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Structural and Wear and Characteristic of Low Temperature Nitrided Stainless Steel

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Abstrak

Paper ini menjelaskan karakteristik struktur dan keausan baja tahan karat AISI 316 L hasil nitridisasi temperatur rendah menggunakan dapur perlakuan panas fluidized bed. Hasil penelitian menunjukkan, peningkatan ketahanan aus baja AISI 316 L berkaitan dengan terbentuknya lapisan expanded austenite (S phase) pada permukaannya dengan kekerasan mencapai ~1350 $HV_{0.5}$, hasil nitridisasi temperature 450 ℃ selama 6 jam. Observasi SEM menunjukkan, sampel uji keausan yang permukaannya tidak terbentuk S phase menampilkan jejak keausan besar akibat cabikan dan deformasi lokal. Dari penelitian ini disimpulkan, perlakuan nitridisasi temperatur rendah dibawah temperatur 500 ℃ terhadap baja tahan karat, untuk membentuk struktur lapisan permukaan S phase, dapat dilakukan menggunakan dapur perlakuan panas fluidized bed.

Kata Kuncl. *Baja Tahan Karat, Nitridisasi, Struktur dan Keausan*

Abstract

An investigation to structural and wear behaviour of nitrided AISI 316 L stainless steel resulting from low temperature fluidized bed nitriding has been made in the present work. It was found that the wear resistance of nitrided specimens was related to the formation of a precipitation-free hardened layer on the austenitic surface. In the present laboratory experiments, the precipitation-free or S phase layer with a surface hardness of ~1350 $HV_{0.5}$ was produced at a nitriding condition of 450 ℃ for 6h. The formation of this S phase layer significantly improved wear resistance of the stainless steel. Wear track observation by SEM revealed that the specimens without formation of S phase layer produced heavy scars due to tearing and local plastic deformation. The present work also suggests that fluidized bed heat treatment furnace can be utilised for nitriding the austenitic stainless steels at low temperatures below 500 ℃ to produce S phase nitrdid layer without losing the stainless feature of this material.

Keywords: *Stainless Steel, Nitriding Structure and Wear*

1. Backgrounds

Nitriding is a thermochemical treatment in which nitrogen is diffused into a steel surface to improve wear resistance and fatigue performance of the steel components. This surface heat treatment method is usually employed at temperatures 515-550 °C to the components made of low alloy steels composed of Cr, Al, Ti, Mo, to cause strengthening effect by the formation of alloy nitrides in nitrogen diffusion zone [1].

Austenitic stainless steel is Fe-Cr-Ni based alloys with Cr composition from 16 to 26 % and Ni minimum 8%. This is the most widely used among the stainless steel grades due to its good ductility and weldablity during fabrication along with its inherent corrosion resistance in acid solutions. This type of stainless steels is widely used in chemical and oil-gas industries as components for example: heat exchangers, piping, and tanks or containers. However, this material has poor tribological properties due to the inherent austenitic structure and low hardness. Earlier attempts on nitriding this material which aims to improve its surface hardness and thus enlarging a possible wider application resulted in appreciable loss of its corrosion resistance [2-4]. This phenomenon is caused by sensitivity effect, where, diffusional reaction in forming chomium nitride will lead to the depletion of Cr in the austenitic solid solution and consequently unable to produce Cr_2O_3 passive layer to make stainless feature. Since the mid of 1980's, attempts have been made to surface harden these materials without compromising their good corrosion resistance. These led to the development of the low temperature nitriding process [2,5-9], which is carried out at temperatures less than 500°C. At such low processing temperatures, it was found that the nitrided layer is free from chomium nitride precipitation, and is supersaturated with nitrogen in solid solution. Such a layer is termed expanded austenite (γ_N) [5,6] or S phase [5,7,10] by some investigators. Bell [11] argued that applying a low temperature nitriding can eliminate the formation of chomium nitrides but at the expense of strengthening effects made by CrN precipitates. Alternatively, the strengthening effect will be replaced by supersaturation of the interstitial nitrogen species in austenite matrix which leads to the hardening of the surface region several tens μm thick. This precipitation-free nitrided layer not only exhibits high hardness but also possesses good corrosion resistance due to the availability of retaining chomium in solid solution for corrosion protection.

