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Effect of N_2+H_2 gas mixtures in plasma nitriding on tribological properties of duplex surface treated steels

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ABSTRACT

Thermo-reactive diffusion chromizing followed by pulsed plasma nitriding were carried out on AISI 52100 and 8620 bearing steels. The chromized samples were pulse-plasma nitrided for 5 h at 500 °C in various N_2–H_2 gas mixtures. The coated steels were characterized using scanning electron microscopy, X-ray diffraction and microhardness testing. The unlubricated wear behaviors of only chromized and duplex treated steels were investigated in ball-on-disc system tests at room temperature. X-ray diffraction patterns of the duplex treated samples containing H_2 indicated the formation of dominant CrN and Cr_2N nitrides as well as the formation of Cr_3C_2 and Cr_7C_3 carbides. Gas mixtures in the plasma nitriding, which was performed after chromizing, have a significant influence on the wear rate of the duplex treated steels. The wear and friction tests showed that the lowest friction coefficient and wear rates were observed for the samples duplex treated in a $50\%N_2+50\%H_2$ plasma. Conversely, the lowest wear resistance was observed on the samples duplex treated in a gas mixture of $75\%N_2+25\%H_2$, probably due to formation of a hard and brittle layer.

1. Introduction

The requirements to increase the service life of machine components have prompted the development of various surface treatment techniques and their combination. Duplex diffusion treatments make it possible to change the chemical and phase composition of the layers easily and effectively, to reduce the number of such defects as porosity, and to increase the strength of their adhesion with the substrate [1]. In many tribological applications, hard coatings of metal nitrides are now commonly used. Promising coatings for tribological use as an alternative to TiN are the chromium-base nitrides, CrN, Cr_2N. Chromium nitride (CrN) has become recently very popular as a coating material for tools and similar products due to their excellent mechanical properties [2]. Chromium nitride coatings have been successfully made using various types of physical vapor deposition (PVD) processes, such as cathodic arc deposition [3], reactive ion plating [4], magnetron sputtering [5] and duplex treatments of chromizing, electroplating and plasma nitriding [6,7].

Thermo-reactive deposition/diffusion (TRD) and plasma nitriding (PN) processes are diffusion-based surface treatments that are widely used in industrial applications. In the TRD process, the diffusing carbon or nitrogen atoms in steel react with the alloying powders at high temperature to form a dense and metallurgically bonded carbide or nitride coating at the steel surface. In the TRD chromizing process, a chromized layer consisting of various chromium carbides such as $Cr_{23}C_6$, Cr_7C_3 and Cr_3C_2 [8–10] are formed. The plasma nitriding process involves diffusional addition of nitrogen into the surface of materials. As a result, a compound layer and diffusion layer are formed on the surface [11,12].

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Table 1 – Chemical composition of test steels

Steel	Chemical composition, % by weight							
	C	Cr	Ni	Si	Mn	Mo	S	P
AISI 52100	0.95	1.45	0.107	0.194	0.318	0.014	0.0076	0.0143
AISI 8620	0.2	0.62	0.52	0.3	0.83	0.15	0.041	0.039

The combination of TRD chromizing and plasma nitriding has been evaluated by a number of investigators. In previous studies, Cr_7C_3 and $Cr_{23}C_6$-dominated layers obtained by TRD chromizing were plasma nitrided in gas mixtures of $25\%N_2+75\%H_2$ and $50\%N_2+50\%H_2$ at different conditions. It was observed that multi layers consisting of chromium nitrides and carbides exhibited better wear resistance than chromium carbides alone [7,13–15]. The available literature does not report on the effect of plasma gas mixtures on TRD chromized steels, and is strictly devoted to the structure of chromium nitride layers produced by fixed gas mixtures. In the present study, to improve the service life of steels for tribological applications, the duplex treatment of pack chromizing and pulsed plasma nitriding in various gas mixtures was performed. The structural characteristics, mechanical properties and unlubricated wear properties were investigated.

