TRIBOLOGICAL PERFORMANCE OF PLASMA NITRIDED 42CrMo4 STEEL AT ELEVATED TEMPERATURE

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Abstract

This paper is focused on the tribological behaviour of the plasma nitrided 42CrMo4 steel at elevated temperature to 300 ºC. The hardened steel was treated in plasma in two variants of nitriding atmosphere: $24H_2$:$8N_2$ and reverse ratio $8H_2$:$24N_2$, which is greater nitrogen concentration. This leads to forming the nitriding layers with different phases, therefore, to distinct mechanical and tribological properties of nitride layers. The "ball on disc" wear test was performed at the variation of temperatures. A profilometer was used to investigate the wear track on the disk surface. Experimental results indicated that the coefficient of friction changed over the test time and temperature, and plasma nitriding in the atmosphere of 8 H_2 : 24 N_2 improved wear resistance better than nitriding in the atmosphere of 24 H_2 : 8 N_2. The results of wear test were further supplemented by measurement of surface roughness, metallografical documentation, layer thickness measurements, surface hardness and microhardness test.

Keywords: Plasma nitriding, wear resistance, coefficient of friction, elevated temperature, 42CrMo4

1. INTRODUCTION

The wear mechanism is classified in some types of wear like abrasive, adhesive, erosive, fatigue, cavitation, etc. The meaning of improving the wear resistance of materials and surface layers/coating become more important because the machinery tolerance at this time and in the future is lower than in past [1][2]. A small change of measurement by wear can affect the operation of machines, to accelerate degradation process, to loss masses, energies and fuel and at the end to lead to shortening service life.

The "ball on disc" wear test is widely used in studying the tribological performance of materials. During studying the wear mechanism of surface layers or coating, their behaviour is not like their steel substrate [3]. Their wear process can be divided into 4 periods [4]:

- Initiation of wear - growth of the friction coefficient and wear
- Contact surface adaptation - high coefficient of friction and wear
- Steady-state regime - low coefficient of friction, low wear
- Destruction of layer - great coefficient of friction, great wear.

A useful method to increase the wear resistance of material is to use the thermochemical treatments, their representative is nitriding. Nitriding, a favourite solution was studied parallelly from the later of 19. century by pioneers in Euro, Russia and America with their specific strong points like using alloy elements or using nitriding atmosphere to control properties of nitrided layers [5]. Based on the saturation of nitrogen to steel surface under anomalous glow discharges to create a nitride layer with valuable properties, plasma nitriding is a very effective technology to enhance surface hardness, corrosion resistance, wear resistance and fatigue limit [6][7][8][9][10]. The nitride layer consists of the compound layer (lower than 8 μm), which is formed of ε-$Fe_{2-3}N$ and γ-Fe_4N phases, and beneath diffusion layer, which is formed of dispersive iron nitrides and nitrides of alloying elements with high affinity to nitrogen so-called nitalloy, like Al, Cr, Mo, W, etc. [11].

The properties of nitride layer are determined by the chemical composition of steel, surface conditions and parameters of nitriding process such as voltage, duration, pressure and nitriding gas composition. Some authors had been studied the positive influence of ε-phase ($Fe_{2-3}N$) to corrosion and wear resistance [8][12], this article is also aimed to create nitride layer with ε-phase by regulation of enriched nitrogen atmosphere.

This article focuses on evaluating tribological characters of tempered and nitrided 42CrMo4 steel, which is widely used in automobile and weapon manufacturing. The "ball on disc" wear test was realised on tribometer BRUKER UMT-3 at various temperatures (21 ºC, 150 ºC and 300 °C).

2. EXPERIMENTAL

The samples for wear tests were manufactured in the shape of round disk with a diameter of 70 mm and the height of 6 mm, the roughness and shape tolerance according to ASTM G99-95a standard [9]. The samples were normalised (850 ºC), quenched (850 ºC) and air tempered (550 ºC) to attain the relevant structure and mechanical properties for next nitriding process.

