Comparison of microstructure and tribological behaviour of plasma nitrided and nitrocarburized AISI 4140 steel

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Abstract

The structural and tribological properties of plasma nitrided and nitrocarburized AISI 4140 steel were characterized using X-Ray diffraction, SEM, microhardness tester and pin-on-disc tribotester. It was observed that case depth and surface hardness obtained in the plasma nitriding were higher than nitrocarburizing. However, it was found that the plasma nitrocarburized specimens showed high surface roughness and lower wear rate because of ε phase presented at the outer surface in nitrocarburizing treatments. The lowest wear rate was obtained after austenitic plasma nitrocarburizing.

Key words: plasma nitriding, nitrocarburizing, tribology

1. Introduction

Plasma nitriding and nitrocarburizing are thermochemical surface treatments which improve mechanical and tribological properties of steel. Nitriding and nitrocarburizing can be performed in different media: solid, liquid, gaseous and plasma atmospheres. Extensive studies were carried out in salt bath and gaseous atmosphere. However, owing to its characteristics of faster nitrogen and nitrogen-carbon penetration, simplicity in application, economic and easier control of thickness of compound and diffusion layers, attention moved to plasma nitriding and nitrocarburizing methods [1–7].

Plasma nitrocarburizing treatment is classified into two groups as ferritic and austenitic according to process temperature. While treatment realized under eutectic point (592 ℃) at Fe-N diagram is named as ferritic nitrocarburizing, the treatment over this temperature is called austenitic nitrocarburizing [8–9].

After nitriding and nitrocarburizing, the hardened layer produced onto the steel surface can be subdivided into compound and diffusion layers. The compound layer is responsible for the good tribological and anticorrosive properties of the surface. Whereas, the diffusion layer with nitrogen and nitrogen-carbon atoms dissolved interstitially in ferritic matrix leads to improve the fatigue resistance. Wear resistance and load bearing capacity are related to the compound layer and case depth of hardened materials [10].

In this study, AISI 4140 steel was plasma nitrided and nitrocarburized under different conditions. The process variables investigated include treatment time (1–12 h), temperature (500–640 ℃) and different gas mixtures, and the effect of these process parameters on the structural, mechanical and tribological properties was investigated by using XRD, SEM, microhardness tester and pin-on-disc tribotester.

2. Experimental details

A series of experiments was carried out to investigate the plasma nitriding and nitrocarburizing responses of AISI 4140 steel. Plasma nitriding and nitrocarburizing were carried out in the same equipment, which was developed by the authors of this paper [11]. The specimens were normalized initially at 850 ℃ for 30 minutes, and then cooled in air. After cleaning with alcohol, the specimens were placed into the chamber, which was evacuated to 2.5 Pa. Prior to the processes, the specimens were subjected to cleaning by hydrogen sputtering for 15 minutes under a voltage of 500 V and a pressure of 500 Pa to remove surface contamin-

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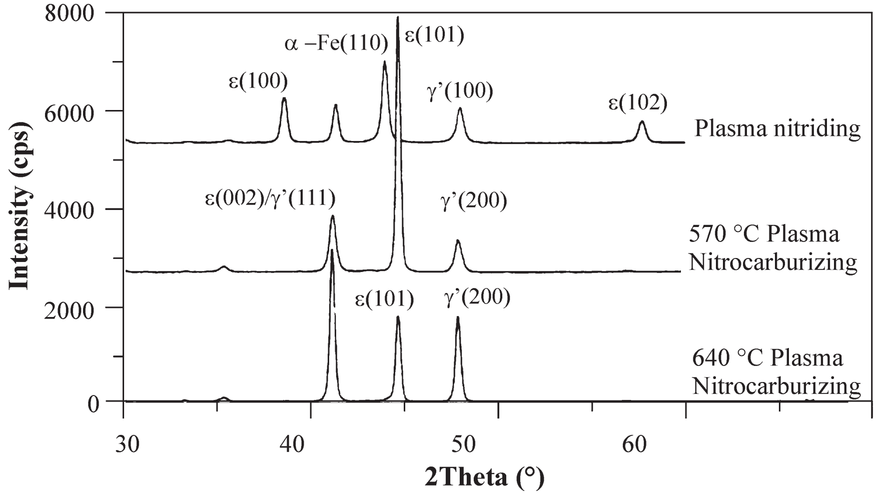

