**PHYSICAL AND MECHANICAL PROPERTIES EVALUATION OF INJECTION MOULDED 316L STAINLESS STEEL USING NEW DEVELOPED THERMOPLASTIC NATURAL RUBBER (TPNR) BINDER SYSTEM**

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**SYNOPSIS:** This paper discussed the sintering characteristics of injection moulded 316L stainless steel powder using new developed binder system based on natural rubber. Natural rubber has been estimated to possess great potential application as a binder system as it is based on natural resources which are locally available. It is believed that this research outcome can assist future research in Metal Injection Moulding (MIM) process in design and development of a new binder.

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**RINGKASAN:** Di dalam kerja penyelidikan ini, ciri ciri pensinteran bahan 316 keluli tahan karat dengan menggunakan sistem pengikat getah asli termoplastik (GATP) telah dikaji. Model penyelidikan telah menggunakan bahan lilin dan bahan sawit biopolymer yang telah diadun dengan bahan GATP. Bahan suapan berbagai formulasi dengan 65 % isipadu bahan logam telah diacukan kepada sampel ujian. Penyahikatan dengan menggunakan proses penyahikatan larutan dan pirolisis terma telah berjaya menyingkirkan bahan lilin dan bahan sawit dengan pantas. Bahan ujian kemudian di sinter di dalam keadaan vakum pada suhu 1320 °C hingga 1380 °C. Penyelidikan tentang pensinteran seperti keadaan fizikal, evolusi mikrostuktur serta ciri ciri mekanikal dikaji dan dibahaskan. Hasil ujikaji menunjukkan apabila suhu pensinteran meningkat, ciri ciri pensinteran juga meningkat dan dapat mencapai sehingga kepada keadaan ketumpatan penuh. Ciri ciri mekanikal bahan ujian pada suku pensinteran 1320 °C hingga 1380 °C memenuhi Standard MPIF 35 MIM.

**ABSTRACT**: The present study investigates and evaluates the sintering characteristics of injection moulded gas atomised 316L stainless steel powder using new developed binder system based on thermoplastic natural rubber (TPNR). Model experiments were conducted with new wax based and palm-based biopolymer binder mixed with TPNR. The feedstock having 65 vol. % of metal powder with different formulations were injection moulded into a test bar. A rapid two stage debinding process involving solvent extraction and thermal pyrolysis was successful in removing the palm stearin binder in short time. The specimens were then sintered under vacuum atmosphere in the temperature range of 1320 °C to 1380 °C. The sintering studies were conducted to determine the extent of densification which corresponds to microstructural changes. In addition, the properties of the sintered specimens such as physical appearance, microstructure evolution and mechanical properties were presented and discussed. The results showed that as the sintering temperature increased, the sintered properties improved and the powders could be sintered to near-full density. The mechanical properties of the samples sintered at 1360 °C and 1380 °C complied with the MPIF Standard 35 MIM Specimens.

**INTRODUCTION**

The mastering issue in Metal Injection Moulding (MIM) business is associated with the feedstock. Indeed, feedstock formulation is the area of greatest patent coverage in MIM (Anwar *et al*., 1995; Cao *et al*., 1992; Henmi *at al*., 1981; Johnson *et al*., 1988; Rivers & Kokomo, 1978; Strivens, 1960; Wiech and Calif, 1983; Weich and Calif, 1984). In particular the binders in the feedstock strongly determine the MIM quality. They provide adhesion among powdered particles and improve the mechanical properties of feedstock and prevent separation phenomena among binders and powders (Rhee *et al*., 1998). However, the performance of binders depends on the content of the backbone polymer, which governs the strength of green parts in the injection moulding phase and the shape of the article in the debinding phase (German & Bose, 1997).

In general, the thermoplastic polymers systems are the most studied and are used as the backbone polymer in industry. The high density polyethylene-wax binder is one of the simplest systems used in Powder Injection Moulding (PIM) (German 1990). The main advantages are low cost, low molecular weight and good lubricating properties. Usually, multicomponent binders, comprising of polymer and other additives, are prepared because they improve the debinding process. The main advantage of these binders is the gradual elimination of different components preventing cracks and shape loss. The combination of low molecular weight and different polymer components allows a progressive removal as the temperature slowly increases. As reported by German and Bose (1997), a combination of LDPE and HDPE, in the LDPE/HDPE backbone polymer, can eliminate the article defect formed by the evaporated gas, and prevent the mass degradation of the backbone polymer in the articles, which frequently occurs when a single backbone polymer is present.

