Sintering Behavior of Elemental Powders with FeB Addition in the Composition of Martensitic Stainless Steel

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The effect of sintering additive for the development of high-strength martensitic stainless steel from elemental powders was studied. The sintering parameters investigated were: sintering temperature, sintering time, and wt.% of FeB. In vacuum sintering, effective sintering took place between 1300 and 1350 °C with 1-1.5 wt.% FeB addition. The maximum sintered density and ultimate tensile strength (UTS) were achieved after sintering at 1350°C for 60 min with 1 wt.% FeB. Secondary pores were observed in samples containing more than 1.5 wt.% FeB sintered at 1350 °C for 60 min. More than 1.5 wt.% FeB content and temperature above 1350°C caused slumping of the specimens. Maximum UTS of 505 MPa was achieved with 1 wt.% FeB content. Above 0.5 wt.% FeB content, maximum increase in density was observed. Fracture morphologies of the sintered samples are reported.

Keywords	FeB, 465 martensitic stainless steel, sintering, ultimate
	tensile strength (UTS)

1. Introduction

Liquid phase sintering process is used for obtaining higher densities for P/M steels. Sintering aids are one of the effective methods to get higher sintered densities by lowering the sintering temperature and reducing sintering time. For liquid phase sintering, different sintering aids can be added to lower the sintering temperature and to reduce sintering time to get higher density (Ref 1, 2). These sintering aids form liquid phase. In two-phase systems involving mixed powders, liquid formation is possible because of different melting ranges for the components. This liquid phase provides a high-diffusivity pathway that causes densification (Ref 3).

Custom 465 stainless steel (Custom 465 is a registered trademark of Carpenter Technology Corporation) is a premium melted, martensitic, and age hardenable alloy. Due to its high strength, excellent notch tensile strength, and fracture toughness, 465 stainless steel has wide spread applications especially in automotive and aerospace industry (Ref 4). The present study is aimed at investigating the effect of using FeB on the sintering temperature, sintering time, and the mechanical properties of elemental powders in the composition of 465 stainless steel.

2. Experimental Procedures

Elemental powders in the composition of 465 stainless steel were blended in "V" shell blender for 4 h. The composition of prepared 465 stainless steel is given in Table 1. The 465 Stainless steel powders were prepared with 0.25, 0.5, 1.0, 1.5, 2, and 3 wt.% FeB content. The blended powders were poured into a tensile specimen die and uniaxially pressed at a pressure of 600 MPa. Sintering of all prepared green samples was carried out in a high-temperature vacuum furnace at a vacuum level of $1 * 10^{-2}$ Pa. The sintering was carried out in the temperature range of 1250-1400 °C and the samples were held for 20, 40, 60, and 80 min. The sintering cycle applied to the samples was: samples were heated to 1000 °C at a rate of 10 °C/min and held at 1000 °C for 10 min, then the samples were heated to various sintering temperatures 1250, 1300, 1350, and 1400 °C at a rate of 5 °C/ min (total heat-up times of 160-190 min depending on sintering temperature) and samples were held for 20-80 min at the sintering temperature. The densities of the sintered samples were measured by Archimedes water immersion method. For metallographic examination, the samples were cut and metallographically prepared to 0.5 µm diamond finish. The microstructure of the sintered samples was studied by scanning electron microscope (SEM) Kevex Leo-1450. Sintered samples were subjected to tensile test. All tensile tests were performed using an Instron mechanical tester at a constant crosshead speed of 0.5 mm/min on 25 mm gauge length.

3. Results and Discussion

The 465 stainless steel samples were prepared by conventional powder metallurgy. The measured density of the green compact was approximately 6.62 g/cm³. Prepared samples of 465 stainless steel powders were sintered at 1400 °C for 1 h, a maximum sintered density of 6.89 g/cm³ was achieved. A set of sintering experiments were conducted in the temperature range from 1250 to 1400 °C for 20-80 min to study the effect

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of FeB addition on the final sintered density and results are shown in Fig. 1(a). The 465 stainless steel samples without FeB additions have produced a maximum sintered density of 6.89 g/cm^3 . At higher FeB contents such as 1 wt.% sintered density has increased to 7.4 g/cm³ as shown in Fig. 1(a). FeB activates the sintering process of the iron powders by the formation of liquid phase. The liquid phase has low solubility in the iron and remains to favor the phenomenon of liquid phase sintering. If the amount of FeB and the sintering temperature are correctly chosen, density 7.4 g/cm³ (94% of the full density) may be obtained. Note: Carpenter Technology lists a density of 7.84 g/cm³ for 465 alloy in the H1000 condition.

The effect of sintering temperature and sintering time on the sintered density with FeB addition is shown in Fig. 1.

