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LOW PRESSURE INJECTION MOLDING OF BORON ADMIXED 316L STAINLESS STEEL FOR ENGINEERING APPLICATIONS

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ABSTRACT

This study was conducted to evaluate the effects of elemental boron on densification of 316L stainless steels. Elemental nano size boron (nB) powder of 0.5, 1 and 1.5 wt. % was blended with 316L SS to produce homogeneous feedstock for low pressure powder injection molding (LPIM) process. LPIM molded samples were sintered at 1230^0C using high vacuum 10^{-5}Torr. The Results showed that addition of elemental boron has favorable effects on densification. Addition of 0.5 wt% nano size elemental boron in 316L SS has enhanced densification up to 98.5%. Defect free gears were injection molded and sintered successfully using optimal sintering cycle.

Keywords: 316L SS powder, Solvent debinding, scanning electron microscopy, n-heptane, sintering, densification, gear.

INTRODUCTION

In order to improve the density of sintered parts, external pressure can be applied on parts during sintering process or highly active kinetics path should be provided during sintering through addition of second phase particles[1, 2]. So there are two non-pressure based approaches that can be utilized to enhance the kinetics of sintering process named: liquid phase sintering and activated sintering. Activated sintering is a special process used for lowering the sintering temperature and reducing the sintering time to improve densification of compacts. Although several studies have been conducted on activated sintering on many metals and alloys like: tantalum [3], molybdenum [4], hafnium [5], beryllium [6], aluminum oxide [7] and copper [8] but now main focus of researchers is to modify the sintering cycle for iron and stainless steels [9-12].

In activated sintering, additives are used in small quantity to modify the sintering behavior of base metals and sometimes it is only added in the fraction of 1 wt.% [13]. Additives have exhibited the key role in improving the diffusion process and mobility of grain boundaries [14, 15]. For generalized presentation, German *et al*.[16] considered two powders one named additive denoted by A and second was named base denoted by B. Additive A was mixed with B for sintering of base material B. They concluded that additive base material B should have higher solubility for additive A to achieve the better diffusion rate. Low solubility of A in B correlates the segregation of layer between the diffused particles grain boundaries and low solubility of A in B minimizes the need for additive required for activation sintering [17]. This consideration is very important and demands the unipolar solubility relationship. Mathematically it can be expressed as:-

$$\frac{S_B}{S_A} \gg 1 \qquad (1)$$

Where S_B is solubility of *B* in *A* and S_A is solubility of *A* in *B*.

In current research work, loading effects of nano size elemental boron powder on densification of 316L SS was studied and conclusion was made on practical observation during sintering process.

RESEARCH METHODOLOGY

In order to develop modified PWA 316L SS samples, an experimental protocol was followed as shown in Figure-1 and details are as under:-

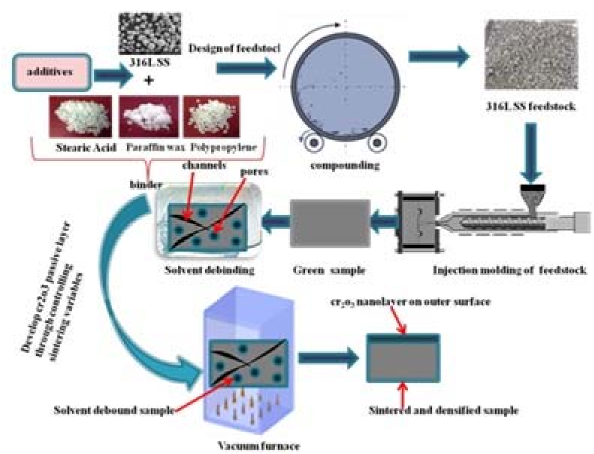

Figure-1. Schematic diagram of methodology.

Feedstock preparation

First, Feedstock preparation is an important process in powder injection molding (PIM). The quality of feedstock established the quality of molding, debinding and sintering process. Feedstock must be homogeneous and has maximum metal powder loading with shape stability and strength after molding. Four formulations

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were prepared as shown in Table-1. Elemental nao boron powder of different wt% (0.5wt%, 1wt%, 1.5wt%) was mixed with SS powder using tubular mixer for 30 min. Then blended powder was mixed with a multicomponent binder of composition 70vol% paraffin wax (PW), 25vol% Polypropylene (PP) and 5vol% stearic acid (SA) in z-blade mixer. A mixture of powder was compounded for 90 minutes at 170^0C. After compounding, mixture was granulated as shown in the Figure-2.