Fig. 1. XRD results of plasma nitrided and nitrocarburized AISI 4140 steel for 4 h.

ates. The nitrocarburizing treatments were performed at 570 and 640 ℃.

After the thermochemical processes, the compound layer formed on the surface was removed by polishing prior to metallographic examination and microhardness testing. The diffusion layer and the maximum surface hardness were measured by using a Buehler Omnimet MHT1600-4980T instrument at a constant load of 50 g and a loading time of 15 s. The maximum surface hardness at 25 μm depth was chosen for comparison so that any possible effects from a compound layer would be negligible. The case depth is defined as the depth at which the hardness is 10 % higher than the core hardness [12]. X-Ray diffractometer Rigaku operated at 30 kV and 30 mA with CuKα radiation was used for XRD analysis. The compound layer thickness was also investigated using a scanning electron microscope (SEM) Jeol 6400.

The wear tests were carried out on Teer POD-2 pin-on-disc tester, using a 5 mm diameter tungsten carbide ball as the pin. The friction force was monitored continuously by means of a force transducer. Unlubricated wear tests with a sliding distance of 141 m were carried out at room temperature ($\approx$ 18 ℃) and a relative humidity of about 50 %, a sliding speed of 0.078 $\mathrm{m \cdot s^{-1}}$, a normal load of 10 N and a wear track diameter of 10 mm. To calculate the wear volume, the profiles were recorded before and after the wear tests by a profilometer Mitutuyo. Then, from the superimposed profiles, the wear volume was calculated. The worn regions after the wear tests were examined using SEM.

3. Results and discussion

Figure 1 shows XRD patterns for the specimens that were plasma nitrided and nitrocarburized for 4 h. As seen in Fig. 1, polyphases (ε and γ') nitrides were formed at the surface. The intensity of ε phase is high in the plasma nitrocarburized specimen. While the intensity of ε (101) carbonitride reaches the maximum values for plasma nitrocarburized at 570 ℃, the intensities of ε (002) / γ' (111) and γ' (200) have high values in nitrocarburizing treatments.

Figure 2 shows SEM micrograph of plasma nitrided and nitrocarburized AISI 4140 steel. The thickness of the compound layer formed during nitrocarburizing is thicker than that of nitriding, from which it can be concluded that the change of compound layer thickness increases in the nitriding as treatment duration increases while the compound layer in the nitrocarburized samples is thicker than that of the nitrided specimens.

When the data given in Table 1 is examined, the hardness increased with increasing time up the 4 h, but then decreased because of tempering effect realizing in material at long treatment times. It was seen that the highest surface hardness was obtained in plasma nitriding performed for 4 h, and the diffusion layer is thicker in the nitriding treatment. The reason of high thickness, especially in plasma nitriding, may be attributed to the effect of diffusion reduction of the carbide compound formed primarily in the nitrocarburizing treatment [13]. As shown in Fig. 3, unlike ferritic nitrocarburizing and plasma nitriding, the maximum hardness in the austenitic plasma nitrocarburizing at 640 ℃ does not exist in the region near the surface. Maximum hardness was obtained with the transformation of austenite to bainite and/or martensite during cooling of α phase, which is entitled as austenite layer and includes the rich nitrogen content [5].

