Songklanakarin J. Sci. Technol.
32 (1), 91-95, Jan. - Feb. 2010

Songklanakarin Journal of Science and Technology
http://www.sjst.psu.ac.th

Original Article

Sintering activation of 316L powder using a liquid phase forming powder

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Received 28 September 2007; Accepted 18 November 2009

Abstract

It was found that the addition of a liquid forming powder (up to 6 wt.% of a gas-atomized tin powder) to 316L powder could activate the sintering process. Sintering activation could be observed by an increase of the sintered density and selected mechanical properties. When optimized tin powder content was used, shorter sintering time and lower sintering temperature could produce sintered 316L+tin materials with excellent mechanical properties. Electron dispersive spectroscopy analyses across 316L-tin-316L grains indicated that Ni transportation during the sintering process was enhanced by the presence of liquid tin.

Keywords: 316L Powder, tin powder, liquid phase forming

1. Introduction

Powder metallurgy (PM) involves forming processing of metal powders into useful metallic parts. In a common PM process, metal powders are compacted into green parts using high pressure, whose strength is just sufficient for handling only. The green parts are then heated up to a certain temperature below the melting point of the principal powdered metal under suitable reducing atmosphere. During heating, sintering phenomena (weldment of contact interfaces between powder particles) causes densification and strengthening of the parts. The basic PM process has been employed for producing engineering metallic parts, which are used in many applications including aerospace, agriculture, appliances, automotive, building and construction, chemical, electrical and electronic, hardware, industrial, jewelry, marine, medical office equipment, recreation and leisure (Reinshagen *et al.*, 1989).

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Sintered 316L stainless steel has been considered as a replacement for sintered ferrous alloys due to its superior mechanical properties and corrosion resistance. Demand for higher performance sintered 316L stainless steels exists. Attempts have been being carried out to improve the performance of sintered 316L stainless steel. The addition of nickel and copper powders to austenitic 316L stainless steel powder resulted in modified densification behaviors (Tosangthum *et al.*, 2006). The composition of the admixed powder, which exhibited optimum mechanical properties, was 316L+6wt. %Ni. The addition of Cu powder to the admixed 316L+Ni powders resulted in a decrease of the sintered density, mechanical properties, and shrinkage after sintering. Although Ni powder is a promising alloying material for the improvement of the sintered 316L stainless steel properties, it is not economically feasible due to its high cost.

High strength sintered 316L stainless steel could be obtained by modification of the sintering process. Increasing sintering time for the 316L alloy resulted in a slightly improved strength (Vetayanugul *et al.*, 2002). However, prolonged sintering time caused detrimental effects to the sintered materials, such as grain growth and particle coarsening (German *et al.*, 1998). Modification of sintering

atmosphere composition by varying the nitrogen content resulted in an improvement of ultimate tensile strength (UTS) and yield strength of the sintered materials (Samal *et al.*, 2001). An increase in the strength after applying nitrogen is attributed to the solid solubility of nitrogen at the interstitial sites in the powder particle matrix (Simsons *et al.*, 1995).

Liquid phase sintering of 316L admixed with liquid forming powders could change the sintered material property (Rosso *et al.*, 1996). The transient liquid phase reaction between iron-nickel and aluminum formed intermetallic $(Fe, Ni)_3Al$, which modified the densification of 316L stainless steel powder and resulted in a change of its macrohardness and corrosion resistance.

During solid state sintering, various phenomena, for example surface diffusion, vapour transport and plastic flow, occured (Exner *et al.*, 1996). Among these phenomena, surface diffusion is the most important one. Atomic diffusion of alloy powder constituents from bulk to the points of contact between powder particles (the areas where powder particles weld with one another) controls the sintering process. The presence of a liquid phase, formed before the commencement of the solid-state sintering of the base powder particles, may ease the diffusion of the alloy powder constituents. With this assumption, some liquid phase forming powders have been developed. One of the liquid phase forming powders, namely nitrogen-atomized tin powder, was employed for the investigation of the sintering activation by a liquid phase forming during heating of the powdered metal parts.

2. Materials and Methods

The powders employed for this investigation included a water-atomized 316L stainless steel powder and a nitrogen-atomized tin powder. Particle sizes of the tin powder were less than 32 μm. The as-received 316L powder was used without further sieving. Experiments were designed to examine the effects of three parameters, namely tin powder content, sintering temperature, and sintering time.

