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Supersolidus Sintering of Boron Doped Stainless Steel Powder Compacts

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Abstract

Supersolidus liquid phase sintering was used to attain a high density in austenitic 316L stainless steel. The effect of boron additions and sintering temperatures on the densification and mechanical properties has been investigated. Higher densification was achieved with compacts with higher boron content. Ultimate tensile strength upwards of 450 MPa was obtained for 316L powders with 0.8 wt.% boron sintered at 1225°C for 30 minutes. The amount of boron and sintering temperature needs to be controlled in order to achieve full densification without distortion and optimal mechanical properties.

Introduction

The formation of liquid phase during sintering is a common technique to enhance densification. Coarse alloy powders can be processed to full density by a process named supersolidus liquid phase sintering (SLPS) [1, 2]. In this process, the alloy powder is heated between the solidus and the liquidus temperature leading to the formation of a liquid phase inside the particles. The liquid spreads to the particle boundaries resulting in a capillary force acting on the semisolid particles. Once sufficient liquid forms to fragment the particles into individual grains, rapid densification ensues due to capillary induced rearrangement [1] and enhanced diffusion in liquid phase. Figure 1 shows the densification process that occurs when a polycrystalline prealloyed powder (a) is heated to a temperature between the solidus and liquidus. Upon heating, liquid forms at the interparticle neck, along grain boundaries inside the particles, and at the pockets located inside the grains (seen in Figure 1b). The liquid located at the interparticle necks provides the capillary force for densification, while the liquid located on the grain boundaries lubricates grain sliding during densification (seen in Figure 1c). Hence the location of liquid formation is critical to densification process. The optimal liquid volume fraction for supersolidus liquid phase sintering varies from 5 to 65 vol. % based on the material system and the initial powder microstructure [2]. Densification in the presence of a liquid phase is analogous to viscous phase sintering [3]. Liquid films at the grain boundaries soften the particles allowing densification in response to the capillary forces at the particle contacts. However, the liquid films also reduce the structural rigidity of the compact to a point where it slumps under its own weight. Thus, the condition necessary for full densification is often close to that resulting in shape loss. Hence temperature and microstructure control are paramount in obtaining desired final properties.

SLPS has been widely applied to processing of ferrous alloys [4-15]. In this study supersolidus sintering of boron doped stainless is investigated in regards to densification and mechanical properties. Addition of boron lowers the solidus allowing a wider temperature range for processing.

Experimental Procedures

In this research water atomized austenitic stainless steel 316L powder was used. The characteristics of the 316L powder are listed in Table 1. As shown in Figure 2, the powder has an irregular shape which is characteristic of powders produced by water atomization. The specimens were prepared by mixing the steel powders with elemental boron of -325 mesh size in Turbula mixer for 30 minutes. The different weight percentages of boron added were 0.2, 0.4 and 0.8. The solidus and liquidus temperatures were identified by performing differential thermal analysis on the admixed powders.

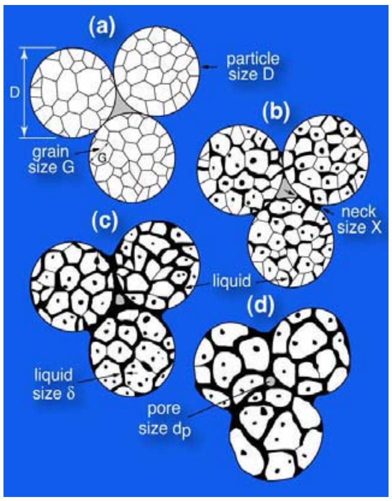


Figure1: Schematic of supersolidus liquid phase sintering stages

Dilatometry was used to observe the *in situ* shrinkage, shrinkage rate, and melt formation during sintering. Dilatometry experiments were conducted on hand pressed samples of 66 ±1 % green density at a constant heating rate of 5°C/min. The experiments were performed using a vertical push rod dilatometer in hydrogen. The temperature inside the dilatometer (model: 1161V, Anter, Pittsburgh, PA, USA) was accurate to ± 2°C.

Mechanical properties were measured using standard flat tensile bars of 6.45 cm^2 projected area pressed using a uniaxial hydraulic press to 61±2% green density. Sintering was done at 1200, 1225, 1250 and 1275°C in a CM horizontal tube furnace (CM Furnaces, Bloomfield, NJ) in dry hydrogen with dew point below –40°C. The hydrogen flow rate of 2000 cm^3/min was maintained in the furnace throughout the sintering cycle. The compacts were heated at 5°C/min to the sinter temperature and were sintered at the peak temperature for 30 minutes. Tensile tests were performed at a constant cross head speed of 2.54 mm/min. The sintered density was measured using the Archimedes water immersion method. Fracture surfaces were observed in a scanning electron microscope.