2. Experimental

AISI 52100 and 8620 bearing steels were used as the substrate material for duplex treatment. The bulk compositions of these steels are provided in Table 1. The bearing steels were sectioned as cylindrical coupons that were 25 mm in diameter and 6 mm in thickness and abraded and polished down with 1 μm Al_2O_3 powder. TRD carbide coatings were prepared by a pack method at 1000 °C for 5 h. [The details of the TRD chromizing were explained in a previous paper [15]]. Having completed the TRD heat treatment, test materials were removed from the box and quenched in air. The TRD chromium carbide-coated steels were washed in distilled water, ultrasonically cleaned in acetone and rinsed in alcohol. The nitriding was carried out in a dc-pulsed plasma nitriding system with power supply frequency of 5 kHz and duty cycle of 75%. The specimens were placed into the plasma nitriding chamber and the chamber was evacuated to 8.2×10^{-4} mbar. Prior to plasma nitriding, the chromized steels were subjected to cleaning by hydrogen sputtering for 30 min and the specimens were then plasma nitrided at 500 °C under a constant pressure of 10 mbar for 5 h at various gas mixtures of nitrogen-hydrogen atmospheres ($100\%N_2$, $25\%N_2+75\%H_2$, $50\%N_2+50\%H_2$ and $75\%N_2+25\%H_2$). The glow discharge was operated with a potential difference of 475–660 V to obtain the prescribed nitriding temperature. The plasma nitriding temperature was measured using a chromel–alumel thermocouple, placed at the bottom of the nitrided sample. Two plasma nitriding experiments were performed for each sample.

Cross-sections of duplex treated steels were prepared metallographically to observe morphological details using a BX60 Olympus microscope and a Leo 1430 VP scanning electron microscope. X-ray diffractograms were obtained using a copper tube source in the conventional Bragg–Brentano (θ–2θ) technique having symmetric geometry with monochromatized radiation (Cu Kα, $\lambda=0.15418$ nm). The thickness of the layers formed on the steels was measured by an optical micrometer attached to the optical microscope. The hardness of the layers was measured on the cross-sections using a Micro-Knoop indenter (Shimadzu HMV-2) with 25 g loads.

Wear tests of the duplex treated bearing steels were performed using a ball-on-disc test device. In the present study, the bottom movable flat surface was the treated steel and the upper fixed surface was the WC–Co ball with diameter of 8 mm. The ball was fixed and the disk sample was rotated at the speed of 300 rpm (0.3 m/s). The applied loads on the ball were 5 and 20 N and the sliding time was 60 min for each test. Wear tests were carried out in the unlubricated condition at room temperature in air. The friction force was detected by a load cell through a friction force measurement arm. The friction coefficient was continuously recorded during the tests. Surface profiles of the wear tracks on the discs were measured by a surface profilometer. Wear volume of the disc specimen was determined from the cross-sectional area of the wear track, obtained perpendicularly to the sliding direction. Two wear experiments were conducted for each sample and three measurements were performed using the surface profilometer. Each wear track and the average value is reported. Worn surfaces of the duplex treated samples were characterized by optical microscopy and scanning electron microscopy and the results were compared with those obtained for nitrided-only specimens.

3. Results and Discussion

3.1. Characterization of Coatings

Fig. 1 shows the cross-section micrographs of the samples which were chromized and the ones also additionally treated by plasma nitriding (PN) in varying gas mixture atmospheres. Table 2 indicates the layer thicknesses of only chromized and duplex treated (TRD+PN) steels in various plasma gas mixtures. Layers obtained by TRD and duplex treatment had a relatively flat morphology. Cracks and fractures occurred at the outermost layers of the samples duplex treated in a $75\%N_2+25\%H_2$ plasma as seen in Fig. 1e and f. This might be caused by rubbing with emery paper during the metallographic procedure due to its brittle characteristic.

As seen in Table 2, the chromized layer on the TRD AISI 52100 bearing steel, having approximately 1 wt.% C, was thicker than that on AISI 8620. It was reported that the thickness of the chromized layer increases with carbon content of steels [16]. Plasma nitriding of the chromized steels increased the total compound layer with the increase in nitrogen content of gas mixtures. However, layer thicknesses of duplex treated steels in a 100% N_2 plasma was nearly the same as those of the TRD-treated steels. Fig. 2 shows XRD results for duplex treated 52100 and 8620 steels as a function of the various gas mixtures used. TRD chromized steels were composed of major Cr_3C_2 and minor Cr_7C_3 phases. X-ray diffraction patterns of the duplex treated samples indicated

