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Original Article

Effect of heating rate on sintered series 300 stainless steel

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Abstract

Stainless steel powders (303L, 304L, 310L and 316L) were formed into tensile test bars using the "press and sinter" process. Most processing parameters, except heating rate, were kept constant. During the heating of the experimental specimens from 700°C to the sintering temperature of 1300°C, heating rates were varied, e.g., 2.5, 5.0, and 10.0°C/min. Experimental results showed that a material heated with a low heating rate tended to have higher sintered density and tensile strength. However, the low heating rate caused grain growth in the sintered material. These results are in contradiction with the improved densification of some ceramics by ultra rapid heating. The reasons for contradiction are as follows. First, the heating rates employed in this work are not very different. The second is attributed to small thermal gradients generated in the thin metal powder compacts. Because of these reasons, densification of the sintered stainless steels series 300 is controlled by an isothermal condition. The low heating rate allows longer time for atomic diffusion, which is an important sintering factor. This means more atoms move to points of contact between powder particles to form necking and to cause necking growth. This results in better sintering. However, the low heating rate means that the materials are exposed to heat for longer time and thus their grains have a tendency to grow.

Keywords: heating rate, sintering, series 300, stainless steel

1. Introduction

In a normal sintering practice using a batch furnace, a powder compact is heated from room temperature to a debinding temperature, which is held for a certain period. After debinding the temperature is subsequently increased to a sintering temperature, which is held for a certain period before the sintered material is cooled down to room temperature. Usually, one sintering cycle (from powder compact loading to the sintered material discharging) takes some time. Sintering in a batch furnace does not only take time but also comes with considerable costs. Thus the effort to shorten the time of a sintering cycle is concerned here. When the whole sintering cycle is considered, shortening the time for heating of the debinded compact (from a specified temperature to the sintering one) is technically possible.

Heating rate is one of many factors, such as powder mixed composition, average particle size, green density, maximum temperature, holding time, atmosphere, support substrate and final density, concerned by powder metallurgists. However, there have been a few studies of the effect of heating rate on densification and properties of the sintered metal alloys. With rapid heating, improved densification of some ceramics is reported (Bumgartner, 1988; Landin and Schulze, 1990). Ultra rapid heating (Young and McPherson, 1989) leads to thermal gradients in a sintering compact. The gradient can create larger diffusional fluxes, especially if the compact is porous. Thus, by subjecting a material to rapid heat input, increase of sintering rate is possible. The driving force for sintering is sensitive to the thermal gradients

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induced by fast heating (Beruto *et al.*, 1989).

However, rapid heating can damage a compact. Along with the thermal gradient is a stress gradient. One consequence is that corners heat faster than flat faces, leading to more stress near the corners and the propensity for cracks occurring near the corners in the sintered product (Dorri *et al.*, 1987). Macro-defect generation, which is minimized with slow heating (Ohji and DeJonghe, 1994), may arise when fast heating is applied.

Because of the importance of the heating rate as mentioned above, this investigation aims to explore new heating schemes. With varied heating rates, densification and property of sintered stainless steels series 300 are expected to change. Densification of sintered materials is indicated by the sintered density. Tensile strength and elongation are representatives of the sintered material properties.

2. Experimental Procedure

The water-atomized 303L, 304L, 310L, and 316L stainless steel powders were obtained from Coldstream, Belgium. Composition of the powders is shown in Table 1. Each powder was compacted into tensile test bars (TTBs), following ASTM E8-04, with green densities of 6.62, 6.52, 6.50, and 6.49 ± 0.05 g/cm^3 for 303L, 304L, 310L, and 316L powders, respectively. The green TTBs were delubricated at 600°C for 60 minutes under argon atmosphere and then sintered at 1300°C for 45 minutes under 100% H_2 atmosphere. Heating rates, between 700-1300°C, were varied from 2.5 to 10°C/min. Densities of green and sintered samples were determined using the Archimedes method. A universal testing machine (Instron model 8801) was employed to measure the mechanical properties of the sintered TTBs. Hardness of the sintered TTBs was carried out using a hardness tester (Rockwell scale B). Microstructures were observed and grain size was measured using Abrams Three-Circle procedure (ASTM E112-96).

3. Results and Discussion

3.1 Sintered density

Sintered density of the sintered stainless steels series 300 decreased with increasing heating rate (Figure 1(a)). Generally, sintered density is directly related to densification level. The results presented in Figure 1 indicate that with a low heating rate densification of stainless steels series 300 tends to improve. These experimental results are in contradiction to some reports. Some sintered ceramics showed improved densification with rapid heating (Bumgartner, 1988; Landin and Schulze, 1990). Rapid heating generated thermal gradients, which drove large diffusional fluxes for sintering process (Young and McPherson, 1989). The sintered stainless steels series 300 responded to the heating rate in a different way compared to the reported ceramics. This is attributed to some reasons. First, the heating rates employed in this work are not very different. Thus sintered density or densification is not sensitive to the heating rate range determined. The second reason is attributed to small thermal gradients due to thin dimensions and high thermal conductivities of metal powder compact. Because of these reasons, densification of the sintered stainless steels series 300 is controlled by an isothermal condition. The low heating rate allows longer time for atomic diffusion, which is an important sintering factor. This means more atoms move to points of contact between powder particles to form sintering necks. When the necks grow, the pore sizes are reduced. This causes an increase of the final density of the sintered products.

