
Morphological and wear behavior of low temperature thermochemical gaseous nitriding process

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HIGHLIGHTS

- Hard particles of γ N, CrN and Fe_3N phases were produced after low temperature gaseous nitriding
 - The surface hardness of nitride layer increased from 400.5 $HV_{0.5}$ to 513.3 $HV_{0.5}$ after gaseous nitriding
 - The thickness of the nitride layer on duplex stainless steel was about 32.7 μm to 60.5 μm .
 - The wear resistance of nitride layer duplex stainless steel was excellent than that of the untreated DSS
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ABSTRACT

In this study, the nitride layer based on incorporation of nitrogen diffusion via low temperature thermochemical nitriding process were prepared. Nitriding temperature, holding time and gas flow rate during nitriding process was used as the parameters to assess morphology and wear behavior. Nitriding holding time was varies for 4, 8 and 12 hours, meanwhile temperature and glass flow rate were keep constant. The thickness and hardness of nitride layer varied with the holding time with the thickest layer of 60.5 μm and surface hardness of 513.3 $HV_{0.5}$. The nitride layers, containing expanded austenite, Fe_3N and CrN phases, as well as solid solution in the stainless steel were developed during low temperature thermochemical gas nitriding process. The expanded austenite formation on the nitride layers demonstrates that the best wear behavior due to the highest hardness. The wear rate and CoF improved by 0.40 and 0.37 compared to untreated DSS, respectively. Furthermore, it was revealed that the worn surface showed very mild striation of wear for the 12 hours compared to 4 hours nitrided sample which demonstrated deeper grooves on the shorter nitriding process. It was demonstrated that nitriding process is capable and feasible to produce hard surface layer on DSS with higher hardness and wear resistance properties which can be used for tribological applications.

Keywords:

| Thermochemical | nitriding | Morphological | Hardness | wear resistance |

1.0 INTRODUCTION

Thermochemical process via diffusion mechanism is mainly applied to steels and consist of enriching the surface of the material to be treated with other element such as gaseous compound containing of nitrogen, carbon and boron gaseous. These gaseous are

supplied and deposited to the surface of material via diffusion mechanism. Generally, the treatment is heated to a temperature ranging from 500 °C to 1000 °C to diffuse on the surface of material. Steels that have been through this treatment are characterized by good resistance to adhesive wear, abrasion and fatigue (Takadoun, 2008). The main advantages of this process is the hardening of the entire surface of the treated sample. However, the process must be controlled and ensure the process temperature is not too high to avoid the reduction of corrosion resistance. Several researchers have explored the opportunity to perform this process in the past (Haruman et al., 2006, Maleque et al., 2019, Tamil-Moli et al., 2016, Paijan et al. 2012 and Liang et al. 2018).

The improvement of the surface layer with the presence of expanded austenite provide better surface hardness and wear resistance without decreasing the corrosion resistance. The hardening of the nitride layer is due to incorporation of nitrogen in the austenite lattice, forming expanded austenite. The growth of expanded austenite has been studied by previous researchers using various surface treatments by nitriding, carburizing and nitrocarburising. Fewell et al., (2000) had explored that the expanded austenite is the crystallite structure and not mixed phase with some grains having different lattice parameters from others.

The most popular technology used to produce expanded austenite is plasma technology, namely plasma nitriding, plasma carburizing and plasma hybrid as reported by the previous research (Haruman et al., 2012). However, this process require high cost for establishment compared to gaseous nitriding using tube furnace. Therefore, the approach for other alternative method is needed with simple and cheaper operation. In order to meet this purpose, employing a low temperature of thermochemical gaseous surface treatment is a possible method to improve tribological properties and wear resistance of the material. This has been proved by previous researchers (Christiansen & Somers, 2005; Christiansen & Somers, 2006; Sun & Haruman, 2008; Wang et al., 2008 and Maleque et al. 2017). Most of the finding was correlated well and have a strong influence with the processing parameters on the outcome of the surface modification treatment. The combination and compatibility of process time, temperature and gas composition are important to produce expanded austenite without precipitation of chromium nitride in nitride layer and chromium carbide in carbide layer. Baranowska (2004) also demonstrated that the formation and growth of the surface layer depends on the relationship between the nitrogen supply and nitrogen demand. In this case, nitrogen demand mainly depends on the diffusion process (temperature and holding time).

Tamil-Moli et al., (2016) analyzed the effect of low temperature gaseous nitriding of AISI 4140 alloy steel on hardness properties. The results found that the surface hardness has increased due to diffusion of nitrogen into steel and formed a nitride layer. Surface hardness of the samples enhanced with the increment of nitriding temperature and holding time in the range of 250–300 $HV_{0.5}$. The highest thickness of nitride layer with a value of 438.6 μm was recorded when the sample was nitrided at 550 °C for 60 minutes.