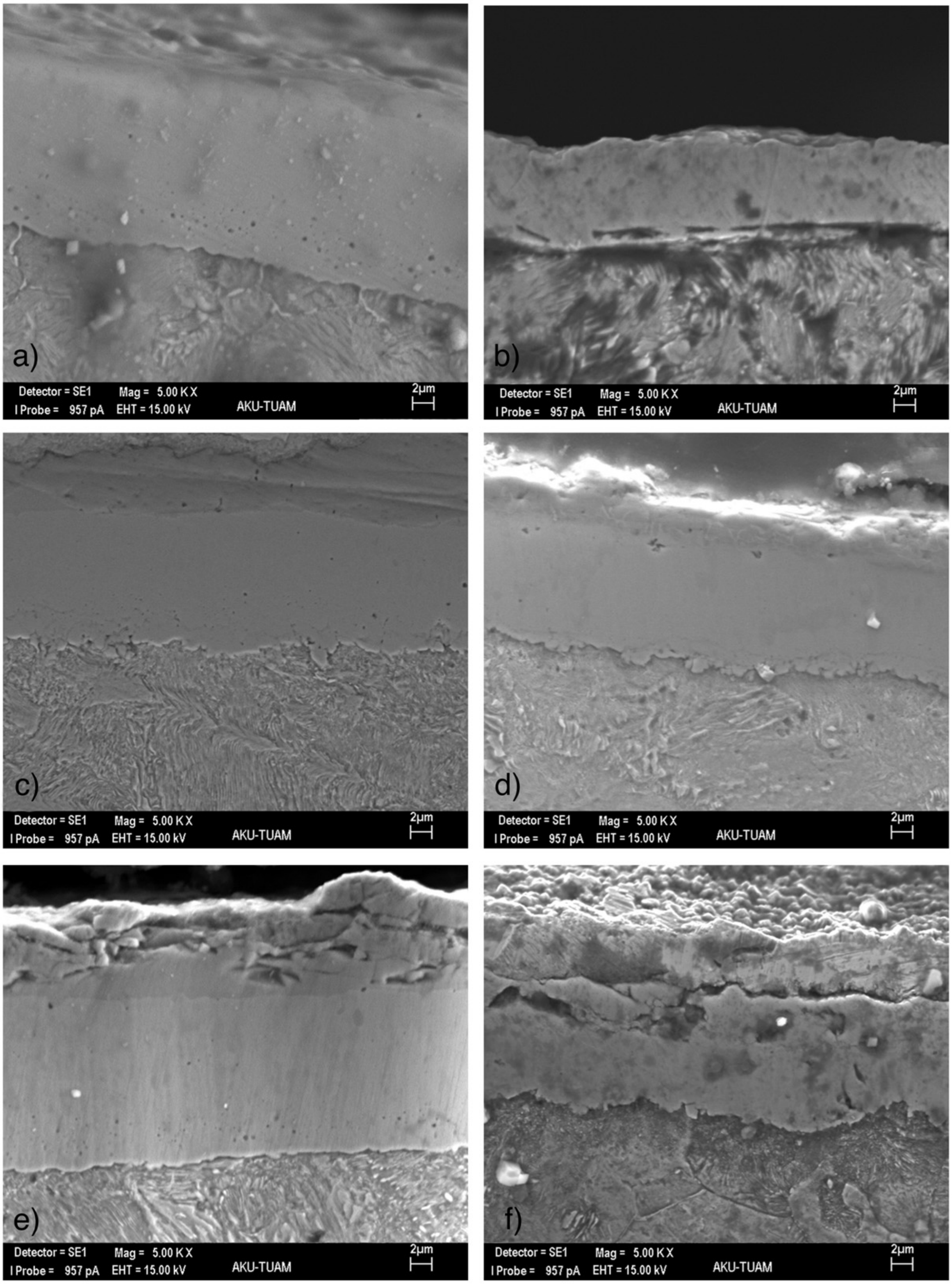

Fig. 1 – SEM Image of cross section microstructure of TRD chromized; a) 52100, b) 8620, duplex treated at 50%N_2+50%H_2 plasma; c) 52100, d) 8620 and duplex treated at 75%N_2+25%H_2 plasma; e) 52100, f) 8620 steels.

the formation of dominant CrN and Cr_2N nitrides as well as the formation of Cr_3C_2 and Cr_7C_3 carbides. No significant difference was observed for X-ray diffraction patterns of 8620 and 52100 steels. In case of both steels duplex treated in 50% N_2+50%H_2 and 75%N_2+25%H_2 plasmas, the peak intensity of chromium nitride increased compared with the samples

Table 2 – Layer thickness of TRD chromized and duplex treated (TRD+PN) AISI 52100 and 8620 steels

Treatment type	Plasma nitriding gas compositions	Layer thickness, (μm)	
		AISI 52100	AISI 8620
TRD	–	14.3±1.66	10.5±1.27
TRD+PN	25%N_2–75%H_2	15.4±0.78	11.6±1.2
TRD+PN	50%N_2–50%H_2	17.4±1.5	12.8±1.05
TRD+PN	75%N_2–25%H_2	19.2±1.6	14.6±1.35
TRD+PN	100%N_2	14.8±0.68	10.5±0.75

duplex treated in a 25%N_2+75%H_2 plasma. However, no peaks that belong to chromium nitride phases were observed in the X-ray diffraction patterns of samples that were duplex treated in a 100%N_2 plasma.