The elemental weight percentages of the used material were investigated by GDOES method (Glow Discharge Optical Emission Spectral) using the LECO SA 2000 device. The chemical composition according to ISO steel standard and measured results by GDOES/BULK method are in agreement (see **Table 1**).

Table 1 Chemical composition of 42CrMo4 steel (wt%)

Element	C	Mn	Si	Cr	Ni	Mo	Cu	P	S
ISO standard	0.38-0.45	0.5-0.8	0.17-0.37	0.9-1.2	≤ 0.5	0.15 - 0.3	≤ 0.3	≤ 0.03	≤ 0.03
GDOES/Bulk	0.42	0.6	0.20	1.0	0.02	0.18	0.01	0.001	0.001

**Parameters of GDOES/Bulk analysis: U = 800 V, I = 30 mA, p(Ar) = 314 Pa*

The tempered samples were nitrided in the RUBIG nitriding equipment. The plasma nitriding was treated in two steps: cleaning in plasma and creating nitride layers. In this article, the samples were nitrided in two variants of nitriding atmosphere. Plasma nitriding PN1 was treated in the gas mixture of 24 H_2 : 8 N_2 (classical) and plasma nitriding PN2 in a gas mixture of 8 H_2 : 24 N_2. Process parameters are given in **Table 2**.

Table 2 Plasma nitriding process parameters

Process	Temperature (ºC)	Duration (h)	Pressure (Pa)	Bias (V)	Gas flow (l/h)	
					H_2	N_2
Plasma cleaning	480	0.5	80	800	20	2
Plasma nitriding PN1	500	10	280	530	24	8
Plasma nitriding PN2	500	10	280	530	8	24

A profilometer was used to measure parameters of surface roughness. The cross-structure observation and documentation were performed with a magnification of 200x and 500x using the optical microscope OLYMPUS DSX 500i. The total thickness of the nitride layer (case depth) was investigated using the LECO LM 247 AT microhardness tester in accordance with DIN 50190 standard. This microhardness test allows to establish microhardness in direction from the surface to the core at 50 g load and 10 s dwell time. The surface hardness was measured on the LECO LV 800AT device.

The “ball on disc’’ wear test, corresponded to ASTM G99-95a [12] was carried out on the BRUKER UMT-3 tribometer with an indenter made of carbide wolfram with the diameter of 6.3 mm and hardness of 92 HRA. Measurement parameters were set as following: a normal load of 20 N, the rotary speed of 500 rpm, track radius of 20 mm, test duration of 27 min, the trajectory of 1696 m. As results of wear test, a relationship between the coefficient of friction and time was established. In order to measure at the required temperature, the sample (disc) and indenter (ball made of carbide wolfram) were heated in the thermal chamber for 25 minutes and kept at this temperature 10 minutes yet, so that these contact pair had a similar temperature. After the wear test, the sample was cooled in the air with a fan, and then cleaned with alcohol using an ultrasound cleaner. The wear track was graphically documented using a laser confocal microscope. Thereafter, the TALYSURF CLI 1000 profilometer was used to evaluate the wear depth and area of wear profile, from which wear rate was calculated.