Inspired by that, natural rubber (NR) and LDPE were combined to produce a thermoplastic natural rubber (TPNR) backbone polymer. TPNR is classified as thermoplastic elastomer as it absorbs solvent and swell, but do not dissolve; furthermore, it cannot be reprocessed simply by heating. The molecules of thermoplastic rubber, on the other hand, are not connected by primary chemical bonds. Instead, they are joined by the physical aggregation of parts of the molecules into hard domains. TPNR has been estimated to possess great potential application as a binder system as it is based on natural resources which are locally available.In this study, TPNR backbone polymer was applied with palm stearin (PS) and paraffin wax (PW). Both PS and PW binder possess attributes as binder such as low viscosity, high decomposition temperature, lower molecular weight to avoid residual stress and distortion, environmentally acceptable, inexpensive and easily dissolved inorganic solvent (Huang & Hsu 2009) and (Istikamah 2010). In this paper, the optimum sintering performance of physical and mechanical properties of the sintered specimen using newly developed TPNR binder was evaluated in order to determine the best binder composition on the system for 316L stainless steel powder as a model material.

**MATERIALS AND METHOD**

In this study, 316L stainless steel powders with 22 µm particle size which was gas atomized and manufactured by Sandvik Osprey were used. Stainless steel was chosen because of its availability and high demand. The chemical composition of the steel provided by manufacturer is presented in Table 1.

Table 1 Chemical composition (wt-%) of 316L stainless steel powder

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| C | Cr | Ni | Mo | Mn | Si | S | P | Fe |
| 0.026 | 16.4 | 10.4 | 2.08 | 1.43 | 0.58 | 0.012 | 0.01 | balance |

The cumulative particle size distribution was determined using CILAS 1190 Dry Particle Size and the result shows that the powder had a median particle size around 5.97 μm.

The binder components used in this investigation included paraffin wax (PW), palm stearin (PS), low density polyethylene (LDPE) and natural rubber (NR) to produce thermoplastic natural rubber (TPNR) as well as stearic acid (SA) as a lubricant. The thermoplastic natural rubber (TPNR); Polyethylene:Natural Rubber (PE:NR) were compounded out in an internal mixer ThermoHaake Polylab OS Rheodrive 4 at 140 °C and speed of 50 rpm for 12 minutes according to the adequate time for the components to melt and homogenise. TPNR contains a thermoplastic component which is polyethylene (PE) and a Natural Rubber (NR) component. The best composition of TPNR (40NR/60PE) was subsequently added together with different formulation in (vol.%) of 50PW/35TPNR/10SA (PW/TPNR/SA-1), 55PS/35TPNR/10SA (PS/TPNR/SA-1), 55PW/45TPNR (PW/TPNR) and 55PS/45TPNR (PS/TPNR) binder systems at the same parameter used for TPNR and mixed for 1 hour. Initially the steel powder mixture was kept constant at 65 %. The granulated feedstock of both formulations were injected into tensile bars using a simple, vertically aligned and pneumatically operated plunger machine, MCP HEK-GMBH and the results have been discussed elsewhere (Hassan *et al.,* 2015a; Hassan *et al.,* 2015b). The green strength of the moulded specimens were determined with a 3-point bend test according to MPIF Standard No.15 using an Instron 3369 universal testing machine (UTM).

Debinding was performed in two stages of solvent debinding by heptane solvent at 60 oC for 5 hours followed by thermal debinding under vacuum with the heating rate of 3 oC/min to 450 oC and soaking for 30 min. A heating rate of 5 oC/min was used for the sintering temperature of 1320 °C to 1380 oC in vacuum. The specimens were soaked for 240 min and then cooled at the natural rate of the furnace with no power supplied. The sintered specimens were cut into slices using diamond saw cutter. The density of each slice was measured using water immersion technique. The sintered density reported here is the average of five measurements taken from the sintered specimens of each formulation reported. The tensile strength and elongation of all binder formulations were measured by using Instron 3369 universal testing machine (UTM) according to ASTM E8M-00. The extensometer was used to measure the elongation. The reported tensile strength and elongation is the average of three measurements. The sintered test specimens were observed under optical microscopy to examine the microstructure and porosity.