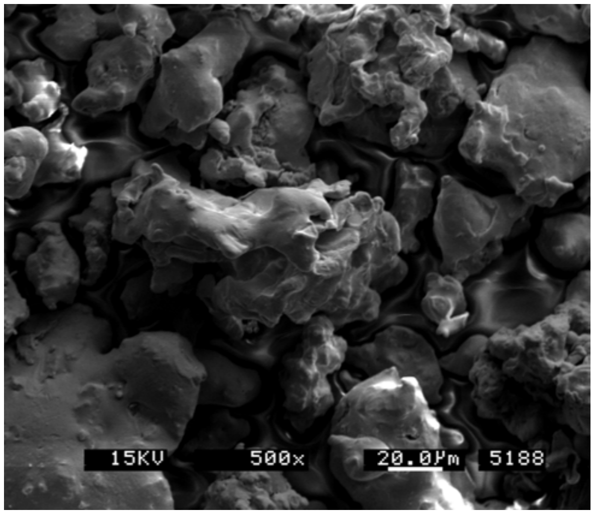


Figure 2: Scanning electron micrograph of water atomized 316L stainless steel powder

Table 1: Powder characteristics of water atomized stainless steel powder

Composition	SS 316L water atomized (Fe, 16-18 Cr, 10-14 Ni, 2 Mn, 1Si, 2-3 Mo)
Designation	Ancor 316L (Hoeganaes)
Apparent density, g/cm^3	3.11
Tap density, g/cm^3	3.60
Pycnometer density, g/cm^3	7.87
Hall flow rate	NFF
Size Distribution, µm D_{10} D_{50} D_{90}	 22 49 93

Results and discussions

Densification, shrinkage, and shrinkage rate were monitored by performing dilatometry on compacts heated at 5^{o}C/min to a different temperature depending on the amount of boron added. A plot of shrinkage versus temperature for different amounts of boron additives is shown in Figure 3. The amount of shrinkage at a given temperature is higher for a compact with higher boron content. Liquid formation temperature was identified using DTA analysis and is shown in Table 2. Even with 0.2 wt. % boron the liquidus is lowered substantially from 1441 to 1233 ^{o}C and the working range for sintering is wider than the base alloy. Figure 4 shows the shrinkage rate versus temperature for different amounts of boron content. The peak shrinkage rate indicates the maximum sintering activity. The temperature of maximum activity is close to liquidus and is similar for different amounts of boron whereas no activity occurs in the undoped compact due to its higher solidus temperature.

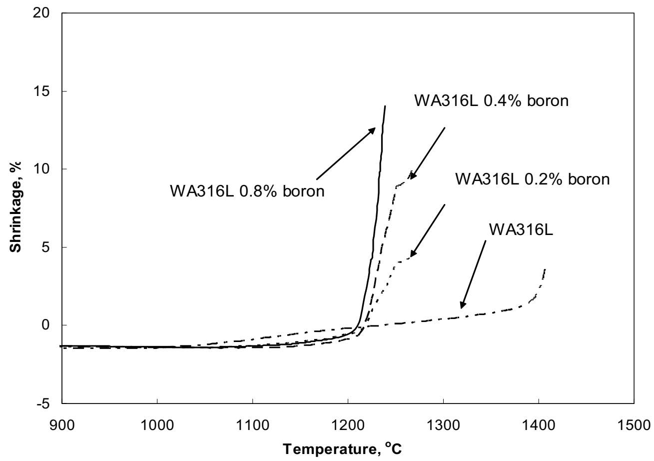


Figure 3: Shrinkage of water atomized 316L with different amounts of boron plotted as a function of temperature.

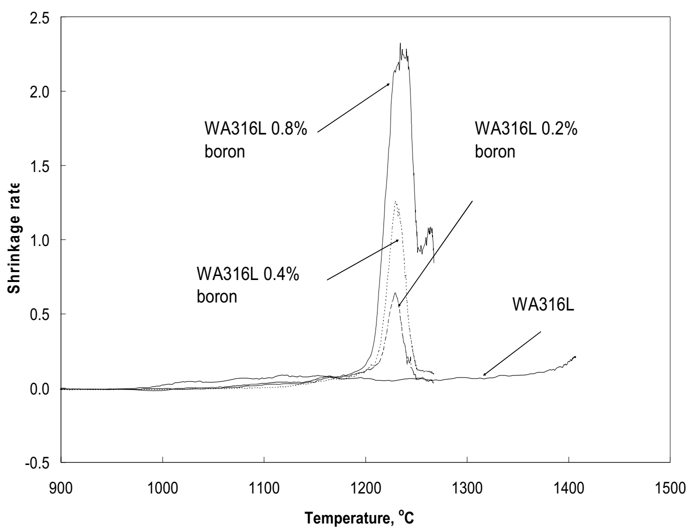


Figure 4: Shrinkage rate of water atomized 316L powder with different amounts of boron plotted as a function of temperature.