TRD chromized samples showed a single layer (Fig. 1a and b) whereas two distinct layers were found for the duplex treated steels (Fig. 1c, d, e and f). The formation of the outermost layer by plasma nitriding may be explained by the fact that the nitrogen atoms both substitute the carbon atoms in the chromized layer and overlay on the chromized layer. Only at the start of the sputtering treatment can decomposition of the carbides take place. Immediately thereafter a nitride layer is formed and further layer growth is controlled by nitrogen diffusion. In a 100% N_2 plasma, no increase was observed in the layer thicknesses on the TRD+PN samples. Chromium nitride surface coatings were not present on these steels. This can be attributed to formation of strongly reducting species by hydrogen gas [17]. The presence of hydrogen in the gas mixture used for plasma nitriding has several effects. The first is the removal of absorbed oxygen on the surfaces. The second is the control of the nitrogen chemical potential that controls the growth of different nitride phases. The third effect is the enhancement of nitrogen atom concentration in the plasma [18,19]. In addition, it is proposed that the NH radicals as well as the H radicals play an important role in nitriding. The presence of numerous H radicals may more effectively convert the NH radical to an active nitrogen atom on the nitride surface. In the case of nitriding in pure N_2 gas, no NH radical exists in the plasma [20].

Surface morphologies of AISI 52100 steel, chromized and duplex treated in gas mixtures of 50%N_2+50%H_2 and 75%N_2+25%H_2, are shown in Fig. 3a–c. The coaxial grain surface structure was observed for the specimen chromized in Fig. 3a and grain boundaries were revealed on the surface. On the other hand, plasma nitriding influenced the microstructure of the samples, producing much smaller grain sizes in the case of a 50%N_2+50%H_2 gas mixture. When the % N_2 increased to 75%, coarse grains appeared in the microstructure as seen in Fig. 3c.

Results of the surface microhardness and roughness measurements are given in Table 3 for the TRD chromized and duplex treated steels at various plasma gas mixtures. In the chromized layer on AISI 52100, relatively higher microhardness values were obtained compared to chromized AISI 8620 steel. This could be attributed to the high concentration of carbon in the 52100 steel [16]. The microhardness values in the duplex treated layers were higher than those of chromized layers, due probably to the Cr_xN phase. It was noted that the microhardness values of duplex treated (TRD+PN) steels in this study were almost equal to the reported data for CrN layers produced by physical vapor deposition, which is approximately 1600–2000 HV [21]. In N_2+H_2 gas mixtures, the microhardness of the duplex treated steels increased with an increase in nitrogen content. However, duplex treated steels,