 Table 1
 Particle size of elemental powders and composition of 465 stainless steel used in current study

Element	Wt.% 0.25	Particle size (μm) >10
Aluminum		
Silicon (Max.)	0.25	>37
Nickel	11.00	>74
Titanium	1.80	>37
Chromium	12.00	>30
Molvbdenum	1.10	>37
Iron	Balance	>74
FeB	0-3	>37
100	0.5	251

Figure 1(b) shows that at a sintering temperature of 1350°C, all samples, after sintering for 60 min with 1 and 1.5 wt.% FeB addition showed a maximum sintered density of 7.4 g/cm³. The liquid phase attacked particles contact and pore regions with capillary forces to cause densification (Ref 5). FeB content greater than 1.5 wt.% showed a decrease in sintered density when sintered at 1350 °C for 60 min. This decrease in density can be attributed to the formation of secondary pores inside the grains. More than 1.5 wt.% FeB content and temperature above 1350 °C caused slumping of the samples. Sintered density was increased with the sintering temperature such as 1250, 1300, and 1350 °C and FeB addition. The results of the effect of sintering time and FeB content at 1350 °C on the resultant sintered density are shown in Fig. 1(c). The samples containing 1 wt.% FeB reached to a maximum sintered density of 7.4 g/ cm³ after sintering for 60 min, however higher sintering time did not improve sintered density. The maximum ultimate tensile strength (UTS) of 358 MPa was reached with 465 stainless steel after sintering without FeB addition. The maximum UTS of 505 MPa was reached with samples containing 1 wt.% FeB sintered at 1350 °C for 60 min. The variation of UTS with FeB content is given in Fig. 1(d). It can be seen from Fig. 1(b) that samples sintered at 1300 °C show 7.4 g/cm3 with 1 and 1.5 wt.% FeB addition and the corresponding values of the UTS are 487 and 495 MPa, respectively (Fig. 1(d)). The corresponding values of the UTSs for 1 and 1.5 wt.% FeB at 1300 °C are lower than the maximum UTS of 505 MPa



Fig. 1 (a) Effect of FeB addition on the sintered density; (b) effect of sintering temperature on the sintered density (sintering time 60 min) with 0.25-1.5 FeB addition; (c) effect of sintering time on sintered density with 0.25-1.5 FeB addition at sintering temperature of 1350 $^{\circ}$ C; and (d) effect of FeB addition and sintering temperature on the ultimate tensile strength (UTS)

achieved with 1 wt.% FeB samples sintered at 1350 °C for 60 min. This may be because of the modification of residual pore structure at higher sintering temperature of 1350 °C.

Figure 2 shows the microstructure of the samples with and without FeB addition. Figure 2(a) shows the microstructure of additive free samples that was sintered at 1400°C for 60 min. The microstructure exhibits sintered particles and pores inside. Figure 2(b), (c) and (d) shows the microstructures of 465 stainless steel samples sintered at 1350 °C for 60 min with 0.5, 1, and 3 wt.% FeB addition, respectively. Sufficient amount of liquid phase was formed with 1 wt.% FeB addition to achieve higher sintered density of 7.4 g/cm³. It is well known that in liquid phase sintering process rearrangement owing to capillary forces entered by a wetting liquid (Ref 6-8). The specimen containing 3 wt.% FeB addition as in Fig. 2(d) shows the secondary pores inside the grains that are responsible for the decrease in the sintered density.

The morphologies of fracture surface of the addition free 465 stainless steel after sintering at 1350 °C is given in Fig. 3(a). It shows the fracture surface of sample sintered without FeB addition; there are a number of pores between the particles, although the particles have fused together. The morphologies of fracture surface of 465 stainless steel with 1 wt.% FeB sintered at 1350 °C for 60 min are given in Fig. 3(b). It can be seen that samples show dimpled rupture and porosities. The dimpled rupture corresponds to ductile fracture. The variation in elongation with the amount of sintering additive (FeB) in 465 martensitic stainless steel is given in Fig. 4.

4. Conclusions

The 465 stainless steel made from elemental powders were produced through conventional press and sinter. The effect of

sintering time and temperature and the addition of 0-3 wt.% FeB on density and UTS were investigated for 465 stainless steel compacts produced using elemental powders. It was found



Fig. 3 (a) Fracture morphologies of sample sintered at 1350 °C for 60 min with 0 wt.% FeB and (b) fracture morphologies of sample sintered at 1350 °C for 60 min with 1 wt.% FeB addition



Fig. 2 Sintering mechanism of 465 stainless steel (a) 465 stainless steel without FeB addition sintered at 1400 °C for 60 min; (b) with 0.5 wt.% FeB addition sintered at 1350 °C for 60 min; (c) with 1.0 wt.% FeB addition sintered at 1350 °C for 60 min; and (d) with 3 wt.% FeB addition sintered at 1350 °C for 60 min



Fig. 4 Variation of % elongation with the addition of FeB as a sintering additive

that FeB content higher than 0.5 wt.% enhanced the sintering process and densification of 465 stainless steel. A maximum sintered density of 7.4 g/cm³ and UTS of 505 MPa was achieved with 1 wt.% FeB, when sintered at 1350 °C for 60 min. Secondary pores were observed in the microstructure

of the samples containing more than 1.5 wt.% FeB addition sintered at 1350 °C, which were responsible for the decrease in the sintered density for higher amounts of FeB addition.

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