Table 2: DTA results showing the solidus and liquidus temperatures for water atomized 316L powder with different amounts of boron additive

Composition	Onset of liquid formation (Solidus ^{o}C)	Liquidus (^{o}C)	Melting range
WA 316L	1441	1459	18
WA 316L +0.2 Boron	1233	1445	212
WA 316L +0.4 Boron	1230	1427	197
WA 316L + 0.8 Boron	1224	1393	169

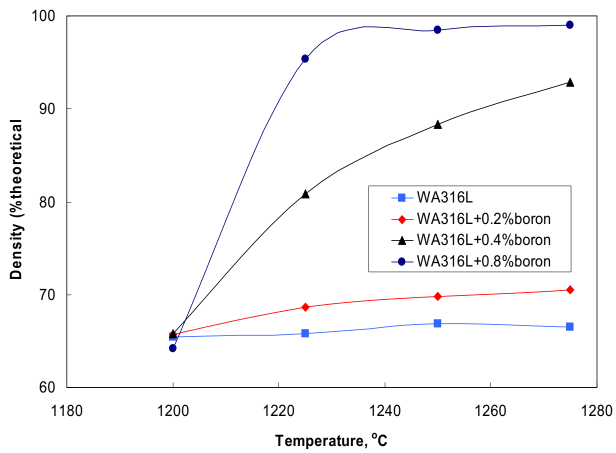


Figure 5: Sintered density of water atomized 316L powder with different amounts of boron sintered at 5°C/min.

The effect of temperature and boron on the sintered density is shown in Figure 5. The sintered density increases with increases in amount of boron additive and sintering temperature. With an increase in boron content in the alloy and sintering temperature, the amount of liquid phase formed increases. Hence, compacts with 0.8 wt. % boron and sintered to 1275°C show the highest density. It is noted that although the compacts achieve full density they also distorted. Hence, there exists a narrow sinter window in which one needs to operate to achieve full density without distortion.

The mechanical properties of boron doped stainless steel are not only dependent on final sintered density but also the microstructure. Boron additives are useful in enhancing the densification at relatively low temperatures and in increasing strength, ductility and hardness of the sintered material. The amount of boron additive has to be optimized to give a desirable microstructure and final mechanical properties. At a sintering temperature of 1200°C the ultimate tensile strength is not significantly enhanced but with increase in sintering temperature to 1225°C the increase in ultimate tensile strength is clear as seen in Figure 6. For compacts with 0.8 wt. % boron, excessive liquid forms as sintering temperature increases and this liquid solidifies at the grain boundaries during cooling. This solidified liquid phase, being brittle provides an easy path for crack propagation as can be seen from the fracture surfaces shown in Figure 7. The fracture surfaces indicate that at 1200°C (Figure 8a) the fracture propagates completely through the pores. With increase in temperature to 1225°C (Figure 8b) the fracture mode changes from predominantly ductile indicated by the dimples to brittle type at 1250°C (Figure 8c) due to the brittle phase at the grain boundaries. Hence the optimum sintering temperature for 0.8 wt. % samples is 1225°C. The yield strength plotted as function of temperature for different amounts of boron additive is shown in Figure 8. The yield strength increases with an increase in sintering temperature and boron content in the alloy. For given boron content the yield strength will reach a maximum at a critical temperature beyond which point the brittle failure occurs. Future work will try to correlate the microstructural parameters such as liquid film thickness, grain size, contiguity and connectivity to mechanical properties.

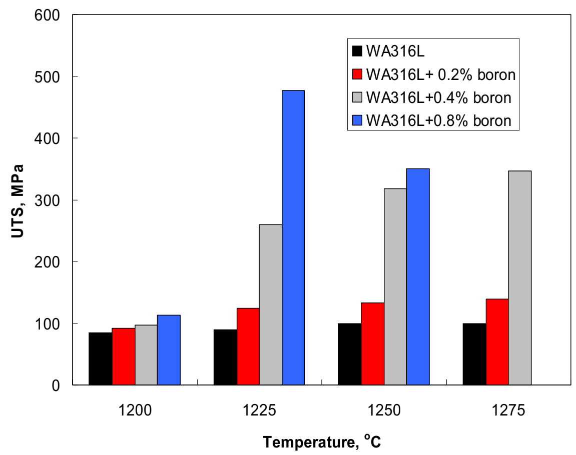


Figure 6: Ultimate tensile strength of boron doped stainless steel alloys at different sintering temperatures.

