Asian Journal of Scientific Research

ISSN 1992-1454

Asian Journal of Scientific Research 6 (2): 307-314, 2013
ISSN 1992-1454 / DOI: 10.3923/ajsr.2013.307.314

Development of Defects Free Stainless Steel Parts Using Powder Injection Molding

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ABSTRACT

Austenitic 316L Stainless Steel (SS) is widely used in aerospace, automotive, sports and medical industries due to its mechanical properties and corrosion resistance. Defects free molding of parts depends upon the features of feedstock. In powder injection molding, feedstock preparation is critical step and any deficiency at this stage cannot be retrieve in latter steps. The objective of this research work is to optimize solid loading for defects free injection molded parts. During the present research work five formulations having solid loading 60-71 vol% were prepared by using multi component binder system. The degradation temperature of feedstocks was determined by using Thermogravimetric analysis (TGA) and flow behavior through rheometer. Homogeneity of the feedstock was verified by using Scanning Electron Microscopy (SEM). Finally, injection molding was done and it was found that the feedstocks having solid loading up to 69 vol% were successfully injection molded and components were without physical defects.

Key words: 316L stainless steel, feedstock, injection molding, binder system, rheology, SEM images

INTRODUCTION

Powder Injection Molding (PIM) is fabricating technique that fulfills the gaps of other conventional techniques. It has advantage to produce metal and ceramic parts with complex shape, dimensional accuracy and low cost (Mutsuddy and Ford, 1995; German and Bose, 1997; Zlatkov *et al.*, 2008). PIM process is completed into four steps. i.e, feedstock preparation, molding, debinding and sintering (Zlatkov *et al.*, 2008). Feedstock is a combination of metal powder and polymeric binder system. Polymeric binder systems consist of two or more than two polymers to provide the easy flow of metal particles within mold cavity, ejection of part from mold and enough strength to handle. Feedstock preparation is very important step in PIM. Feedstock must be homogeneous having maximum metal powder loading with part shape stability and provide enough handling strength after molding and minimum residue upon debinding. Any deficiency during feedstock preparation cannot be recover in latter steps and it affects the molding and final properties of the parts (Supati *et al.*, 2000).

The 316L stainless steel is used in various industries due to ease of availability, low cost as compared to other metal alloys and excellent combination of mechanical properties with corrosion resistance (Zlatkov *et al.*, 2008).

Suitability of feedstock for PIM can be determined by rheological test and degradation temperature of polymeric binder was determined by using TGA.

Previously, authors studied the effects of particle shape, powder loading, various binder systems, debinding and sintering temperature on final properties of PIM 316L SS (Ohk, 1997; Liu *et al.*, 2002; Amin *et al.*, 2009; Jorge, 2008; Matula *et al.*, 2008; Raza *et al.*, 2011). They concluded that particle shape and powder loading has significant effects on flow behavior as compared to temperature. Liu *et al.* (2002) used the commercially available binder and found that feedstock showed pseudo plastic behavior which is suitable for PIM. The authors concluded that the viscosity of the feedstock depends upon temperature and shear rate and increased by decreasing temperature or shear rate. Matula *et al.* (2008) used polyethylene as binder with M2 HSS reinforced with carbide and measured torque to optimize solid loading and concluded that low viscosity is responsible to achieve maximum solid loading. Amin *et al.* (2009) and Jamaludin *et al.* (2008) investigated the effects of particle size on rheological properties of 316L SS . The authors used two different particle sizes and shapes with a binder system composed of PEG, PMMA and SA. They found that large size particle has better rheological behavior over a wide range of temperature as compared to small particle size. So, the large particle size is suitable to produce parts without physical defects. Jamaludin *et al.* (2008) also investigated the rheological behavior of bimodal feedstock of 316L SS and found that bimodal particles are helpful to reduce the viscosity as compared to the monomodal due to the distribution of particles. Supati *et al.* (2000) mixed 316L SS with Ti carbides by using a patented binder and results showed that feedstock with 54 vol% powder loading showed better rheological properties when mixed at 90°C with rotation speed 30. Khakbiz *et al.* (2005) investigated the effects of 3 wt.% TiC on rheological properties of 316L SS. Various formulations were prepared and showed pseudo plastic behavior.

During the present research work five formulations with solid loading 60, 65, 67, 69 and 71 vol% were prepared by using paraffin wax based polymeric binder system that consists of Paraffin Wax (PW), polypropylene (PP) and Steric Acid (SA) in volume ratio 12:5:1, respectively. All feedstocks were characterized for degradation and flow before molding.

