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Effect of Nitrogen on Mechanical Properties of Sintered 409L Stainless Steel

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Abstract
Sintering of ferritic stainless steels is necessary to be carried out in a proper atmosphere. Normal atmosphere, namely cracked ammonia containing 75%H_2 and 25%N_2, employed for sintering of iron and steels, causes detrimental effect on properties of sintered ferritic stainless steels. The sintered alloys significantly lose their elongation. Nitrogen in the atmosphere is the main cause of embrittlement. Sintering of these alloys in pure hydrogen is the perfect way to prevent nitride formation. However, the cost of sintering is expensive so it causes less competitiveness of the sintered ferritic stainless steels in the P/M markets. Mixed H_2 + N_2 atmosphere with suitable content would reduce sintering cost while keep desirable properties of the sintered products. It was found that the sintered 409L stainless steel exhibited good elongation (about 15%) and increased strength and hardness when the 80%H_2 + 20%N_2 atmosphere was employed. Better strength and hardness were attributed to hardening effect caused by nitrogen.
Keywords: Nitrogen, mechanical property, sintered 409L stainless steel,

1. Introduction

There are various parameters controlling sintering process of metal powders. The parameters include (i) processing parameters (sintering temperature and time), (ii) chemical composition (atmosphere and powder composition) and (iii) sintering methods (solid-state, liquid-phase and supersolidus sintering methods). Preliminary study on compacting and sintering of 409L showed that under a certain atmosphere (pure hydrogen) both sintering temperature and time affected mechanical properties of the sintered 409L stainless steel [1]. Optimization of the processing parameters only is not complete information required for design and manufacturing of sintered 409L stainless steel. To obtain the optimized mechanical properties of the P/M 409L stainless steel, other sintering parameters should be examined.

Sintering atmosphere is one of the prime processing parameters for producing powder metallurgy (P/M) parts. There are various kinds of sintering atmospheres [2]. The atmosphere is required because of its multiple functions, which include removal of undesirable species (oxygen, humid, organic vapour and oxide), prevention of oxidation, protection of clean metal powder surfaces (inert atmospheres) and reaction with metal powder surfaces to form a hard surface layers. According to its functions the sintering atmosphere should be selected with careful considerations. Normal sintering atmosphere is inert, purified and reducing. Some typical sintering atmospheres, except the case of vacuum, usually contain reducing species, such as hydrogen (H_2) and carbon monoxide (CO), which are capable to react with oxygen (O_2) and undergo oxygen exchange with metal oxide in a sintering zone.

The atmospheres employed for sintering of stainless steels, are pure H_2, mixed H_2 + N_2 and vacuum. Vacuum sintering has to be carried out in a special furnace, which is normally a batch type. Therefore, it is a typical time-consuming process with low productivity. The vacuum-sintered stainless steel parts showed, comparing with dissociated ammonia (75% H_2 + 25% N_2)-sintered ones, higher value for impact resistance (unnotched bar test specimen) and lower value for other mechanical properties [3]. Ductility and toughness were superior. Better ductility means, in principle, better bonding between particles, therefore better corrosion resistance. Sintering of 316L stainless steel powder compacts under pure H_2 produced parts with lower tensile and yield strengths and hardness but higher elongation and corrosion resistance, compared to the same material sintered in cracked (dissociated) ammonia (75% H_2 + 25% N_2) atmosphere [3-5].

The best sintering atmosphere for P/M ferritic stainless steels is pure H_2. However, cost of sintering under pure hydrogen is high. This would cause the sintered ferritic stainless steels be less competitive in P/M markets. In this investigation, three different atmosphere compositions, 100% H_2, 90% H_2 + 10% N_2 and 80% H_2 + 20% N_2, has been employed for sintering of parts from the 409L stainless steel powder. Effects of N_2 content on mechanical properties and microstructures of the sintered 409L stainless steel are discussed in this article.

