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Physical and Mechanical Evaluation of Injection Moulded of 17-4PH Stainless Steel

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Original Research Article

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Abstract: The 17-4PH stainless steel (17-4PH SS) parts were fabricated by using a metal injection moulding (MIM) route that utilizes three binder systems comprising of palm stearin (PS), thermoplastic natural rubber (TPNR), polyethylene (PE), paraffin wax (PW) and stearic acid (SA). The 17-4PH SS powder was mixed with binders at powder loading of 65% vol by using a z-blade mixer for 2 hours at a temperature of 180°C to produce a homogeneous feedstock. The feedstock completely filled the mold cavity at a temperature of 180°C and pressure of 400 bars. A two-stage debinding process (solvent and thermal) was performed to remove binders in green parts. Debound parts were sintered at temperature ranging from 1300°C to 1380°C for 2 hours. The microstructures, physical and mechanical properties of the sintered parts were evaluated in order to determine the feasibility of binder. Among the three binder systems considered herein, PW/PE/SA appeared to produce optimum sintered properties while PS/TPNR produced the lowest. Experimental results have also demonstrated that comparable sintered properties could be obtained from the PS/PE at higher sintering temperature. In summary, this work has indicated that bio-composite binder systems, PS/TPNR and PS/PE could be considered as a promising binder in MIM of 17-4PH SS.

Keywords: Metal Injection Moulding; 17-4PH Stainless Steel; Bio-Composite Binder; Solvent Extraction; Sintering.

INTRODUCTION

The 17-4PH stainless steel generally has high strength and high apparent hardness while exhibiting superior corrosion resistance [1]. The wrought 17-4PH precipitation–hardening stainless steel is found extensively in applications in the aerospace, chemical engineering, medical and metal working industries, where a higher level of corrosion resistance, strength and hardness must be maintained. The high hardness characteristic owned by this steel resulting from precipitation hardening, however, creates a high volume of metal scrap waste which in turn increased the cost of machining process.

In recent years, the potential of producing 17-4PH components by powder injection moulding which may aid to eliminate the restriction of the poor machinability have been reported [2-7]. Previous work by Jeefferie *et al.*, [5] had demonstrated that by using waste rubber binder combined with high sintering temperature of 1360^{0}C, tensile strength of 853.5 MPa and homogenous grain distribution can be achieved. Meanwhile, the capability to assure a combination of strength and ductility at a level which are challenging to realize for austenitic, ferritic and martensitic stainless steel grades by adopting powder metallurgy route has been reported by Jan Kaziour [7].

The fundamental of powder injection moulding (PIM) technology that has emerged as a cost effective manufacturing technique for relatively small, complex and high performance components have been described in several books [8, 9]. The aforementioned literatures provide comprehensive acquaintance on the four sequential stages of the PIM process which comprised of mixing, injection moulding, debinding and sintering. Additionally, the literature also provide insight of the current state-of-the-art and useful practical guidance for the practitioners to consider PIM as an alternative route for producing precision metallic parts at high production volumes.

Among many stages in PIM, feedstock preparation is crucial due to fact that shortcomings such as inhomogeneity of feedstock, metal particles segregation and metal powder-binder seperation cannot be corrected by subsequent processing adjusment[8-9].

One key component at this stage is the selection and formulation of binder as the binder promotes fluidity and rigidity of the feedstock particularly during mixing, injection moulding and debinding. The characterictics of the binder greatly affected PIM parameters such as particle packing, agglomeration, mixing, rheology, moulding, debinding, dimensional accuracy, defects and properties of the sintered PIM part.

Experimental Procedure

MATERIALS

The Sandvik Osprey Powder was used to produce the gas atomised 17-4PH powder required in this study. The particles were approximately spherical in shape with an average size of 22 µm. Some particles are large while others small. Overall, the powder showed a relatively wide particle size distribution. This is essential for efficient particle packing in MIM powder.