Table-1. Formulations compositions.

Sr. No	Formulations name	316L SS (wt%)	Elemental Boron (wt%)
1	PWA316L SS-0B	100	0
2	PWA316L SS-0.5B	99.5	0.5
3	PWA316L SS-1B	99	1
4	PWA316L SS-1.5B	98.5	1.5

Figure-2. Granulated feedstock of PWA-316LSS.

DSC (Differential Scanning calorimeter) and TGA (Thermo gravimetric analysis) is thermal analysis techniques that were used to observe the melting and degradation temperature of the binder ingredients which is helpful for feedstock preparation for injection molding.

Green samples were produced according to the MPIF 10 standard. The injected samples were examined physically and no defects were observed. Samples dimension and weight was measured to calculate the shrinkage and densification of green samples.

Solvent debinding process was used to remove the binder from green test samples. In order to avoid the damage of green samples during debinding process, a multicomponent binder system was used for preparation of feedstock. Samples were immersed in n-heptane at 60 ^{o}C for 5h.

Thermal debinding and sintering processes were integrated in high vacuum furnace using optimal sintering cycle. Scanning electron microscopy (SEM) was used to observe the evolution of microstructure at sintering temperature 1230^oC. EDX analysis was carried out to analyze the elemental composition of sintered samples.

XRD was used to detect the phases developed during sintering process.

RESULTS AND DISCUSSION

Materials characterization

Irregular shaped powder water atomized 316L stainless steel (PWA 316L SS) (PF-20R) used during this research work was supplied by Epson Atmix Corporation Japan. The PWA 316L SS was characterized using MASTERSIZER 2000, manufactured by Malvern Worcestershire United Kingdom, and its morphology was determined using scanning electron microscopy. The SEM micrograph of PWA316L SS shows that particles were irregular in shape as shown in Figure 3. Surface weighted particle size of PWA 316LSS determined by particle size analyzer was 10µm. Spherical shaped nano sized elemental boron (nB) powder was purchased from Xuzhou Jiechuang new material technology co. ltd hong Kong and has average particle size 60 nm as shown in Figure-4.

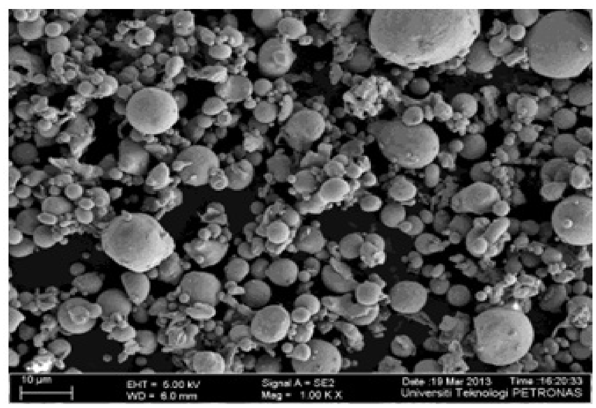

Figure-3. FESEM image of PWA 316L SS powder.

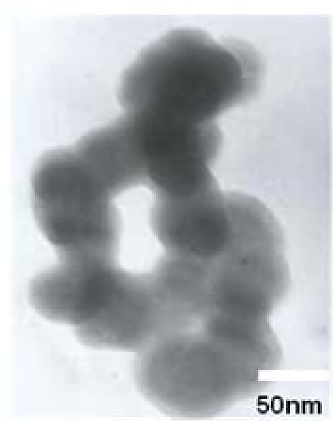

Figure-4. TEM image of elemental nano boron.

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FESEM analysis of feedstock

Feedstock was analyzed using FESEM to observe homogeneity of 316L SS powder within the plastic binder. From the micrograph shown in the Figure-5, it is clear that the prepared feedstock has homogeneous dispersion of powder particles in plastic binders.