**RESULTS AND DISCUSSION**

**Physical Properties of Sintered Specimens**

The shrinkage observed after sintering process at various temperatures (1320-1380 oC) under vacuum condition shows the same anisotropic shrinkage that occurred during moulding. As evidenced by Figure 1, the shrinkage of PW/TPNR/SA-1 and PS/TPNR/SA-1 specimens was larger than the PW/TPNR and PS/TPNR system, relatively. This is due to a higher loading of binder, which leaves behind more porosity after debinding resulting in the larger shrinkage during sintering. The linear shrinkage of the sintered specimens at different composition of binder system and sintering temperature shown in Figure 1 indicates that the percentage of shrinkage increases with increasing sintering temperature for all specimens. The sintered specimen PS/TPNR made with 55 vol.% of PS and 45 vol.% of TPNR backbone polymer showed the lowest shrinkage compared to the others. The result was proportional to the sintered density which showed an increase as the temperature increased (Figure 2). As the temperature increased, it reduced the pore volume by reduction in binding energy that led to compact densification and an increase in the shrinkage of specimen.

***Figure 1.*** *Linear shrinkage for sintered specimen at various sintering temperature with*

*different binder formulation in vacuum atmosphere*

Moreover, shrinkage is inversely dependent on the green density. High green density of specimen resulted in lower shrinkage. Sintering is improved by a high initial packing density in specimen because there is more particle contact involved in the bonding process. As a consequence, it is desirable to maintain a high and uniform powder packing density in the feedstock to decrease the net shrinkage and thus eliminate one source of distortion.

Figure 2 shows the relationship between sintering temperature and sintered density for specimens made with different binder system composition sintered at temperature ranging from 1320 oC to 1380 oC. It clearly shows an increase of 10 vol.% of TPNR in binder system by eliminating the SA content; and approximately 1 % to 2 % increase in sintered density for binder system containing PS. The sintered specimen of PW/TPNR/SA-1 possessed the highest density at all sintering temperatures. It explains that incorporation of TPNR backbone binder gives better effect with PW as the soluble binder and can provide better sintered part integrity. As the binder components were not present during sintering process, it is assumed that the increase in density resulted from the more favourable way in which the particles were packed, as the TPNR content increased in the moulded material and after binder removal.

*MPIF Standard 35*

***Figure 2.*** *Density for sintered specimen at various sintering temperature with*

*different binder formulation in vacuum atmosphere*

The theoretical density of the 316L stainless steel is 7.90 g/cm3. As the sintering temperature increased, the density of specimen also increased. For example, a change of temperature from 1320 °C to 1340 oC (specimen PW/TPNR/SA-1) caused the density to increase by 0.1 %, from 7.674 to 7.679 g/cm3. The enhancement in the density from 1320 °C to 1340 oC indicates that the progress of the first stage sintering (the formation of inter-particle necks) at 1320 oC to the second stage at about 1340 oC resulted in significant transport among the particles. The sintering temperature of 1320 oC is only at the intermediate stage of sintering where pores begin to close up and densification starts. The relative density is 97.14 % of theoretical density.

At a sintering temperature of 1340 oC, the sintering process reached the intermediate stage where a transport mechanism such as bulk transport played a dominant role. The densification of the part has reached 97.20 % of the theoretical density. The density is also believed to be influenced by volume diffusion, grain boundary diffusion and plastic flow at high temperatures, notably at 1360 oC. At that temperature, sintering is likely to be slowed down, as during this final stage of sintering isolated pores start to form. Hence, densification of the specimen will reach a certain level with respect to increasing sintering temperature. At 1360 oC, the specimen showed a smaller increase of 1 % in density, from 7.67 to 7.75 g/cm3. At a density of 7.75 g/cm3, it achieved 98.11 % of the theoretical density. Further sintering at 1380 oC depicted that the density of the sintered specimen increased to 7.85 g/cm3 and the relative density reached 99.47 % that is really close to theoretical density. Results obtained were comparable with other researchers (Heaney *et al.,* 2004; Huang & Hsu, 2009) as they reported densities of 316L SS sintered parts are in the range of 95 % - 99 %.

**Mechanical Properties of Sintered Specimens**

The variation of the mechanical properties with the sintering temperatures is shown in Figure 3 and Figure 4. The figures show the result of tensile and elongation conducted on the specimens made with different composition of binder systems. All results were comparable to MPIF Standard 35 (2000) for metal injection moulded part. Standard 35 states that the minimum Ultimate Tensile Strength (UTS) and per cent elongation are 450 MPa and 40 % elongation. It can be seen from Figure 3 that specimen with PW/TPNR/SA-1 binder system showed a significant linear increment of strength obtained with increasing sintering temperature. However, for the other three binder formulations, PW/TPNR, PS/TPNR and PS/TPNR/SA-1 the strength varied exponentially with sintering temperature respectively.