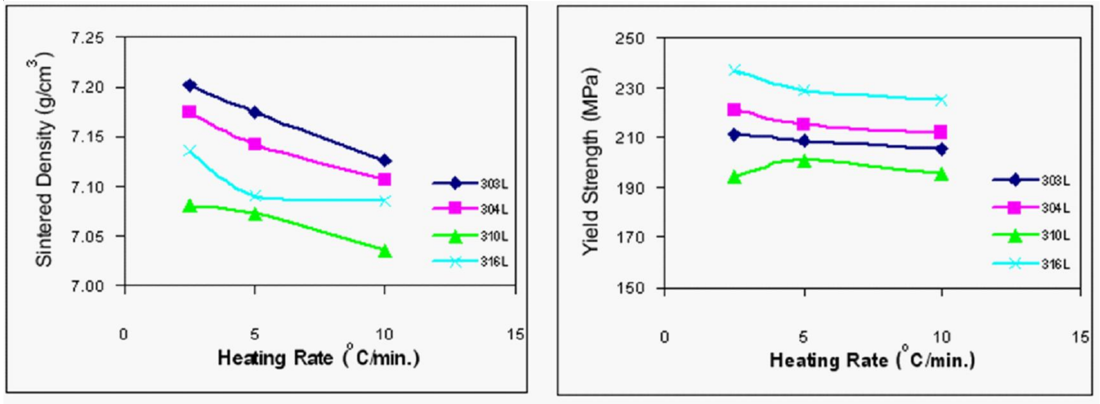
3.2 Mechanical property

Most of the sintered stainless steels series 300 showed a dependency of the tensile properties on the heating rates. With a low heating rate, the sintered materials exhibited higher strength and ductility (Figure 1(b)-(d)). The dependency of the tensile properties on the heating rate was in same trend than that of the sintered density. In a previous study (Coovattanachai, 2006) it was found that sintered density exhibited a linear relationship with tensile properties (ultimate tensile strength, yield strength, and elongation) for the sintered 316L and 304L materials. Therefore, the reason for a better tensile property with a low heating rate is the same for a better sintered density with a low heating rate.

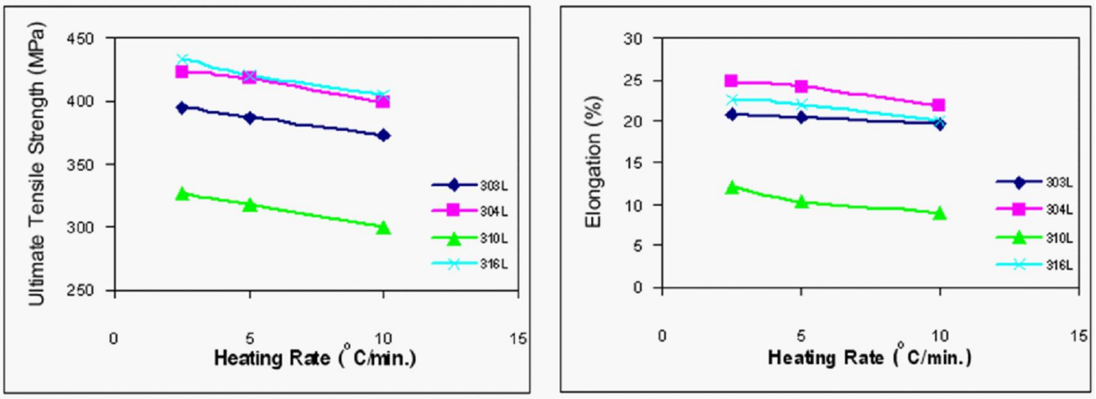
In general, tensile properties of sintered materials are controlled by both quantity (number and size) and quality of the sintered necks. When one particular grade of stainless steel is considered, the quantity of sintered necks may be the

Table 1. Composition of stainless steel powder series 300.

Grade	Composition, wt. %										
	C	Ni	Cr	Si	Mn	S	O	N	Mo	Cu	Fe
303L	0.01	12.9	17.2	0.8	0.07	0.20	0.161	0.037	-	2.0	Bal.
304L	0.01	11.2	18.5	0.9	0.20	0.01	0.253	0.049	-	-	Bal.
310L	0.01	20.1	24.1	1.1	0.40	-	-	-	-	-	Bal.
316L	0.02	12.8	16.9	0.8	0.13	0.01	0.266	0.027	2.3	-	Bal.



(a) Sintered density (b) Yield strength



(c) Ultimate tensile strength (d) Elongation

Figure 1. Properties of sintered stainless steels series 300 at various heating rates.

