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Sintering

In Situ Evaluation of Viscosity during Sintering of Boron Doped Stainless Steel using Bending Beam Technique

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Abstract

Beam bending analysis is used to evaluate the viscosity of boron doped water atomized 316L powder compacts during sintering process. The viscosity of the beam decreases with an increase in sinter temperature. Upon liquid formation the compact becomes soft. The effective viscosity of water atomized 316L with 0.2 wt. % boron at the onset of densification is ~250 MPa.s.

Introduction

Powder metallurgy (P/M) allows fabrication of high quality, complex parts to close tolerances in an economical manner. Monitoring of the sintering process can be done by tracking bulk properties such as density; microstructural parameters such as neck size; mechanical properties such as hardness or other parameters such as X-ray absorption [1]. Despite these wide varieties of techniques, the *in situ* measurement techniques prove most valuable.

Supersolidus liquid phase sintering (SLPS) is a process for full densification of coarse alloy powders. Prealloyed powders are heated to a temperature between the solidus and the liquidus. Upon heating, liquid forms along the grain boundaries within the particles, between the particles and inside the particles. The liquid spreads and rearrangement of the grains due to capillary forces causes densification [2, 3, 4]. The amount of liquid formed depends on the system and the temperature. The liquid content also dictates the dimensional stability of the sinter compact. Viscosity of the semisolid compact is an important factor that determines the densification and distortion rates. Hence, it is necessary to understand how viscosity evolves during sintering and correlate it to the microstructure.

Viscosities of ceramic materials undergoing densification are typically measured by applying compressive or tensile stress to the specimen, which causes creep deformation and densification. Loading dilatometry has been used to measure the viscosity during sintering of certain ceramics [5, 6, 7]. Another technique is to measure viscosities with the bending beam technique. Hagy [8] reported a beam-bending method similar to measure viscosity of glasses between 10^7 to 10^{14} Pa.s. The viscosity was calculated from the rate of deflection of a simple beam using a sapphire loading rod. This method has been adopted as an ASTM standard to measure glass viscosity between the softening point (~ 10^6 Pa.s) and annealing points (~ 10^{12} Pa.s) [9]. Bending beam analysis has been performed on ceramics and ferrous alloys to calculate the effective viscosity and the results were consistent with the data obtained using dilatometry [10, 11, 12].

In this paper bending beam analysis is performed on boron doped stainless steel 316L powder compacts to calculate the viscosity during constant heating rate sintering experiments.

Experimental Procedures

Water atomized stainless steel 316L powder mixed with 0.2 wt. % boron was selected for these experiments. The particle size information for 316L steel powder is provided in Table 1. The boron powder was of 99.9% purity and -325 mesh size. The water atomized 316L and boron powders were mixed in Turbula for 30 minutes to obtain a homogenous blend. The mixed powders were pressed in a hydraulic press to a green density of 58±2 %. The green beams were presintered to 500^{o}C for 1h to impart some holding strength. The dimensions of the presintered beams were 10.45 mm in width, 57.60 mm in length and 1±.05 mm in thickness.

The bending beam geometry during the tests is shown in Figure 1. The span length was 35 mm and the supports were fully dense alumina and resting in an alumina boat to contain any melting or contamination. The setup was placed in a horizontal tube furnace with a quartz window to observe bending. The bending experiments were conducted at 5 K/min to a maximum temperature of 1450 ^{o}C. The *in situ* bending was observed using the system shown in Figure 2. The sample was illuminated with the strobe light while a camera takes images at times synchronized with the strobe flash.

Viscosity Analysis

Bending beam analysis allowed the calculation of the viscosity of the solid-liquid mixture during sintering. In the case of uniform loading of elastic beams, the general deflection equation is expressed as [13]

$$\frac{d^2\delta/dx^2}{\left[1+(d\delta/dx)^2\right]^{3/2}} = \frac{M}{EI} \qquad (1)$$

where M is the bending moment, I is the moment of inertia, E is elastic modulus at temperature, δ is the deflection of the beam, and x is the distance from the left support (see Figure 1). For a beam of rectangular cross section bending under its own weight

$$M = \frac{qlx}{2} - \frac{qx^2}{2} \qquad (2)$$

$$I = \frac{bh^3}{12} \qquad (3)$$

Where l is the span length, q= ρgbh is the distributed load due to the beam's own weight, g the gravitational constant, and ρ, b, and h are the density, width, and thickness of the specimen, respectively.