There are another low temperature treatment for surface modification namely cryogenic treatment. In this process, the treatment is carried out below the sub-zero temperature until -180°C. It is increasing wear resistance, dimensional stability, fatigue behavior, hardness, machinability, microstructure, residual stress and toughness of steel material. Previous research by Cicek et al. (2015) demonstrated that the cryogenic treatment placed in nitrogen gas at -145°C for 24 hours on AISI H13 tool steel resulted in

improvements in the wear resistance and mechanical properties. It is found that the improvement was contributed from the formation of small-sized and uniform distribution of carbide particles and transformation of retained austenite to martensite microstructures. Other research work by Kara et al. (2021) found out that deep cryogenic process exposed at lower temperature of -180°C for 24 hours on Sleipner high alloyed tool steel has increased the macro hardness about 30 HV with more homogeneous microstructures at the inner structures of the steel and reduce the residual stresses on the surface material. However, the caution need to be addressed on this process especially on the thermal shock that may contribute to the microstructural changes resulted in decrease of hardness and material deterioration.

Although many works have been made to improve the tribological behaviour of stainless steel, several possibilities remain unexplored. Among them is the incorporation of nitrogen atom on the duplex stainless steel in the atmosphere environment with addition of ammonia and nitrogen gaseous at different nitriding holding time. Accordingly, this study takes on the task of examining the possibility of using tube furnace with addition of nitrogen atom to improve the wear properties of duplex stainless steel.

2.0 MATERIALS AND METHODS

2.1 Materials

In the present work, the experimental substrate was duplex stainless steel (grade of AISI Duplex-2205). The chemical composition of this material is given in Table 1. The specimen size was cut into smaller size of 50 mm x 33 mm and 10 mm using EDM wire cut. The samples were ground using SiC papers ranging from 240 to 1200 grade. Then, the specimens were immersed in concentrated hydrochloric acid (2M) for 15 minutes. This procedure is carried out to remove native oxide film on the surface of material to promote the effectiveness of the nitrogen diffusion during the nitriding process. After immersion, the samples are placed onto a quartz boat with vertical position and inserted into the tube furnace immediately. The sample is put in the middle of the tube furnace, to ensure the sample obtain the optimum heating zone.

Table 1: Chemical composition of duplex stainless steel (AISI Duplex-2205)

Steel grade	Chemical Composition %						
	C	Cr	Si	Mo	Mn	S	Fe
AISI Duplex-2205	0.026	22.06	0.69	2.58	1.74	0.008	Balance

2.2 Thermochemical gaseous nitriding process

A horizontal tube furnace system was performed for thermochemical low temperature gaseous nitriding process. Figures 1 and 2 show the tube furnace for nitriding process and schematic diagram of the nitriding experimental setup, respectively. Meanwhile, the example of nitriding heat cycle treatments at 450°C for 4 hours is shown

in Figure 3. The system in the was purged to about 30 minutes using nitrogen gas to remove any contaminants in the furnace environment. After that the ammonia gas supply was generated by opening the valve of the gas piping flow. When ammonia gas (NH_3) was introduced, the system commenced to heat up the temperature required. During the heating process, the ammonia gaseous decomposed into nascent nitrogen and diffused to the surface sample. The nitriding temperature for this experiment was keep constant at 450°C for different holding time of 4, 8 and 12 hours. The gas flow rate of NH_3/N_2 was supplied consistently to the system with $0.5 \text{ NH}_3/\text{N}_2$. Finally, the nitrided samples placed in the system were cooled down to room temperature.



