waste rubber was utilized, formulated and evaluated as a new binder system for the MIM processing due to its advantageous.

2.0 EXPERIMENT

2.1 Materials

Basically, every process involved in Metal Injection Molding (MIM) has significant impacts to the characteristics of starting materials since the powder and the binder are critically important to the overall success of this process. In this research, the main raw material used is 17-4PH stainless steel and the binder systems consist of paraffin wax (PW), thermoplastic waste rubber (TPWR) and stearic acid (SA) combination, while the feedstock consists of 65 vol. % of powder loading and 35 vol. % of the binder system refers to the proportion selected based on previous study (Omar and Subuki, 2006).

2.2 Specimen Preparation

The specimen samples were prepared through MIM process which involved the mixing, injection molding, debinding and sintering process. Prior to the MIM operation, the mixing process must be undertaken to produce a homogenous feedstock. By using Z-Blade mixer type, the powder and binder were mixed together all in one batch. The mixing process was operated at temperature condition of 175^0C for about two hours to produce a granulated feedstock according to the parameters used on the previous study (Omar and Subuki, 2006; Gulsoy *et.al*., 2007). This granulated feedstock was injected into a tool with tensile bar-shaped cavity using a vertically aligned and pneumatically operated plunger machine (MCP HEK-GMBH Vertical

Injection Molding) operating at temperature of 170^0C, 6 bar of injection pressure, and 0.25 seconds of cycle time in order to produce good surface finish of green body.

The two stage operations, namely solvent extraction and thermal pyrolisis (Figure 1a), must be performed in order to remove the binder system through the process of debinding within the feedstock. Solvent extraction was first performed using water bath brand Memmert and then the samples were immersed in the heptane in order to remove all soluble binder. The dimension of green body specimens were measured prior to the solvent extraction process. Thereafter, the green body specimens were arranged in a glass container containing heptanes that must be heated at 60^0C and half immersed in the tap water (Omar and Subuki, 2006). The specimens were then immersed for about five hours. To prevent the evaporation of the heptane solvent during the extraction operation, the glass container should be covered. When the extraction completed at about five hours, the specimens were removed and left for drying in the oven at the temperature of 40^0C for four hours to remove the remaining solvent.

Next, the thermal pyrolisis or debinding is carried out in Linn furnace to remove the remaining binder that cannot be completely removed during the solvent extraction operation. Thermal debinding operation used two different heating rates which are 5^0C/min up to temperature 250^0C for the first stage and 5^0C/min up to temperature 450^0C for the second stage. About one hour of soaking period is allocated for each stage, before the cooling operation.

2.3 SINTERING

Sintering is a processing technique to produce density-controlled materials and components from the metal powder by applying thermal energy (Kang, 2005). Therefore, the study on the effect of heating rate and soaking period of sintering was based on the operation in hot wall multi-atmosphere sintering furnace in the vacuum condition with sintering temperature at about 1360^0C. The sintering profile can be found in the Figure 1(b).