For small deflections, i.e. dδ/dx < 0.15, the second order term $(d\delta/dx)^2$ can be neglected and Equation 1 can be expressed as

$$\frac{d^2\delta}{dx^2} = \frac{M}{EI} \qquad (4)$$

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

Table 1: Powder characteristics of water atomized stainless steel 316L

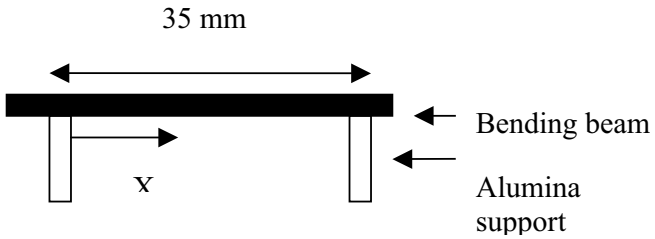


Figure 1: Simplistic view of the bending beam on supports

Solving Equation 4 with appropriate boundary conditions [10] gives the maximum deflection occurring at the middle of the span as

$$\delta_{\max} = \frac{5\rho g L^4}{32 E h^2} \qquad (5)$$

In sintering experiments, materials undergoing densification and distortion demonstrate linear viscous rather than linear elastic behavior. Using the analogy between these deformation modes, one simply replaces E by uniaxial viscosity, η, and deflection, δ by the deflection rate, $\dot{\delta}$.

Assuming pure Newtonian viscous flow allows Equation 5 to be rearranged to give the analogous viscous form; *in situ* viscosity is calculated as

$$\eta = \frac{5\rho g L^4}{32 \dot{\delta} h^2} \qquad (6)$$

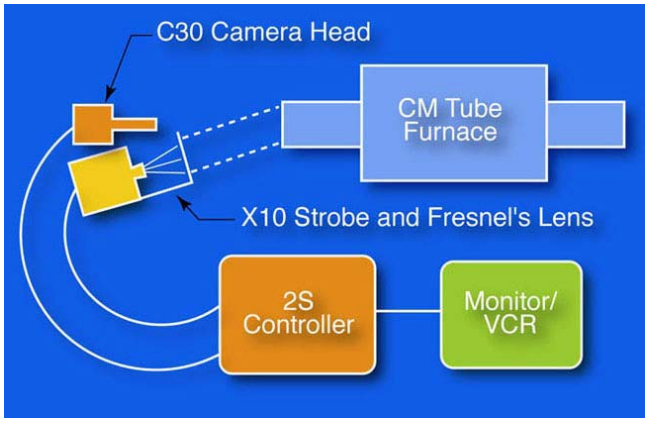
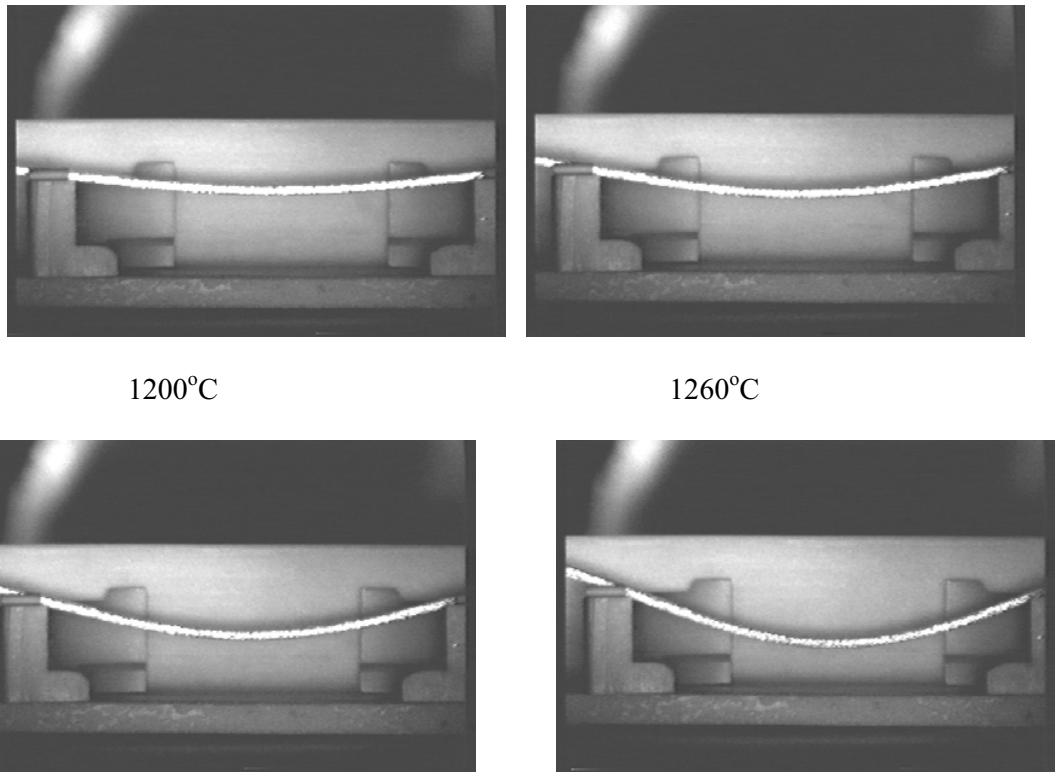