Figure 1: Tube furnace for nitriding process

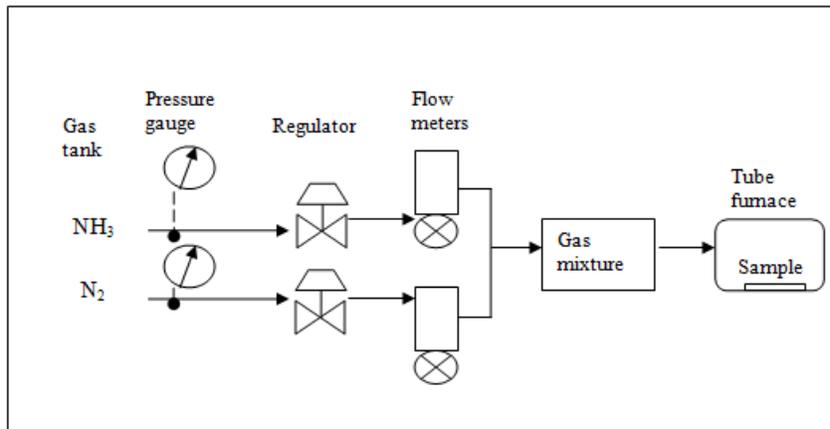


Figure 2: Schematic Diagram of the Nitriding Process Experimental Setup

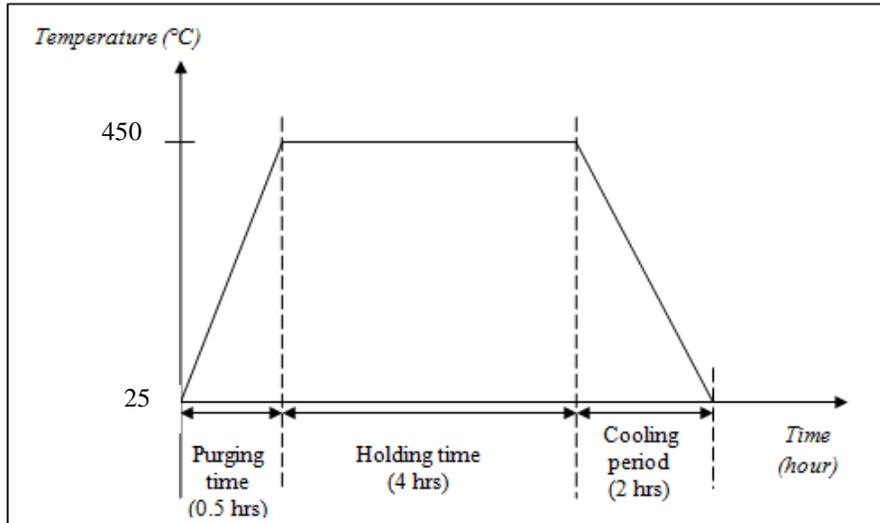


Figure 3: Nitriding Heat Cycle Treatments at 450 °C for 4 hours

2.3 Microstructural characterization and micro-hardness profile

The nitrided sample was cut into cross-section to examine the quality of the thermochemical gaseous nitriding process at different nitriding holding time. The cross-sectional sample was manually ground, polished and etched to reveal the morphology of the nitride layer. Then, the sample was examined by using scanning electron microscopy to observe and measure the thickness of nitride layer. The phase composition was determined through X-ray diffraction (XRD) with Cu-KU radiation for diffraction angle from 35° to 90°. Furthermore, the micro-vickers hardness was conducted for hardness profile measured from the surface towards substrate material using a weight of 500 gf load, pyramid diamond indenter and 10 second indentation time. The hardness measurement was carried out using Vickers hardness tester based on ASTM E38.

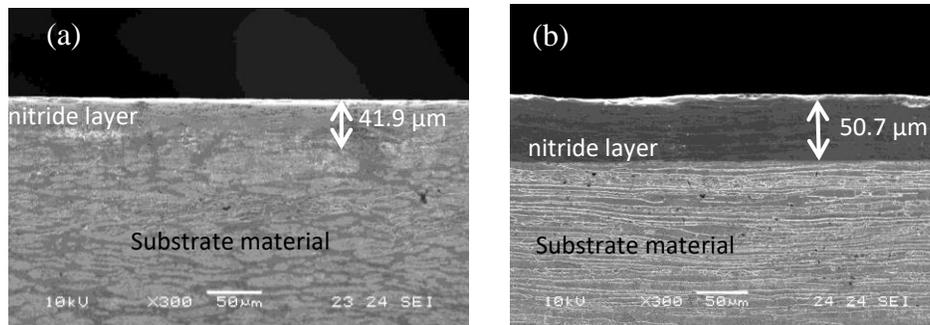
2.4 Reciprocating Wear Testing

The reciprocating dry wear test was used to assess the surface performance of the nitride layer using alumina ceramic ball. This test method was according to ASTM D6079. An alumina ceramic ball with 6 mm diameter was chosen as a counter-body due to high hardness of this material (2000 HV_{0.5}). The nitrided sample (dimension of 15 mm × 15 mm × 6 mm) was placed to the steel holder to grip the sample while for alumina ceramic ball was fixed to the ball holder. The tests were performed at a constant load of 30 N for 10 minutes, moving frequency of 5 Hertz, a stroke 10 mm and ambient temperature of 23 ± 2 °C. To ensure the accuracy of the results, the wear tests with the same experimental parameters were carried out at least three times. After wear test completed, the weight loss of the sample was measured using weight balance to determine the wear rate of the sample. The winducom software from the reciprocating wear test equipment was produced the result directly for coefficient and friction value. Moreover, the SEM micrographs of the wear tracks were analyzed using scanning electron microscopy.