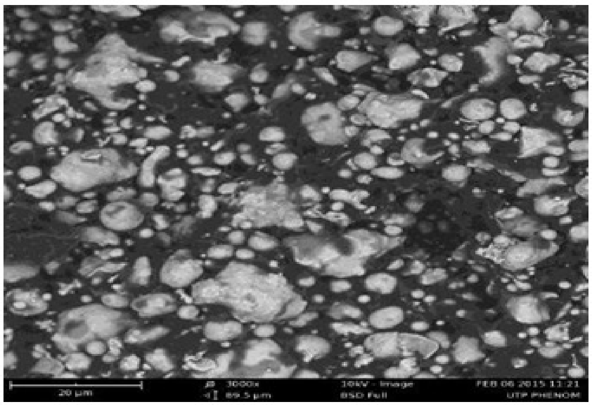

Figure-5. FESEM image of nao size boron admixed 316L SS.

After preparation of feedstock, its rheology is measured using rheometer. Rheology shows the flow behaviour of the material applied under force. Flow behaviour of feedstock was studied at 160 °C, 170°C and 180 °C to investigate the effect of temperature on viscosity. It is clear from Figure-6 that as shear rate increases the viscosity decreases. Feedstock shows the pseudo plastic behaviour and it is considered appropriate for the powder injection molding process. Results are consistent with the findings [18].

Feedstock was successfully molded using vertical injection molding machine and defect free dogbone shape samples and gears are developed as shown in Figure-8. The injection temperature to inject the feedstock was kept 170± 5 °C. The injection pressure was 0.45MPa and injection time was kept 10-15 seconds depending on boron loading. The optimal integrated thermal debinding and sintering cycle as shown in Figure-7 was used to produce defect free dogbone shape and gears.

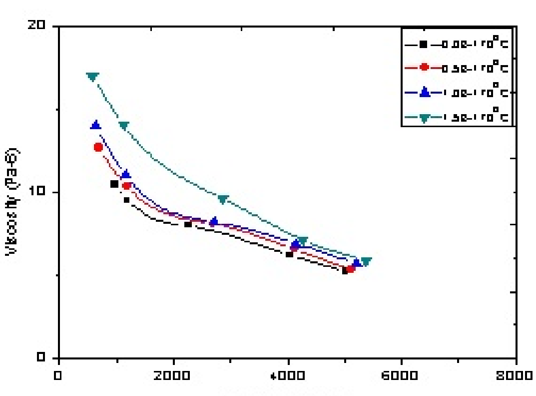

Figure-6. A graph between shear rate and viscosity.

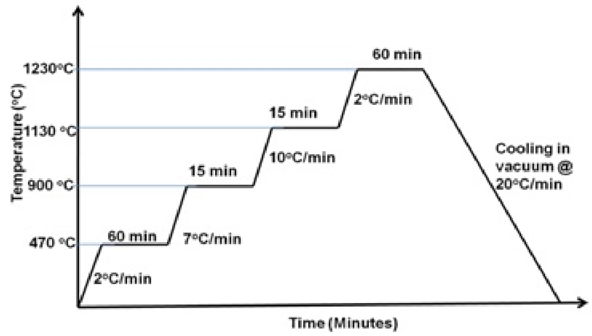

Figure-7. Optimal integrated thermal and sintering cycle.

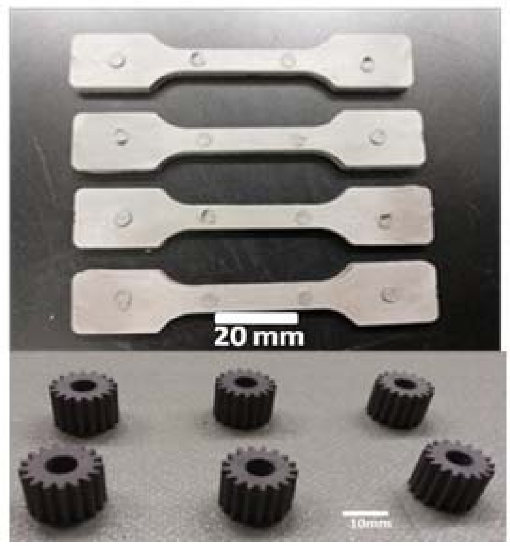

Figure-8. Defect free injection molded (a) dogbone shape sample (b) gear part.