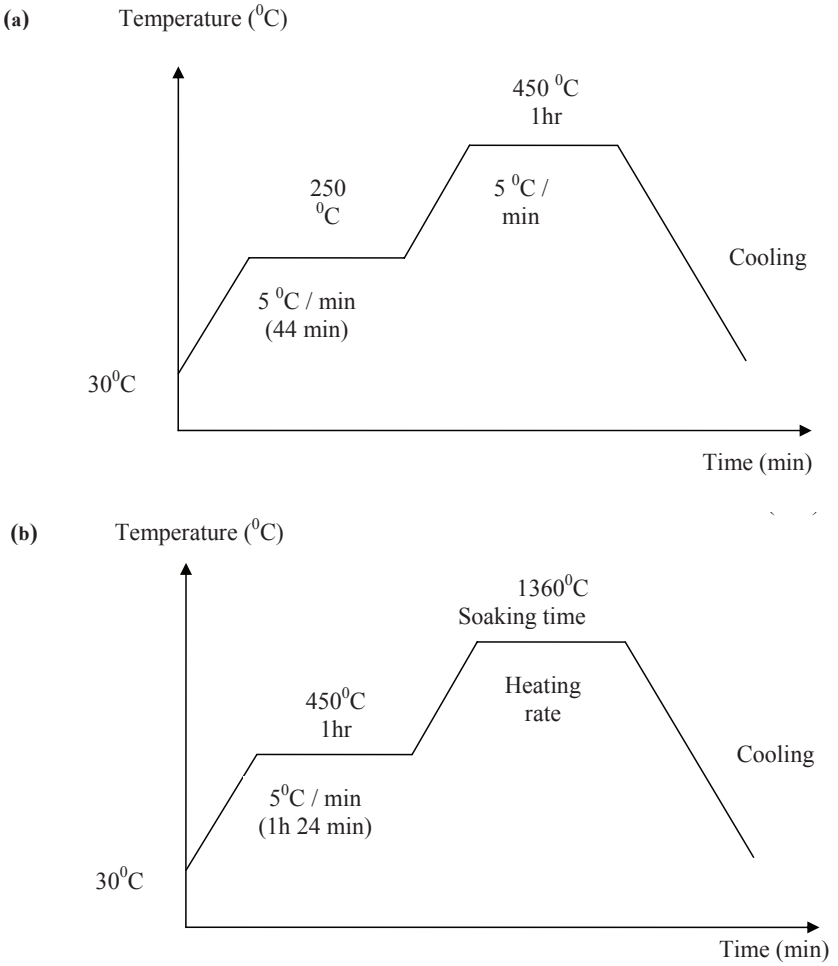


FIGURE 1: (a) The heating profile of thermal pyrolisis process (b) The heating profile of sintering operation

2.4 Density Test

ASTM 328 is a density determination standard to measure the density of sintered specimens by determining the specific gravity using the Archimedes principle. The density measurement was carried by taking the weight of sintered specimens in the air and in the water, without impregnation done to the samples, prior to the measurement. Later, the calculation of the density was done by referring to the following formula.

Density, ρ = [*Wair / (Wair – Wwater)] X ρwater* [1]

Where:

W*air*	= Weight of specimen in air
W*water*	= Weight of specimen in water
ϱ*water*	= Density of water

2.5 Tensile Test

The tensile properties of sintered samples are measured using the Universal Testing Machine UTM model AG–1/100 kN (Shimadzu Corporation, Japan). The standard test for tensile testing of metallic materials is based on ASTM E8. Prior of the test, specimen's initial length were marked and measured. Then, the specimens were gripped and tensile stress was imposed onto the sample until the fracture occurs. The tensile strength and tensile modulus were measured to determine the mechanical properties of the prepared specimens.

2.6 Hardness Test

Micro Vickers Hardness Test with diamond indenter was used as the indentation hardness test against the surface of the material, by using an established machine setup to force a diamond spheroconical indenter under the specified condition. This test is to measure the difference in depth of the indentation according to the MPIF Standard 51 (under the specified conditions of preliminary and total test forces) to determine the micro hardness of the sintered specimens.

2.7 Scanning Electron Microscope (SEM)

To understand the properties of 17-4PH stainless steel, the microstructures studies were carried out. This was done by observing through the Scanning Electron Microscope (SEM). The cross sectional surface of the highest density fractured samples was selected and the SEM micrographs were taken at 1000-times, 3000-times and 50000-times of magnifications by using a secondary and backscattered electron mode image detector.

3.0 RESULTS AND DISCUSSION

3.1 17-4 PH Stainless Steel Powder and Green Body Characterization

In this study, the metal powder used is 17-4 PH stainless steel

(precipitated hardened martensitic stainless steel with Cu and Nb/ Cb additions). The 17-4PH stainless steel metal powder had nearly spherical particle shape with broad size distribution within 1μm into 22μm, as depicted in the SEM micrograph of Figure 2. This powder shape exhibits that gas atomization powder had higher possibility to be packed in higher density. The micrograph shows that the particles are polydispersed in nature where the surface of particles looks smooth. The particles size with less than 22μm indicates largest particles size (Figure 2a). Figure 2b shows the morphology of green body at fracture surface with 1000x of magnification using the SEM observation. It can be clearly seen that the binders are homogeneously filled at all the interstitial spaces between the powder particles. The formation of pores occurred because of air trapped during the injection molding or binder system shrinked during the cooling process. The powder particles are linked to each other by a network of binders. This proved that the function of binder system in MIM processing gives a temporary vehicle for a homogenous powder packing into desired shape and then holding the particles in that shape until the debinding process carried out to remove the binders (German, 1990; German and Bose, 1997).

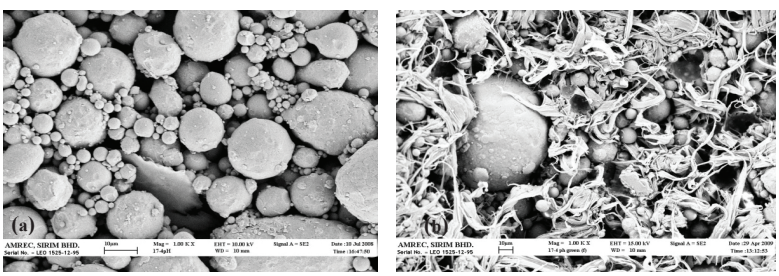
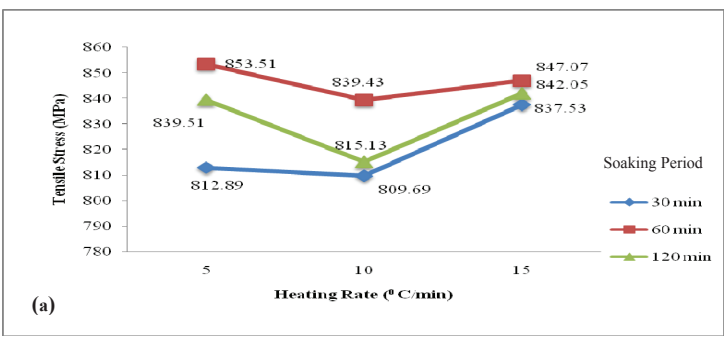