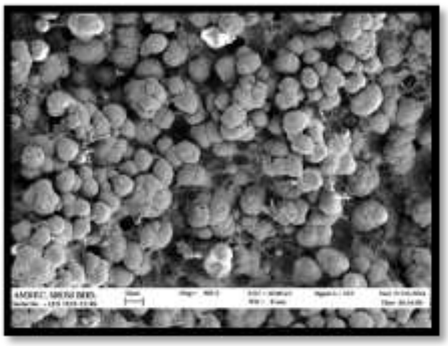

Fig-1: Scanning electron micrograph of 17-4PH Stainless Steel

Feedstock Formulations and Mixing Process

The 17-4PH powder was mixed with three proprietary multicomponent binder formulations comprising of various concentration of paraffin wax (PW), polyethylene (PE), stearic acid (SA), palm stearin (PS) and thermoplastic natural rubber (TPNR) in order to prepare the feedstock. The feedstock composition is as shown in Table 1. The Z-Blade mixer was used to mix the feedstock formulations containing 65 % powder loading at a temperature of 170ºC for a duration of 2 hours at a speed of 50 rpm in order to obtain a homogeneous paste. Henceforth, these feedstocks are identified as F1, F2 and F3 respectively. These feedstocks are then granulated into pellet form for easy feeding into the injection molding machine.

Table-1: The composition of the feedstock (vol%)

Paraffin Wax (PW)	Polyethylene (PE)	Stearic Acid (SA)	Palm Stearin (PS)	Thermoplastic Natural Rubber (TPNR)	Feedstock Tag
55	35	10	-	-	**F1**
-	-	-	70	30	**F2**
-	30	-	70	-	**F3**

The MCP HEK-GMBH vertical injection moulding machine was used to mold the granulated feedstock into the form of tensile bars. An appropriate set of molding parameters were set in order to obtain defect-free molded parts.

Debinding and sintering

A two-stage debinding process was applied in producing the green molded parts. The injection moulded part was leached in a bath of n-heptane at a temperature of 60°C for 5 hours without stirring. This solvent debinding process removed the soluble portion of binders. A glass container was used to cover the bath in order to prevent evaporation of the n-heptane. After completion of the solvent extraction process, the brown parts were dried in an oven for 2 hours at a temperature of 40°C to remove the remaining n-heptane. Subsequent thermal pyrolysis was carried out in a Lynn furnace. The thermal debinding cycle involved heating to 450°C at a heating rate of 1°C/min. This was followed by soaking for one hour before the furnace cooled.

Sintering was conducted at optimum sintering temperatures of 1380°C in a vacuum environment. To prevent crack formation, the heating rate applied was 1°C/min up to a temperature of 450°C. This was followed by 3°C/min up to the sintering temperature for the second stage. About one hour of soaking time was allotted for each stage before the part was subsequently furnace cooled.

The tensile properties of the sintered samples were assessed using an Instron Series IX Automated Materials Testing System. The fracture surfaces were observed by scanning electron microscopy and sintered densities were calculated by Archimedes' method.

RESULTS AND DISCUSSION

Feedstock Formulations

The MIM process starts with preparing a feedstock that could be injected into a mould cavity and allowed to solidify. The formulation of the feedstock (metal-binder mixture) is one of the most crucial aspects due to its effect on every step of the MIM process. Low amount of binder used in formulation of the feedstock resulted in high viscosity feedstock which makes the moulding process difficult. On the other hand, a large amount of binder provides low strength and may produce heterogeneous green parts. Past researchers have established that good quality of feedstock is essential to ensure adequate mouldability, dimension stability of the moulded part during debinding and dimensional precision of the sintered part [8, 9].

The conventional binder, F1, comprise of primary binder parafin wax (PW), polyethelene (PE) as the backbone and stearic acid (SA) as the surfactant. The F2 is formed by replacing both the primary and secondary binder with palm stearin (PS) and thermoplastic natural rubber (TPNR). A study conducted by Omar *et al*., [14] has shown that PS which contains glyceride and possessed beneficial attributes as a binder such as low viscosity, high decomposition temperature and lower molecular weight was found to be compatible with polyethelene and stainless steel powder. Norita *et al.*, [12]_reported the potential of TPNR as a good polymer backbone in MIM of 316l stainless steel owing to its higher degradation temperature that may provide better shape retention of the MIM parts. Meanwhile F3 was developed by maintaining the backbone PE and replacing the primary binder PW with PS. In the current work, mixing of the 17-4PH powder with the three multicomponent binders to form a homogenous feedstock was really easy.

Moulding was performed by feeding the granulated feedstock through the vertical metal injection moulding machine into a small tensile test specimen according to MPIF Standard 50. At this stage, it is very crucial to select appropriate moulding parameters to ensure formation of defects-free injection moulded part. Too high injection speed leads to an increase in the peeling of the part surface whilst too low injection speed may result in crumbled edges defects. Appropriate injection pressure on the other hand is required to ensure sufficient amount of feedstock is allowed to flow and completely filled up the mould cavity, hence avoiding formation of imperfections such as void, porosities and internal cracks.

