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Density and Strength Improvement of Sintered 316L Stainless Steel

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ABSTRACT

Admixing of Ni powder was found to improve some mechanical properties of sintered 316L stainless steel. However, Ni powder addition is not economically feasible due to its high cost. A metal powder, designated as PMTEC1, was employed as an alternative admixing powder for performance improvement of the sintered 316L stainless steel. In this investigation, 316L, 316L+6wt.%Ni and 316L+6wt.%PMTEC1 powders, were processed using a 'press and sinter' method. Properties of the sintered 316L+6wt.%Ni and 316L+6wt.%PMTEC1 alloys were compared to the sintered 316L stainless steel. Sintering was carried out in different atmosphere compositions. It was found that the factors, such as N₂ content in the atmosphere, Ni and PMTEC1, showed some effects on some properties. Particularly, addition of the PMTEC1 significantly increased sintered density, yield strength and hardness with severely sacrificed elongation. The PMTEC1 powder caused grain growth, no matter the sintering atmosphere compositions employed. Nitrogen content in the atmosphere caused formation of nitride. The presence of PMTEC1 not only accelerated grain growth, but also activated nitride eutectoid formation.

Keywords: 316L stainless steel, powder admixing, mechanical property.

1. INTRODUCTION

Attempt has been being carried out to improve performance of sintered 316L stainless steel. Addition of nickel and copper powders to austenitic 316L stainless steel powder resulted in modified densification behaviors [1]. The composition of the admixed powder, which exhibited optimum mechanical properties was 316L+6wt.%Ni. Addition of Cu powder to the admixed 316L+Ni powders resulted in decrease of sintered density, mechanical properties and shrinkage after sintering. Although Ni powder is a promising alloying material for improvement of the sintered 316L stainless steel property, it is not economically feasible due to its high cost.

High strength sintered 316L stainless steel could be obtained by modification sintering process. Increasing sintering time for the 316L alloy resulted in slightly improved strength [2]. However, prolonged sintering time caused detrimental effects, such as grain growth and particle coarsening, to the sintered materials [3]. Modification of sintering atmosphere compositions by varying nitrogen content resulted in improvement of ultimate tensile strength (UTS) and yield strength of sintered materials [4]. Increase of strength with nitrogen content is attributed to solid solubility of nitrogen at the interstitial sites in the powder particle matrix [5].

Liquid phase sintering of 316L admixed with a liquid forming powders showed evidence of material property change [6]. The transient liquid phase reaction, between ironnickel and aluminium to form an intermetallic (Fe, Ni)3Al, modified densification of 316L stainless steel powder and resulted in change of its macrohardness and corrosion resistance. During solid state sintering, various phenomena, for example surface diffusion, vapour transport and plastic flow, occur [7]. Among these phenomena, surface diffusion is the most important. 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 sintering process. The presence of a liquid phase, formed before the commencement of solid-state sintering of the base powder particles, may ease diffusion of the alloy powder constituents. With this assumption, some liquid phase forming powders have been being developed. One of the liquid phase forming powders, designated as PMTEC1, was employed for improvement of sintered 316L stainless steel. The outputs resulted from Ni and PMTEC1 additions and sintering atmosphere compositions are presented and discussed in this article.

2. MATERIALS AND METHODS

The powders employed for this investigation include 316L, 316L+6wt.%Ni and 316L+6wt.%PMTEC1. Powder particle sizes of Ni and PMTEC1 were less than 32 μ m. For each experimental conditions, 15 tensile test bars were produced by compacting the powders into standard tensile test bars (MPIF standard 10, ASTM B783) with green density of 6.58 \pm 0.05 g/cm³. The green tensile test bars were debinded at 600°C in argon and sintered at 1300°C for 45 minutes in different atmosphere compositions (100%H₂, 25%H₂+75%N₂, 50%H₂+50%N₂ and 100%N₂). To ensure the atmosphere composition control, high purity H₂ and N₂ gases were mixed and packed in a cylinder by a gas supplier.

Dimensional changes along selected dimensions, with respect to tooling component dimensions, of the tensile test bars (Figure 1) were measured. Sintered density was measured by using MPIF standard 42. Microstructural observation was performed by using optical microscopy. Mechanical (tensile) properties of the sintered tensile test bars were measured by using a universal testing machine. Hardness of the sintered specimens was carried out using a hardness tester (Rockwell scale B).