Microstructure analysis of sintered test samples

FESEM analysis of nano size boron additive added in 316L SS shows that boron made the complex compound of iron boride on grain boundaries and this compound has exhibited low melting temperature. During LPIM process, the boron nano size particles are dispersed in base materials and make a diffusive path on grain boundaries. The base material flows across the flux path and contributes in growing of neck, strengthening and pore elimination. It results in densification of sintered parts. Segregation of second phase particles is necessary for densification process. Figure-9 shows the optical micrographs for boron free and boron containing samples. It is clear from this figure that boron containing samples shows almost full densification and pores are eliminated. The spots inside the boron containing samples of Figure-9 (b) are actually the segregation of some boron particles that has diffused inside the base materials. However, boron reacts with iron and form iron boride low eutectic compound on grain boundaries and it acts as flux path for base material. Iron atoms diffuse across the grain boundary and densification occurs because of atoms

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diffusion, bulk diffusion, dislocation movement and volume diffusions process. During diffusion process, it is evident from the Figure-9 (b) that grain boundaries contributed to mass transportation. The boron additives played an effective role between the interparticles interfaces through segregation during sintering. Thus, the role of boron as diffusion rate enhancer is proven by calculating the sintered density of samples. However Figure-9(a) shows the boron free samples optical micrographs and it shows lot of pores inside the grains.

Figure-10 (a) shows the FESEM micrographs for boron free sample and it is clear that these sample exhibited twin boundaries at 1230 °C and pores are also present inside the grains and on the grain boundaries. Austenitic stainless steel exhibits twinning phenomenon on low sintering temperatures. However, Figure-10(b) shows large grain size and pores are totally eliminated. Moreover, iron boride is also visible on the grain boundaries that help in densification of 316L SS. The visible texture in Figure-10(a) is simply the result of different random crystal orientations and different rates of etchant attack during the etching process. It is well known that activated sintering rearranges the particles and enhances densification owing to the capillary action of the liquid phase on grain boundaries. Thus small size particles of 316L stainless steel enhanced the capillary action because of high capillary stresses. Moreover, high densification is considered due to higher solubility of SS for additive boron in order to achieve the better diffusion rates that result in high densification.

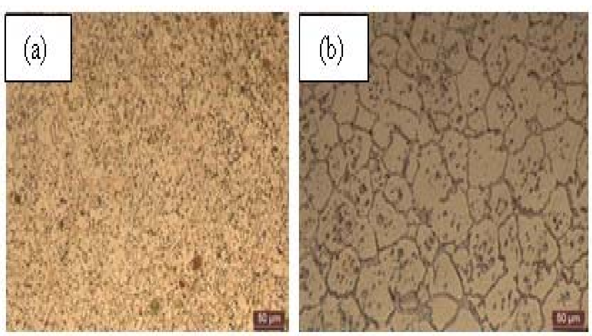

Figure-9. Optical microscope micrographs for (a) PWA-0B (b) PWA-0.5B sintered at 1230 °C.

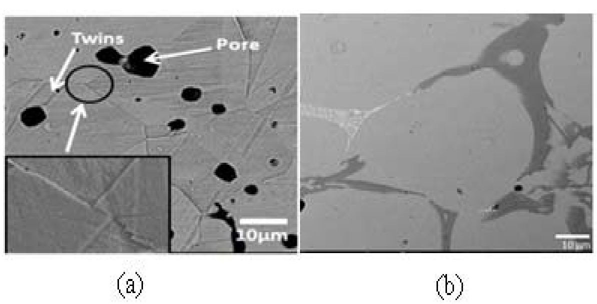

Figure-10. FESEM micrographs for (a) PWA-0B (b) PWA-0.5B sintered samples at 1230 °C.