Table-2: Injection Moulding Parameters

Injection Pressure (bar)	**Nozzle Temperature (ºC)**	**Mold Temperature (ºC)**	**Cycle Time (s)**
400	180	Room Temperature	18

As tabulated in Table-2, the four feedstocks were successfully moulded at optimum moulding parameters after several trials. The green injection molded parts were found to be fairly good and free from normal injection moulding imperfections such as short shot, flashes at the parting surface and binder separation.

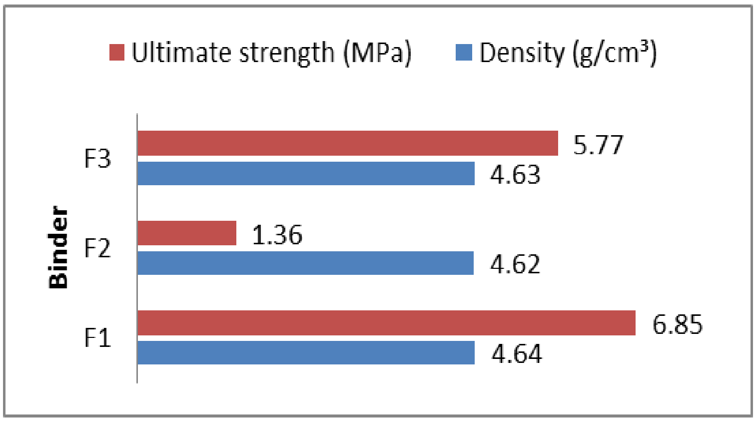

Fig-2: Density and strength of green part

The density and strength of the green injection moulded part are presented in Figure-2. According to German and Bose [9], it is crucial to achieve green density near to 5 g/cm^3 for ferrous based feedstock as the green injection molded part is subjected to handling prior to debinding and sintering process. The green density of all MIM parts was near 5 g/cm^3 suggesting that the three feedstocks had allowed the molding pressure to be transmitted uniformly during the injection molding process, and resulted in better packing of the feedstock into the mold cavity. As depicted in Figure 2, the green 17-4PH MIM part for binder formulation F1 was the strongest. This is attributed to the presence of surfactant, stearic acid

(SA) that strengthens the adhesion between binder and 17-4PH stainless steel particles.

Debinding and Sintering Process

Solvent extraction was employed to remove the soluble binder components from the green MIM part. The formation of open pores channel that will aid rapid removal of non-soluble binder component in the subsequent thermal debinding stage was discussed elsewhere [14, 13, 10].

Table-3: Mechanical Properties of Sintered Part.

Binder Formulation	Tensile Strength (MPa)	Young Modulus (MPa)	Hardness (HRC)	Density (g/cm^3)
F1	809.7	71828.9	45	7.34
F2	543.8	60612.3	50	6.61
F3	791.2	68751.6	40	7.33

The mechanical properties of MIM 17-4PH stainless steel were investigated in order to demonstrate that the parts produced by this route are capable to meet the rigorous demand of various applications. Table 3 depicts the results of optimum tensile strength from all the binder formulations and other related mechanical properties attained at the corresponding sintering temperature. The mechanical properties of sintered parts for the three binder systems were lower compared to the properties of 17-4PH sintered injection moulded part as specified by MPIF Standard 35 for 17-4PH material [11]. When comparing mechanical properties of the sintered MIM 17-4PH for each binder system, the data suggests that the bio-composite binder systems, F2 and F3 are feasible in MIM of 17-4PH stainless steel although the attainable sintered properties were lower compared to the conventional binder system F1.

Among sintered part with different binder system, the highest tensile strength of 809.7 MPa was attained for binder formulation F1, containing 55 vol% paraffin wax (PW), 35 vol% polyethylene (PE) and 10 vol% stearic acid (SA). The average hardness of that sintered part was approximately 45 HRC whilst the Young's Modulus and sintered density were 71.8 GPa and 7.34 g/cm^3 respectively. Comparable tensile strength of 791.2 MPa and density of 7.33 g/cm^3 were achieved for binder formulation F3, containing 30 vol% polyethylene (PE) and 70 vol% palm stearin (PS) whilst the attainable microhardness and Young's Modulus are 40 HRC and 68.8 GPa respectively. The findings suggested that a blend of PW/PE/SA and PS/PE could be used effectively as a binder in MIM of 17-4PH stainless steel.