FIGURE 2: SEM micrograph of the (a) 17-4PH stainless steel used in the feedstock preparation and; (b) the green body after injection molding at fracture



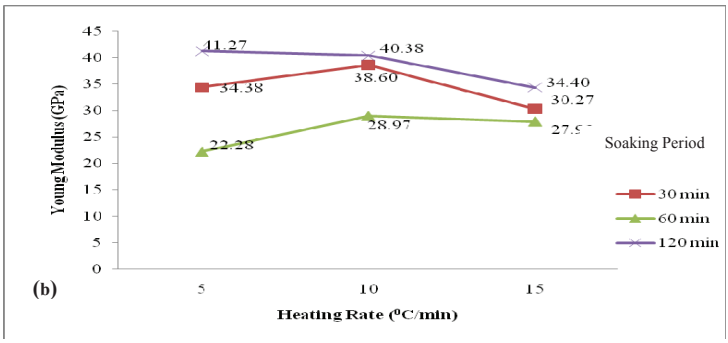


FIGURE 3: (a) Tensile strength and (b) Young modulus for each sample set

3.2 Tensile Test Results

There were nine sample groups tested with the tensile test to obtain the tensile strength and Modulus of elasticity (Young's modulus). The maximum strength and Young's modulus are clearly depicted as in the Figure 3. The lowest value of tensile strength is samples that soaked for 30 minutes. The samples sintered using 15^0C/min shows the higher tensile strength for each difference soaking period, except for the sample that soaked at 60 minutes. At the initial of sintering process, a constant heating rate of 50C/min is used for varied soaking period of 30, 60 and 120 minutes. Thus, the increasing of soaking period increased the tensile strength before it reduced at 120 minutes of soaking period. This is likely due to the presence of pores in the sintered parts which tend to allow further growth of grain boundaries as a result of excessive or abnormal grain growth during the long period of sintering time. Excessive grain growth will result grain coarsening which will lower the strength of the sintered parts (Klar and Samal, 2007).

3.3 Fracture Surface Morphology of Tensile Specimens

Tensile properties obtained from tensile testing are correlated with the morphological observation, where it provides direct evidence of the strengthening or reinforcing mechanism to the fabricated metal composites. Figure 4 depicts the SEM micrographs of vacuum sintered specimens. Through the observation by using 1000x magnification on the samples soaked for 30 minutes, there are large numbers of pores presence between the particles, where the original shape of the powder can still be discerned even though the particles have been fused together. When the soaking period or sintering time increased to the 60 minutes, there is less porosity of microstructure, existed. This is near to fully densification of sintered specimens, although the original shape

of powder can still be detected, where the powder boundaries were replaced by grain boundaries development. However, if the soaking period was increased into 120 minutes, the microstructures began to coarse and the grains began to grow excessively. There are small pores still occurred in that specimens with the dimple rupture phenomenon obviously revealed.

The fracture morphology of 17-4 PH stainless steel specimens sintered at 10^0C/min and 15^0C/min of heating rates for three different soaking periods are relatively having similar microstructure evolution to specimens that were treated at 5^0C/min. However, most of specimens exhibit dimple rupture and pores. This is caused by the δ-ferrite formation occurred during the sintering that decreased the amount of porosity and exhibits ductile fracture, where the morphologies of the tensile fracture surface of gas atomized exhibits the dimple rupture with spherical shape of pores (Gulsoy *et.al*., 2007). The pores existed in the sintered parts tend to provide extra space for further growth of the grain boundaries that resulted the excessive or abnormal grain growth during the long period of sintering time. Excessive grain growth will result larger grain formation which particularly lower the strength of the sintered parts (Klar and Samal, 2007).

