Binder Removal from Powder Injection Molded 316L Stainless Steel

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Abstract: This study reports the results of preparation of 316L stainless steel polymer based injection molded feed stock, rheology of feed stock and injection molding. The plastic binder was extracted from molded samples. The binder extraction was carried out in two steps: by solvent and thermal techniques. The results showed that feed stock prepared was suitable for injection molding and this was confirmed by rheology data measured by using capillary rheometer. The test samples were injection molded without physical defects. Paraffin Wax (major binder) was extracted by using solvent extraction for 300 min. The thermal debinding was performed four different heating rates range 1-7°C min⁻¹. The SEM results showed that the PW was completely extracted from the test samples after 300 min.

Key words: 316L stainless steel, Powder injection molding, Rheology

INTRODUCTION

Complex shaped high performance and more précised metal or ceramic components can be produced economically with conventional injection machine and this process is known as Powder injection molding (PIM) (German, 1990; German and Bose, 1997). Powder injection molding consists of four sub sequential steps mixing, injection molding, debinding and sintering (Zlatkov et al., 2008; Jamaludin et al., 2007). The PIM process starts with the mixing of powder and binder. The final mixture is known as feed stock. Feed stock is very important step in PIM process any deficiency in feedstock can not be recovered latter (Supati et al., 2000). The role of binder systems like transporter, which is helpful for the homogeneous distribution of metal powder into the desired shaped. These systems also hold the particles in the beginning of sintering process (German and Bose, 1997). Due to this reason binder system has key role for the success of injection molding process. Binder played important role to achieve the shape and strength of the product. Several binder systems are available but the formulation depends upon the metal powder size, shape and size (Quan et al., 2008). The binder system has low viscosity, small molecular weight, surfactant inexpensive and environment friendly (German, 2007). Zaky et al. (2009) investigated different binder systems and found that the binder system contained 62wt.% of paraffin wax is an excellent one. They also concluded that solvent immersion is suitable technique to remove the major binder. They removed the major binder completely heating at 40°C for 5 h. Feed stock is than converted into granules followed by injection molding. After the injection molding the green product is debinded. Finally the sintering is done to obtain optimum properties.

A large number of researchers investigated the different aspects of 316L stainless steel. German (2007) suggested that the powder should have size less than 20 μm, tap density less than 50% of theoretical density, spherical in shape and free from agglomeration. Quan et al. (2008) and Barriere et al. (2003) suggested that for better deformation resistance vacuum environment is more beneficial than gas (N₂) atmosphere. They also conclude that particle size, shape, solid loading, heating rate and atmosphere also affect the deformation defects. Quan et al., 2008. Nylund et al. (1995) found that sintered density is more important to achieve excellent mechanical properties and good corrosion resistance. Various techniques are employed to improve the density. Loh et al. (2001) used TiC with 316L stainless steel to improve the mechanical properties. The also found that sintering temperature and heating rate also effect the mechanical properties. The packing density also increased by using different size powders (German, 1992; German and Bulge, 1992). Huang and Hsu (2009) also studied the effects of back bone polymer (LDPE and HDPE) on

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dimensional changes and mechanical properties of MIM 316L components. Ji et al. (2001) suggested that the factor such as sintering temperature, sintering time, heating rate and atmosphere affect the sinter density. They got maximum 96% of sinter density. Becker et al. (2000) adopted the PM route and achieve the 98% sintered density of water atomized 316L stainless steel. According to them the liquid phase is responsible for high density. Omar (1999) obtained the 96% sintered density of FIM 316L stainless steel using PEG and PMMA binder system under argon atmosphere, while Jamaludin et al. (2008) obtained 99% sintered density by using same system except the sintering atmosphere (Vacuum) between temperature range 1340-1400 with heating rate 10°C/min and dwell time 4 h.

Yimin et al. (1999) studied the effect of solid loading on MIM gas atomized 17-4 Stainless steel on density and mechanical properties. The study concludes that 68Vol% is the best solid loading with binder 65%FW, 30% EVA and 5%SA.

During the present study two formulation of the feed stock with solid loading 60 and 65%vol% were prepared using paraffin wax based binder system (PW 70vol%, PP 25vol% and SA5vol%). The feed stock was characterized by TGA and Rheometer. The injection molded samples were debinded and analyzed by using SEM/FESEM.