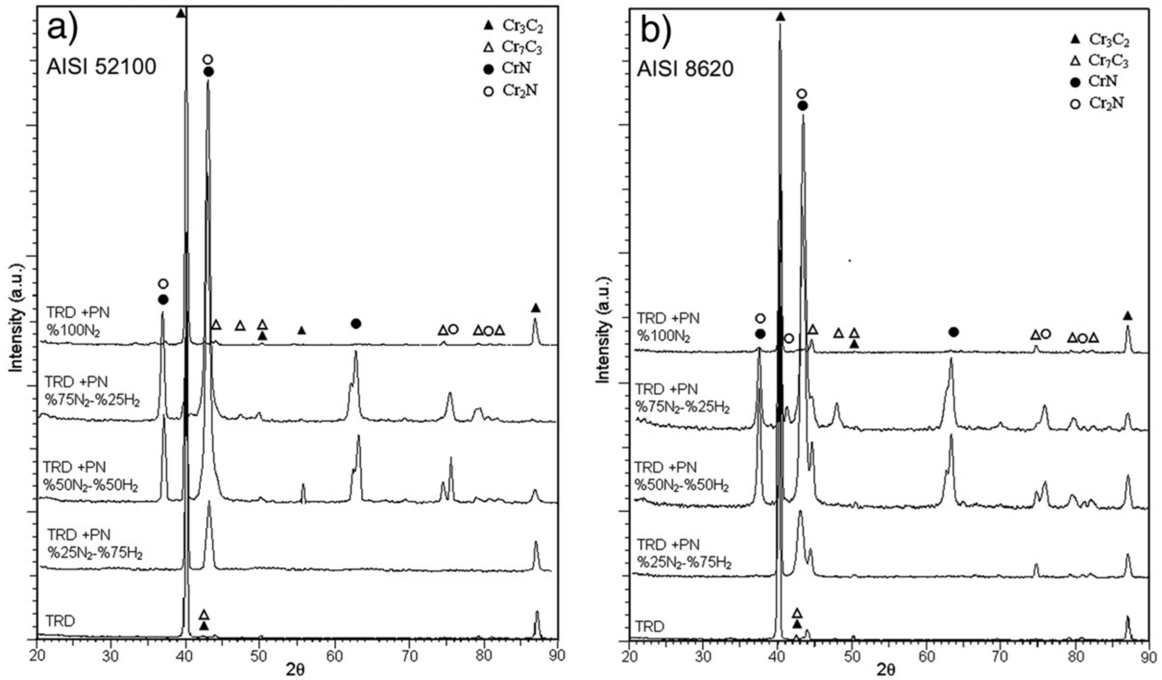

Fig. 2 – XRD patterns of AISI 52100 and 8620 steels only TRD chromized and duplex treated at various gas mixtures.

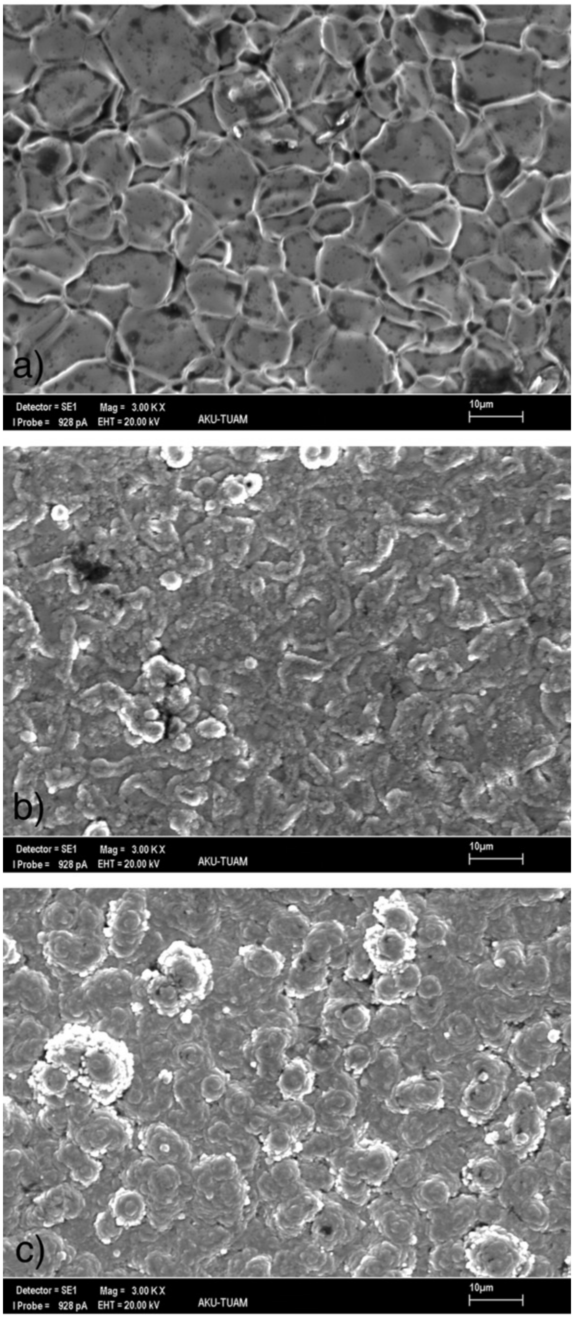

Fig. 3 – Surface morphologies of AISI 52100 steel a) only TRD chromized b) duplex treated at 50%N_2 + 50%H_2 plasma and c) duplex treated at 75%N_2 + 25%H_2 plasma.

which were nitrided at 100% N_2 plasma, showed lower hardness compared to steels duplex treated at gas mixtures containing H_2. This might be due to the absence of chromium nitride phases in the surface layer; this result is in agreement with the X-ray diffraction results. As seen in Table 3, the surface roughness of steels is affected by surface treatment steps. A polished surface roughness of 0.03 μm Ra increased to about 0.4 μm after the chromizing process, and then increased with increasing nitrogen content in the plasma nitriding process.

3.2. *Wear and Friction*

Steady state friction coefficient values for loads of 5 and 20 N are illustrated for the steels chromized (TRD) and duplex treated (TRD + PN) at various gas compositions in Fig. 4. Steady state friction values were calculated by summing and averaging the friction coefficient values. The error bars indicate the variation of the friction coefficient after steady state was reached. Friction results indicated that, for both steels, the lowest steady state friction coefficient was observed for the samples duplex treated in a gas mixture of 50%N_2 + 50%H_2 followed by the samples duplex treated in the gas mixture of 25%N_2 + 75%H_2. This might be attributed to the lower friction coefficient of chromium nitride [22]. However, contrary to expectations, the samples treated in a 75%N_2 + 25%H_2 plasma exhibited a similar friction coefficient to that seen for the samples TRD chromized and treated in a 100% N_2 plasma.