For the first experimental work, varied amounts of tin powders (1-6 wt.%) were admixed with 316L before being compacted into green tensile test bars (TTBs) with green density of 6.50±0.05 g/cm^3. The green TTBs were debinded/delubricated at 600°C in argon and sintered at 1300°C for 45 minutes in pure hydrogen atmosphere. The sintered materials were tested and characterized as following. Sintered density was measured by using MPIF standard 42. Mechanical (tensile) properties of the sintered TTBs were measured by using a universal testing machine. Hardness of the sintered specimens was determined using a hardness tester (Rockwell scale B). Microstructural observations were performed using optical microscopy.

In the second experimental work, varied sintering temperatures, 1150, 1200, 1250, 1300, and 1350 °C, were performed while the optimum tin powder content and other processing parameters were kept constant. In the third experimental work, sintering of the material containing optimum tin powder content at optimum sintering temperature was performed with varied sintering times.

3. Results and Discussion

3.1 Effect of tin powder content

The sintered 316L+tin materials showed improved sintered densities when tin powder contents of more than or equal to 4 wt.% were added (Table 1). These materials also showed improved ultimate tensile strengths, yield strengths, and hardness, when the same amounts of tin powder were added. However with improved strengths and hardness, the sintered 316L+tin materials lost their ductility when tin powder of 5 and 6 wt.% were added. Addition of the tin powder with contents of more than 6 wt.% resulted in a distortion of the sintered 316L+tin TTBs. In addition to shape distortion, excessive liquid tin was observed to ooze out the TTBs resulting in the formation of solid tin on the TTBs surfaces during cooling.

In general, sintered materials exhibit superior mechanical properties when they have (i) higher sintered density, (ii) higher volume fraction of sintered necks or sintered bonds between metal powder particles, (iii) chemical homogeneity, and (iv) strengthening factors. Superior tensile strengths of the sintered 316L+tin materials may indicate that they have larger volume fraction of sintered necks. Increased sintered density results from either decreased pore volume fraction or increased sintered neck volume fraction or

Table 1. Dependence of sintered 316L+tin properties on tin powder content.

Properties	Tin powder content (wt.%)						
	0	1	2	3	4	5	6
Sintered density (g/cm^3)	7.08	7.10	7.12	7.20	7.69	7.44	7.46
Ultimate tensile strength (MPa)	445.87	395.31	375.79	410.17	481.46	470.51	471.36
Yield strength (MPa)	249.56	228.42	195.27	254.60	297.34	353.83	352.16
Elongation (%)	24.43	17.17	16.42	19.03	22.86	15.58	10.51
Hardness (HRB)	41.56	41.16	43.40	47.47	67.90	67.62	71.13

Table 2. Effect of liquid tin volume fraction on grain size.

Material	Liquid Sn volume fraction (V_L)	Grain size increase (%) compared to averaged 316L grain size
316L	0.0000	0.00
316L+1wt.%Sn	0.0093	9.70
316L+2wt.%Sn	0.0187	40.65
316L+3wt.%Sn	0.0278	47.12
316L+4wt.%Sn	0.0391	212.31
316L+5wt.%Sn	0.0466	216.11
316L+6wt.%Sn	0.0591	220.05

increased shrinkage. When both, tensile strength and sintered density, were taken into account, it might be assumed that the sintered neck is the prime factor governing the sintered density and mechanical properties.

Admixing of 316L powder with tin powder also caused grain growth in the sintered 316L+tin materials (Table 2). Grain growth was frequently observed in the materials sintered for prolonged times (Tosangthum *et al.*, 2006). Surprisingly, grain coarsening in the sintered 316L+tin materials even occurred at normal sintering conditions for 316L powder. This may indicate that liquid tin accelerates atomic diffusion-related processes occurring during heating of 316L+tin materials. The grain size increased with increasing liquid tin content of up to 6 wt.%. This phenomenon contradicted the behaviors of the sintered materials, such as W-Ni, VC-Co, W-Ni-Fe, and Pb-Sn (German *et al.*, 1998). Grain size of the sintered W-Ni, VC-Co, W-Ni-Fe, and Pb-Sn materials increased with increasing liquid volume fraction.

3.2 Effect of sintering temperature

Sintering is a process based on diffusion of atoms to form necks and cause neck growth at the contacting areas between powder particles (Exner *et al.*, 1996) tomic diffusion is a temperature-dependent process. It is thus necessary to investigate the effect of sintering temperature. The admixed 316L+ 4 wt.% tin powder was selected for the investigation because it had the optimum composition.