MATERIALS AND METHODS

Materials: The metal powder used in this study was stainless steel 316L (PF-10R) water atomized supplied by PACIFIC SOWA Japan. The mean particle size D50 is 5-7μm. The chemical composition and morphology of the powder is given in Table 1 and Fig. 1a, respectively. To make the easy flow of metal powder into mold cavity, polymeric based binder system was used. In the current research work a paraffin wax (PW) based system was prepared. The ingredients with paraffin wax (PW) were Polypropylene (PP) and Steeric Acid (SA). The composition of the binder system was PW 70vol%, PP 25vol% and SA 5vol%, minor components of the binder system acts as surface active agent. The binder system was thermally characterized by using TGA (Perkin Elmer). Testing was done under nitrogen atmosphere with heating rate of 20°C min⁻¹. The flow behavior was studied by using capillary rheometer CFT 500D with die 1×10mm at different loads and temperatures.

**Feed stock preparation:** Two formulations F1 and F2 were prepared with solid loading 60 and 65vol%, respectively. The stainless steel powder was mixed with binder using Z-blade mixer at temperature 165°C for 90 min at speed 60 rpm. After mixing, the paste was converted into granules. The characterization of the feed stock was done by using TGA and capillary rheometer.

**Characterization:**

**Table 1: Chemical composition of 316L Stainless Steel**

<table>
<thead>
<tr>
<th>Element</th>
<th>Standard wt.%</th>
<th>Measured wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.03</td>
<td>0.024</td>
</tr>
<tr>
<td>Si</td>
<td>0.5</td>
<td>0.36</td>
</tr>
<tr>
<td>Mn</td>
<td>0.5</td>
<td>0.07</td>
</tr>
<tr>
<td>P</td>
<td>0.04</td>
<td>0.029</td>
</tr>
<tr>
<td>S</td>
<td>0.03</td>
<td>0.002</td>
</tr>
<tr>
<td>Ni</td>
<td>10-11</td>
<td>10.53</td>
</tr>
<tr>
<td>Cr</td>
<td>16-17.2</td>
<td>16.57</td>
</tr>
<tr>
<td>Mo</td>
<td>2-2.4</td>
<td>2.1</td>
</tr>
<tr>
<td>Cu</td>
<td>6.1</td>
<td>6.02</td>
</tr>
<tr>
<td>N</td>
<td>-</td>
<td>0.002</td>
</tr>
</tbody>
</table>

**Molding:** The tensile specimens were molded by using 100KSA vertical injection molding machine. The samples were molded at temperature 164°C and pressure 4.5 bar. The molding time varies from 0.3-0.5 min.

**Physical examination:** In powder injection molding the common molding defects are cracks, powder-binder separation, short shots, sink mark, internal voids etc. these defects may be due to the bad material or improper molding conditions (Namko et al., 1993). In molded samples no defects were observed.

**Debinding:** The debinding process is most appropriate for removal of binder from Powder injection molded green parts. It consists of (1) solvent extraction and (2) thermal debinding.

Solvent extraction process the soluble component of the binder removed from the molded parts (Johnson, 1988; Wiech, 1980, German, 1990). Different organic solvents such as n-hexane, n-heptane and iso-octane were at different temperature ranging from 30-60°C (Fu et al., 2005).

During the present study solvent extraction process was followed by thermal debinding process. Solvent extraction process was carried out by immersing test samples in n-heptane at 60°C. To study the effect of time on loss of PW the test specimens were heated up to 5 h. The debinding ratio was measured (Zaky, 2004; Omar et al., 2003). Finally, the results were verified by using SEM.

During thermal debinding process test specimens were heated at 450°C for 1 h with four different heating rates (1°C/min, 3°C/min, 5°C/min and 7°C/ min) to optimize the suitable debinding rate.

**Sintering:** The test specimens will be sintered under vacuum to observe the effect of different heating and cooling rate on density, mechanical properties and corrosion as well.
RESULTS AND DISCUSSION

Material characterization: Scanning electron micrograph of Stainless steel 316L showing in Fig. 1a, while the particle size analysis is shown in Fig. 1b. From Fig. 1a it is clear that powder particles have round shape and particle size between 4-7 μm.

TGA analysis of binder and feedstock: TGA analysis of binder and feedstock was done. The data given in Fig. 2 indicate that no residue was left after the decomposition of binder. The decomposition of binder started about 250°C. From the graph shown in Fig. 2, it is clear that after the decomposition of feedstock F1 about 91 wt. % residue left. This amount of residue is same as SS powder calculated in feedstock.