Figure 2: *In situ* video imaging using Synchrotron system

Results

The constant heating rate experiments were repeated 4 times to check the repeatability of the bending experiments. The results were repeatable and the average of the 4 bending curves was considered for further analysis. Figure 3 shows the *in situ* images of the bending beam during sintering in one such experiment. Figure 3 shows the bending of the beam increases with temperature during sintering. This is due to several factors such as increasing liquid content with temperature, grain growth, and decreasing viscosity. The deflection of the beam was calculated using image analysis software. The mid point deflection of the beam as a function of temperature is shown in Figure 4 for four replica runs. The plot indicates that the bending increases with temperature, but there is a ± 25% variation in bending between runs.



1200°C 1260°C

1320°C 1380°C

Figure 3: *In situ* bending pictures of 316L with 0.2% boron showing beam deflection at different temperatures along the sinter cycle.

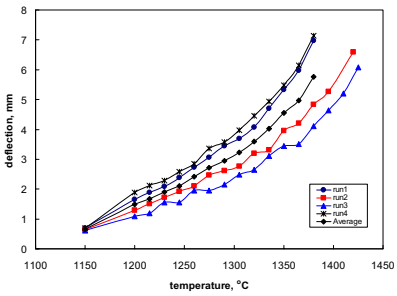
Discussion

From the knowledge of the heating rate, the deflection can be plotted as a function of time during sintering. Curve fitting the data gives the deflection as a function of time. Deflection rate is calculated as a function of time by taking the time derivative of the deflection curve. Equations 7 and 8 show the regression analysis expressions for average deflection and deflection rate with respect to time.

$$\delta = 1.228 + 0.0021 \times t^2 \quad \text{mm/min} \qquad (7)$$

$$d\delta/dt = 7 \times 10^{-6} t \quad \text{cm/s} \qquad (8)$$

t= 0 in the above equations corresponds to 1150^{o}C. Dilatometer runs were performed at 5 K/min to sinter temperature to determine shrinkage as a function of temperature. Using the continuity equation along with shrinkage data, the changes in density and thickness were calculated as a function of temperature. The deflection rate, density and thickness changes were input into Equation 6 along with the gravitational constant and span length. The apparent viscosity was calculated as a function of temperature and is shown in Figure 6. The magnitude of viscosity values obtained in this study ~ 0.2-0.6 GPa.s are lower than those reported by with the results from Lame et al. [12] who reported effective viscosity values of 60-80 GPa.s during bending experiments on steel powders.



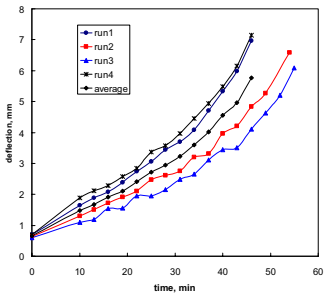


Figure 4: Mid point deflection versus temperature during sintering of stainless steel 316L with 0.2% boron during heating at 5K/min.
Figure 5: Mid point deflection versus time during sintering of stainless steel 316L with 0.2% boron during heating at 5K/min.

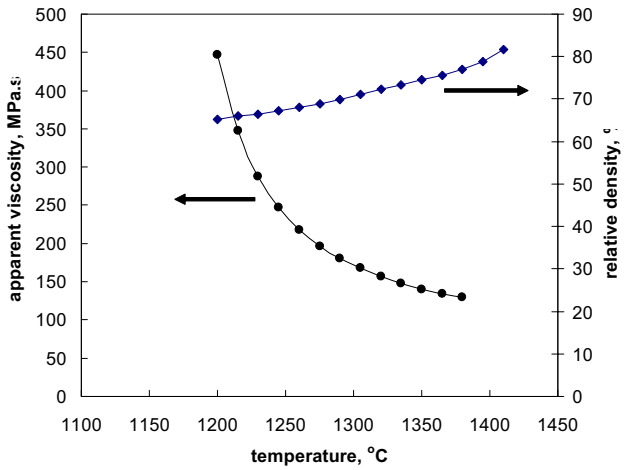


Figure 6: Apparent viscosity and relative density as a function of temperature during sintering at 5K/min.

Conclusions

Bending beam method offers a unique way of determining viscosity of material during sintering. The technique was successfully demonstrated with boron doped stainless steel 316L powder compacts. The results give an effective viscosity of ~250 MPa.s at the onset of densification and decreases as the compact densifies. These are in reasonable agreement with prior studies on other steel systems. Further experiments are underway to investigate the influence of factors such as liquid content, liquid film thickness and grain growth on the evolution of viscosity and to correlate the microstructure to viscosity evolution.

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