Figure 4 shows the variation of wear rates, at which the lowest wear rate was obtained in austenitic plasma nitrocarburizing treatment because of the hard austenite region below the compound layer in-

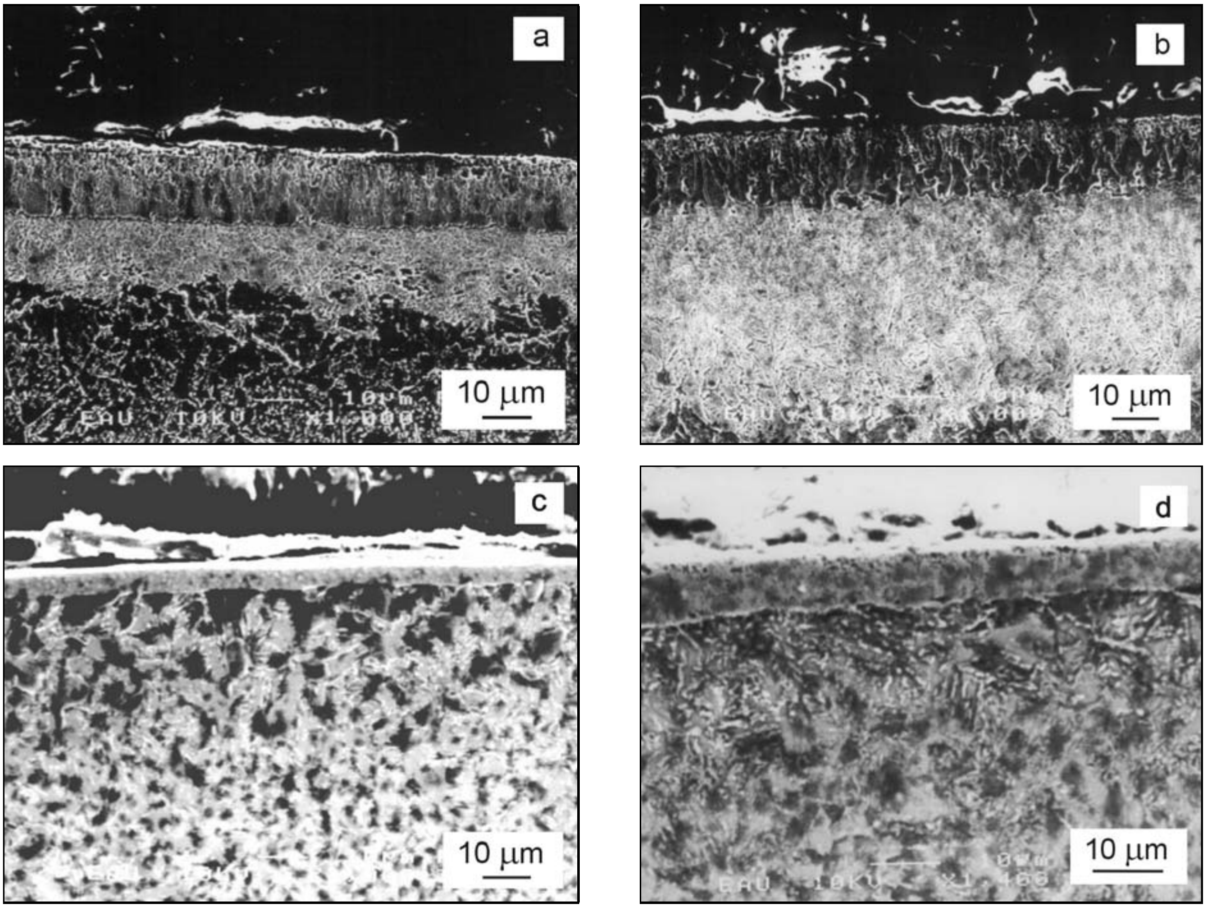

Fig. 2. SEM micrographs of surface treated AISI 4140 steel: a) plasma nitrocarburized for 1 h at 570 ℃, b) plasma nitrocarburized for 4 h at 570 ℃, c) plasma nitrided for 1 h at 500 ℃, d) plasma nitrided for 4 h at 500 ℃.