3.0 RESULTS AND DISCUSSION

3.1 Morphological and structural aspects of the nitride layers

Surface layer cross-section micrographs obtained after thermochemical gaseous nitriding process is shown in Figures 4 (a-c). Low temperature gaseous nitriding has developed a thin nitride layer along the sample surface of DSS. The nitride layer thickness increased gradually from 32.7 μm , 50.7 μm to 60.5 μm with the increasing of nitriding period from 4, 8 to 12 hours, respectively. At longer nitriding holding time, the nitride layer thickness increased, might be related to the higher nitrogen concentration on the substrate surface and continuous diffusion of nitrogen atom on the sample with nitriding holding time. From the results, it can be seen that the nitriding holding time have strong influence on the thickness of nitride layer. It can be seen that the nitride layer increased with the longer nitriding holding time has occurred similarly to that obtained by previous research on grey cast iron using gaseous nitriding process (Wang & Liu, 2013). However, it can be observed that there is a formation of dark phases namely chromium nitride (CrN) for sample nitided for 12 hours as shown in Figure 4(c). The disadvantage of this CrN formation is that it can cause a decrease in corrosion resistance. The relationship between the formation of the dark phases and the precipitation of chromium nitrides in the nitride layer is confirmed by X-ray diffraction analysis in Figure 5 (d). Similar finding by Sun & Haruman (2008) also demonstrated that the increasing treatment time to 12 hour and 24 hour at 420 $^{\circ}\text{C}$ leads to the formation of a small amount of CrN in the alloyed zone of AISI 321 stainless steel with dark phases.



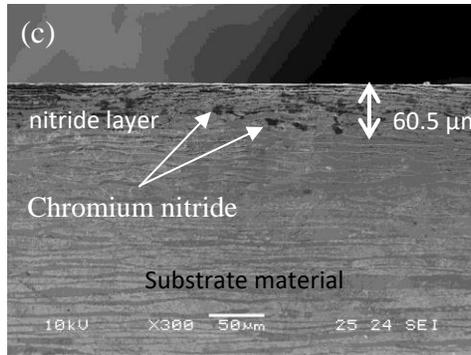
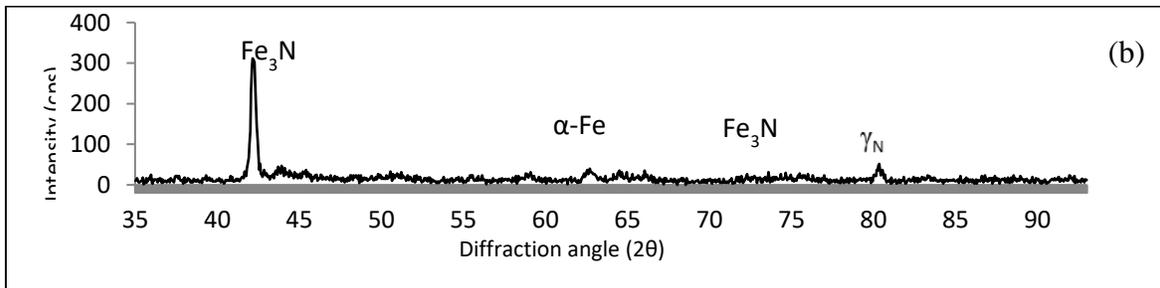
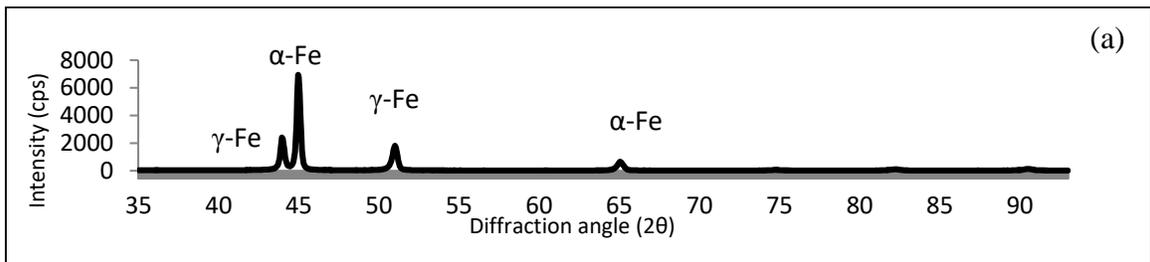


Figure 4: SEM images for surface nitride layer treated at 450 °C for (a) 4 hours (b) 8 hours and (c) 12 hours

