Asian Journal of Scientific Research

ISSN 1992-1454

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Asian Journal of Scientific Research 6 (2): 323-330, 2013
ISSN 1992-1454 / DOI: 10.3923/ajsr.2013.323.330

The Influence of Nitriding Time of AISI 316L Stainless Steel on Microstructure and Tribological Properties

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ABSTRACT

An investigation on microstructure and tribological properties of low temperature nitriding on 316L austenitic stainless steel with varies time treatment has been made in the present work. The improvement of wear resistance and surface hardness of nitrided steel were identified related with the increasing of layer thickness due to the extended time of treatment, where the hardness significantly improved with 754 $Hv_{0.01}$ after 8 h treatment as well as the tribological properties. The nitrided layer properties improvement in this investigation shows that the wear mechanism was confirmed by the results of coefficient of friction measurements that were obtained from pin on-disc wear test ranging averagely from 0.3-0.5 on 8 h nitrided sample compared to untreated (1.4-2.0) and 2-5 N (0.6-0.8) with shorter nitrided time, respectively. This is agree with theoretical approach where minimum value is achieved and represents high wear resistance. Surface morphology were characterized by using FESEM and the micrograph of worn region showed that untreated 316L experienced severe wear, while nitrided steel presented only slight abrasion with shallow and narrower wear track.

Key words: Structural properties, characterization, expanded austenite, wear resistance, sensitization

INTRODUCTION

Austenitic stainless steel type is widely used in the engineering application because it has excellent oxidation and corrosion resistance. However, there are some limitation in industrial application for this type of steel where exposing wear mechanism, for instances like camshafts, cam followers in automotive parts and also in chemical and oil and gas industries. The introduction of nitrogen for thermal diffusion process such as nitriding treatment is recognized where the treatment could improve the wear resistance and surface hardness of the steel during the operation (Triwiyanto *et al.*, 2009).

Austenitic stainless steel wear mechanism begins when the protective oxide layer is break and active boundary lubrication is poor (Ashby and Jones, 2005). However, AISI 316L stainless steel become the chosen material in the engineering industry because of its good ductility, weldability and better corrosion resistance. Due to the inherent austenitic structure, this material is poor due to the effect of wear and low hardness, which result in poor tribological properties (Subbiah and Rajavel, 2005). One of the approaches to improve wear resistance and surface hardness of the steel is by nitriding treatment where its offer the benefits of high dimensional stability (Haruman *et al.*, 2006).

Nitriding is a surface hardening technique by the diffusion of nitrogen into the surface layers and the change of chemical compositions of the steel (Elgun, 2012). The new phase compositions also known as S-phase and formed nitrided layer (Toshkov *et al.*, 2007) with the term of expanded austenite γ_N (Subbiah and Rajavel, 2005). This phase is characterized with its good mechanical properties and acceptable corrosion resistance (Hamdya *et al.*, 2011). In order to accelerate diffusion on austenitic stainless steel, the steel will be treated at relatively high temperature, about 570°C (Bell and Li, 2002).

However, the formation of chromium nitride/carbide could occur during the diffusion at high temperature. As the results, chromium nitride/carbide might be precipitated into the grain boundary and the passive layer , which is chromium oxide, Cr_2O_3 will unable to be produced and reduced the corrosion resistance property of stainless steel (Triwiyanto *et al.*, 2009). This phenomenon is known as sensitization effect. The effort to avoid the sensitization effect relatively with low temperature treatment, according to Zhang and Bell (1985). They have investigated the low temperature nitriding technique where it was found that a nitrided layer was able to form on surface of AISI 316L with plasma nitriding technique. The thickness of the layer could be formed up to 20 μm at temperature around 400°C. The characteristics of the layer show that it has very high hardness and excellent wear resistance, as well as very good corrosion resistance Zhang and Bell (1985).