2. Materials and Method

The water-atomised 409L stainless steel powder was obtained from Coldstream of Belgium. The powder was compacted into tensile test bars (TTBs) with green densities of 6.50 ± 0.05 g/cm^3. The green TTBs were then delubricated at 600 °C for 60 minutes under argon atmosphere and sintered at 1350 °C for 45 minutes under different atmospheres, such as 100% H_2, 90% H_2 + 10% N_2 and 80% H_2 + 20% N_2. Dimensional changes of the TTBs, along different dimensions shown in Fig. 1, after compaction and sintering were measured. Densities of green and sintered samples were determined using the Archimedes method. A universal testing machine (Instron model 8801) was employed to measure mechanical properties of the sintered TTBs. Hardness of the sintered TTBs was carried out using a hardness tester (Rockwell scale B).

1 = Grip
2 = Gauge
3 = Length

Figure 1. Positions on a TTB measured for dimensional change.

3.Results and Discussion

(3.1) Dimensional change after compaction

During compaction process, the powder particles are pressed to form a green compact in a rigid die. The load exerting to the powder particles is divided into two types, namely axial and radial loads. Both loads cause residual stresses, which are stored in the compacts. Radial expansion of the green 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 TTBs was calculated from changes of their dimensions with respect to those of the die.

Plot of spring back against dimension of the green TTB is shown in Fig. 2. Spring back along the TTB gauge showed the highest value, followed by those along grip and length, respectively. Magnitude of the residual stress causing spring back is probably dependent on dimension of the green TTB. During powder compaction, higher residual stress is stored along shorter dimension. Therefore, the residual stress along the TTB gauge compacts is higher than that along other dimensions.

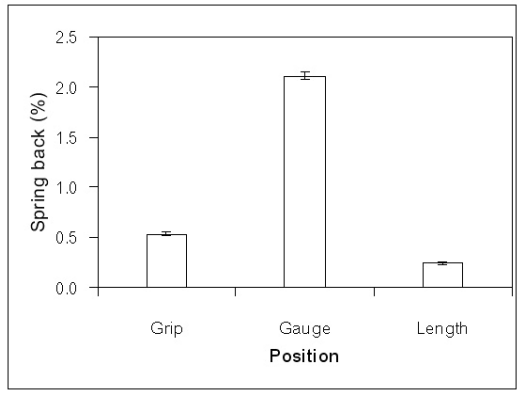


Figure 2. Spring back of the green TTBs along different dimensions.

(3.2) Dimensional change after sintering

Shrinkage after sintering was not dependent on the TTB dimensions and sintering atmospheres (Fig. 3). However, total dimensional change (spring back + shrinkage) depended on the TTB dimensions. The TTB gauge showed the highest total dimensional change due to the effect of spring back.

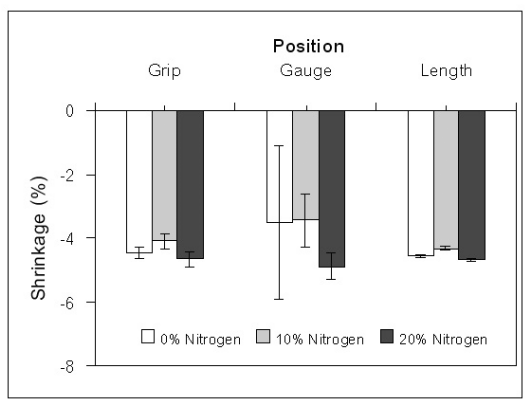


Figure 3. Shrinkage of the sintered TTBs along different dimensions.

(3.3) Sintered density

There was no relationship between sintered density and nitrogen content in the atmosphere (Fig. 4). However, sintered density showed close relationship with shrinkage (comparison between Fig. 3 and Fig. 4). The TTBs showed highest shrinkage and highest sintered density when they were sintered in 20% nitrogen atmosphere. Shrinkage or macroscopic volume reduction can be explained by using solid-state sintering mechanism, which can be divided into 3 stages [6]. In Stage 1, the points of

contact form in the necks between the individual particles. Stage 2 is characterized by rapid shrinkage and slow grain growth. In sintering process of loose powders, the transition to Stage 2 occurs at densities of approximately 75% of the theoretical. At densities between 91 and 95% Stage 3 starts, when the interconnected pore channels become unstable forming isolated pores at the grain boundaries or the interior of the grains. Due to microscopic volume change caused by neck formation (materials transport from powder particles to points of contact), neck growth and isolated pore formation, decrease of macroscopic volume (shrinkage) occurs.