Figures 5(a-d) shows the XRD spectra of the untreated and three nitrided DSS for thermochemical gaseous nitriding processes. It can be observed that the untreated samples exhibited of four peaks consist of α -Fe (ferrite) and γ -Fe (austenite) as shown in Figure 5a. Meanwhile, the peak of the spectra moved slightly to lower angle and peak broadening for nitrided sample exposed at 4, 8 and 12 hours owing to the formation of intermetallic compound consist of Fe_3N , CrN and expanded austenite (γN) phases as can be seen in Figures 5(b-d). This hump represents the decomposition of these nitrided phase at 2θ angles which has been identified by these three phases. It can be seen that at holding time for 4 and 8 hours, the nitride layer is precipitation-free from the formation of chromium nitride as shown in Figures 3(b and c). However, as discussed earlier there is a formation of CrN for longer holding time to 12 hours, leading to deterioration of corrosion resistance. Figure 3d shows the existence of CrN at angle of and 53.8° and 56.8° due to reaction between chromium and nitrogen atoms to form this new phases.



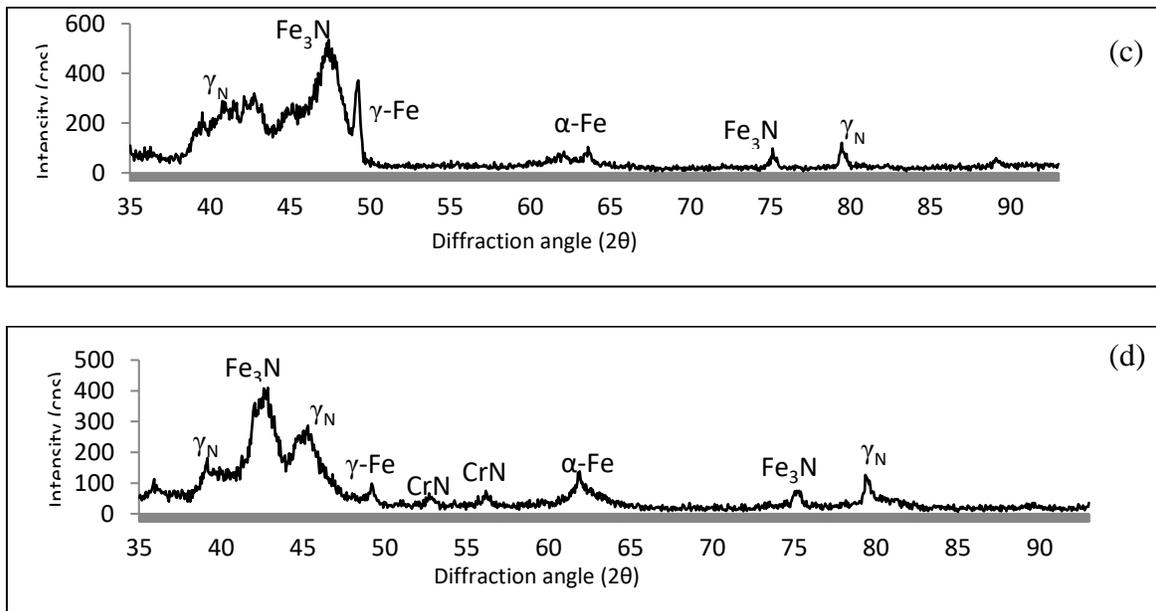


Figure 5: XRD spectra for (a) untreated sample and nitride layer treated at temperature of 450 °C for (b) 4 hours (c) 8 hours and (d) 12 hours

3.2 Vickers micro-hardness profile of surface coated DSS

The micro-hardness depth profile across the nitride layer was conducted to measure the attainment of hardness value from low temperature gaseous nitriding process. The hardness depth profile for nitriding process at different nitriding holding time is shown in Figure 6. It clearly indicates that micro-hardness depth profile for all nitride layer DSS has resulted a rapid drop in hardness from the surface to 10 μm depth. Furthermore, the hardness value shows gradual decrease towards substrate material. This proves that the formation of expanded austenite provides hard layer on the surface and the transition towards soft substrate of DSS occurs within a few microns (Christiansen & Somers, 2006).

From Figure 6, it is demonstrated that the sample nitrided at 4 hours experienced lowest hardness value of 400.5 $HV_{0.5}$ compared to other samples. This might be due to insufficient holding time of 4 hours to promote the high amount of nitrogen atom to diffuse into the surface of DSS. Moreover, the highest hardness value of 471.4 $HV_{0.5}$ and 513.3 $HV_{0.5}$ were developed in samples nitrided at longer holding time for 8 and 12 hours, respectively. The expanded austenite in nitride layer DSS produced hardness 2 times greater than the untreated DSS (250 $HV_{0.5}$) that has strong correspondence to the formation of expanded austenite and intermetallic compound as discussed in morphological and structural aspects of the layers. This proves that the temperature of 450 °C and longer holding time is the significant parameter to produce higher hardness of the sample. The highest hardness achieved at 12 hours holding time also contributed by the existence of CrN leading to the increment of hardness.