Time variables of treatment also could be analyzed in this low temperature nitriding treatment. The case depths or thickness of the nitrided layer become the relation with the time variables (Haruman *et al.*, 2006). There are different case depth could be identified under the nitrided layer morphology. Moreover, the time variables may bring us to have the understanding in the improvement of surface hardness for each different time of treatment. For gas nitriding treatment, ammonia gas can be used as the gas resource for nitriding. Nitrogen is introduced into the surface steel by holding the metal at a suitable temperature in contact by ammonia. Thus, ammonia will disassociate into gas into hydrogen and nitrogen on the surface steel (Mridha, 2006). Nitrogen then diffuses from the surface into the core of the material at the certain temperature range.

The typical wear mechanism characteristics for austenitic stainless steels sliding against steels are adhesive and abrasive wear mechanism (Li and Bell, 2004). During nitriding steel, previous investigations done by Li and Bell (2004) and Hashemi *et al.* (2011) showed that the characteristics of wear mechanism through the surface morphology analysis were shallow, narrower and superficial wear track, as well as abrasive wear which was dominated on the surface. This is due to the formation of layer that provide a convenient support to the protective layer of oxides, which may introduce an increasing of wear resistance (Gallo, 2009).

This study describes characteristics of austenitic stainless steel 316L nitrided by low temperature thermochemical treatment with different treatment durations.

MATERIALS AND METHODS

The material used is AISI 316L stainless steel and the steel supplied in the form of rod with the diameter of 50.3 mm and thickness of 40 mm. Then the samples of pin and disc are cut into 50×6 mm for disc and 6×12 mm for pin. Those four pins and discs are required for undergo the pin on-disc wear test as be standardized by ASTM G99-95a. Then all the samples surface are ground on 120, 220, 500, 800, 1000, 1200 grit SiC papers and then polished using 1 μm Al_2O_3 pastes to the mirror finish, followed by cleaned using ultrasonic cleaning and immersed in HCl (2 M) solution for 15 min duration to remove the native oxide film that commonly forms on austenitic stainless steel.

Table 1: Sample classification with different treatment

Sample	Type	Treatment
UN	Pin	Untreated
	Disc	Untreated
2N	Pin	2 h nitriding
	Disc	2 h nitriding
5N	Pin	5 h nitriding
	Disc	5 h nitriding
8N	Pin	8 h nitriding

Nitriding treatments were performed at 450°C in a Carbolite CTF Tube Furnace. The specimens were heated by electrical resistance heating. Prior to treating, the specimens were soaked in concentrated HCl (2 M) solution for 15 min duration with the purpose to remove the native oxide film that commonly forms on austenitic stainless steel and protects the metal matrix from corrosion. This oxide layer is believed to act as a barrier for diffusional nitrogen transport.

Furthermore, the ammonia gas will be purged together with nitrogen. The amount of ammonia and nitrogen gas is set to be 50% or 0.3 Standard liter per minute (0.3 SLPM) each. Those gases will mix in the mixing chamber before purge together into the tube furnace. After nitriding treatment, pin on-disc wear test at dry sliding condition is proceed according to the standard; ASTM G99-95a. Ducom TR-701-M6 Multi Specimen Tester machine is used. The load values for the test are 17 N with the speed of 75 rpm and sliding distance of 300 m (for 30 min time of operation). The temperature is the room temperature and the atmosphere is set to be the laboratory air. Characterization of the nitriding product were performed by using a wide range of instruments. Wear resistance required data of coefficient of friction from the wear test. Vickers hardness test instrument; Model HV-1000A Micro Hardness Tester with 10 gf load and 15 sec well time is used. The thickness of nitrided layer for each samples is measured using Field Emission Scanning Electron Microscope (FESEM) instrument; Carl Zeiss AG-SUPRA 55VP. The cross-sectional samples are required using standard metallographical technique and Marble's etching (4 g $CuSO_4$+20 mL HCl+20 mL distilled water). Surface morphology of worn region also is investigated by FESEM instrument. Table 1 presents the classification of samples with different treatments.