T a b l e 1. Experimental results obtained after plasma nitriding and nitrocarburizing

Process parameters			Experimental results			
Temperature (℃)	Time (h)	Gas mixture	Compound layer (μm)	Diffusion layer (μm)	Surface hardness HV 0.05	Surface roughness Ra
500	4	A	13–15	220–240	550–590	0.15–0.25
570	1	A	12–15	150–170	390–430	0.15–0.25
	4	A	16–18	210–230	530–570	0.62–0.72
	12	A	14–17	280–300	460–500	0.70–0.80
640	4	A	17–19	230–250	520–560	0.20–0.30
500	1	B	5–7	190–200	630–670	0.06–0.16
	4	B	9–12	270–290	640–680	0.08–0.18
	12	B	11–15	380–400	560–600	0.12–0.22
Unnitrided					300–320	

A: 49% N_2+49% H_2+2% CO_2; B: 50% N_2+50% H_2

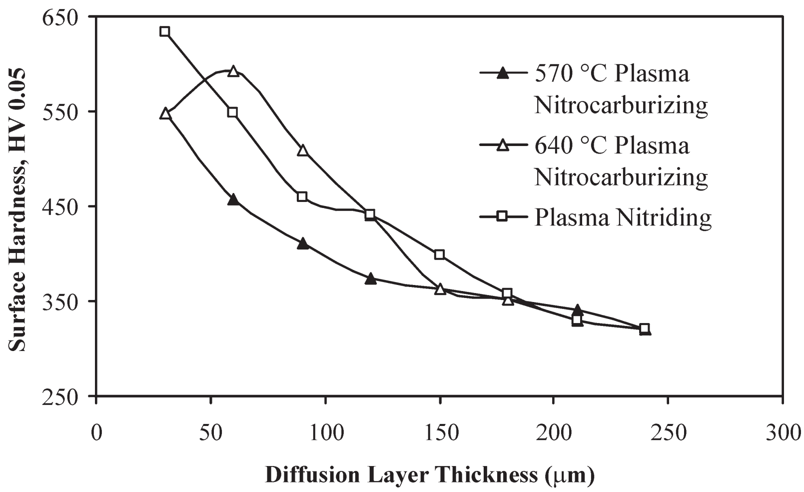

Fig. 3. The variation between surface hardness and diffusion layer thickness of plasma nitrocarburized and plasma nitrided AISI 4140 steel for 4 h.

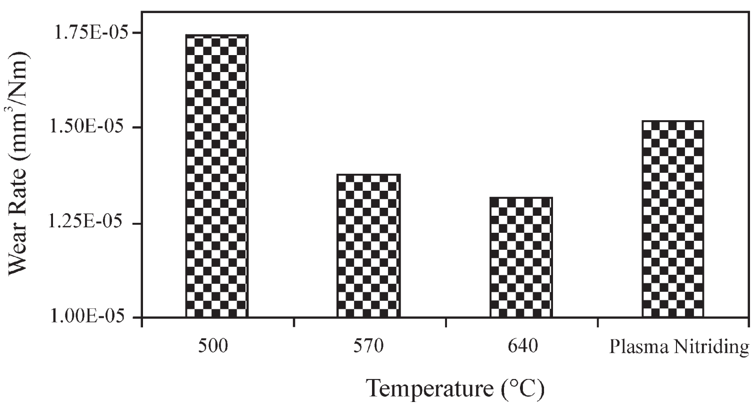

Fig. 4. Effect of process temperature on wear rate of plasma nitrided and nitrocarburized AISI 4140 steel for 4 h.

cluding dissolved nitrogen and occurring in austenitic nitrocarburizing. Furthermore, the wear rate close to this value in ferritic plasma nitrocarburizing at 570 ℃ is due to dominant ε nitrocarbide phase having a beneficial effect on wear. This phase could not occur sufficiently at low temperatures, the highest wear rate was obtained at this temperature (640 ℃). It was shown that the wear rate in plasma nitriding was higher than that of in ferritic and austenitic plasma nitrocarburizing. The reason is the presence of a compound layer with two different phases (ε, γ) causing the appearance of an abrasion wear component, due to breaking down of the compound layer during sliding causing the formation of hard abrasive particles. In the initial period of sliding, the brittle compound layer fractures under high stress, and then transforms into abrasive particles [13]. The wear rate after plasma nitrocarburizing decreases due to the compound layer including major $Fe_{2-3}(C, N)$ phase with higher toughness [14].