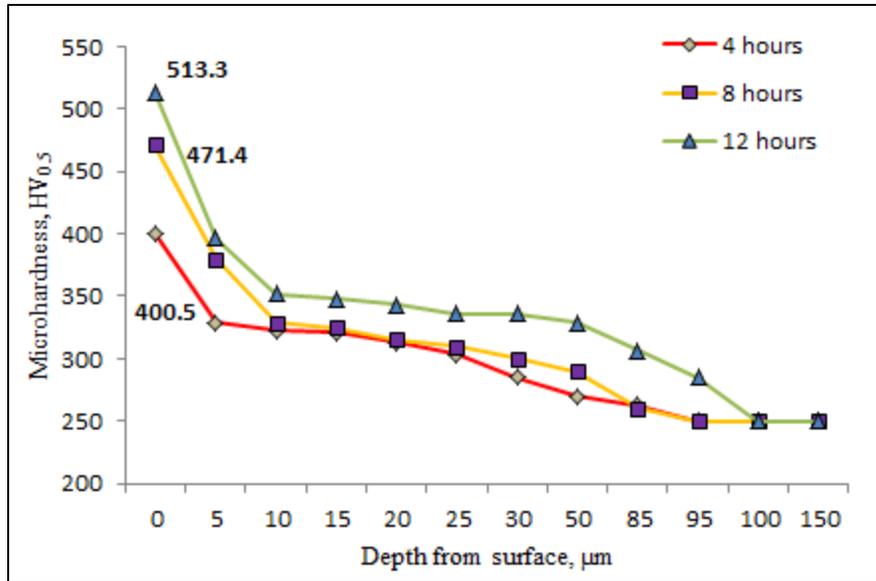


Figure 6: Hardness profile of the nitride layer DSS cross section for nitriding temperature of 450 °C for holding time of 4, 8 and 12 hours

3.3 Liner motion of reciprocating wear

In this study, the wear rate and CoF of nitrided surface DSS were compared at different holding time of nitriding process. The wear behaviour results of the nitrided sample measured against alumina ceramic ball are shown in Figures 7 and 8 for wear rate and CoF, respectively. It was observed that the nitride layer produces improvement of wear properties with the reduction of wear rate and CoF. Table 2 shows the summary of the wear performance value of the nitrided samples and compared with that of untreated DSS in percentage value.

The sample at holding time of 12 hours exhibited the lowest wear rates and friction (Figures 7 and 8) with the value of $4.25 \times 10^{-4} \text{ mm}^3/\text{Nm}$ and 0.535, respectively. In comparison with the untreated DSS, the wear rate and CoF properties have improved about 67.5 % and 23.6 %, respectively. The second lowest wear rate and friction are observed in sample at holding time of 8 hours with $4.625 \times 10^{-4} \text{ mm}^3/\text{Nm}$ and 0.545, respectively. The wear properties improved about 35% for wear rate and 22.1 % for CoF. This behavior could be explained by the higher hardness of the sample with 513.3 HV_{0.5} and 471.4 HV_{0.5} at the surface which are able to withstand the applied load during wear testing. This observation confirms with previous result on the formation of expanded austenite that contributes the increment of hardness as discussed in section 3.1 and 3.2.

Furthermore, the sample nitrided at holding time of 4 hours exhibited the highest wear rate and CoF with value of $4.77 \times 10^{-4} \text{ mm}^3/\text{Nm}$ and 0.57, respectively. This results shows the improvement about 33 % for wear rate and 18.6% for CoF as compared with that of untreated DSS. This is correlated well with lower hardness value owing to

ineffective nitrogen diffusion in this sample. For a given load during reciprocating action, the amount of plastic deformation at contact area with alumina ceramic ball is greater for a lower hardness sample. This suggests that the deformation facilitates disruption of the oxide film for lower hardness sample, thus produce greater oxide-free surface and produce higher friction. With lower hardness, the sample is unable to withstand the rubbing friction from the counterface alumina ceramic ball and promotes the adhesive wear of the sample.