RESULTS AND DISCUSSION

Wear resistance analysis: As shown in Fig. 1 which is the comparison results of coefficient of friction vs. time profile, UN sample yield the highest coefficient of friction, which averagely ranging from 1.4-2.0. Then, the coefficient of friction followed by the sample of 2N with 0.8-1.4, 5N with 0.6-0.8 and 8 h 0.3-0.5 nitrided steel. Theoretically, lowest coefficient of friction gives highest wear resistance during wear mechanism on the surface. The presence of nitrided layer on the samples of 2, 5 and 8N could be explained that the formation of nitrided layer provide a convenient support to the protective layer of oxides, which may introduce an increasing of wear resistance (Gallo, 2009). According to investigation by Subbiah and Rajavel (2005), formation of nitrided layer on steels is increased in thickness as the time of nitriding treatment is extended. This is why the sample of 8N achieved high wear resistance.

Microhardness measurement: From Fig. 2, surface hardness of the nitrided steel is significantly improved from 2-8 h nitriding treatment. The 2N sample formed nitrided layer with maximum hardness of about 480 $Hv_{0.01}$, which is much lower than the hardness of 720-754 $Hv_{0.01}$

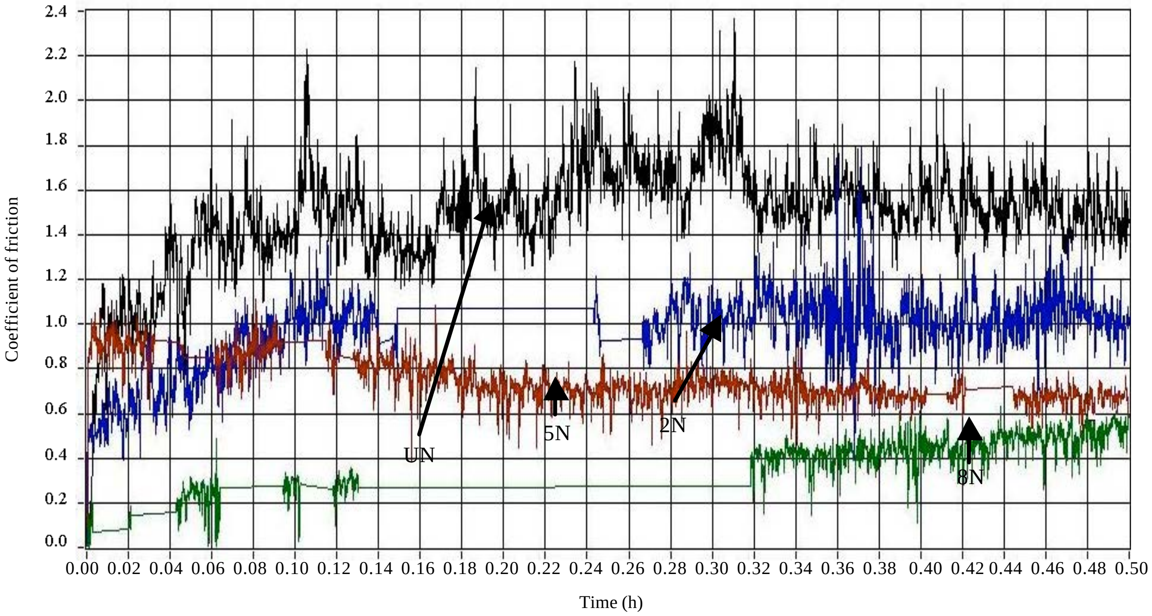

Fig. 1: Coefficient of friction vs. time profile for UN, 2N, 5N and 8N samples

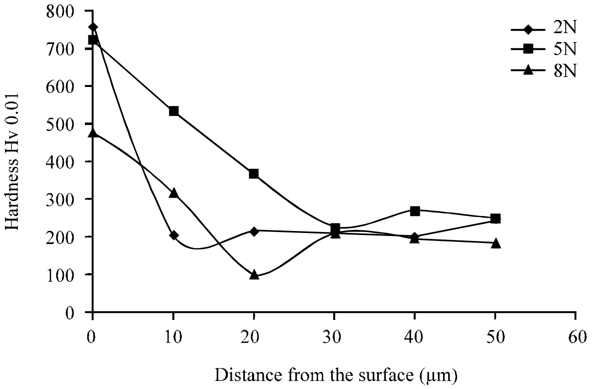

Fig. 2: Depth profiles of microhardness