The use of fluidised bed furnace in heat treating operation has been introduced elsewhere [12] that this type of furnace offers several advantages, including: faster treatment time, precise control of treatment parameters together with its economic benefits of low investment and operational costs. Nitrocarburising in the fluidized bed furnace produced thicker compound layers due to higher kinetic of nitrogen and carbon transfers compared to other conventional gaseous furnaces [13]. Previous works on low temperature nitriding was particularly based on plasma environment. Derived from the nitrocarburising experiments [13], low temperature nitriding of austenitic stainless steel using the fluidized bed furnace has been performed at temperatures between 400 and 500 °C [14]. The present work discusses structural charactersistics and wear behavior of the nitrided layers produced by low temperature nitriding in the fluidiesd bed atmospheres.

2. Experimental Method

The hot rolled AISI 316L austenitic stainless steel specimens with a dimension of 2 mm x 20 mm x 50 mm were ground and polished, followed by cleaning in concentrated HCL with the purpose to remove the native oxide film on the surface. The nitriding treatments were carried out at a constant gas composition of 25% NH_3 + 75% N_2 and subsequently the specimens were cooled in the furnace. The whole series of nitriding experiments are given in Table 1.

The nitrided specimens were characterised by surface microhardness measurements, and X-ray diffraction (XRD) analysis using Cu-Kα radiation with a vanadium filter. Scanning Electron Microscopy (SEM) was used to investigate the surface topography of the worn nitrided specimens and cross sectional pictures of the marbles etched nitrided specimens. TEM investigations were undertaken to the cross-sectional cut of the nitrided surface. The TEM specimens were prepared by mechanical thinning to a thickness of 0.08-0.1 mm, dimpling, followed ion bean thinning prior insertion into the electron microscope. A Gatan Model 691 Ion Polishing System was used to eliminate the difference in thinning rates between the nitrided layer and the substrate

Before the wear tests, the surface hardness of nitrided specimens were measured using a microhardness method. The wear tests were performed using a Pin-on-Disk tribometer at non lubricated condition with an applied normal load of 15 N, a sliding speed of 10 cm s^{-1} over a total sliding distance of 200 m. The pin radius used during the course of tests was 3 mm with a track radius of 5mm. The Wear rate of the specimens after the tests was calculated using the ASTM G 99.

3. Results and Discussion

The surface microhardness of nitrided stainless steel specimens is presented in Figure 1. It can be seen that the surface hardness of the steel is significantly improved after nitriding at 500 °C with 3 h and 6 h duration. An improved surface hardness of ~1350 $HV_{0.5}$ is also shown with the specimen nitrided at 450 °C for 6 h. The increase in the surface hardness of specimens nitrided at 500 °C can be explained due to a combined hardening effect by S phase and chomium nitride (CrN) formation as supported by X-ray diffraction data in Table 1. Whereas, in the case of specimen nitrided at 450 °C for 6 h, the hardness improvement on the stainless steel surface is caused by the formation of mono S phase layer. Nitriding at 400 °C did not produce hardened layer although the treatment was prolonged to 6 h. At a nitriding temperature of 450 °C, the precipitation-free nitrided layer, or S phase, began to form after 3 h. However, at this treatment duration the layer was very thin, and did not significantly increase the surface hardness of the stainless steel.

The TEM investigation to selected untreated and nitrided specimens corroborated the XRD results, which confirmed the formation of the S phase resulting from low temperature fluidised bed nitriding at 450 °C for 6h. Figure 2 shows micrograph and diffraction patterns of these two selected specimens. It can be seen that the diffraction pattern of S phase layer reveals substantial diffuse scattering at each spot compared to that of untreated AISI 316 L stainless steel with a fcc single structure of austenite.

Earlier work on anodic polarisation tests of the fluidised bed nitrided layers [14] indicated that the stainless steel surface nitrided at 500 °C exhibited higher susceptibility to the corrosion attack than the specimens nitrided at 450 °C. This evidence is in good agreement with the present X-ray diffracftion data given in Table 1, which indicates that nitriding at 500 °C leads to the formation of CrN precipitates resulting the sensitivity effect of the material. Cross sectional SEM micrographs of the specimens nitrided at 450 °C for 6h showing a ~30 μm thick S phase layer and 500 °C for 6 h showing a dual mixed S phase and CrN precipitates layer are presented in Figure 3.

The wear test results for various nitrided specimens are summarised in Table 2. Wear rates for a selection of specimens performing low wear rate during the tests is given in Figure 4, which demonstrate that the specimen nitrided at 450 °C with a 6 h duration exhibit a closely similar wear rate to those treated at 500 °C. Thus, the present works suggests that nitriding the austenitic stainless steels at a proper low temperature condition using fluidised bed furnace can improve surface hardness and wear resistance of the austenitc stainless steel without causing the sensitivity effect to the material.