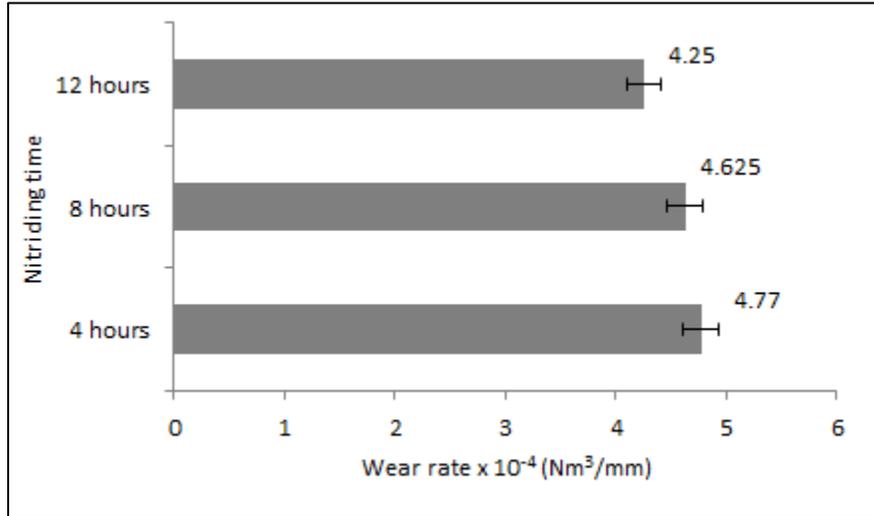


Figure 7: Wear rate of the nitride layer DSS cross section for nitriding temperature of 450 °C for holding time of 4, 8 and 12 hours

It can be seen that the COF result having a similar trend with wear rate result whereby the CoF reduced for longer nitriding holding time of for 8 and 12 hours of gaseous nitriding process. This phenomenon could be attributed to the higher hardness of these samples which obtained the hardness within the range of 400.5 $HV_{0.5}$ to 513.3 $HV_{0.5}$. The lower CoF produced is related to the higher hardness value and high amount of nitrogen element in the nitride layer which make the surface smoother after nitriding process. This is similar to the previous finding by Rosales & Martinez, (2007) where the plasma nitriding process used on molybdenum silicides has improved the wear resistance of the material in which the nitride surface decreased the CoF of molybdenum silicides reinforced with niobium addition.

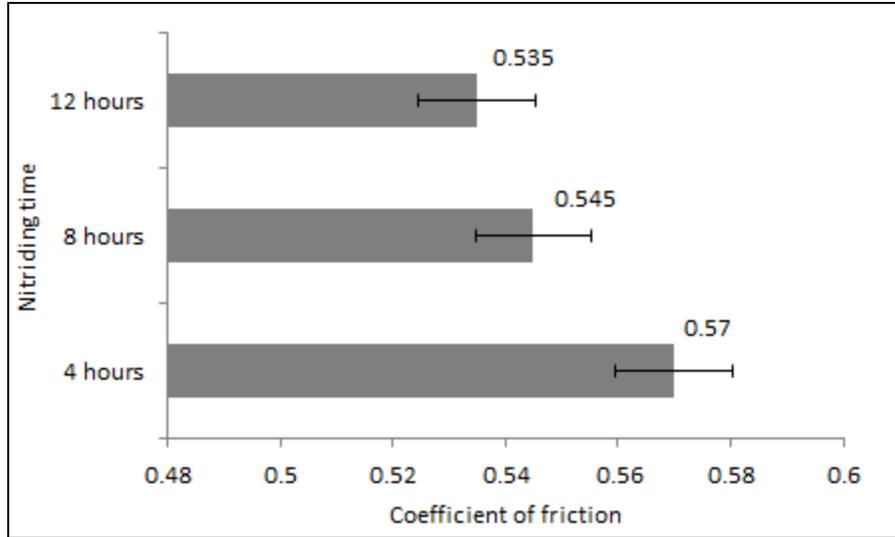


Figure 8: Coefficient of friction of the nitride layer DSS cross section for nitriding temperature of 450 °C for holding time of 4, 8 and 12 hours

Table 2: Wear rate and CoF value of the nitrided samples and compared with that of untreated DSS in percentage

Nitriding holding time	Wear rate x 10 ⁻⁴ (Nm ³ /mm)	CoF	Compared with that of untreated DSS in percentage	
			Wear rate (%)	CoF(%)
Untreated DSS	7.12	0.7	-	-
4 hours	4.77	0.57	33	18.6
8 hours	4.63	0.55	35	22.1
12 hours	4.25	0.54	67.5	23.6

3.4 Worn surface analysis

SEM images taken from the worn surfaces of the untreated DSS and treated samples are shown in Figure 9. The characteristic of the worn surfaces reveals a different wear behaviors between untreated and nitrided samples. Figure 9a shows the untreated sample suffers severe wear with ploughing and grooves. On the contrary, a basically smooth appearance with mild abrasive wear can be seen on the worn surfaces of the nitrided samples producing a smooth oxidized surface, as shown in Figure 9(b-d). This adherent oxide layer formed on the surface of nitrided samples acts as a lubricating layer, preventing metal-to-alumina contact and reducing adhesive wear thus reducing the wear mechanism. Mild ploughing can be seen at sample for holding time of 4 hours due to formation of nitrided layer has improved the abrasive wear of the sample suggesting the strong bonding energy with the surface are separated during the wear test as shown in Figure 9b. Clearly, there are an improvement on the abrasive wear in the worn surface for 8 hours sample with moderate striation, suggesting stronger interaction between nitride layer and counterpart