An increases of the sintered density, the mechanical properties (Table 3), and the grain size (Table 4) of the sintered 316L+tin materials with increasing temperature was observed. The experimental results are not uncommon. However, it is worth noting here that because of the presence of tin lower sintering temperatures (1200 and 1250°C) compared to the normal sintering temperature (1300°C) for straight 316L powder (Vetayanugul *et al.*, 2002) are con-

Table 3. Dependence of sintered 316L+tin properties on sintering temperatures.

Properties	Sintering temperature (°C)				
	1150	1200	1250	1300	1350
Sintered density (g/cm^3)	7.10	7.22	7.32	7.69	7.58
Ultimate tensile strength (MPa)	346.20	418.79	452.82	481.46	583.10
Yield strength (MPa)	275.65	308.54	314.41	297.34	373.43
Elongation (%)	4.11	8.17	11.63	22.86	22.38
Hardness (HRB)	43.38	44.32	49.18	67.90	71.30

Table 4. Effect of sintering temperature on grain size of 316L + 4 wt.%Sn.

Sintering temperature (°C)	Grain size increase (%)compared to averaged grain size of the material sintered at 1150°C
1150	0.00
1200	20.69
1250	62.07
1300	244.83
1350	320.69

Table 5. Dependence of sintered 316L+tin properties on sintering times.

Properties	Sintering time (min)					
	15	25	35	45	55	65
Sintered density (g/cm^3)	7.00	7.00	7.56	7.69	7.15	7.13
Ultimate tensile strength (MPa)	451.48	497.75	603.45	481.46	537.66	524.45
Yield strength (MPa)	272.78	309.51	380.85	297.34	333.59	320.69
Elongation (%)	17.78	20.31	23.52	22.86	21.64	20.50
Hardness (HRB)	48.55	49.62	65.80	67.90	50.60	50.20

siderably sufficient for the sintering of 316L powder. The 316L+tin materials sintered at 1200 and 1250°C showed properties (Table 3) comparable to those of the 316L material sintered at 1300°C (Table 1). Lower temperatures for sintering of stainless steel powders series 300 may be feasible for P/M parts manufacturing.

3.3 Effect of sintering time

In general, sintering time is one of the factors controlling the number of diffused atoms during a sintering step (German *et al.*, 1998). A longer sintering time means a higher number of diffused atoms. Increases of sintered density and mechanical properties of the sintered 316L+tin are shown in Table 5. Because of the prolonged time of high temperature exposure and higher number of diffused atoms, a grain size increases with increasing sintering time was observed (Table 6).

3.4 Chemical analysis by EDS

EDS line scanning was performed across 316L-tin-316L grains (Figure 1). During EDS line scanning, the intensity of the characteristic energy of the specified element was counted and recorded against the scan distance. It was found that there were fewer amounts of Fe, Cr, and Mo elements in the tin phase compared to those in the 316L grains (Figure 2). In contrast, an abundance of Ni and tin elements was observed in the tin phase (Figure 2). During heating the powder constituent atoms diffuse from the 316L grains to their surfaces. Some of the grain surfaces are contacting areas between 316L grains. The sintered necks are formed and

Table 6. Effect of sintering time on grain size

Sintering time (min)	Grain size increase (%)
15	0.00
25	75.00
35	100.00
45	116.67
55	133.33
65	150.00

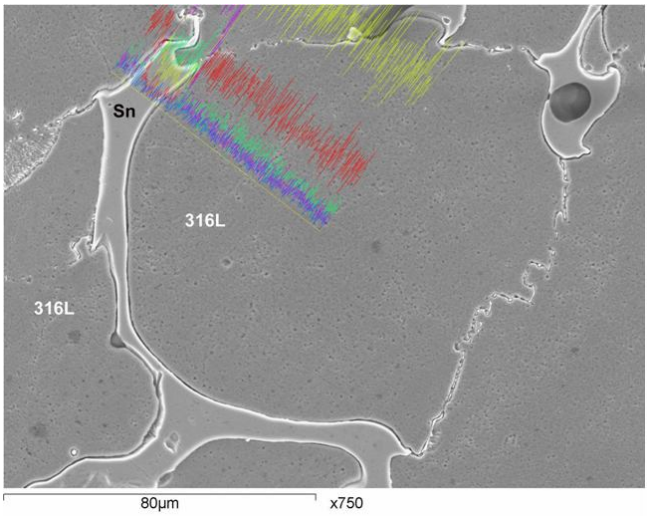

Figure 1. SEM micrograph showing EDS scan across 316L-tin-316L grains.

grown. For the surfaces contacting directly to the liquid tin, the diffused atoms are resolved in the liquid. Because of the presence of the liquid phase, transportation of elemental atoms is supposed to be enhanced. In other words, the liquid tin is acting like a path for elemental atom diffusion.