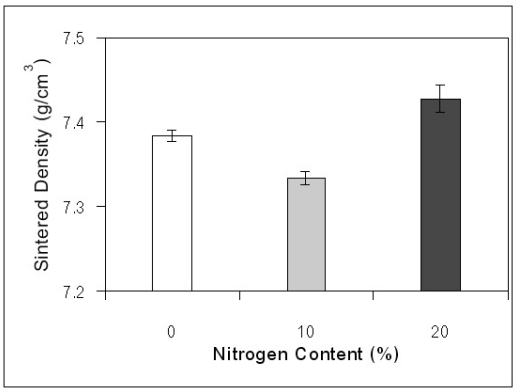


Figure 4. Sintered density of the 409L stainless steel sintered in different atmospheres.

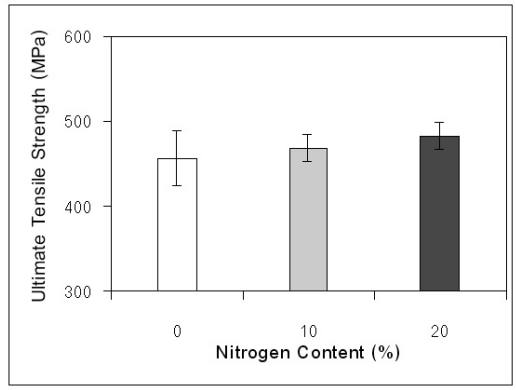
(3.4) Mechanical property

There was no clear relationship between sintered density (Fig. 4) and mechanical properties (Fig. 5). However, when mechanical property and nitrogen content were considered, relationship between these two factors existed (Fig. 5). UTS, yield, and hardness were increased with increasing nitrogen content. In contrast, elongation showed opposite trend. It was decreased with increasing nitrogen content.

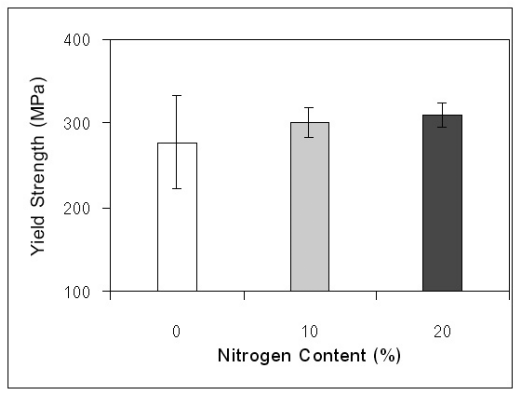
Better strengths and hardness was probably attributed to hardening effect caused by nitrogen. Microstructural observation indicates that two possible phenomena occur during cooling of the sintered 409L in nitrogen-containing atmospheres. First, the absorbed nitrogen dissolved in the 409L matrix. The solid solution of nitrogen in the matrix is the cause of material hardening. Second, the absorbed nitrogen reacts with chromium to form fine precipitates of chromium nitride along grain boundaries. In this case precipitate hardening takes responsibility for improved strength.

The nitrogen–containing atmospheres, compared to vacuum and pure hydrogen, when employed for sintering caused increase of UTS, yield and hardness of P/M stainless steels [3-5, 7]. The possible cause of hardening effect was chromium nitride formation. Evidence of eutectoid nitride formation at grain boundaries of sintered 316L stainless steel was clearly observed, when the material was sintered in an atmosphere with high nitrogen content [7].

