Investigation of Boron addition and compaction pressure on the compactibility, densification and microhardness of 316L Stainless Steel

S Ali 1,2, A M A Rani 1, K Altaf 1, Z Baig 1
1 Mechanical Engineering Department, Universiti Teknologi PETRONAS, 32610 Bandar Seri Iskandar, Perak, Malaysia
2 Department of Design and Manufacturing Engineering, National University of Sciences & Technology (NUST), H12, Islamabad, Pakistan

Email: majdi@utp.edu.my

Abstract. Powder Metallurgy (P/M) is one of the continually evolving technologies used for producing metal materials of various sizes and shapes. However, some P/M materials have limited use in engineering for their performance deficiency including fully dense components. AISI 316L Stainless Steel (SS) is one of the promising materials used in P/M that combines outstanding corrosion resistance, strength and ductility for numerous applications. It is important to analyze the material composition along with the processing conditions that lead to a superior behaviour of the parts manufactured with P/M technique. This research investigates the effect of Boron addition on the compactibility, densification, sintering characteristics and microhardness of 316L SS parts produced with P/M. In this study, 0.25% Boron was added to the 316L Stainless Steel matrix to study the increase in densification of the 316L SS samples. The samples were made at different compaction pressures ranging from 100 MPa to 600 MPa and sintered in Nitrogen atmosphere at a temperature of 1200°C. The effect of compaction pressure and sintering temperature and atmosphere on the density and microhardness was evaluated. The microstructure of the samples was examined by optical microscope and microhardness was found using Vickers hardness machine. Results of the study showed that sintered samples with Boron addition exhibited high densification with increase in microhardness as compared to pure 316L SS sintered samples.

1. Introduction

Powder Metallurgy (P/M) is one of the most diverse and continually evolving technologies among the various metal working methodologies for producing parts of various sizes and shapes. The main advantages of P/M include high productivity and dimensional accuracy of the parts with minimal use of energy and owing to material savings as there is minimal loss of raw material[1]. The attraction of P/M for producing functional parts originate from several attributes ranging from high volume production of precise parts, difficult to process metal materials and specialty alloys containing mixed phases.

The manufacturing of a sintered part using P/M starts with the compaction of the metal powder in a rigid die of the desired size and shape. In this process, the die cavity is filled with metal powder and a high compaction pressure is applied on the powder in the die cavity. The powder particles get squeezed and a certain amount of cold welding takes place. The powder particles get interlocked and
remain intact with the surrounding particles leading to the development of interparticle bonds. Once
the part is ejected from the die, the compact green part owns significant strength to withhold its shape
and size. The next stage in this process is the sintering of the green compact parts where in the parts
are placed in a furnace and heated to a temperature slightly below the melting point of the metal. The
sintering cycle is designed accordingly depending upon the metal composition and the additives used.
The heating rate is kept low to minimize the chances of crack initiation in the parts[2]. This step
provides the thermal energy required to strengthen the powder particles and initiate the interparticle
bond that helps in improving the mechanical properties of the parts.

Stainless Steel parts produced by P/M play an important role in engineering sector due to their low
production cost and reduced need for post processing[3]. AISI 316L Stainless Steel is one of the
promising materials used in P/M. It has low carbon concentration with high nickel and chromium
contents that make it an attractive industrial material combining good corrosion resistance and
ductility in a number of applications[4]. It’s superior mechanical properties, high ductility, average
strength and corrosion resistance makes it an ideal material for use in several applications including
automotive, industrial, electrical and electronic, chemical, pharmaceutical, surgical and medical tools,
aerospace, structural components, bio-applications and others[5-8]. Although P/M has been there for
a quite long time but there has been research going on to improve the densification behavior of the P/M
materials. To cope with, improvement of processing steps of compaction and sintering and
modification of the powder has been developed to obtain high density P/M parts[5, 8].