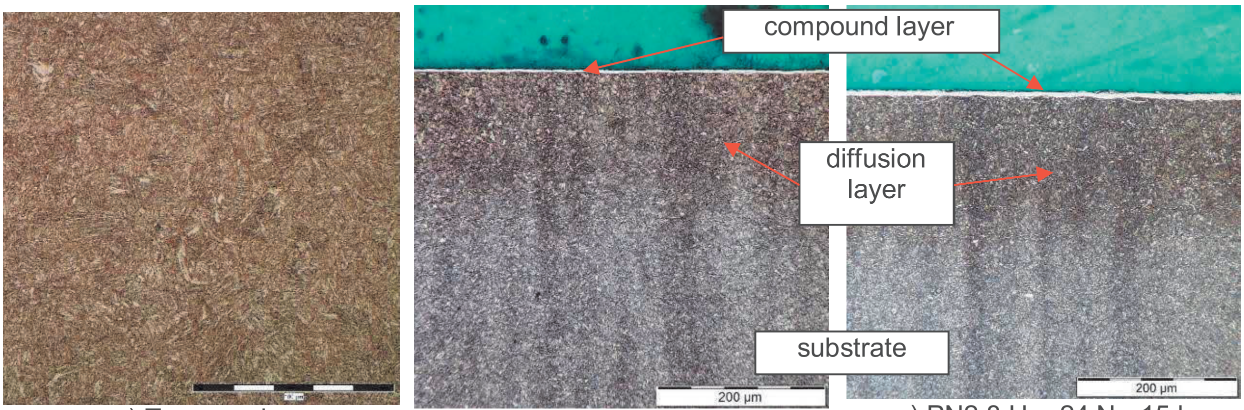
3. RESULTS AND DISCUSSIONS

The measured parameters of surface roughness are shown in **Table 3**. In general, after plasma nitriding process PN2, all chosen parameters of surface quality are higher than tempered while the surface roughness Ra and Rt after plasma nitriding PN1 are smaller than tempered sample.

Table 3 Parameters of surface quality

Sample	Ra (µm)	Wa (µm)	Rt (µm)	Rq (µm)	RSm (µm)
Tempered	0.52 ± 0.05	0.10 ± 0.03	3.69 ± 0.39	0.66 ± 0.06	0.028
PN1	0.41 ± 0.09	0.22 ± 0.09	3.44 ± 0.44	0.52 ± 0.12	0.037
PN2	0.57 ± 0.10	0.17 ± 0.04	4.02 ± 0.53	0.71 ± 0.12	0.031

The bainitic-sorbitic microstructure of heat treated sample and structure of nitride layers can be observed in **Figure 1**. The nitride layer consistes of compound and diffusion layer. The thickness of the compound layer of nitriding PN1 is smaller (2.86 µm) than nitriding PN2 (5.09 µm).



a) Tempered b) PN1 24 H_2 : 8 N_2, 15 h c) PN2 8 H_2 : 24 N_2, 15 h

Figure 1 Cross-sectional microstructure (500x)

The border between the diffusion layer and the substrate of this steel is not obvious, therefore the total thickness of nitride layer should not be measured on photo captured by an optical microscope, but from the microhardness test. The results of microhardness test are given in **Table 4**. The difference of gas mixture leads to distinct microstructure and nitride layer properties. It is seen that the thickness of nitride layer (case depth) of nitriding PN2 is lower than nitriding PN1 but the microhardness is in contrary.

Table 4 Properties of nitride layer

Type of nitriding	Compound layer (µm)	Case depth (mm)	Microhardness (HV 0.05)
PN1	2.86	0.3055	744
PN2	5.09	0.3042	766

The descending of microhardness to case depth is given in **Figure 2**.

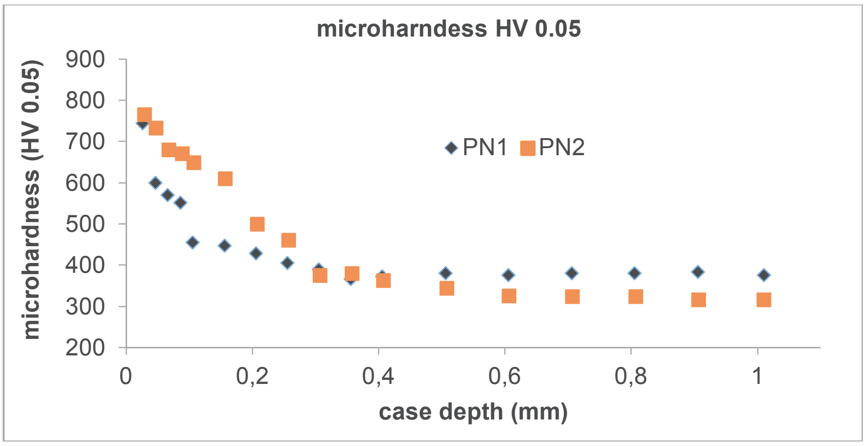


Figure 2 Microhardness of nitrided sample

The surface hardness measured by various loads is given in **Table 5**. With the growth of used load, the value of hardness goes down because the depth of indenter imprint is greater, the effect of nitride layer gets smaller than lower load.