The wear profiles of the plasma nitrided and nitrocarburized AISI 4140 are given in Fig. 5. Abrasive wear occurs when hard debris particles are indented and make a groove in the rolling surface of the materials. Therefore, abrasive wear is convincingly related to the mechanical strength of the compound layer and to the adhesion between the compound layer and the substrate [15]. Initially, the wear begins as plugging, and then the debris separate from track and embed in wear track during moving, and as a result, it can be said that their particles facilitate abrasive wear. When compared to Figs. 5a,b,c, it is obvious that the wear track of plasma nitrided specimen is untidy, and debris occurs in the wear track. The profiles of plasma nitrocarburized specimen are smoother and more stable. Especially, the nature of the wear profiles of plasma nitrocarburized specimen tends to severe increase. The wear track of plasma-nitrided specimen is larger than that of nitrocarburized specimens.

The friction coefficients measured against a tungsten carbide ball are shown in Fig. 6 for the plasma nitrided and nitrocarburized specimens. It was observed in each case that the initial friction coefficient is ap-

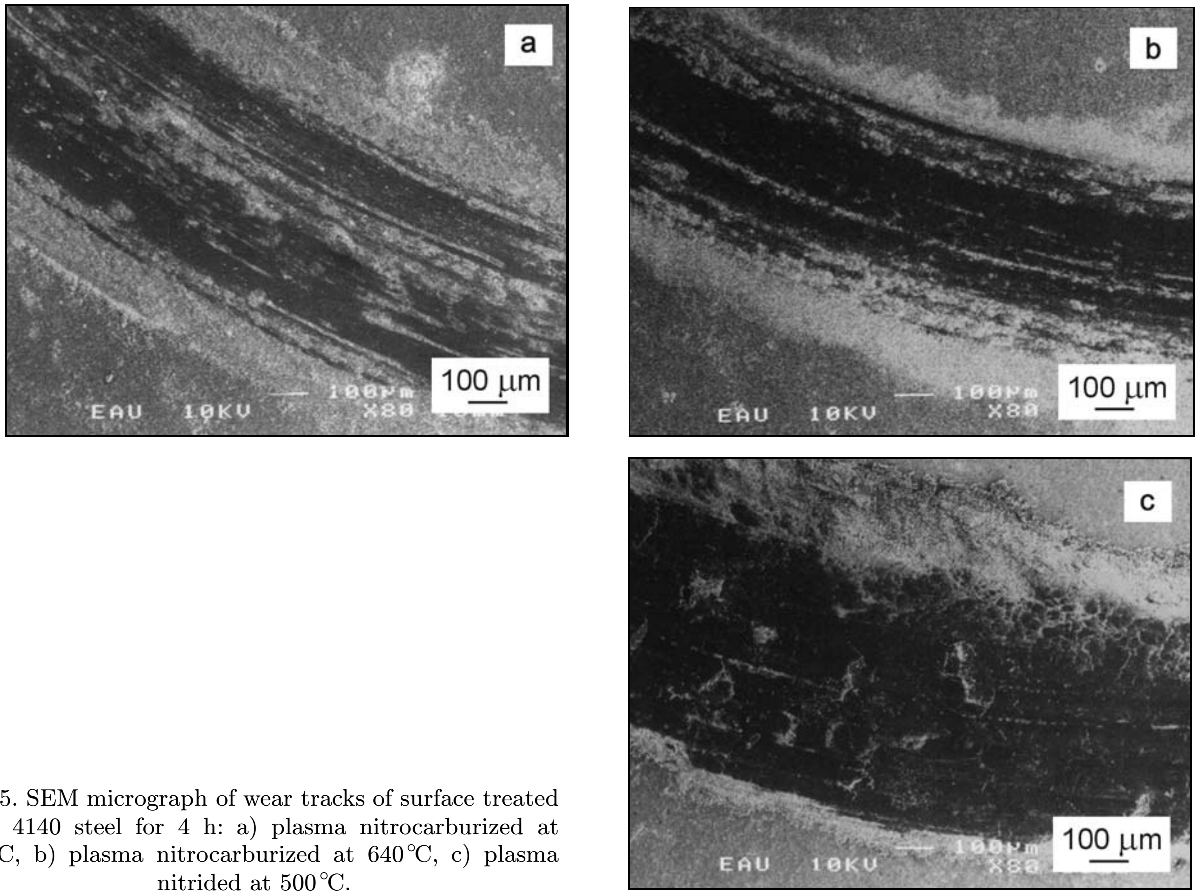

Fig. 5. SEM micrograph of wear tracks of surface treated AISI 4140 steel for 4 h: a) plasma nitrocarburized at 570 ℃, b) plasma nitrocarburized at 640 ℃, c) plasma nitrided at 500 ℃.

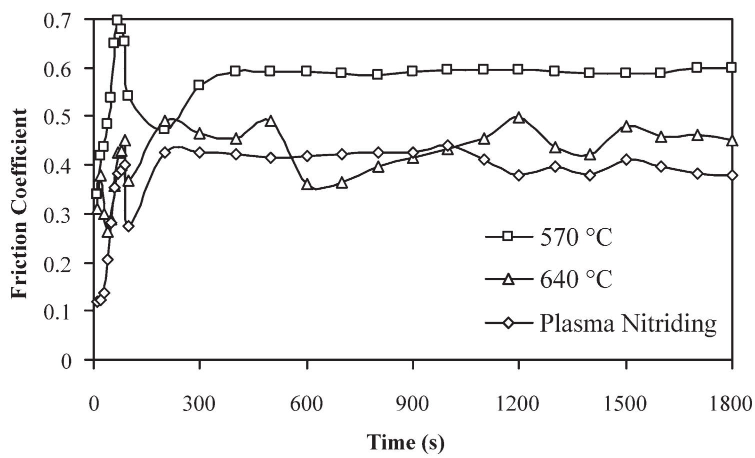