Several Studies on sintered Stainless Steels have revealed that there are pores present inside the
sintered parts which have a deleterious effect on the mechanical properties of the parts[9, 10]. This
porosity is a cause of reduction in the tensile strength, impact resistance, ductility and corrosion
resistance of the parts[11, 12]. Porosity thus hinders the above mentioned properties and researchers
all over the world have tried to address this issue by incorporating additives in the stainless steel
matrix to get fully dense parts. Several studies on improved densification of Stainless Steels have
suggested the use of elemental Boron powder as an additive to get nearly fully dense parts for ferrous
alloys[7, 13-19]. The analysis of Iron-Boride (FeB) diagram reveals that Boron is an active sintering
additive for Iron based materials. The eutectic reaction takes place between Iron and Boron at a
temperature higher than the temperature required for eutectic transformation. At this stage, a liquid
phase appears which helps in improved sintering of the parts[1, 10]. Boron has also a good tendency to
form complex borides with Iron resulting in FeB, with Nickel forming Ni2B and with Chromium it
forms CrB and segregate at the grain boundaries if dispersed in the Stainless Steel matrix[10].

The addition of Boron depends not only on the density of the Stainless Steel but also on the
mechanical properties and its microstructure[1]. To improve densification, microhardness and other
mechanical properties, the amount of Boron addition plays an important role. In this article, 0.25% Boron
was added to the 316L Stainless Steel matrix to investigate the effect of its addition on the
compactibility, densification, sintering characteristics and microhardness at different compaction
pressures ranging from 100 MPa to 600 MPa.

2. Materials and methods
In this study, six samples from pure 316L Stainless Steel powder and another six samples from 0.25%
Boron added Stainless Steel powder were produced by Powder Metallurgy method. The 316L
Stainless Steel Powder used in the study was supplied by Zhe Jiang Bai Nian Yin Industry & Trade
Co. Ltd Su Xi, Yongkang, Zhejiang, China and was produced by high-pressure gas atomization
method. The chemical composition of 316L SS is given in Table 1. Nano size elemental Boron powder
used in this study was supplied by Xuzhou Jiechuang New Material Technology Co. Ltd. Hong Kong
and had particle size of 90 nm. The morphology of both the powders, observed by Scanning Electron
Microscope (SEM) is shown in Figure 1.
Table 1. Chemical composition of 316L Stainless Steel powder

| Element | Cr  | Ni  | Mo  | Si  | Mn  | C   | P   | Fe  |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|
| wt. %   | 17.04 | 12.01 | 2.4 | 0.9 | 1.5 | 0.028 | 0.045 | Bal |

Figure 1. SEM micrograph of (a) 316L Stainless Steel powder and (b) Boron powder

The samples were made in disk shape at different pressures ranging from 100 MPa to 600 MPa as shown in Figure 2. In order to produce samples from Boron added Stainless Steel, the Boron and Stainless Steel powders were mixed together using the Turbula mixer device at 40 rpm for two hours. The Green Compact samples were then produced by pressing the powder uniaxially in a hardened steel die to obtain 30 mm diameter with 10 mm thick disk shape samples. The green compact samples were then sintered in Nitrogen atmosphere at 1200°C for 1 hour as shown in Figure 3.

Figure 2. Green Compacts made from (a) pure 316L Stainless Steel (SS) powder and (b) Boron added Stainless Steel (BSS) powder
Figure 3. Sintered Samples in Nitrogen atmosphere at 1200°C

The density of the Green Compacts was measured by geometrical method and density of the sintered samples was measured by the water displacement method using Archimedes’ Principle according to Standard ASTM B962-17. Optical Microscope (OM) studies were carried out to observe the microstructure and morphology of the sintered samples. For this, all the samples were prepared by mounting them in Bakelite and then polishing the surfaces. The samples were then placed in the Glycergia etching solution for 15 seconds to reveal the microstructure. Hardness of the sintered samples was measured using Vickers Hardness Tester according to Standard ASTM E92-82.