Table 5 Properties of nitride layer

Sample	HV 1	HV 3	HV 5	HV 10	HV 30
Tempered	317 ± 9	315 ± 8	320 ± 8	310 ± 9	307 ± 2
PN1	602 ± 9	717 ± 26	699 ± 37	619 ± 18	515 ± 15
PN2	740 ± 20	772 ± 24	722 ± 14	704 ± 15	588 ± 15

The change of coefficient of friction (next time just COF) to a number of cycles at 21 °C is presented in **Figure 3**. At 21 °C, three samples don't have an obvious difference of COF. The COF of nitriding PN1 rose at the end of the measurement (about 1300 cycles). It can be explained by that, after a running-in period, the brittle compound layer was chipped. The debris particles transferred along wear track made the COF relatively low. Till the compound layer was peeled off, the indenter contacted to diffusion layer, the COF was increased. Although nitriding PN2 obtained thicker compound layer, the COF of nitriding PN2 began rose sooner, after about 500 cycles.

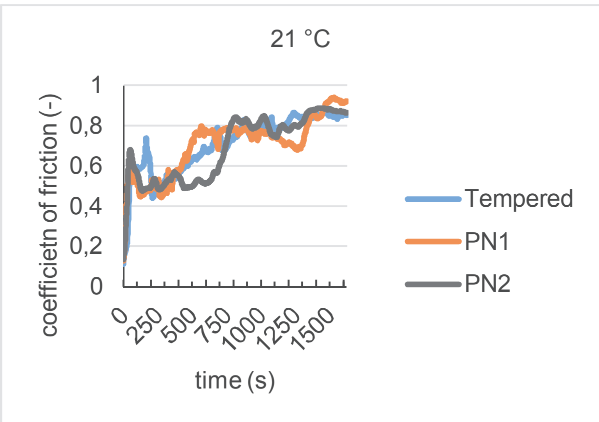


Figure 3 Coefficient of friction at 21 °C

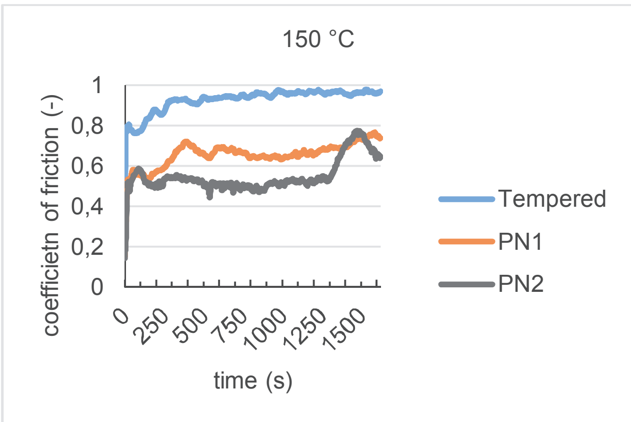


Figure 4 Coefficient of friction at 150 °C

At 150 °C, the tempered sample showed a high COF after a while from the beginning of the measurement. The COF of nitriding PN1 rises until 300 cycles then the COF is stable. The COF of nitriding PN2 became stable the first time after 250 cycles, and the second time after 1300 seconds, then the COF rose (see **Figure 4**).

At 300 ºC, the COF of nitrided samples is much lower than tempered and the amplitude of COF changes more than at 150 ºC and 21 ºC (see **Figure 5**).