MATERIALS AND METHODS

Water atomized 316L SS (PF-10R) powder supplied by PICIFIC SOWA Japan having particle size D_{10} = 1.83 μm, D_{50} = 4.42 μm and D_{90} = 7.63 μm. The chemical composition provided by the company and morphology of the powder is same as described in earlier work (Raza *et al.*, 2011). Feedstocks were prepared by using wax based polymeric binder system. The binder system consists of paraffin wax 70%, polypropylene 25% and stearic acid 5%.

Five formulations F1, F2, F3, F4 and F5 were prepared with different solid loadings as given in Table 1. Feedstocks were prepared by using Z-blade mixer at temperature 180°C for 90 min at speed 60 rpm. After mixing, the paste was dried and converted into granules. The characterization of the feed stock was done by using TGA and capillary rheometer. Vertical injection molding

Table 1: Composition of feedstock formulations

Formulation	Powder loading (vol%)	Binder (vol%)
F1	60	40
F2	65	35
F3	67	33
F4	69	31
F5	71	29

machine modal 100KSA was used to mold the test samples. All formulations except F5 were molded at temperature 175±5°C. The molding time varies from 15-20 sec. No physical defects were observed on the surface of the test samples.

RESULTS AND DISCUSSION

TGA analysis of binder and feedstocks: TGA analysis of binder and feedstocks is shown in Fig. 1. The results showed that degradation of the binder started at 200°C. At this temperature the degradation of the PW and SA started and rest of the binder component (PP) starts its degradation at 375°C and at the end of the experiment the amount of residue left is approximately zero that is amount of carbon content after the degradation of binder.

In all formulations, TGA analysis showed same degradation behavior except the difference in amount of residue. It was observed that after the decomposition of feedstock F1 about 91 wt.% residue was left. This amount of residue is same as SS powder calculated in feedstock. Similarly, after the decomposition of feedstock F2, F3, F4 and F5 the residue left was 93.3, 94.8, 95 and 93 wt.% , respectively shown in Fig. 1. The amount of residue is equal to the amount of stainless steel powder used to prepare these formulations. While in case of F5 the residual weight was 2% less as compared to the calculated amount of powder which means the binder is not homogeneously mixed or air is tapped within the binder that causes to reduce the residual weight.

Rheology of feedstocks: The rheological behavior of all feedstocks was studied at different temperatures ranging from 140-180°C to investigate the effects of temperature on flow behavior of feedstock. This selected range of temperatures was between melting and degradation temperature of the PW. The results showed that variation of flow behavior depends upon the applied force and the material composition (Bhattacharya, 1997).

From Fig. 2-5, it is clear that shear rate and viscosity of feedstocks showed non Newtonian flow. Commonly this type of flow is known as pseudo plastic flow which is recommended for PIM. In pseudo plastic behavior the viscosity and shear rate are opposite to each other. It may be due to change in structure of the solid particles or binder. For successful metal injection molding the shear rate ranges from 10^2-10^5 S^{-1}. The maximum suitable viscosity for feedstock is 10^3 Pa s at molding temperature (Shah and Nunn 1987; Mutsuddy and Ford, 1995; Jorge, 2008). The rheology results showed that by increasing the solid loading shear rate was decreased due to the interaction between the particles increased and the presence of plastic binder in feedstock resulted the non Newtonian behavior (Bhattacharya, 1997). It was observed that by increasing the temperature the viscosity of the feedstock was decreased. The feedstock having low viscosity at higher temperature