material on this sample, as can be seen in Figure 9c. Furthermore, it can be seen that the worn surface in the sliding direction are continuously improved when the nitriding holding time increases to 12 hours with mild striation as depicted in Figures 9d. As a result of the presence of a harder surface, increasing the concentration of nitrogen diffusion to longer holding time reduces the contact between the coating surface and the ceramic ball, enhancing the wear resistance properties. There is no visible spalling on the coating, which is consistent with its wear rate. This is because this sample has the highest load-bearing ability with better homogeneous distribution of expanded austenite and higher microhardness values in the sample. Moreover, the increase in hardness can be explained by the existence of chromium nitride as reported by number of researchers (Sun et al., 2008, Christiansen et al., 2005 and Tsujikawa et al. 2005).

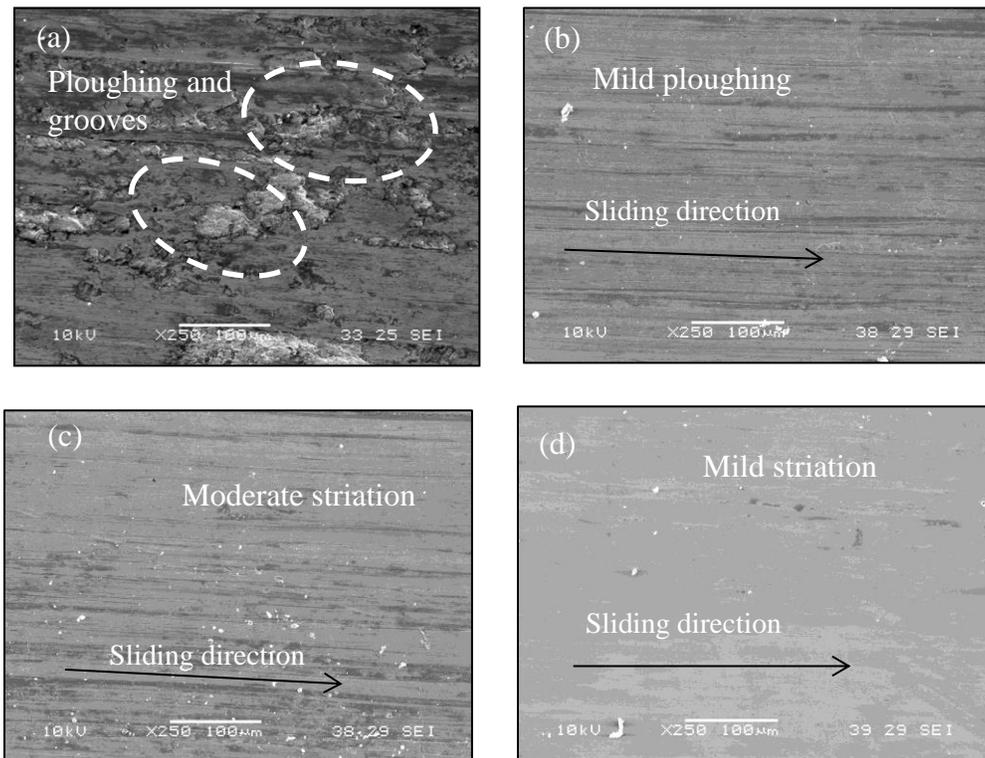


Figure 9: Worn surface morphology for (a) untreated DSS, nitride layer treated at temperature of 450 °C for (b) 4 hours (c) 8 hours and (d) 12 hours

CONCLUSION

In conclusion, this work examined the influence of low temperature thermochemical gaseous nitriding on morphological, hardness properties and wear reciprocating resistance of duplex stainless steel. The research results presented in the work lead to the following conclusions remarks:

1)The formation of nitided layer to produce hard nitride layer is successfully established with thickness layer ranging from 32.7 μm to 60.5 μm .

- 2) The nitride layer formation consists of new intermetallic compound namely Fe₃N, CrN and expanded austenite (γ N) phases.
- 3) The hardness value of 471.4 HV_{0.5} was developed in samples nitrided for 8 hours and free from chromium nitride without affecting the corrosion properties.
- 4) The wear rate value for nitrided sample is 4.25×10^{-4} mm³/Nm and CoF is 0.535, showing that the nitriding holding time for 12 hours presents the excellent wear performance.
- 5) Low temperature gaseous nitriding is a feasible process for wear properties enhancement on duplex stainless steel, provides a promising application for high load and long lifetime wear industry.

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