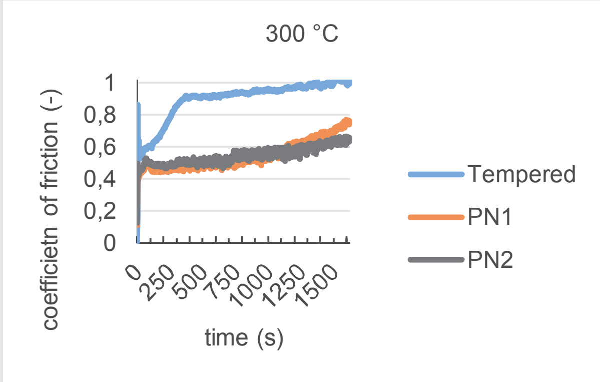


Figure 5 Coefficient of friction at 300 ºC

It can be explained by the thickness of the compound layer. Nitriding PN1 has a thinner compound layer, the COF rises until the compound layer was peeled off. The nitriding PN2 has a thicker compound layer, then the COF rises when at the time of 1300 seconds, the compound layer was completely removed, the COF become stable.

The value of friction coefficient is considered of the stable section of friction coefficient. For example, the coefficient of friction of blue curve of the tempered sample is averaged from 400th cycles to end of the measure. The calculated coefficient of friction is showed in **Table 6**.

Table 6 The average of friction coefficient at various temperatures

Sample	21 °C	150 °C	300°C
Tempered	0.85	0.95	0.96
PN1	0.79	0.66	0.55
PN2	0.83	0.55	0.55

The COF of tempered samples slightly increased to temperature, but still very high. On the contrary, the COF of nitrided samples decreased to temperature.

The wear depth and profile area of wear track, as result measured 5 places on the wear track is summarised in **Table 7**. The wear depth and profile area of wear track of nitrided samples rose to the temperature. But the tempered showed a lower wear depth and profile area at elevated temperature than ambient temperature, and at 150 ºC their values are lower than at 300 ºC.

It is amazing that at elevated temperature, the wear depth and profile area are lower than nitrided samples. It can be explained by the continuous formation of oxide layer on tempered surface at elevated temperature. And this oxide layer can play a role as a self-lubricant, which protects against penetration of the indenter.

As seen in **Table 7**, the wear depth at 21 ºC is lower than compound layer thickness, and at elevated temperature, the wear penetrates to the diffusion layer.

Table 7 The depth and profile area of wear track

Sample	Wear depth (µm)			Profile area (µm^2)		
	21 °C	150 °C	300 °C	21 °C	150 °C	300 °C
Tempered	5.52 ± 1.12	2.39 ± 0.73	3.95 ± 0.83	2158 ± 879	745 ± 111	1561 ± 392
PN1	2.94 ± 0.46	4.46 ± 0.56	9.38 ± 3.34	867 ± 182	1369 ± 269	5814 ± 2584
PN2	3.3 ± 0.42	8.57 ± 2.45	10.53 ± 2.51	1021 ± 107	1332 ± 241	5090 ± 1124

The next useful result of profile measurement is area of the wear profile, from which it can be calculated the wear volume by a simple equation: V = 2Π.R.A, where R is the rotary radius of indenter (mm), A is profile area (mm^2). Then the wear rate is calculated by this equation [1]:

$$w = \frac{V}{F_N . s} \qquad (1)$$

Where: w - wear rate ($mm^{-3}.N^{-1}.m^{-1}$), V - removed volume (mm^{-3}), F_N - normal load (N), s - trajectory of motion (m).

Figure 6 shows the relationship between wear rate and coefficient of friction at various temperatures. It is an amazing trend that the nitrided samples showed a reduction of friction coefficient with the growth of temperature, but on the contrary, the wear rate is increased. For the tempered sample, the coefficient of friction increased to the growth of temperature. But at elevated temperature, the wear rate is lower than at ambient temperature.