3. Results and discussion

3.1. Green density

The density of the Green Compact samples was measured from geometrical dimensions, applied load, weight of the sample and area of the used die. It was found that the green density increased with increasing the compaction pressure for pure Stainless Steel powder as well as the boron added Stainless Steel powder. The increase in density with the increase in compaction pressure has been depicted in Figure 4.

Figure 4. Graph representing Green Density as a function of Compaction Pressure for pure Stainless Steel and Boron added Stainless Steel
From Figure 4, it can be seen that the green density of the compacted samples is increasing with increasing the compaction pressure. However, the green density of Boron added Stainless Steel shows variation as compared to the pure Stainless Steel. It can be seen that Boron addition in stainless steel samples reduced the green density at all compaction pressure. This can be attributed to the presence of Boron particles between the SS particles that provide hindrance during compaction and avoid particles to come close even at high pressures.

3.2. Sintered density

Figure 6 demonstrates the sintered density of both pure Stainless Steel and Boron added Stainless Steel samples sintered under Nitrogen atmosphere. It can be clearly seen that all 316L Stainless Steel samples exhibited significant increase in sintered density at all compaction pressures. Moreover, the samples added with Boron powder show a notable increase in the densification of the sintered samples beyond 200 MPa. The schematic diagram of Boron addition to the Stainless Steel particles and the densification mechanism has been shown in the figure 5 below.

![Figure 5. Schematic diagram of Boron diffusion and densification mechanism](image)

A maximum densification of 92.5 % was achieved for Boron added Stainless Steel sample at 600 MPa. It thus validates that Boron addition promotes densification along with increase in compaction pressure for 316L Stainless Steel. The behaviour of the boron addition in Stainless Steel shows that it facilitates the sinterability of the samples by formation of liquid phase that is produced by the eutectic reaction that takes place between Iron and Boron. This reaction takes place at a temperature higher than the temperature required for eutectic transformation. At this stage, a liquid phase appears which helps in improved sintering of the parts by reducing the number of pores. Boron tends to segregate by forming a layer on the grain boundaries which helps in rapid densification of the powder by providing high diffusivity rate. This helps in reducing the number of pores inside the surface supporting improved densification. The amount of Boron addition and the sintering temperature also have a high impact on densification and if these parameters are correctly chosen, nearly full dense parts can be produced.
3.3. **Densification parameter**

The compressibility of powder is an index of its densification behaviour under the application of an external pressure. The compressibility of powder compacts during compaction process is normally measured by green density which is the measure of its densification.

The fractional density of compacts can also be measured by Densification Parameter. It is an index of fractional densification achieved as a result of compaction as compared to theoretically maximum attainable densification. The Densification Parameter for all the samples is given in Table 2.

**Table 2.** Densification Parameter for pure Stainless Steel and Boron added Stainless Steel

| Powder Composition | Densification Parameter at different Compaction Pressures |
|--------------------|---------------------------------------------------------|
|                    | 100 MPa | 200 MPa | 300 MPa | 400 MPa | 500 MPa | 600 MPa |
| 316L SS            | 0.30     | 0.32     | 0.33     | 0.345   | 0.49    | 0.52    |
| Boron added 316L SS (BSS) | 0.31     | 0.38     | 0.47     | 0.49    | 0.62    | 0.66    |

3.4. **Microhardness**

The microhardness measurements of the sintered samples were evaluated using Vickers Hardness testing to determine the effect of the sintering atmosphere. Figure 7 shows the hardness values calculated for all the specimens sintered at different compaction pressures ranging from 100 MPa to 600 MPa. It was observed that microhardness increased with increasing Compaction Pressure. Boron addition also favoured the increase in microhardness and the samples sintered with Boron added Stainless Steel showed a notable increase in the microhardness. A maximum hardness of HV 195.25 was observed for the Boron added Stainless Steel sample.