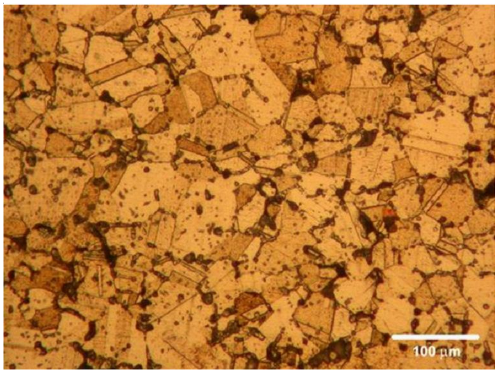
most important factor controlling the tensile properties. The experimental results indicate that a low heating rate tends to increase the quantity of sintered necks. When all of the sintered stainless steels are compared, at the same heating rate, the quality of the sintered necks has to be taken into account. The experimental results indicate that the quality of the sintered necks is seemingly dependent on the metal powder composition. The high-alloyed 310L seems to have poor sintered necks compared to other series 300 alloys investigated.

3.3 Microstructure

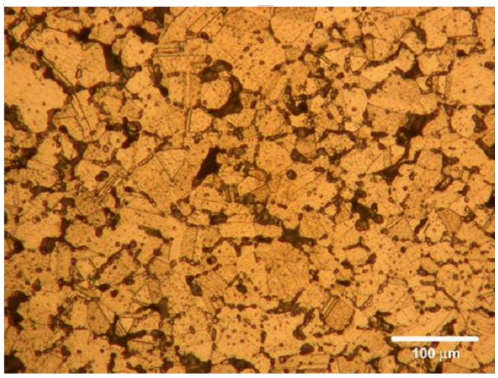
The sintered 304L material was chosen as a representative for determination of the effect of heating rate on microstructural changes in the sintered stainless steels series 300. Microstructures of sintered 304L material heated with different rates are shown in Figure 2. For the microstructures, pores were observed between and inside the 304L grains. The pores results from the voids between particles initially contain lubricants, binders, and contaminants. As heating occurs, these species evaporate, leaving pores between particles. In the sintered material heated with a slow rate, round pores were observed along grain boundaries and inside grains. In the fast heated material, there were more irregular pores. With progressing of the sintering process, the initially irregular pores become rounded and change size. With further sintering, pore expansion occurs, which results from a coarsening process, so called *Ostwald ripening* (German, 1998). It happens more when the pore size distribution is wider, with the larger pores growing at the expense of the smaller pores.

Twinning was clearly observed in the 304L grains. The twinned structures, generated during compaction steps using at high pressure to form a powder compact, still existed in the sintered materials. The austenitic structure of the 304L powders is susceptible to twin formation. This is because the austenitic 304L has a high number of slip planes. Recently, it was found that when the grain size of the sintered 316L exceeded 200% of the original size, twinning disappeared (Tosangthum, 2006). The disappearance of twinning associated with grain growth is attributed to atom re-arrangement within the structure of the grains.

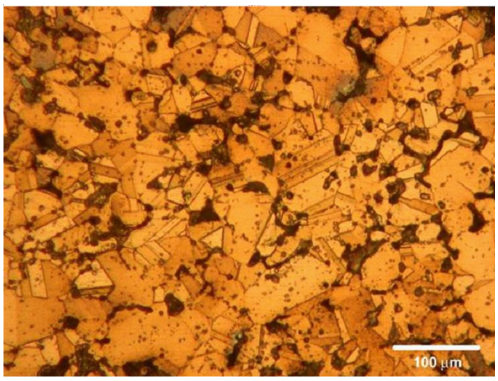
The grain size of the sintered specimen was measured



(a) 2.5°C/min.



(b) 5°C/min.



(c) 10°C/min.

Figure 2. Microstructures of sintered 304L specimens heated with various rates; (a) 2.5°C/min. (b) 5°C/min., and (c) 10°C/min. (Glyceregia etched).

Table 2. Grain size of sintered 304L specimens.

Heating Rate (°C/min)	Grain Size (mm)	
	304L	316L
2.5	38	37
5	34	34
10	31	30

by using Abrams Three-Circle procedure. It was found that a slow heating rate caused coarser grains in the sintered materials. The decrease of the grain size with increasing heating rate for the sintered 304L and 316L materials is shown in Table 2. The reason of coarser grains in the sintered material heated with the low rate is as follows. The low heating rate allows longer time for atomic diffusion, which controls the sintering process. Longer time for atomic diffusion means more atoms move to points of contact between powder particles to form sintering necks. In the cases of sintered necks forming between bigger and smaller particles a grain boundary is removed, with the larger grains growing at the expense of the smaller grains.

4. Conclusions

The exploration of new sintering schemes, by varying heating rates, indicates that the heating rate range of 2.5-10°C/min provides small differences in the densification and properties of the sintered stainless steels series 300. A slow heating rate tends to improve densification and property of the studied alloys. The experimental results are contradicting the improved densification of some ceramics by ultra rapid heating. The reasons for this contradiction are as follows. First, the heating rates employed in this work are not very different. The second is attributed to small thermal gradients generated in the thin metal powder compacts.

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