Fig. 6. The variation between friction coefficient and time for nitrided and nitrocarburized AISI 4140 steel for 4 h.

proximately 0.1, and the friction coefficient rises to a steady value during the test time. It was shown that the friction coefficients of plasma nitrocarburized specimens were generally higher than those of the plasma nitrided specimens due to ε phase having a porous structure open to the surface at the outer surface in nitrocarburizing treatments, and highest surface roughness value obtained after ferritic nitrocarburizing.

4. Conclusion

The following main conclusions can be derived from the above results and discussions:

– In the compound layer, ε-(Fe_{2-3}(C-N)) and γ'-(Fe_4(C-N)) phases in both the nitrocarburizing and nitriding were observed, the nitrocarburizing treatments dense ε-(Fe_{2-3}(C-N)) phase and also in nitriding dense γ'-(Fe_4(C-N)) phase were determined.

– The case depth (diffusion layer) formed during plasma nitriding is higher than in the plasma nitrocarburized samples.

– The hardness profiles of the plasma nitrided specimens exhibited maximum hardness in the range of 640–680 HV 0.05 for 4 h nitriding time.

– The wear rate of the plasma nitrocarburized specimens was higher compared to the plasma nitrided samples. The lowest wear rate was obtained after nitrocarburizing at 640 ℃.

– The friction coefficient of the ferritic plasma nitrocarburized specimen was higher than that of the plasma nitrided specimen.

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