for other two nitrided samples. The increasing of surface hardness can be explained due to the extending of time treatment where nitrogen atoms diffused in the surface become more density in the nitrided layer.

Theoretically, formation of nitrided layer due to precipitation-free diffusion layer by nitrogen supersaturated, which is normally, knows as S-phase (Toshkov *et al.*, 2007). This supersaturation of nitrogen in the austenite will cause the expansion of the lattice of the substrate austenite (Triwiyanto *et al.*, 2009). Thus, a new type nitrite phase (S-phase) as a nitrided layer provides extremely high surface hardness.

Nitrided layer morphology: From Fig. 3(a-c), thickness of nitrided layer formed on the 2, 5 and 8 N samples were measured by Field Emission Scanning Electron Microscopy (FESEM)

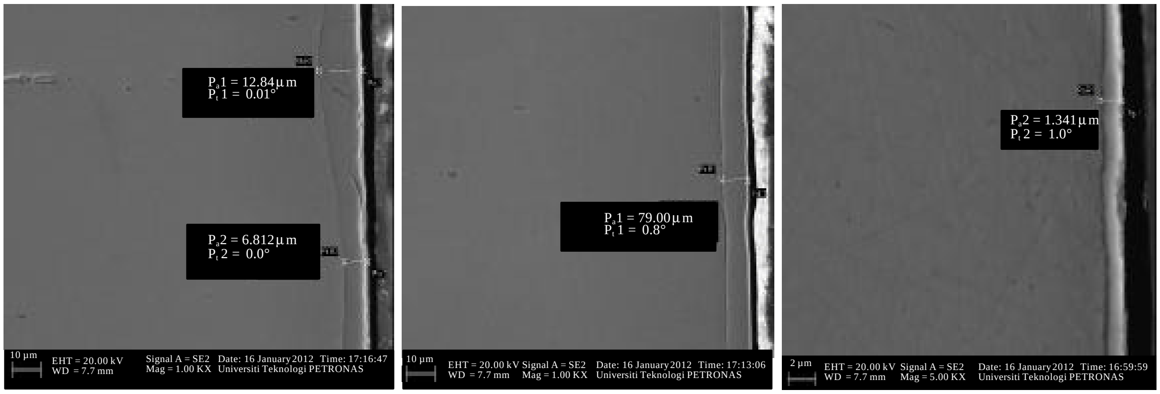

Fig. 3(a-c): Nitrided layer morphology of (a) 8N (b) 5N and samples at 1000x magnifications and (c) 2N samples at 5000x magnifications

instrument at 1000-5000x magnifications. The 8N samples formed highest thickness of nitrided layer with 12.84 µm, followed by 5N with 7.93 µm and 2N with 1.34 µm. Fig. 3(a) showed that nitrided layer formed on 8N samples was not uniform. However, for 2N and 5N samples in Fig. 3(b-c), the layers are uniform. The characteristic of gas nitriding technique more likely to occur with irregular diffusion due to the gas accumulating near to the tempered surface steel and diffuse instead of accelerating directly diffuse on the surface. This might be the reason regarding non-uniform layer formed on the surface steel.