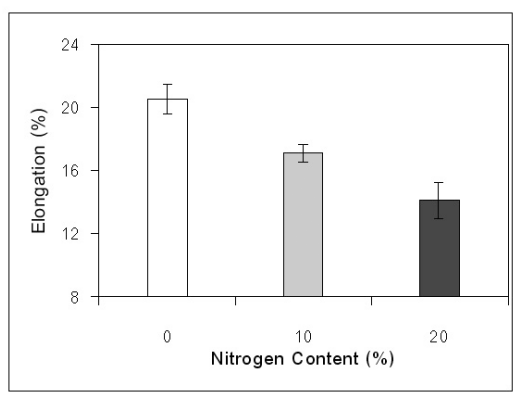
It was not uncommon that the sintered 409L stainless steel gained better strengths and hardness with scarified ductility. However, it still exhibited good elongation (about 15%) when the 80%H_2 + 20%N_2 atmosphere was employed. From the experimental results, it is determined that the 80%H_2 + 20%N_2 atmosphere offers a good combination between strength, hardness and elongation.



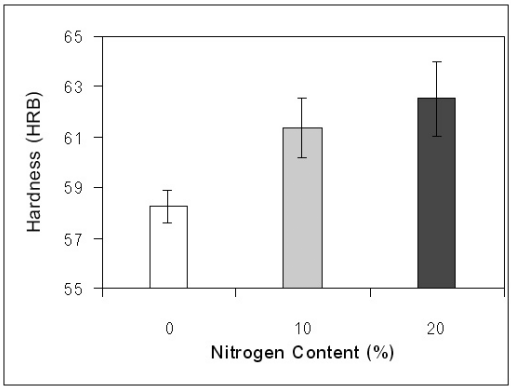
(a) UTS



(b) Yield strength



(c) Elongation



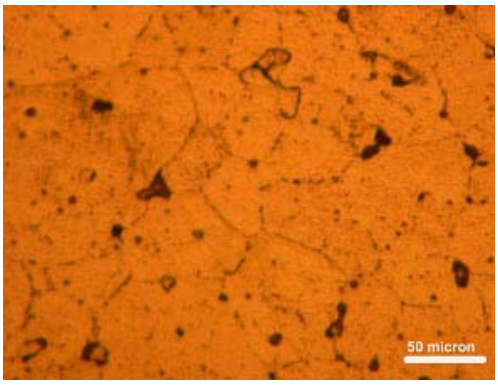
(d) Hardness

Figure 5. Mechanical properties of the 409L stainless steel sintered in different atmospheres.

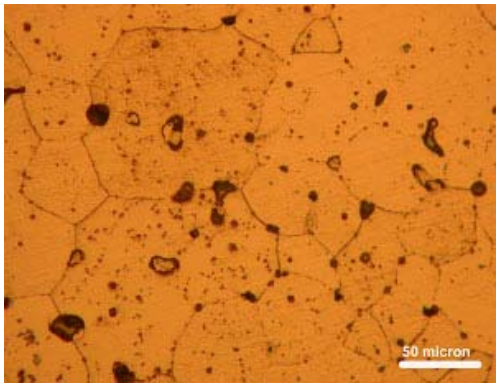
The 409L stainless steel sintered in this atmosphere exhibits sufficient mechanical properties for various applications. However, from the points of view of P/M stainless steel users, only mechanical properties may be not enough for application. Its chemical property, particularly corrosion resistance, is necessarily to be taken into account. Corrosion resistance of the sintered 409L material is recommended for further investigation.

(3.5) Microstructure

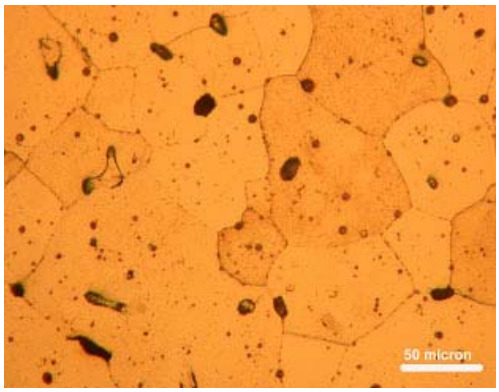
No clear evidence of eutectoid chromium nitride was observed (Fig. 6). Microstructures indicate that two possible phenomena occur during cooling of the sintered 409L stainless steel in nitrogen-containing atmospheres. First, the absorbed nitrogen dissolved in the 409L matrix. The solid solution of nitrogen in the matrix may be the main cause of material hardening. Second, the absorbed nitrogen reacts with chromium to form fine precipitates of chromium nitride along grain boundaries. In this case precipitate hardening takes responsibility for improved strength.