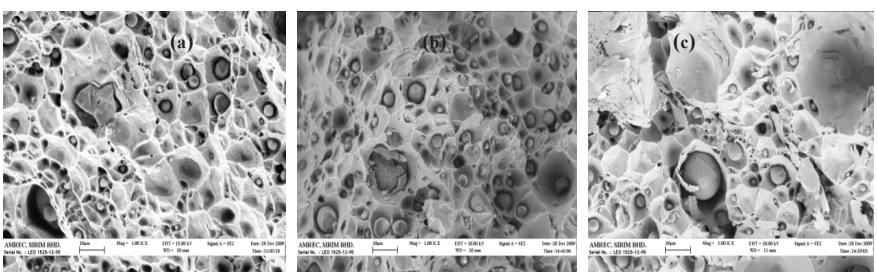
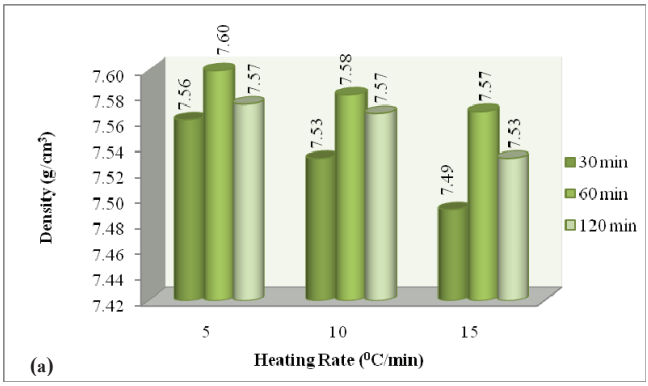


FIGURE 4: The SEM images of fractured surface of the samples used 5^0C/min of heating rate for three different soaking period: (a) 30 minutes; (b) 60 minutes; (c) 120 minutes



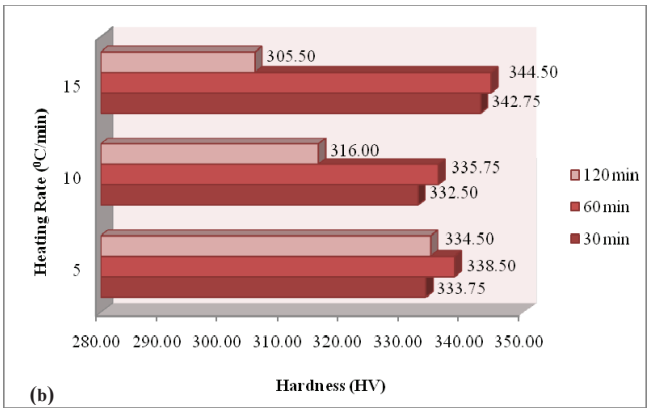


FIGURE 5: (a) actual density and (b) hardness; of 17-4PH sintered samples for different heating rates and soaking periods

3.4 Density and Hardness Evaluation

The theoretical density of sintered 17-4PH stainless steel is 7.5 g/cm3. In this study, the actual densities of sintered 17-4PH stainless steel specimens are shown as in the Figure 5a. The graph shows that samples were treated at 60 minutes of soaking periods for each heating rates gives the higher value of densification, where the sample treated at 30 minutes of soaking periods exhibit the lower density values. Previous studies found that the heating rates for vacuum sintering will affect the density of the sintered specimens (Liu *et.al.*, 2000).

The actual value of hardness for sintered sample of 17-4PH stainless steel at different heating rate and soaking period is presented in Figure 5b. The hardness was increased with the soaking period for the entire heating rate before it reduced at 120 minutes of the soaking period. It was found that the decrease of hardness value at 120 minutes of soaking period applies for specimens treated at 10^0C/min and 15^0C/min of the

heating rate. This phenomenon occurred due to long soaking duration after rapid heating of sample treatment. For the specimens treated at 5^0C/min of heating rate and 120 minutes of soaking period, the grains were constantly growing started from the lower sintering temperature until the temperature of 1360^0C. At this condition, the grain is continuously growing in the constant mode. This phenomenon was caused by the reduction of free energy that concurrently minimizing the interfacial energy per unit volume against the grain growth where the hardness decreased by larger size of grains formation (German, 1996).

4.0 CONCLUSION

Combination of 17-4PH stainless steel metal powder with waste rubber binder provides an increase to the mechanical properties of the injected specimens that sintered at below than 120 minutes of soaking period with optimum heating rate of 5^0C/min. The combination of these parameters during the sintering process gives the higher density, higher tensile strength, less porosity and homogenous grain shape morphology of the sintered samples. Although the introduction of waste rubber as binder system in MIM fabricated product would not affect to the properties of sintered component due to achievement of the nearly theoretical values of the resulted mechanical properties, but the application of innovative and novel binder system is compatible and very promising for the 17-4PH stainless steel metal powder product fabricated through the MIM process. Utilization of waste rubber provides an alternative binder for metal injection molding process. This alternative gives the advantageous of an economical and environmental friendly material as well as to solve the problems of waste rubber disposal.

5.0 ACKNOWLEDGEMENT

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