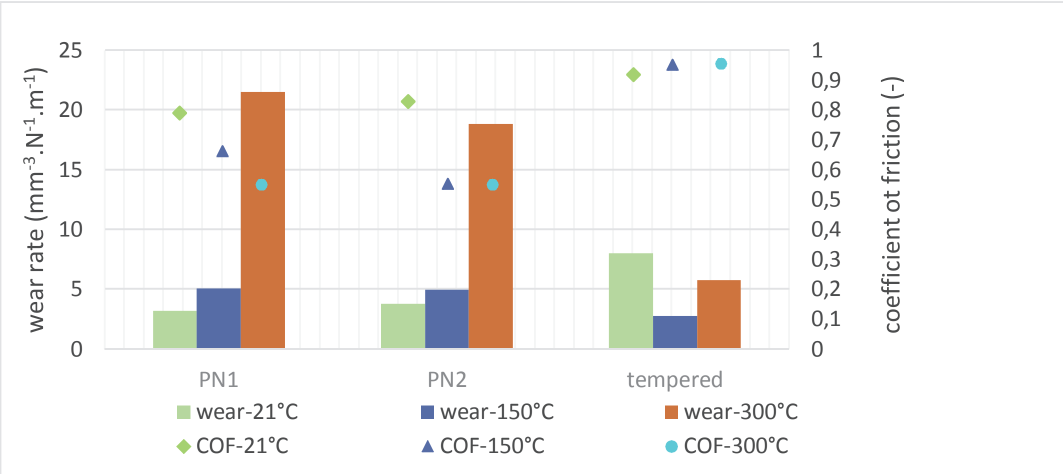


Figure 6 Relationship between the wear rate and the coefficient of friction

4. CONCLUSIONS

This article studied the formation of the nitride layer, its microstructure, surface roughness, hardness and tribological behaviour of nitrided 42CrMo4 steel. The experiments results confirm the presence of ε-phase phase as mentioned by Saïed et al. [12], which makes nitride layer had greater hardness, thicker compound layer.

The wear test was carried out at ambient and elevated temperature (21 ºC, 150 ºC and 300 ºC). At ambient temperature, nitrided sample enhanced wear resistance obviously. But at elevated temperature, the tempered sample showed a smaller wear than the nitrided sample. It is considered that, at elevated temperature, the continuous formation of oxide layer plays a role as self-lubricant, and it decreases wear. But at the point of corrosion protection, Kusmic et al. [14] pointed out that, the corrosion resistance of nitrided 42CrMo4 steel under the same nitriding process conditions exhibited much higher than tempered sample (the NSS corrosion test).

It is showed that the wear depth of plasma nitriding PN2 ($8H_2$:$24N_2$) is greater than nitriding PN1 ($24H_2$:$8N_2$) but the wear rate of nitriding PN2 at elevated temperature is lower than nitriding PN1. Because the shapes of wear profile of two samples were not equal, therefore the wear profile area of nitriding PN2 is smaller than nitriding PN1 and wear rate is proportional to the profile area.

It can be also recognised that, although the PN2 had a greater hardness, thicker and harder compound layer, but at ambient temperature, the wear depth and wear rate were greater than nitriding PN1. It can be explained by the harder but more brittle compound layer may appear tiny fragments and peeled off, which can accelerate the process of removing materials (abrasive mechanism).

The coefficient of friction of nitrided layer decreased according to the growth of temperature. The trend of friction coefficient is opposite for tempered sample.

In general, the nitride layers improved obviously the hardness and wear resistance at ambient temperature, they also decreased the coefficient of friction. But at elevated temperature on the tempered surface, an oxide layer was formed and increased wear resistance, while the nitride layer decreased wear resistance. Netherless, in service, the component surface is affected by various chemical aggression and has to fulfil requirements of strength, hence plasma nitriding is a good treatment that we should consider.

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