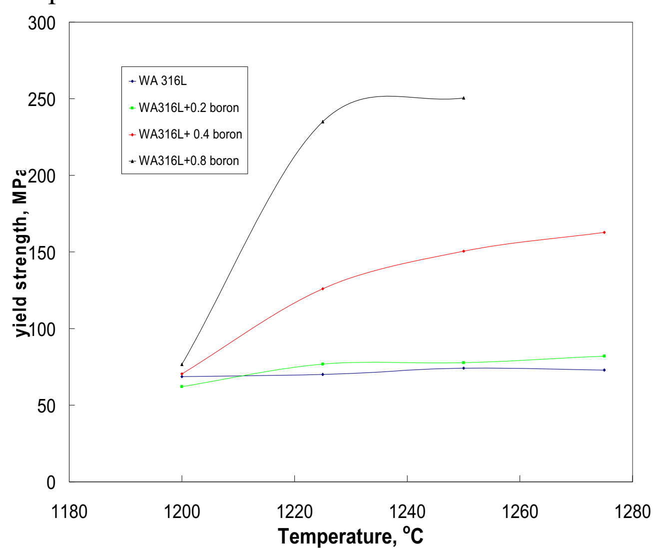
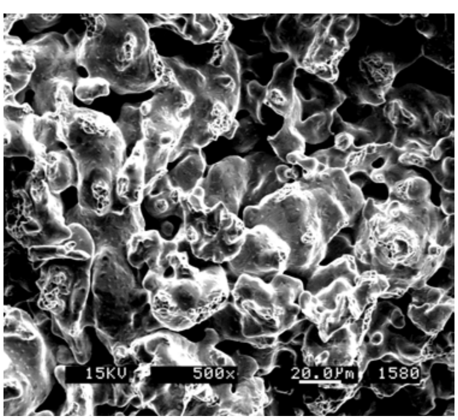
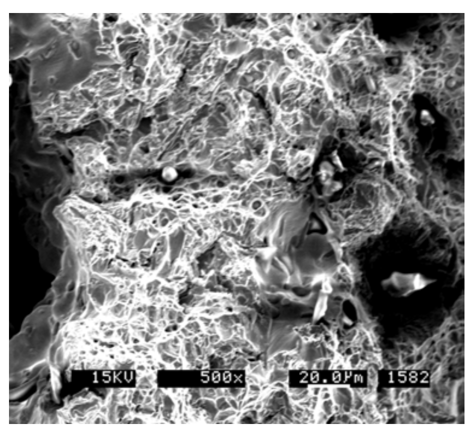


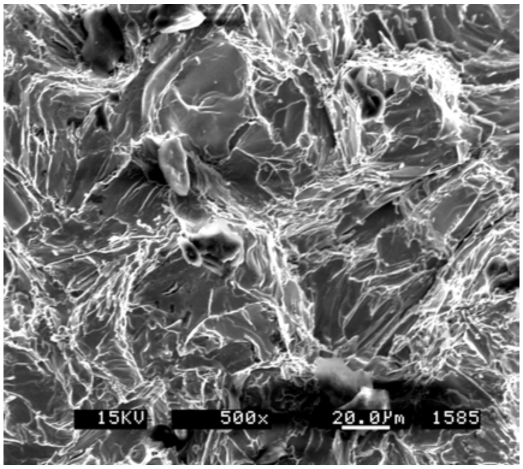
Figure 8: Yield strength of boron doped stainless steel 316L alloys sintered at different temperatures.



(a)



(b)



(c)

Figure 7: Fracture surfaces of water atomized 316L with 0.8 wt. % boron additive sintered at a) 1200^{o}C b) 1225^{o}C c) 1250^{o}C.

Conclusions

It has been shown that adding boron to steel powders lowers the solidus and hence enhances the densification. Ultimate tensile strength of over 450 MPa can be achieved for alloys with 0.8 wt. % boron sintered at 1225 ^{o}C for 30 minutes. The fracture mode changes from ductile to brittle as sintering temperature increases for alloys with 0.8 wt.% boron due to the interparticle brittle boride phase formation. The amount of boron and sintering temperature need to be optimized for desired microstructure and mechanical properties.

Acknowledgements

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