Rheology of binder and feedstock: The rheological behavior of both feeds tocks were studied at different temperature ranging from 100 to 160°C. The results are shown in Fig. 3. From graphs it is clear that shear rate and viscosity showed slightly pseudo plastic behavior. The viscosity of both formulation of feeds stock is lower than 20Pa.s, because the presence of Paraffin wax having viscosity less than 10Pa.s (Zaky et al., 2009). For successful metal injection molding the shear rate ranges

Fig. 1a: SEM micrograph of 316L stainless steel at 3KX

Fig. 1b: Particle size distribution of 316L Stainless steel

Fig. 2: Comparison of TGA scan for PW, PP, SA, binder, and 60vol% solid loading

Fig. 3a: Viscosity of feedstock F1 as a function of shear rate at different temperatures raging 100 to 160°C

Fig. 3b: Viscosity of feedstock F2 as a function of shear rate at different temperatures raging 100 to 160°C

Fig. 4: Comparison of masses of green parts for both formulations F1 and F1 after molding
Fig. 5: Weight percentage of Paraffin Wax removed with respect to debinding time for 60% vol (F1) and 65% vol (F2) solid loading.

Fig. 6a: SEM micrograph of molded sample of F1 showing the well dispersion of powder (surface view).

Fig. 6b: SEM micrograph of molded sample of F1 showing the well dispersion of powder (fracture view).

Fig. 6c: SEM micrograph of F1 showing the top of as molded part after leaching for 300 min.

Fig. 6d: SEM micrograph of F1 surface showing the fracture view of as molded part after 300 min leaching.

Fig. 7a: SEM micrograph of F2 showing the surface of molded part.

Fig. 7b: SEM micrograph of F2 showing the fracture view of molded part.

Fig. 7c: SEM micrograph of F2 showing the top of as molded part for 300 min leaching.

Between 102-105 S-1. The maximum suitable viscosity for feedstock is 103 Pa s at molding temperature (Shah and Nunn, 1987; Mutsuddy, 1983; Jorge, 2008). Fu et al. (2005) suggested that at high temperature low
Viscosity is suitable for injection molding. For ceramic injection molding the shear rate between 100 and 1000 S-1 and viscosity should be less than 1000Pa.s. From the results, it is clear that the feed stocks for both formulation is suitable for injection molding.

Visual examination: The visual examination was carried out, no defects were observed on the samples. The mass and dimensions of all samples were measured. In both formulations the molded samples have almost same masses and dimensions Fig. 4.

Leached samples: From results obtained by debinding ratio are given in Fig. 5. It is clear that for both formulations the major binder PW was completely removed. From the results it is clear that 300 min is the suitable time to remove the major binder from the samples. After the solvent extraction process no swelling or cracks were observed on surfaces of the test samples. The results were also verified by SEM analysis as well.

Thermal debinding: The samples from both formulations were thermally debinded with heating rate 1°C, 3°C, 5°C and 7°C. There was no swelling or cracks on the surface of the debinded samples. The debinded samples have brown colour. The only one sample from formulation 65% vol heated with rate 1°C having dark brown colour.

Binder removal by leaching: Figure 6 shows SEM micrographs for metal and binder (60 vol% solid loading) before and solvent extraction. Fig. 6 (a-b) shows the surface and fracture micrographs magnification 1KX respectively. It is clear that the powder is well distributed in binder system. Fig. 6 (c-d) showed the surface and fracture view of the solvent debinding after 300 min. from both micrographs it is clear that there are pores initiate from surface to the core of the sample. These pores are helpful for thermal debinding process to remove the gasses from the test samples to avoid the cracks on the samples. From these pores are indications that PW is completely removed from the molded parts.

Figure 7 represents the surface and fracture view of the 65% vol solid loading. From Fig. 7a and b it is clear that there is no agglomeration of powder in binder. Figure 7c and d showed the pores on the surface and fracture surface of the test samples which indicate that PW dissolved in n-heptane. From the measurement it is clear that Paraffin Wax entirely removed after 300 min.

CONCLUSION

From the above research work, we can conclude following:

- For solvent extraction suitable temperature is 60 and time is 300 min
- For both solid loading 60% vol and 65% vol solid loading the shear rate showed pseudo plastic behavior
- For both solid loading the viscosity is less than 20 which is suitable for Powder injection molding
- Both formulations are suitable for Injection molding
- The debinding heating rate 1°C is not suit able for solid loading 65% vol

REFERENCES