(a) 0 % N_2



(b) 10% N_2



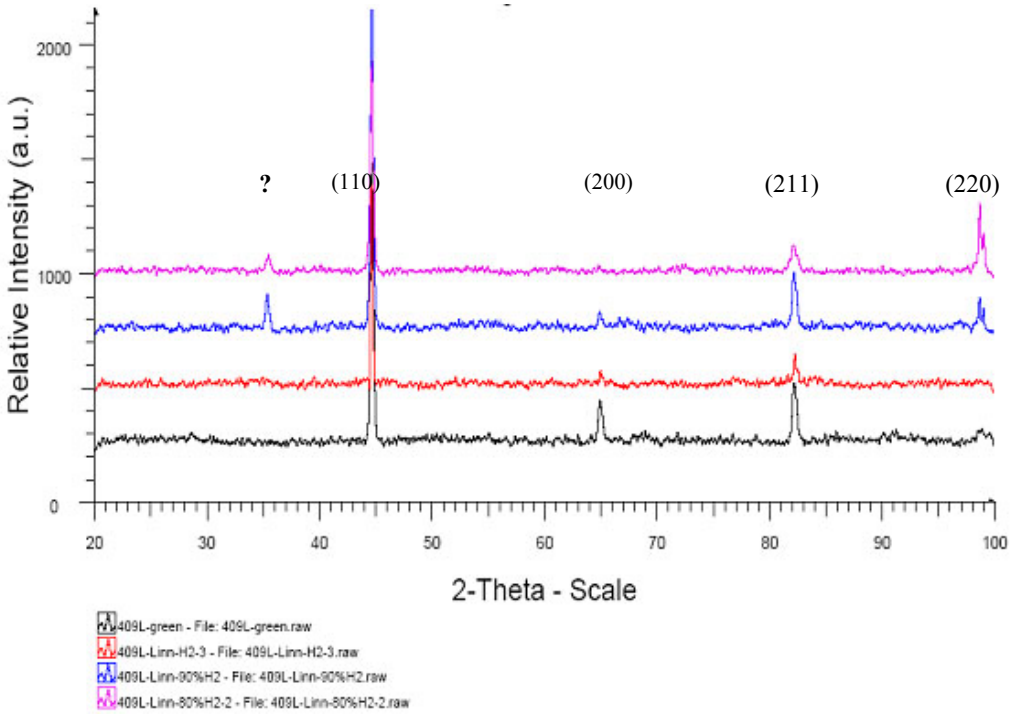
(c) 20 % N_2

Figure 6. Microstructures of the 409L stainless steel sintered in different atmospheres.

(3.6) Characterisation by XRD

Identification of XRD peaks obtained from different materials (green 409L stainless steel TTB, 409L stainless steel TTB sintered in 0%N_2, 409L stainless steel TTB sintered in 10%N_2 and 409L stainless steel TTB sintered in 20%N_2) is shown in Fig. 7. Nitrogen in the sintering atmosphere did not change crystallographic structure of the α-iron, which is the basic structure of the ferritic 409L stainless steel. Indexing of XRD peaks is given in Fig. 7. Although nitrogen, one of the austenitic structure stabilizers, was absorbed during cooling of the 409L stainless steel TTBs, it could not promote transformation from ferritic to austenitic structure.

There was an unidentified peak, designated as '?', appear at low 2θ (about 35 degree). The presence of this peak is not understood yet. Attempt to identified the peak was not successful.



The peaks (from bottom to top) are:

- Green TTB
- Sintered TTB-0% N_2
- Sintered TTB-10% N_2
- Sintered TTB-20% N_2

Figure 7. XRD peaks from TTBs of the 409L stainless steel.

Conclusions

The P/M 409L stainless steel gained better tensile strengths and hardness with scarified elongation when it was sintered in N_2-containing atmosphere. The sintered 409L stainless steel still exhibited elongation of about 15% when the atmosphere containing nitrogen content of up to 20% was employed. Better strength and hardness were attributed to hardening effect caused by nitrogen.

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