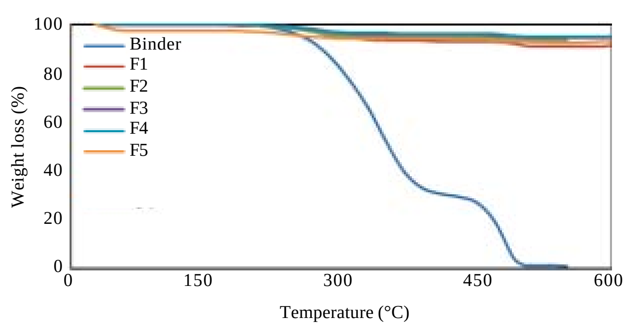

Fig. 1: Comparison of TGA of binder and feedstocks

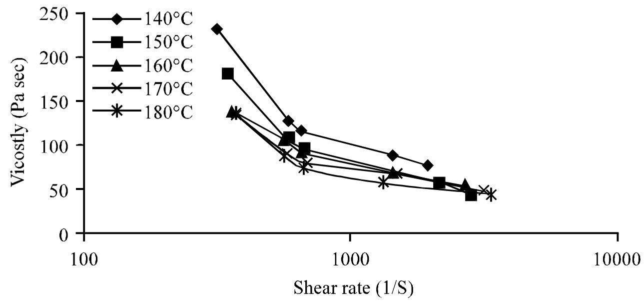

Fig. 2: Feedstock F1 showing pseudo plastic behavior at different temperatures

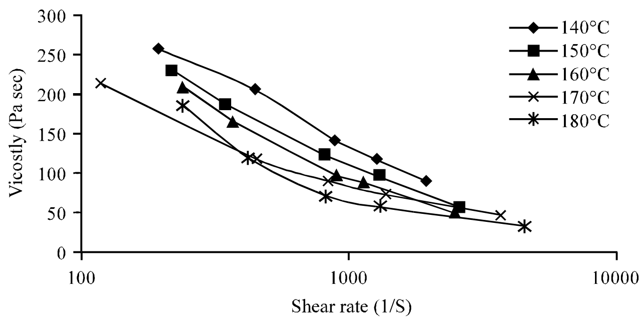

Fig. 3: Feedstock F2 showing pseudo plastic behavior at different temperatures

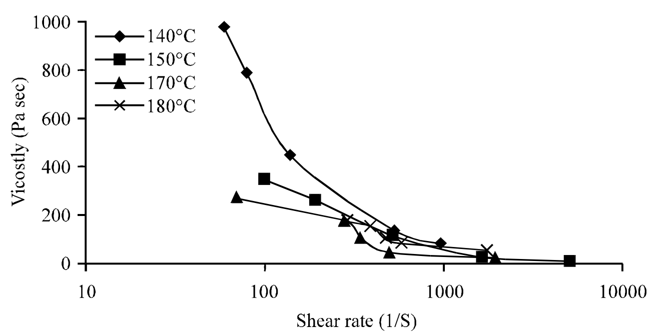

Fig. 4: Feedstock F3 showing pseudo plastic behavior at different temperatures

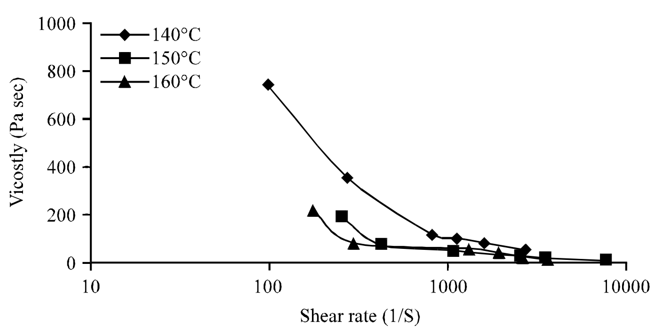

Fig. 5: Feedstock F4 showing pseudo plastic behavior at different temperatures

is suitable for injection molding (Fu *et al.*, 2005). From these results, it is concluded that the formulations from F1-F4 are suitable for injection molding.

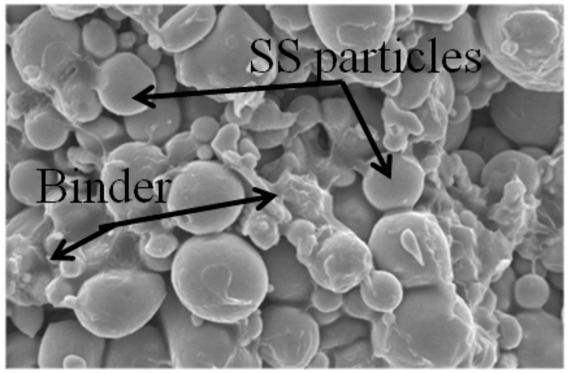

Fig. 6: SEM micrograph of F1 (60 vol% solid loading) showing homogeneous mixing: 3kX

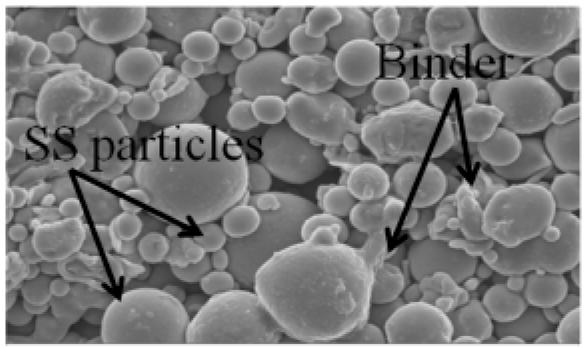

Fig. 7: SEM micrograph of F2 (65 vol% solid loading) showing homogeneous mixing: 3kX