The Nitrogen atmosphere was found suitable for sintering all the samples because it favoured the dissolution of Nitrogen into the samples during the sintering process which had an impact on the improved densification and enhanced microhardness. It has been reported that Nitrogen is a better
sintering atmosphere for austenitic Stainless Steels which helps in strengthening the grain size of the sintered samples[20]. Moreover, the addition of Boron helped in increasing the microhardness of the sintered samples. Boron has a tendency to form borides with Iron, Chromium and Nickel present in the Stainless Steel when dispersed in the matrix during the sintering process. These borides segregate themselves at the grain boundaries and are a source of increasing the strength of the resulting sample which can be distinguished in figure 7.

Figure 7. Microhardness of the sintered samples for pure Stainless Steel and Boron added Stainless Steel

3.5. Microstructure

The microstructures of the samples with and without the addition of Boron are shown in the figure 8 and 9. Figure 8 (a) to (f) shows the microstructure of the pure Stainless Steel samples compacted at different compaction pressures ranging from 100 MPa to 600 MPa. The figure 8 (a) shows that as the compaction pressure is too low (100 MPa), therefore, a number of empty spaces in the form of pores are present. As we increase the compaction pressure, the number of pores keep on decreasing and at compaction pressure of 600 MPa shown in figure 8 (f), there are very less pores left inside the sintered sample which represents good densification. A maximum densification of 89.5 % was achieved for the samples compacted at 600 MPa.
Figure 8. Microstructures of Pure Stainless Steel samples compacted from 100 MPa to 600 MPa

Figure 9. Microstructures of Boron added SS samples compacted from 100 MPa to 600 MPa

Figure 9 (a) to (f) show the microstructure of the Boron added Stainless Steel samples. It can be noted that with the addition of Boron, the number of pores start reducing and at 600 MPa compaction pressure, the Boron added Stainless Steel sample revealed 92.5 % densification. In the samples sintered with Boron added Stainless Steel, sufficient amount of liquid phase formed during sintering process which provided nearly full densification. The grains were well surrounded with eutectic phase indicating that high diffusivity layer was formed at the grain boundaries resulting in nearly full densification. The Boron also helped in smoothing the pore size from irregular to spherical shape. It thus concludes that the microstructure of the sintered samples has improved with the addition of Boron.

4. Conclusion
In this study, Stainless Steel was compacted at different Compaction Pressures ranging from 100 MPa to 600 MPa to study the effect of Compaction Pressure on the densification of the sintered Stainless Steel samples. It is concluded from the results that the densification increases as the Compaction Pressure is increased. A maximum densification of 89.5 % was achieved for the pure Stainless Steel samples compacted at 600 MPa. To improve the densification of the sintered samples, 0.25 % Boron was added to the Stainless Steel matrix. The study concluded that the Boron addition has a notable effect on the density of the sintered samples and a maximum of 92.5 % densification was achieved at Compaction Pressure of 600 MPa. Thus, we conclude that Boron aids in increasing the density of the sintered samples.
using Powder Injection Method and Powder Metallurgy methods have been reported[1, 7, 12, 16, 19] and supports the fundamentals of increase in densification of sintered samples. The effect of Compaction Pressure on the microhardness of the samples was also studied using Vickers Hardness testing which confirmed that increase in Compaction Pressure increases the microhardness of the sintered samples. A maximum of HV 183.76 was achieved for the pure Stainless Steel samples compacted at 600 MPa. The microhardness for Boron added Stainless Steel was further improved to HV 195.25 at Compaction Pressure of 600 MPa. The sintering atmosphere also has an impact on the overall properties of the sintered samples and Nitrogen was found well suitable for sintering the samples at 1200°C. It is believed that this atmosphere favours the dissolution of Nitrogen into the samples during sintering process. This in turn has a notable effect on the densification, microhardness and strength of the sintered samples in terms of strengthening grain size, grain boundaries, pore geometry and porosity ratio. It is thus concluded that Compaction Pressure as well as Boron addition enhances density and microhardness of the sintered samples. It is now recommended to increase the Compaction Pressure to 800 MPa along with Boron addition upto 1.5 wt%. This will further increase the densification and microhardness of the sintered samples and can get nearly 98–99 % dense parts with improved strength.

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