Specimens sintered at 1320 oC showed constant strength for all binder formulation with PW/TPNR/SA-1 binder system having the highest value. The unstable increment in strength value was obtained when specimens were sintered at 1340 oC. These might be due to the changes occurring, from early stage to intermediate stage of sintering that undergoes a transformation to become dense and pore channel closure, with changes in the pore size and shape. Meanwhile, sintering at 1360 oC inherently involved substantial shrinkage with elimination of pores that led to a dramatic increase in strength of all specimens. This phenomenon was also reported by Heaney *et al*. (2004) while sintering 316L SS at 1340 oC and 1360 oC.

*MPIF Standards 35*

*MPIF Standard 35*

***Figure 3.*** *Ultimate tensile strength of the sintered part with different binder formulation at*

*different sintering temperatures in vacuum atmosphere*

As temperature increased from 1360 oC to 1380 oC, small deviational changes of strength was observed especially for binder formulation involving PW either with existence of SA or without SA giving higher value in strength compared to formulation consisting of PS. This can be related to previous studies by Iriany (2002) and Istikamah (2010) in which they had proven that the best sintering temperature for specimen consisting of PS was at 1360 oC as it resulted in excellent strength. In summary, the result showed that all specimens with different binder formulation complied to the international standard MPIF 35 MIM specimens starting at 1360 oC and 1380 oC of sintering temperature.

The trend was polynomial relation and proportional to the elongation of the specimen (Figure 4). High value of tensile strength for the sintered specimen resulted in a subsequent increase in elongation percentage. The graph clearly showed that the percentage of elongation increased with an increase in sintering temperature. The highest elongation observed for the sintered specimen with binder formulation of PW/TPNR and PW/TPNR/SA-1 at 1380 oC of 67.9 % and 64.4 % respectively. As a comparison, the specimen with PS/TPNR and PS/TPNR/SA-1 binder formulations showed lower elongation as expected because of the lower value in tensile strength.

*MPIF Standards 35*

*MPIF Standard 35*

***Figure 4.*** *Elongation of the sintered specimen with different binder formulation at*

*different sintering temperatures in vacuum atmosphere*

The hardness of the sintered specimen is strongly correlated with the density; therefore the hardness can be predicted from the density of the sintered specimen. Vickers microhardness measurements were conducted on the cross-sectional area of the tensile bar, at the core region. The area was polished, and the microhardness of the sintered specimen was measured. The average microhardness results are as plotted in Figure 5 and the MPIF standard 35 of minimum hardness value for 316L stainless steel parts was also indicated in the graph. In general, the microhardness of the 316L stainless steel specimens with binder formulation consisting of PW incorporation with TPNR backbone polymer is significantly higher than the 316L stainless steel parts with PS incorporation of TPNR binder system. A suitable composition of TPNR backbone polymer was expected in improving the microhardness of the sintered specimens. As the sintering temperature increased from 1340 to 1360 oC, the microhardness increased drastically from 196.8 to 257.2 Hv for the PW/TPNR/SA sintered specimen. When the specimens sintered at 1380 oC, the microhardness increased to 258.5 Hv. Increase in hardness value was correlated with densification of sintered specimen; the higher the density the better the hardness properties (Huang & Hsu 2009).

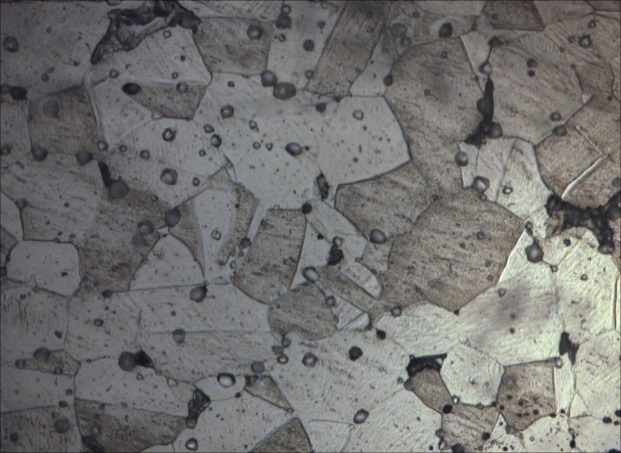


***Figure 5.*** *Hardness of the sintered specimen with different binder formulation at*

*different sintering temperatures in vacuum atmosphere*

**Microstructure of Sintered Specimens**

The optical micrographs of sintered specimen in Figure 6 shows the typical pores structure and typical microstructure (etched), which display the austenitic grain and pores under vacuum sintering conditions at 1380 oC. The pores structure and microstructure of the sintered specimen are relatively influenced by the densification of specimen. It could be seen that the sintered specimens at the optimised sintering condition has the least porosity; the pores are rounded, small and appeared to be isolated in their distribution as the sintering temperature was increased. An increase in sintering temperature promotes neck growth and change in pore morphology. The particle boundaries become thicker and darker indicating that increased sintering temperature resulted in more active changes at the particle boundaries during sintering