The presence of Ni-rich tin phase in the sintered 316L+tin materials may indicate that during cooling some excessive Ni atoms are ejected from the liquid tin and diffuse back to the 316L grains. The remaining high Ni content in the liquid tin phase indicates either high solid solubility of Ni in tin or formation of Ni-Sn compounds. Whatever process occured, it is supposed here that the Ni atoms are able to diffuse faster in the liquid tin. The Ni diffusion acceleration may be the cause of the sintering process activation.

3.5 Optimum sintering conditions for 316L+tin powders

In order to choose the optimum sintering conditions, the sintered properties, including density, tensile strength, and hardness, were taken into account. From the experimental procedure it was observed that the suitable tin powder content for admixing with the 316L powder was 4 wt.%. When the 316L+4 wt.% tin material was experimentally sintered at different temperatures an optimum temperature

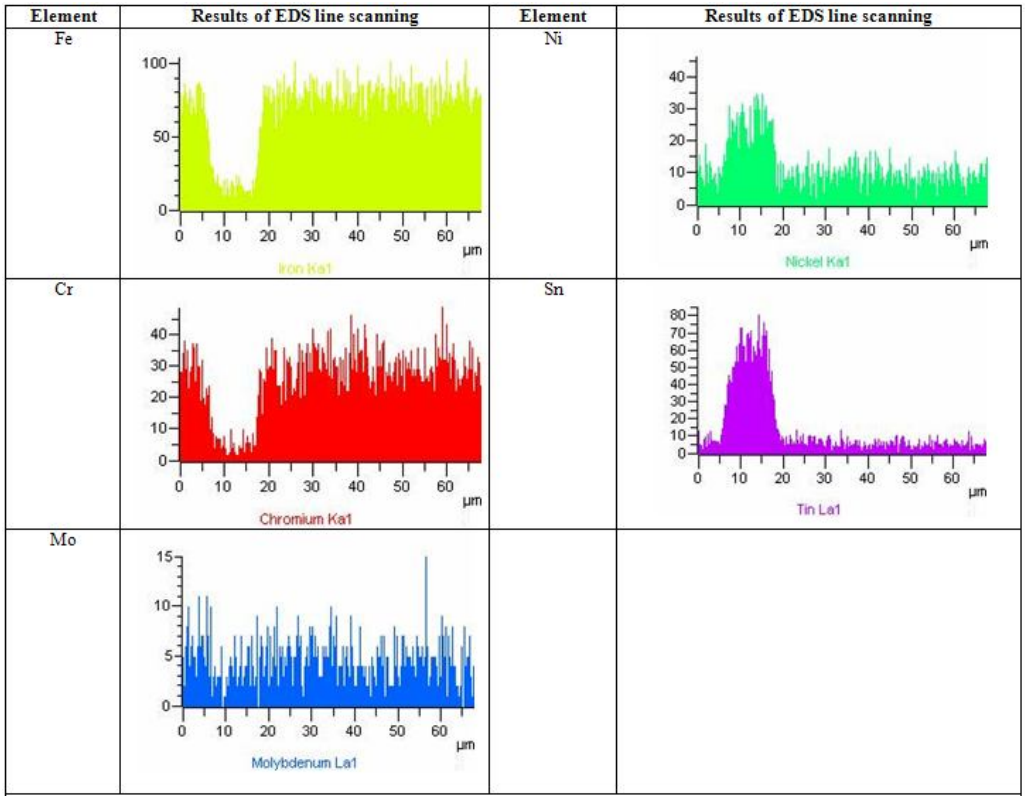

Figure 2. EDS scan results.

of 1300°C was found. When sintering times were tested, the period of 30 minutes was sufficient for producing sintered 316L+4wt.% materials with considerably good properties.

4. Conclusions

Sintered density of 316L+tin material was increased significantly with increasing tin powder content. Tin addition affected the properties and microstructures of the sintered 316L+tin material. Grain growth was an obvious evidence of microstructural change. Superior mechanical properties, such as ultimate tensile strength, yield strength, elongation, and hardness, could be obtained when optimum sintering conditions were employed.

Acknowledgment

The authors would like to express their sincere gratitude to the National Metal and Materials Technology Center (MTEC), Pathum Thani, Thailand, for financial support.

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