Fig. 5 shows wear rate values of AISI 52100 and 8620 steels TRD chromized and duplex treated (TRD + PN) in various gas compositions for loads of 5 and 20 N. The wear rates of samples worn with a 5 N load are only slightly higher than those with a load of 20 N. This is because the rate of increase in the amount of wear volume is less than the increase of applied load. Duplex treated samples in the gas mixture of 50%N_2 + 50%H_2 give the best wear resistance values followed by steels treated by plasma nitriding in 25%N_2 + 75%H_2. It was noted that there was only a small difference between the wear rates of steels only chromized and duplex treated steels in a 100% N_2 plasma. This might be due to the absence of chromium nitride phase formation. The wear rate values of the samples duplex treated in the 50%N_2 + 50%H_2 plasma were about 30% and 36% lower, respectively, compared to only TRD chromized ones and samples duplex treated in a 75%N_2 + 25%H_2 gas mixture. Although the surface hardnesses of both steels duplex treated in a gas mixture of 75% N_2 + 25%H_2 were higher than those of steels duplex treated in a 50%N_2 + 50%H_2 plasma, the most intensive wear occurred in the samples duplex treated in a gas mixture of 75%N_2 + 25%H_2. As seen in the cross-section of Fig. 2e and f, the outermost layer of the samples duplex treated in a 75%N_2 + 25%H_2 plasma was hard and brittle and had poor adhesion.

Fig. 6 shows the wear tracks of the 52100 samples chromized and duplex treated in gas mixtures of 50%N_2 + 50%H_2 and 75%N_2 + 25%H_2 using a 5 N load. An overview of the worn surface of the WC–Co ball is shown in the upper right corner of the scanning electron micrograph. As seen in Fig. 6a, the sliding tracks of the sample TRD chromized were wider and had a relatively rougher morphology with evidence of cracking and flaking compared with that of the steel duplex treated in a gas mixture of 50%N_2 + 50%H_2. The contact surface of the ball rubbed against the TRD sample showed wear debris collected besides the wear-tracked surface. Higher magnification revealed that the chromium carbide layer initially cracked and then broke into pieces (Fig. 6b). The wear track of the sample duplex treated in a 50%N_2 + 50%H_2 gas mixture (Fig. 6c) was relatively shallow and smooth. In addition, there was no transfer of material to the ball and the ball wear scar was very smooth. Higher magnification of this wear track (Fig. 6d) showed a smooth conforming surface and new initiation of micro-cracking. However, the sliding track of the sample duplex treated in a 25%N_2 + 75%H_2 gas mixture had a larger

Table 3 – Microhardness and surface roughness of TRD chromized and duplex treated (TRD+PN) AISI 52100 and 8620 steels

Treatment type	Plasma nitriding (PN) gas compositions	Microhardness, $HK_{0.025}$		Surface roughness, Ra (μm)	
		AISI 52100	AISI 8620	AISI 52100	AISI 8620
TRD	–	1823	1769	0.42 ± 0.04	0.415 ± 0.06
TRD+PN	25%N_2–75%H_2	1867	1835	0.610 ± 0.09	0.604 ± 0.11
TRD+PN	50%N_2–50%H_2	2135	1976	0.634 ± 0.1	0.612 ± 0.08
TRD+PN	75%N_2–25%H_2	2438	2287	0.703 ± 0.08	0.688 ± 0.07
TRD+PN	100%N_2	1854	1795	0.708 ± 0.23	0.727 ± 0.2

surface cracking area due to the combined effects of the brittle compound layer and high stress. The stress developed beneath the contacting asperities resulted in little plastic deformation; the removal of plate particles gradually initiated and then proceeded in the direction of sliding [23]. The contact surface of the ball became very rough and large because of the effects of the abrasive delaminated abrasive coming from the disc. Delaminated area and large flake debris can be seen in