The fractography analysis performed at the respective sintering temperature reveal that fracture surfaces of all sintered parts are a mixed type of ductile and brittle fracture identified by smooth curved surfaces and dimpled features, respectively. It is noted that ductile dimples are larger and shallow when analyzing as sintered part of F2 binder formulation (Figure 3(b)) which account for the lowest attainable mechanical properties as compared to binder formulation F1 and F3.

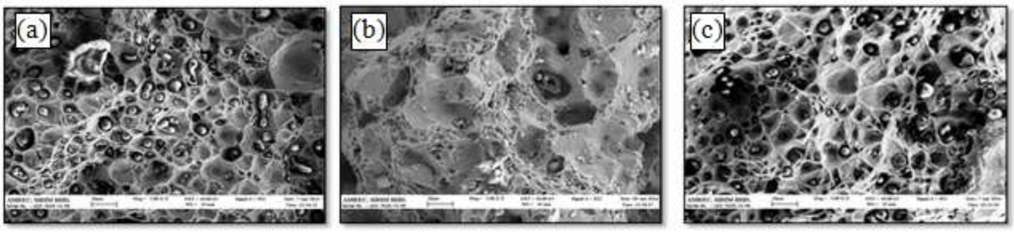

Fig-3: Fracture surface as sintered of (a) F1, (b) F2 and (c) F3.

A comparison of the typical microstructure of the as sintered 17-4PH stainless steel MIM parts is presented in Figure 4. It was noted that the sintered part for all binder formulations depict probably ferritic microstructure with second phase particles homogenously dispersed in the matrix. It must be noted, however, that the F1 parts possess smaller grains with greater amount of second phase particles is in good agreement with the highest attainable mechanical properties.

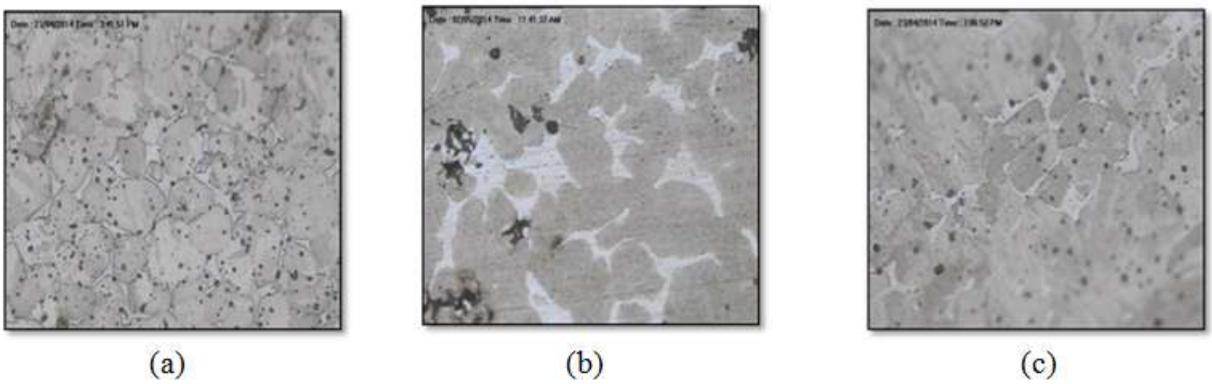
(a) (b) (c)

Fig-4: Optical micrograph of as sintered (a) F1, (b) F2 and (c) F3

CONCLUSION

The experimental results obtained in this study indicate that the multi-component binder system; PW/PE/SA, PS/TPNR and PS/PE work successfully with 17-4PH SS powder at maximum injection pressure of 400 bar and a temperature of 180°C. The injection moulded parts for all binder systems were good and free from normal defects such as short moulding, flashing and parting surfaces. The perfect binder has yet to be discovered and extensive research should be conducted in determining suitable combination of binder components, debinding route as well as optimum sintering parameters. The findings suggest that the sintered properties obtained from binder system PW/PE/SA were superior compared to binder systems PS/TPNR and PS/PE. Nevertheless, the bio-composite binder systems, PS/TPNR and PS/PE could be considered as a promising binder in MIM of 17-4PH SS.

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