SEM investigation on the wear track of the specimens treated at 450 °C for 6 h (Figure 5) revealed that the surface only suffered mild wear although the S phase layer were smeared by relatively high sliding friction. The untreated specimen, on the other hand, suffered heavy scars, identified as galling, which was due to tearing and local plastic deformation (Figure 6). The same tendency of a badly worn surface was also demonstrated by the specimen treated at 450 °C with only a 3 h duration. The very thin S phase layer produced from this treatment duration easily flaked off and thus causing severe wear during the test (Figure 7). The present Pin-on-Disk wear test also indicated that the formation of the S phase layer produced a low coefficient of friction on the steel surface (Figure 8). The coefficient of friction for this type of layer is significantly lower than those demonstrated by the layer containing dual mixed S phase and CrN precipitates as well as the untreated stainless steel substrate Therefore, it seems no doubt that low temperature nitriding by

Table 1.
X-Ray Diffraction Data of The Specimens After Nitridings.

Time (h)	400 °C	450 °C	500 °C	Untreated
1	335 x10^{-3} mm^3 m^{-1}	303 x10^{-3} mm^3 m^{-1}	216 x10^{-2} mm^3 m^{-1}	348 x10^{-2} mm^{3}m^{-1}
3	323 x10^{-3} mm^3 m^{-1}	236 x10^{-3} mm^3 m^{-1}	81 x10^{-2} mm^3 m^{-1}	
6	364 x10^{-3} mm^3 m^{-1}	64 x10^{-3} mm^3 m^{-1}	53 x10^{-2} mm^3 m^{-1}	

Table 2.
Wear Rate of Various Nitrided Specimens

Time (h)	400 °C	450 °C	500 °C
1	γFe,Ni (111) (200) (222)	γFe,Ni (111) (200) (222)	γFe,Ni , [S1, S2, S3 (weak)]
3	γFe,Ni (111) (200) (222)	γFe,Ni, [S1, S2, S3 (weak)]	γFe,Ni, [S1, S2, S3, (strong)] CrN
6	γFe,Ni (111) (200) (222)	γFe,Ni, [S1, S2, S3 (strong)]	γFe,Ni, [S1, S2, S3, (strong)] CrN

formation of a single S phase nitrided layer provides an advantage of reducing friction problems when the steel is to be exposed to sliding wear condition.

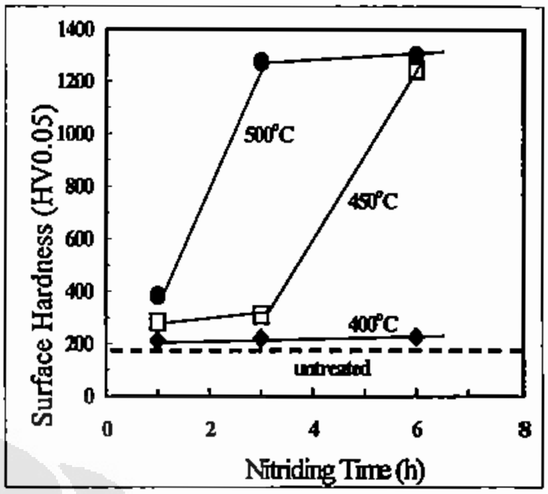
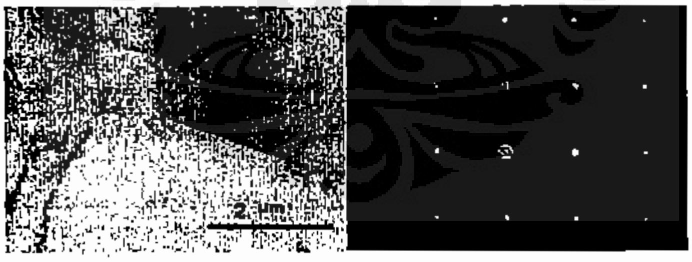


Figure 1.
Surface Microhadness of The Nitrided Specimens.