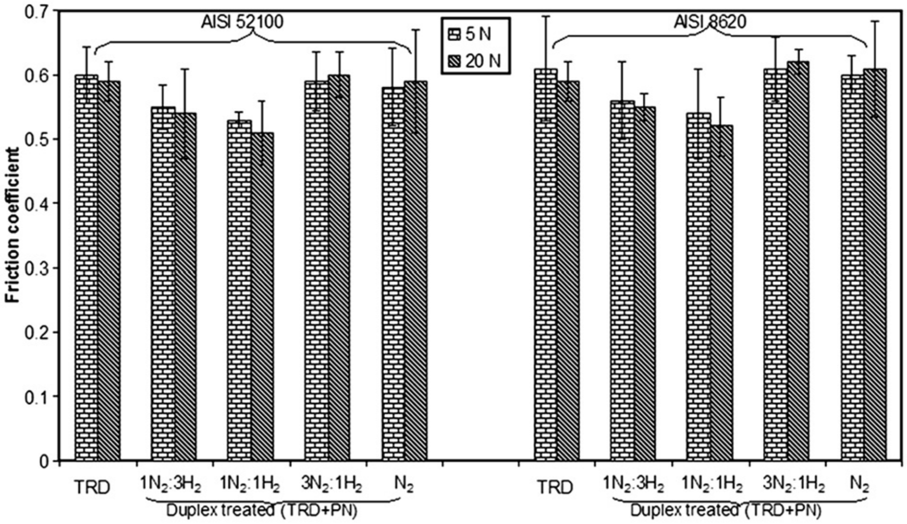

Fig. 4 – Steady state friction coefficient values for AISI 52100 and 8620 steels only TRD chromized and duplex treated at various gas mixtures under loads of 5 and 20 N.

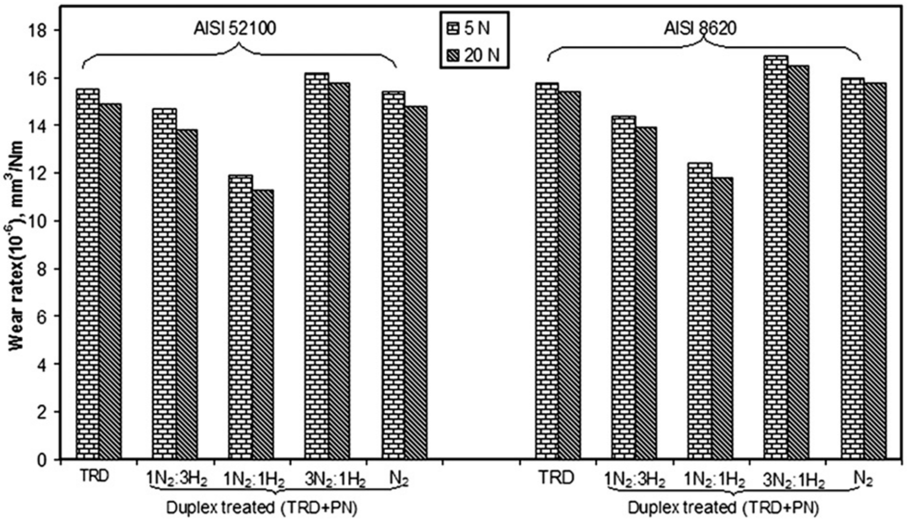

Fig. 5 – Wear rate values of AISI 52100 and 8620 steels only TRD chromized and duplex treated at various gas mixtures for loads of 5 and 20 N.