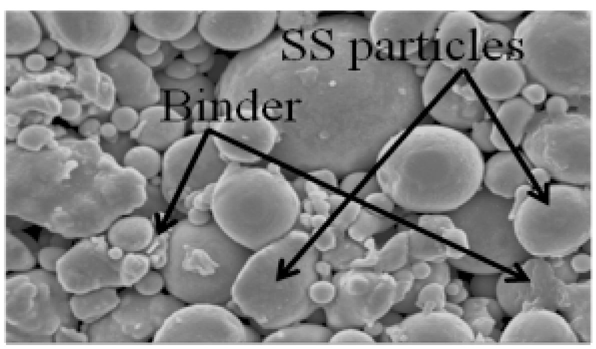

Fig. 8: SEM micrograph of F3 (67 vol% solid loading) showing homogeneous mixing: 3kX

For F5 with 71% solid loading it was not able to flow through the capillary die. So no results were noted for F5 formulation. It is due the less binder between the metal particles which is not enough to provide the transportation to the metal particles through die.

SEM analysis of feedstock: All five formulations were analyzed by using SEM to observe the homogeneity of 316L SS powder and polymeric binder. From the micrographs shown in Fig. 6-10, it is clear that the prepared feedstocks have homogeneous dispersion of binder system between powder particles. From these micrographs it is clear that the amount of binder between the particles

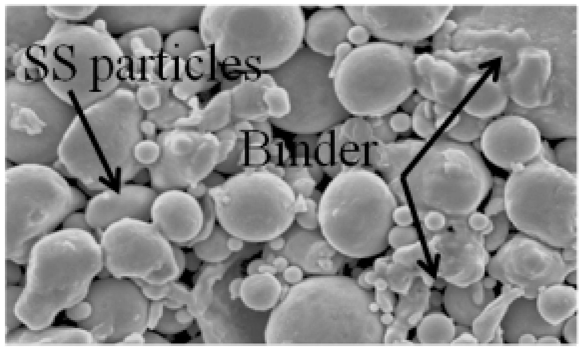

Fig. 9: SEM micrograph of F4 (69 vol% solid loading) showing homogeneous mixing: 3kX

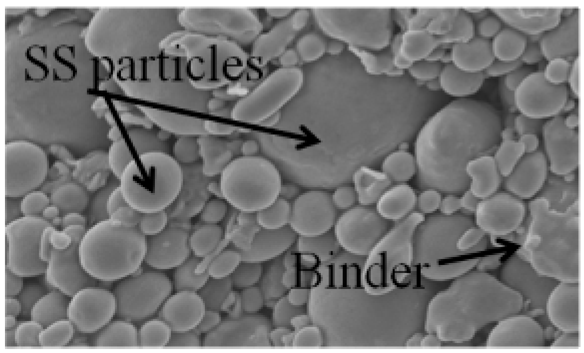

Fig. 10: SEM micrograph of F5 (71 vol% solid loading) showing homogeneous mixing: 3kX

Fig. 11: Injection molded test samples free from physical defects

or coating on the particles is reduced by increasing the solid loading. The micrograph shown in Fig. 10 the amount of solid loading is 71 vol% and the amount of binder between two powder particles is less as compared to the amount of binder with 60 vol% solid loading shown in Fig. 6.

Injection molding: All formulations except F5 were successfully molded by using vertical injection molding machine. The injection temperature was 175±5°C. The injection air pressure was 4.5 bar and the injection time varies from 15-20 sec depending upon the solid loading of the formulation. The injection molded test samples are free from physical defects as shown in Fig. 11.

Fig. 12: Formulation F5 shows short shot defects in test samples of F5 (71 vol%)

While in case of formulation F5 (71 vol% solid loading) feedstock was unable to flow through the mold due to lower amount of binder. Short shot defects were observed during molding as shown in Fig. 12.

CONCLUSIONS

This study concluded that:

- Pseudo plastic behaviour was observed in formulations having solid loading up to 69 vol% and viscosity lies within the range required for PIM
- The solid loading up to 69 vol% was successfully injection molded
- If the amount of binder is not sufficient within feedstock than it is not possible to flow inside mold cavity that result short shot defect (71 vol% Solid loading)

ACKNOWLEDGMENTS

The authors would like to express their appreciation to Universiti Teknologi PETRONAS for providing the fund and laboratory facilities. Authors are also thankful to “Advanced Materials Research Centre (AMREC) SIRIM, Kulim, Malaysia” for providing experimental support.

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