Figure 1. Selected dimensions for dimensional change measurement on the tensile test bar.

3. RESULTS AND DISCUSSION

3.1 Sintered Density

Density of the sintered 316L was improved when Ni and PMTEC1 powders were added (Figure 2). Increase of sintered density, caused by Ni addition, was attributed to dimensional change (shrinkage) during sintering step [1]. In a solid-state sintering, such as the cases of 316L and 316L+6Wt.%Ni, shrinkage is perhaps related to neck radius growth, which is in turn related to material transport as mentioned in [7]. Increase of sintered density with increasing Ni amount may be attributed to diffusion of Ni atoms into the austenitic matrix [8]. Elemental or Ni alloy powders activate sintering of the Nicontaining admixed powder compacts.

The sintered densities, in the range of 7.60 and 7.80 g/cm³, were obtained when the green 316L+6wt.%PMTEC1 tensile test bars were sintered in different atmospheres. The PMTEC1 was produced from a low melting metal so it was melted prior to the solid-state sintering occurred. Presence of a liquid phase may ease material transport, which governs sintering. Diffusion of elemental atoms through a liquid may occur faster than any other routes.



Figure 2. Effect of atmosphere compositions and powder admixing on sintered density of sintered materials.

3.2 Dimensional Change

3.2.1 Spring Back

During uniaxial compaction, the powder particles are squeezed to form a compact in a rigid die. The load exerting to the powder particles may be divided into two types, namely axial and radial loads. Both loads cause residual stresses, which are stored in the compacts. Radial expansion of the compact after being ejected from the die is called "spring back". Spring back is the phenomenon related to residual stress releasing. Spring back of the green tensile test bars was calculated from changes of their dimensions with respect to those of the die.

Addition of small particles of Ni and PMTEC1 to 316L powder decreased spring back (Figure 3). This can be explained using the following hypothesis. Admixing of 316L powder particles with small particles of Ni and PMTEC1 results in that small Ni and PMTEC1 particles occupy interstitial voids



(a)

Figure 3. Dimensional changes of the sintered tensile test bars measured along different dimensions; (a) dimension 1.

(b)

Figure 3 (continue). Dimensional changes of the sintered tensile test bars measured along different dimensions; (b) dimension 2.

(c)

Figure 3(continue). Dimensional changes of the sintered tensile test bars measured along different dimensions; (c) dimension 3.

between larger 316L particles. When the mixed powders are compacted, the interstitial powders are plastically deformed. This prevents the larger 316L powders from being elastically deformed. Few residual stress caused by elastic deformation is created. Therefore, spring back is reduced. Similar phenomena have been observed for the case of admixing of 409L with small particles of Cu powders [9].

3.2.2 Shrinkage

Shrinkage of the sintered materials depends on several factors, such as green compact geometry, green density, powder composition, admixing powder type, sintering atmosphere, sintering temperature and sintering time. For the effect of green compact geometry, shrinkage of the sintered tensile test bars along different dimensions were observed and reported in 316L+Ni+Cu [1] and 409L+Cu [9] alloys.

Addition of small particles of Ni and PMTEC1 caused shrinkage of the sintered 316L+6wt.%Ni and 316L+6wt.%PMTEC1 alloys be higher than that of 316L. The sintered 316L+6wt.%PMTEC1 alloy showed the highest level of shrinkage. As mentioned in (3.1), both Ni and PMTEC1 powders modify sintering in different manners. Ni acts as a solid-state sintering activator while a liquid PMTEC1 acts as a fast diffusion route for atomic transportation. In the case of 316L+6wt.%PMTEC1 alloy, constituent atoms jump from 316L powder particles to a liquid, through which they diffuse quickly to the points of contacts between powder particles (the neck formation sites). This causes neck growth and bring the 316L particle centres to be closer. Accumulation of these microscopic phenomena causes bulk shrinkage of the sintered materials.

Fluctuation of shrinkage was clearly observed when pure H₂ was employed for sintering of 316L, 316L+6wt.%Ni and 316L+6wt.%PMTEC1 alloys (Figure 3). The 316L+6wt.%PMTEC1 alloy exhibited the highest shrinkage when being sintered in pure H_2 atmosphere. It is not understood why the alloys sintered in pure H_2 atmosphere exhibiting fluctuated shrinkage.