Sintered density and shrinkage analysis

Figure-11 shows that PWA-0B formulation sintered at 1230 °C exhibits lower densification as compared to boron added test samples. The sintered density of PWA-0B is observed 91.86 % at 1230 °C. The low densification of PWA-0B at 1230 °C sintering temperatures is due to slow atomic diffusion of atoms, and sinking of pores from inside grains to grain boundaries needs sufficient kinetic energy and dwell time. The test samples PWA-0.5B shows the sintered density 98.5 % at 1230°C. PWA-0.5B test samples sintered density is 7.22 % higher than PWA-0B test samples sintered at same temperature i.e 1230 °C. This increase in densification is associated to the low melting eutectic phase of iron and boron that develop flux path and increase diffusion across the grain boundaries [16]. However, samples PWA-1B showed densification 1.03 % greater than PWA-0.5B test samples when sintered at 1230 °C but geometrical features of test samples were deteriorated. The densification of PWA-1.5B test samples was reduced 0.5 % as compared to PWA-1B due to excessive amount of liquid formed on grain boundaries that trap the gases inside resulting lower densification. Boron additive played an important role in changing the interfacial energy, diffusion rate, mobility of grain boundaries and phase stability that resulted in high density of 316L SS.

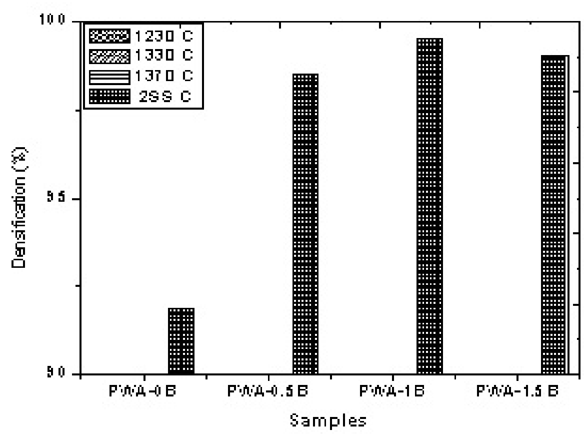

Figure-11. Effect of boron addition on densification.

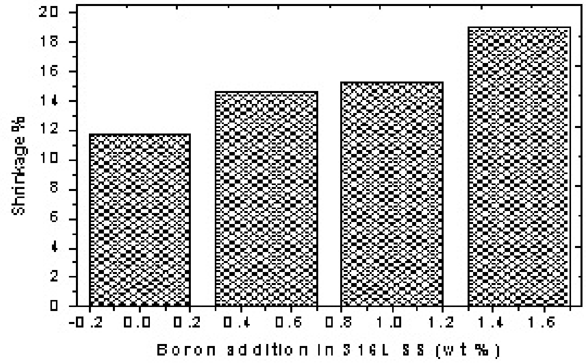

Figure-12. Effect of boron addition on shrinkage.

Dimensional changes at 1230 °C sintering temperatures are measured. The shrinkage rate for PWA-nB formulations was measured in longitudinal direction. Addition of nano size boron powder has shown remarkable effect on shrinkage at 1230 °C temperatures. It is clear from Figure-12 that PWA-0.5B caused shrinkage in the sintered samples up to 14.49% and it is 2.73% higher than PWA-0B . The shrinkage of boron containing samples is due to the influence of the low melted eutectic mixture formed on grain boundaries and it has shown uni-polar solubility. Figure-12 shows that as the addition of boron is increased, the lower melted eutectic mixture is formed on grain boundaries. It helps the atomic diffusion of steel across the grain boundaries and increases the shrinkage of samples. However, samples containing boron more than 0.5 wt. % were not able to retain their shapes due to excessive amount of liquid formed on grain boundaries.

CONCLUSIONS

- Addition of boron in 316L SS is useful as diffusion rate enhancer.
- Optimal amount of boron as additive is o.5 wt% for high densification.
- Linear shrinkage of samples increases as amount of boron increases.
- High densification, 98.5% is achieved through 0.5 wt% of boron addition in 316L SS without deterioration of geometrical features of samples.
- PWA-0.5B feedstock is considered appropriate for low pressure injection molding to produce defect free gears components.
- The sintering temperature for PWA-0.5B injection molded sample is 1230°C and it is appropriate to produce the parts with complicated profiles.

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