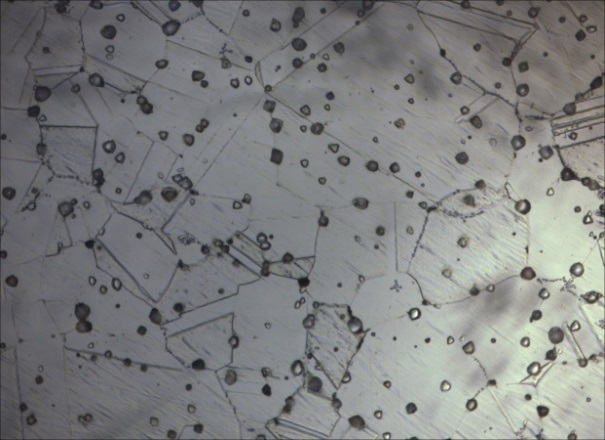


Grain boundary

10µm

10µm

a)PW/TPNR/SA-1 b) PW/TPNR



twinning

10µm

10µm

c) PS/TPNR/SA-1 d) PS/TPNR

***Figure 6.*** *Pores structure and microstructures of sintered specimens with different binder formulation at 1380 oC under vacuum atmosphere. Magnification of all specimen was at 500x*

The optical micrographs show the typical structure of stainless steel with clear austenite grain, grain boundaries and twinning. These microstructures affirmed an austenite stainless steel material. The presence of different colours were due to the different plane of austenite phases. Temperature plays a key role in the interaction of grain growth and densification, since it controls the relative rate of grain growth, pore migration and densification. From the above results, sintering at 1380 oC resulted in better physical and mechanical properties of sintered specimen especially for system with combination of PW and TPNR. Figure 6 shows the optical micrograph of sintered specimen at 1380 oC under vacuum conditions made with different formulation and composition of binder system.

As shown in Figure 6, there are no significant differences in the optical images of specimen sintered at 1380 oC. However, smaller grain size can be observed for PW/TPNR/SA-1 and PW/TPNR system as shown in Figures 6 (a) and (b) which had proved to have higher density, strength and hardness. On the other hand, PS/TPNR/SA and PS/TPNR specimen that exhibit lower density, strength and hardness was believed to be related to larger grain size of the sintered specimen. Nevertheless, the sintered strength does not vary in a systematic manner with any of the common microstructural parameters (German 1996). The fracture strength is controlled by either flaws or the larger grain. At small grain sizes, the flaws control the strength, while at large grain sizes, there is a true grain size dependence.

Referring to the previous studies (Cai & German 1995; Loh and German. 1996; Omar 1999; White & German 1994) sintering of stainless steel was carried out in the range of 1300 oC and 1380 oC. Song *et al*. (2006) also suggested that sintering at a high temperature causes rapid shrinkage and yields a high relative density. The optimum performance of sintered specimen is achieved at sintering temperature of 1380 oC in vacuum atmosphere. The optimum results were also obtained by Huang and Hsu (2009) using 316L SS sintered at 1380 oC in vacuum atmosphere.

**CONCLUSIONS**

In this study, a combination of natural rubber (NR) and polyethylene (PE) was introduced as a back bone polymer and the optimum percentage of the backbone polymer was determined as 40 vol.% of NR and 60 vol.% of PE, which gave the most appropriate composition according to rheological analysis. This combination is called TPNR and classified as thermoplastic elastomer with thermoplastic behaviour. At the sintering process, results obtained showed that the optimum sintering temperature is at 1380 oC in vacuum atmosphere. The specimen that was sintered at the optimum condition possessed similar shrinkage percentage in all four specimen dimensions. This indicated that the homogeneity of all feedstock with different binder formulations having the TPNR backbone polymer were relatively good. The dimensional stability of the sintered parts of the feedstock was also consistent with the associated property of the green parts. The physical properties of the green and sintered specimen were improved by introducing the TPNR backbone polymer, which helps to eliminate the crack defect from sintered specimens, and improve the dimension, density and hardness stabilities of the sintered specimens. The mechanical properties of sintered specimen showed that all specimens with different formulation complied with the international standard MPIF 35 MIM specimens starting from 1360 oC, to 1380  oC, with strength ranges from 470 to 520 MPa and the elongation ranging from 44 % to 68 %.

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