Net dimensional change (spring back + shrinkage) is related to sintered density. It was clearly observed that net dimensional change of the PMTEC1-containing material was the highest (Figure 3). This is perhaps one of the reasons for the highest sintered density obtained in the 316+PMTEC1 material.

3.3 Microstructure3.3.1 Sintering in 100% H₂

The sintered 316L stainless steel (Figure 4(a)) and sintered 316L+6wt.%Ni alloy (Figure 4(b)) exhibited typical twinned austenitic grains with considerably clean grain boundaries and some pores. The clean grain boundaries indicated a few precipitation. It was observed previously that addition of Ni powder could improve microstructure of the sintered 316L stainless steel [1]. Number of open interconnected pores and pore sizes in the sintered 316L+Ni alloys were apparently decreased with increasing Ni powder content. The sintered 316L+6wt.%PMTEC1 alloy (Figure 4(c)) exhibited microstructures consisting of coarse non-twinned grains, large pores and solid PMTEC1 phase. The admixing PMTEC1 powders become a liquid at temperature lower than sintering temperature. The PMTEC1 liquid penetrates along powder particle boundaries. It is supposed to be path for atomic diffusion acceleration. Figure 4(c) may indicate that atomic diffusion acceleration by the presence of PMTEC1 liquid obviously leads to grain growth and metallurgical change of the base 316L material nature.

3.3.2 Sintering in Mixed N₂-H₂

Microstructures of the tensile test bars sintered in $50\%N_2$ - $50\%H_2$ showed microstructural changes (Figure 5). The sintered 316L stainless steel (Figure 5(a)) showed grains surrounded with dark grain boundary phases, which were identified as chromium nitride precipitates [10]. Similar to

(a) 316L

(b) 316L+6wt.% Ni

(c) 316L+6wt.% PMTEC1

Figure 4. Microstructures of the tensile test bars sintered in 100%H₂.

that of the sintered 316L alloy, grain boundaries of the sintered 316L+6wt.%Ni alloy (Figure 5(b)) were thickened. The sintered 316L+6wt.%PMTEC1 alloy (Figure 5(c)) showed the microstructure, within which there were large pores, PMTEC1 phase, lamellar Cr_2N eutectoid and coarse non-twinned grains. Precipitation of the lamellar Cr_2N eutectoid was previously reported to occur in the AISI 316L and AISI 430L materials sintered at 1280 °C in 30%H₂+70%N₂ [11].

(a) 316L

(b) 316L+6wt.% Ni

(c) 316L+6wt.% PMTEC1

Figure 5. Microstructures of the tensile test bars sintered in $50\%N_2$ - $50\%H_2$.

3.3.3 Sintering in Pure N₂

Microstructures of the tensile test bars sintered in 100%N₂ (Figure 6) showed obvious microstructural changes. The sintered 316L stainless steel (Figure 6(a)) showed grains surrounded with thickened grain boundaries. Some areas in the microstructure were converted to the lamellar Cr_2N eutectoid. Compared to the sintered 316L stainless steel (Figure 6(a)), there were less amounts of lamellar Cr_2N eutectoid areas in the sintered 316L+6wt.% Ni alloy (Figure 6(b)). The sintered 316L+6wt.%PMTEC1 alloy (Figure 6(c)) showed microstructure similar to that of the same material sintered in 50% N₂-50%H₂ atmosphere. There were coarse grains with solid PMTEC1 phase at some grain boundaries and some lamellar Cr₂N eutectoid.

3.4 Mechanical Property 3.4.1 Atmosphere Effect

Mechanical properties (UTS, yield strength and elongation) of the sintered 316L and 316L+6wt.%Ni alloys were sensitive to atmosphere composition (Figure 7). Increase of nitrogen content up to 75% resulted in increase of UTS and yield strength but decrease of elongation. Increase of strength is attributed to interstitial solid solution strengthening caused by nitrogen dissolution [5]. Presence of residual nitrogen in solution in the matrix leads to strengthening of the

(a) 316L

(b) 316L+6wt.% Ni

(c) 316L+6wt.% PMTEC1

Figure 6. Microstructures of the tensile test bars sintered in $100\%N_2$.

(a)

Figure 7. Influence of Ni, PMTEC1 and nitrogen on mechanical properties of the sintered tensile test bars; (a) UTS, (b) 0.2% yield strength.