From the image of FESEM shown on the 3c, the thin nitrided layer was formed on the surface after 2 h nitriding with the improvement of surface hardness, which can be referred in Fig. 2 for depth profile of microhardness. Thus, the thin nitrided layer begins to support the protective oxide layer on this sample and resist the wear action (Triwiyanto *et al.*, 2009). However, the mechanism of the abrasive and adhesive wear still took place on the sample slightly compared to untreated sample.

Surface morphology: For UN sample, the large plastic deformation is obviously observed on the worn region which is shown in Fig. 4a-b. The morphology of the worn region for UN sample also showed the deep plow with plate-like wear debris. The deep plow is explains the wear severely occurred on the sample by abrasive mechanism, while the plate-like wear debris are due to the adhesion wear mechanism, where the material or wear debris (particles) transferred on the surface and would then be plastically deformed and compacted by the rubbing action between the slider and the disc (Li and Bell, 2004). For 2 N sample, the morphology characteristic could be obviously seen in Fig. 4c which is 100x magnification where it appears to be less worn and the shallow plow built up on the surface. This indicates that abrasive wear also experienced on the sample but not severely compared to untreated sample. Figure 4d with 500x magnification, the shallow plow and plate-like wear debris built up on the surface clearly seen. In Fig. 4e, the worn region morphology at 40x magnification for 5 h nitrided sample only observable with several surface digging and also with narrower and superficial wear track (Li and Bell, 2004; Hashemi *et al.*, 2011). The structure seems that the mechanism occurred is slight abrasive wear. This could be strongly support by analyzing the morphology in 500x magnification in Fig. 4f. The spot taken on the surface digging