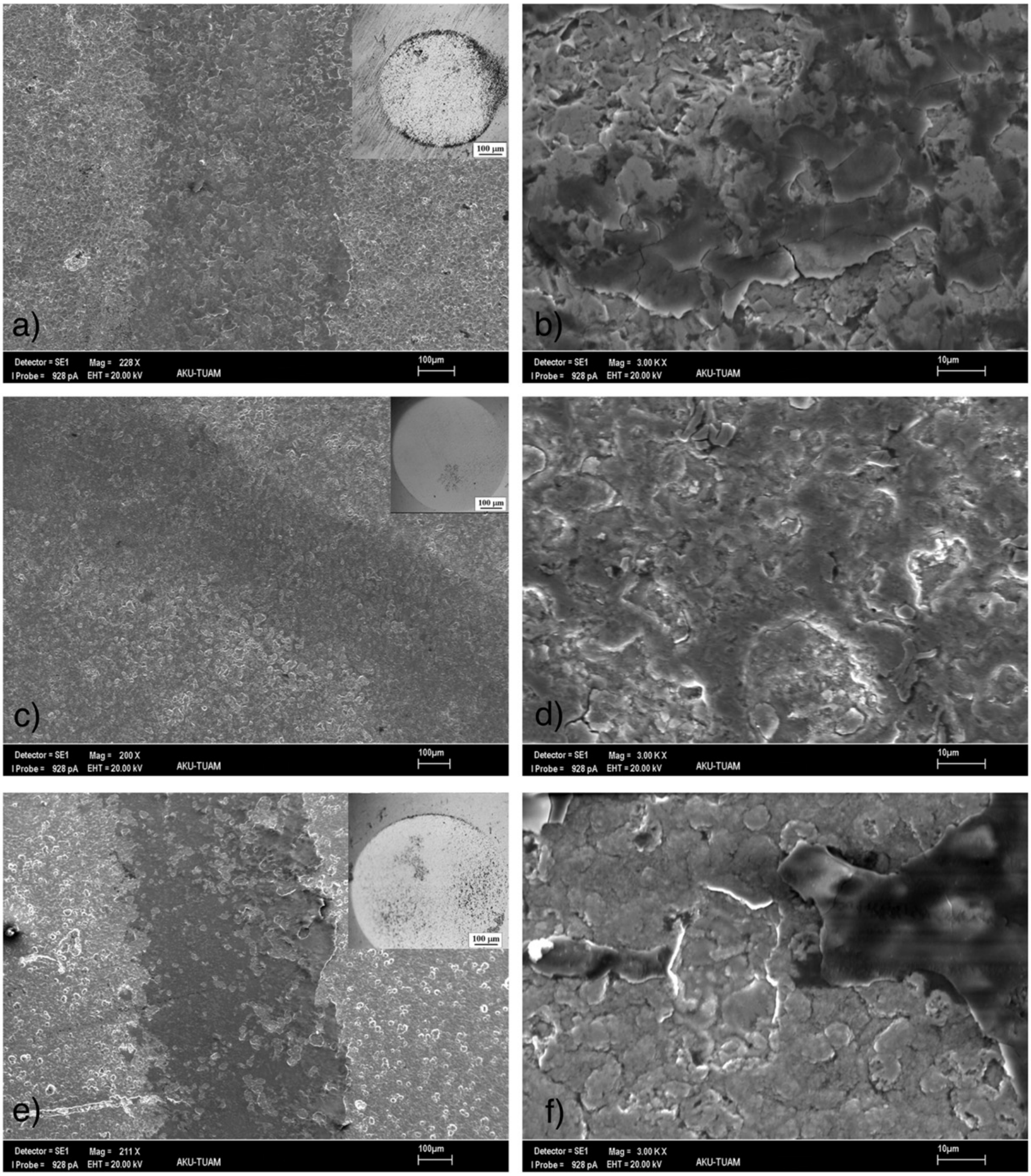

Fig. 6 – SEM micrographs of the wear tracks of 52100 samples and the optical worn ball overview at the upper right corner of the micrographs for 5 N load; a) wear track of only TRD chromized, b) worn surface under high magnification of (a), c) wear track of duplex treated at 50%N_2+50%H_2 plasma, d) worn surface under high magnification of (c), e) wear track of duplex treated at 75% N_2+25%H_2 plasma, f) worn surface under high magnification of (e).

higher magnification of this wear track (Fig. 6d). Initial wear of these samples seems to occur by a fatigue mechanism and thereafter continues by abrasion.

4. Conclusions

Plasma nitriding of the chromized steels showed that the total compound layer increased with an increase in N_2 content of gas mixtures containing H_2. Chromium nitride phases as well as phases of chromium carbides were found on the duplex treated steels. However, in the case of 100% N_2 plasma, no increase was observed in the layer thicknesses of the duplex treated samples; chromium nitride phases were not present in these steels.

Gas mixtures in the plasma nitriding, which was performed after the samples were TRD-chromized, have a significant influence on the tribological properties of the duplex treated steels. The lowest friction coefficient was observed for the samples duplex treated in a 50%N_2+50%H_2 plasma. The samples chromized and duplex treated in either a 75%N_2+25%H_2 mixture or a 100% N_2 plasma exhibited higher friction coefficients.

The samples duplex treated with 50%N_2+50%H_2 plasma exhibited the best wear resistance, whereas the lowest wear resistance was found for the samples duplex treated in a 75%N_2+25%H_2 plasma, probably due to formation of a hard and brittle layer. For all surface treated steels, delamination is the principal wear mechanism. During sliding wear testing, the duplex treated steels prepared in a 50%N_2+ 50%H_2 plasma showed less tendency towards subsurface crack initiation than steel prepared in a 75%N_2+25%H_2 plasma.

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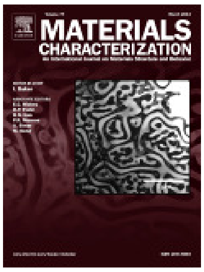

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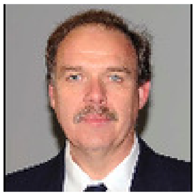

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