(c)

(d)

Figure 7 (continue). Influence of Ni, PMTEC1 and nitrogen on mechanical properties of the sintered tensile test bars; (c) elongation and (d) hardness.

matrix [11]. Although nitrogenating of powders by solid-state processing is limited by the depth of N_2 diffusion into solid material of approximately 10 to 100 μ m, but this is the diameter of most metal powder [5].

When the 316L and 316L+6wt.%Ni materials were sintered in 100% N₂, small decrease of UTS and yield strength, in companion with significant decrease of elongation were observed. Slight decrease of UTS and yield strength may be attributed to grain boundary precipitation/grain boundary precipitate coarsening, lamellar Cr₂N eutectoid formation and its growth and sintering prohibition by oxide film at some points of contact between powder particles. Although sintering of 316L and its admixed powders was successful in 100% N₂, this atmosphere is considered as less effective for oxide reduction [3]. Oxide on metal powder surface has to be removed in order to improve metallic bonding quality during sintering step. When the sintering atmosphere composition is considered, decrease of sintered material elongation is caused by formation of chromium nitride in the nitrogen-penetrating zones. When this compound formed in the sintered material microstructure, it would caused embrittlement. The formation of the nitride was also reported to occur by nitrogen absorption during cooling [4].

The sintered 316L+6wt.%PMTEC1 alloy showed UTS with less sensitive to atmosphere. However, its yield strength and elongation were strongly dependent on nitrogen content.

It was observed that up to 25% nitrogen significantly improved hardness of the sintered alloys. Using nitrogen content beyond 25% resulted in a slight hardness improvement. The increase of hardness may be attributed to formation of nitride on the sintered alloy surface [4] and N_2 dissolution inside grains [10]. Comparison of sintered material microstructures (Figures 4-6) and hardness values (Figure 7 (d)) suggests that N_2 dissolution may exhibit stronger effect on

hardness than the case of surface nitride formation. If this hypothesis is true, nitrogen absorption as solution inside grains is saturated when the 25% N_2 +75% H_2 atmosphere is used.

3.4.2 Admixing Powder Effect

Addition of Ni powder to 316L powder slightly altered UTS, yield strength and hardness compared to those of the base 316L. However, Ni addition caused higher elongation of the sintered 316L+6wt.%Ni than that of the base 316L when the sintering atmosphere contained 25% to 75% nitrogen. In contrast, addition of PMTEC1 powder to 316L powder significantly altered UTS, yield strength, elongation and hardness compared to those of the base 316L. Yield strength and hardness were clearly improved. However, loss of ductility of the sintered 316L+6wt.%PMTEC1 was so large. Loss of ductility is strongly attributed to three factors, i.e. grain growth, metallurgical change (phase change of metallic grains) and chromium nitride formation. Loss of more than 50% of ductility, compared to the sintered 316L alloy, was caused by grain growth and metallurgical change (compared Figure 3 and Figure 6(c)).

It was noticed that Ni and PMTEC1 showed apparently opposite effect on UTS and yield strength. This is attributed to function of these two admixing species. The Ni powders acts as solid-state sintering activator [8]. Solid-state diffusion of Ni atoms may accelerate metallic bonding formation process. However, the presence of admixing Ni powders slightly change of grain size of the sintered 316L. With small grain size difference, the sintered 316L and 316L+ 6wt.%Ni materials exhibits close values of UTS and yield strength.

PMTEC1 becomes a liquid at a temperature lower than the sintering temperature. The PMTEC1 liquid is perhaps a path for atomic diffusion acceleration, which leads to grain growth. Although grain growth leads to poor UTS and yield strength but its effect is compensated by high sintered density (with less pores), which means high numbers of metallic bonds per unit volume. Therefore, the 316L+6wt.%PMTEC1 still exhibits high values of UTS and yield strength.

4. CONCLUSIONS

Admixing of small particles of Ni and PMTEC1 to 316L powder improved sintered density. The 316L sintered density of higher than 7.60 g/cm³ could be obtained when 6wt.% PMTEC1 was added. Addition of Ni powder caused slightly changes of UTS, yield strength and hardness of the base 316L. In contrast, addition of the PMTEC1 significantly increased yield strength and hardness with severely sacrificed elongation. The PMTEC1 caused grain growth, no matter the sintering atmosphere compositions employed. Nitrogen content in the atmosphere caused formation of nitride. The presence of PMTEC1 not only accelerated grain growth, but also activated nitride eutectoid formation.

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