(a)

(b)

Figure 2.
(a) TEM Micrograph and Diffraction Pattern of Untreated AISI 316 L Stainless Steel
(b) TEM Micrograph and Diffraction Pattern of The S Phase Revealed From Specimen Nitrided at 450 C for 6h and its Diffraction Pattern

(a) (b)

Figure 3.
SEM Micrographs of Specimens Nitrided With (a) 450 C, 6h, and (b) 500 C, 6 h

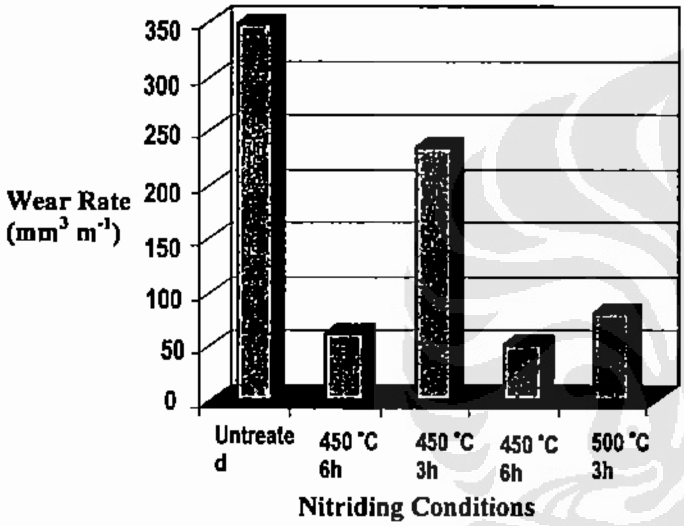
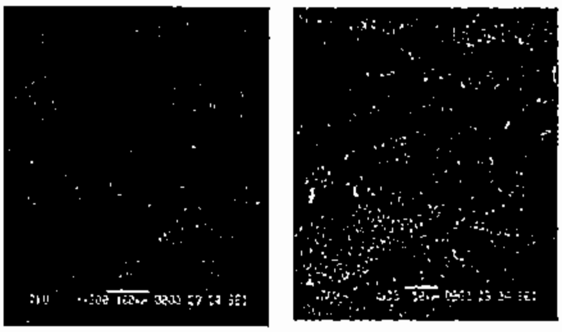
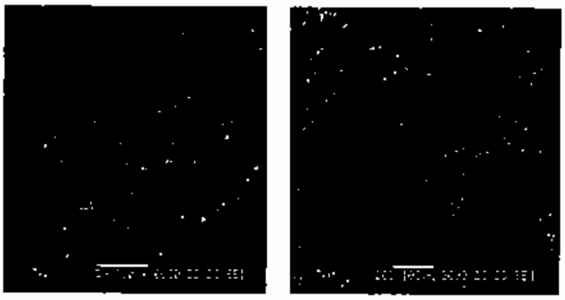


Figure 4.
Wear Rates For a Selection of Nitrided Specimens



(a) (b)

Figure 5.
Wear Track of Specimen Nitrided at 450 °C For 6 h, (a) Low and (b) Increased Magnifications



(a) (b)

Figure 6.
Wear Track of Specimen Nitrided at 450 °C for 3 h, (a) Low and (b) Increased Magnifications

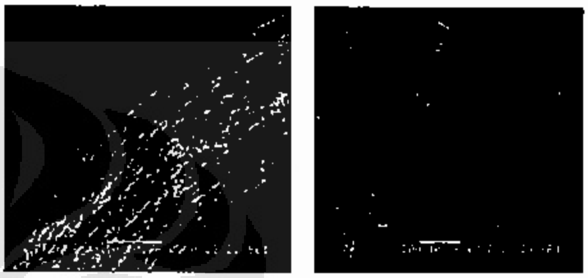


Figure 7.
Wear Track of Untreated Specimen, (a) low and (b) Increased Magnifications

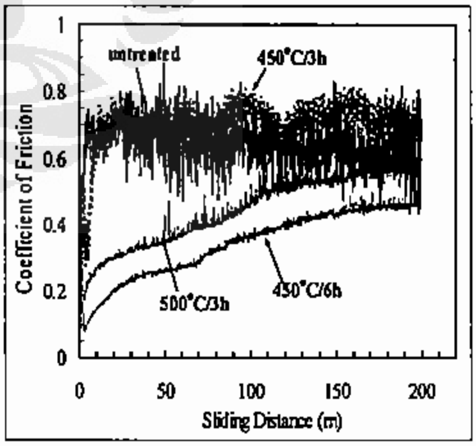


Figure 8.
Coefficient of Friction For a Selection of Nitrided and Untreated Specimens Measured During Pin-on-Disk Wear Test.

4. Conclusions

The present work has demonstrated that a low temperature fluidized bed nitriding process can be used to improve the surface hardness of austenitic stainless steel through the formation of a 30 µm thick precipitation-free nitrided layer with a surface hardness of ~1350 $HV_{0.5}$. The layer has low coefficient of friction and exhibits much improved wear resistance than the untreated stainless steel surface, thus, offering wider applications of the austenitic stainless steels.

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