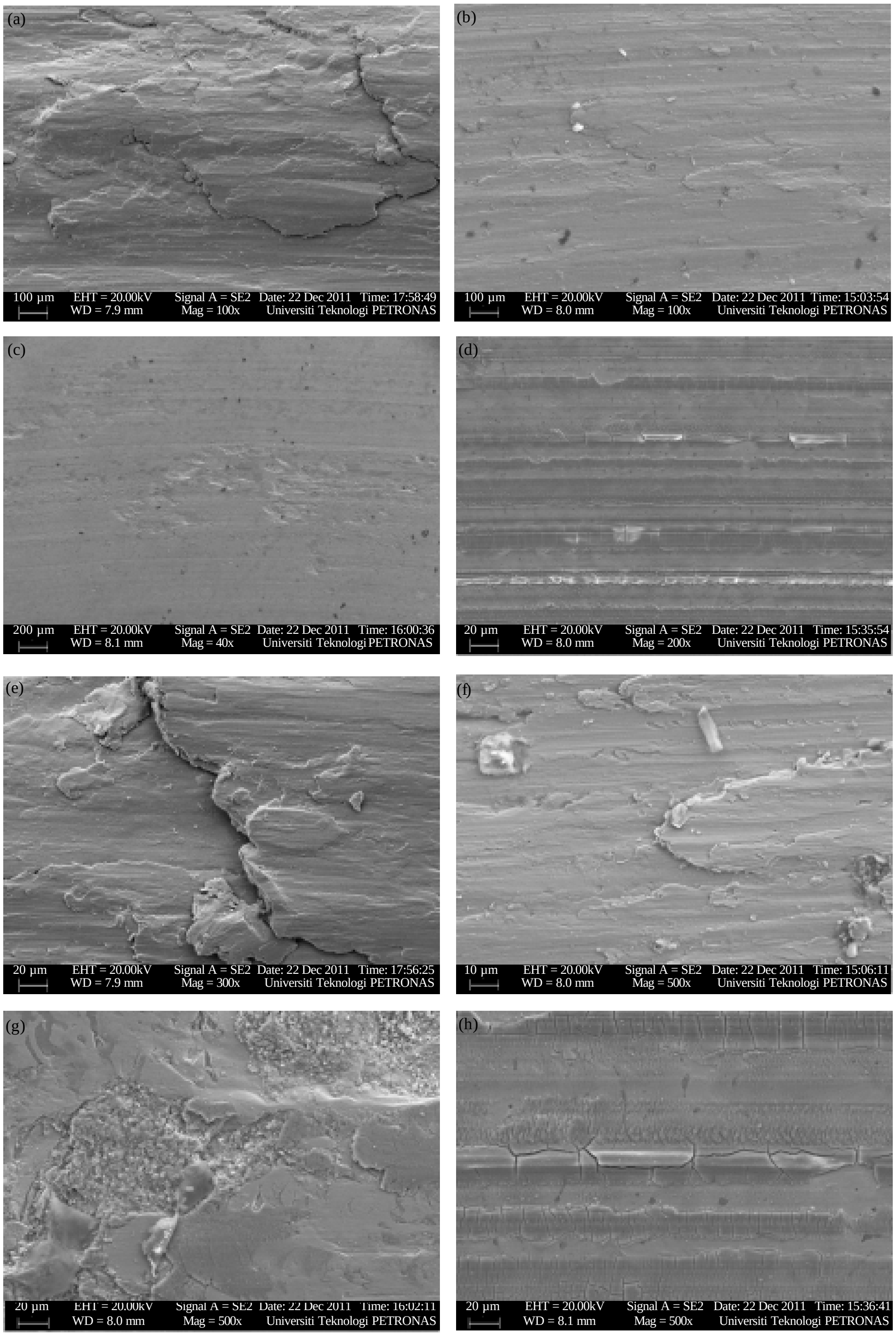

Fig. 4(a-h): Worn morphology of UN at (a) 100x and (b) 300x magnification, Worn morphology of 2N at (c) 100x and (d) 500x magnification, Worn morphology of 5N at (e) 40x and (f) 500x magnification and Worn morphology of 8N at (g) 200x and (h) 500x magnification clearly had shown the abrasion of the samples. This is the characteristic of nitrided steel where only abrasive mechanism dominates the wear process (Li and Bell, 2004; Liang *et al.*, 2000; Li *et al.*, 2012). The thicker nitrided layer formed on this sample combining with protective oxide layer prevent from intimate contact and adhesion on the surface. This is why only abrasive mechanism took place. From the image of wear track obtained on this sample at 200-500x magnifications in Fig. 4g-h show that there was some crack occurred on the worn region. However, the mechanism of the abrasive and adhesive wear still took place on the sample slightly compared to untreated sample.

Supposedly, the wear mechanism characteristic for 8 h nitrided steel after wear test similar with 5 h treatment, which is shallow, narrower and experiencing and slight abrasion. However, from the result of wear track obtained on this sample at 200-500x magnifications in Fig. 4(g-h) show that there was some crack occurred on the worn region.

This is possibly due to the nitrided layer formed on this sample was not uniform, which can be seen on the Fig. 3 previously, in the nitrided layer morphology result. Thus, morphology of this nitrided layer leads to the crack when the slider (pin) slide on the uneven nitrided layer and hit the thicker layer.

CONCLUSION

Wear resistance and surface hardness of nitrided AISI 316L stainless steel is significantly improved through the low temperature gas nitriding treatment with 2, 5 and 8 h time variables. Eight hour treatment achieved the lowest value of coefficient of friction result, which was 0.3 and gave high wear resistance. Meanwhile, maximum surface hardness achieved was 754 $Hv_{0.01}$ after 8 h treatment. These improvements are due to the formation of nitrided layer on the treated steel that successfully formed during low temperature nitriding gas treatment. The maximum thickness layer formed was 12.84 μm at 8 h treatment. Moreover, the formation of nitrided layer also supported the protective oxide layer during the wear mechanism on the surface, where the in contact surface only experienced slight abrasion with shallow and narrower wear track.

ACKNOWLEDGMENT

The authors would like to thank to Universiti Teknologi PETRONAS for financing these findings in the International Conference on Plant Equipment and Reliability 2012.

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