

WEAR RESISTANCE ENHANCEMENT OF CASE-HARDENING STEELS BY UTILIZING PLASMA NITRIDING IN MILITARY APPLICATIONS

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Abstract

The paper presents results of the research focused on testing of wear resistance of a selected case-hardening steel after application of a selected plasma nitriding technology. The steel equivalent 16MnCr5 (i. e. CSN 41 4220) is frequently utilized in manufacturing of large diameter components, in automotive and small arms industry. The chemical composition of tested steel was verified by optical emission spectrometry on the instrument Tasman Q4 Bruker. The microstructure, tribology tracks and tracks obtained by Scratch Test were observed by Opto-digital microscope Olympus DSX500i. The depth of nitrided layers was measured by utilizing of microhardness profiles obtained by the automated microhardness tester LM247 AT LECO equipped by hardness testing system AMH55. The tribometer Bruker UMT-3 TriboLab was utilized for measurement of kinetic friction coefficient by the method Ball-on-Flat. The same instrument was also utilized for Scratch Test. The wear resistance was assessed according to the cross-sectional profiles of wear tracks obtained by profilometer Talysurf CLI1000. The paper provides the results proving benefits connected with the utilization of the plasma nitrided layers on the selected case-hardening steel.

Keywords: Plasma nitriding, case-hardening steel, wear resistance, friction coefficient, microhardness

1. INTRODUCTION

Wear resistance is one of the key material properties of the functional surfaces of components which are exposed to abrasion, a typical mechanical process that leads to degradation of contact surfaces. For a reason to enhance the wear resistance surface technologies in many branches of industry especially in automotive and weapon industry are utilized. In case of barrels of small arms and large-caliber artillery barrels or engine and transmission components such as shafts, crankshafts, gear wheels etc. it is crucial to obtain hard and wear resistive microstructure near the surface while the microstructure of the core must remain tough enough to resist bending, torque or impacts [1-5]. For the reason a heat treatment such as surface hardening and a chemical-heat treatment such as carburizing, which meet mentioned criteria, are used the most commonly. Both processes utilize martensitic transformation of steel's microstructure in a region near the surface. The martensitic transformation is non-diffusional structural transformation induced by quenching from high temperatures typical for austenitic phase. Martensitic microstructure is very hard and brittle after quenching and hence it should be tempered subsequently. A result of the mentioned treatments is a creation of a surface layer with a hardness decreasing inwardly to the core of treated components. Whereas, in case of surface-hardening mainly structural changes as martensitic transformation are taking place, in case of carburizing a diffusion of carbon from liquid, gaseous or solid (powder) atmosphere rich in carbon precede the quenching.

By the carburizing a content of carbon in the region of the surface microstructure is increased. Due to this fact, case-hardening steels predetermined for the carburizing are carbon or low-alloyed steels with a low content of carbon which does not exceed 0.3 wt. % [6]. After quenching the component's core with low content of carbon in its microstructure is composed of low-carbon martensite, bainite, pearlite and ferrite. While such the

microstructure provides the required toughness of the core, the surface saturated by carbon with its content about 0.8 – 1 wt. % allows to obtain much harder martensite in comparison with solely surface-hardened state [7].

Unfortunately, the utilization of carburizing is subjected to some risks. By the rapid cooling within quenching, a diffusion of particles is limited, which results in a residual stress in the microstructure and it can be also remained after subsequent tempering. This phenomenon can be manifested by a distortion leading to a geometrical and dimensional changes of components, brittleness and ruptures in their microstructure [1]. In sphere of military technology a high quality of components, including very precise manufacturing, is demanded [8]. This is the reason of effort to substitute this technology by some alternatives. One such alternative technology is plasma nitriding or very close ferritic nitrocarburizing.

As well as carburizing, plasma nitriding also belongs to chemical-heat treatment technologies. The plasma nitriding is based on a saturation of the component's surface by an atomic nitrogen from a gaseous atmosphere in abnormal glow discharge. By the process a nitrated surface layer with a chemical composition different from an original one is created. In the layer the content of nitrogen in the microstructure decreases in direction inwardly to the core material where the chemical composition is equal to the original one. While in case of surface-hardening and carburizing a creation of martensitic microstructure is responsible for increase of hardness, nitriding utilizes ability of some chemical elements such as Cr, Mo, Al, V act. to create very hard and stable nitrides and carbides. Nitriding temperature can be much lower than in case of carburizing, typically in range of 480 - 540°C. Nitriding technology is also promising for future utilization in sphere of additive technology or for a treatment of medical instruments [7,9,10].

Plasma arises by ionization of a low pressure gas present between nitriding recipient (anode) and treated components (cathode). Contrary to nitriding in a standard gaseous atmosphere, where an only source of nitrogen is ammonia (NH₃) with strictly determined stoichiometry 1:3, utilizing of plasma allows us to use gases N₂ and H₂ for a creation of the nitriding atmosphere [11]. This results in a variability of atmospheres' composition and wide possibility of theirs control. This is an important advantage. It is well-known that chemical composition of steels has a crucial effect on a resulting nitride layer and many of articles are devoted to this issue e.g. [12-14]. By a purposeful control of the atmosphere's composition and other parameters such as pressure, temperature or voltage, current and plasma density, it is possible to appropriately set nitriding conditions according to a selection of the material and also influence a composition of the resulting nitride layer as well as its properties [15].

Nitride layer is mostly composed of two (sub)-layers. On the top of the surface a compound (sub)-layer, also called white layer, is created. Compound layer can be dual-phase composed of iron nitrides γ' - (Fe₄N) and ϵ - (Fe₂₋₃N) or mono-phase created solely by ϵ - (Fe₂₋₃N) in accordance to the parameters of the nitriding process. A distinct interface between compound layer and other microstructure is clearly visible, hence compound layer has a character of a coating rather than a layer. The second (sub)-layer is a diffusion layer composed of the original microstructure and nitrides of iron and alloying elements. This layer transit to the original microstructure.

Although, a minor volume increase of nitrated components was documented, due to absence of quenching, other geometrical deteriorations are suppressed [8]. Therefore, the paper is devoted to plasma nitriding and its possibility to enhance wear-resistance of case-hardening steels. This paper partially builds on the results of the previous research described in paper [16].

2. MATERIALS AND METHODS

The selected case-hardening steel and an influence of plasma nitriding on the steel were observed by optical microscopy and examined by the optical emission spectrometry, microhardness measurement, friction coefficient measurement and Scratch Test.

2.1. Materials

The steel equivalent 16MnCr5 (i. e. CSN 41 4220) frequently utilized in manufacturing of large diameter components, in arms and in automotive industry esp. in manufacturing of shafts, crankshafts, gear wheels etc. was selected for the application of plasma nitriding and for evaluation of its influence on the wear-resistance of the steel.

2.2. Chemical composition

The chemical composition of the steel and its compliance with its datasheet was verified by employing optical emission spectrometer Tasman Q4 Bruker. The chemical composition was determined as an average from five measurements by utilizing Fe110 method. The results are presented in (**Table 1**).

Table 1 Chemical composition of the steel verified by Tasman Q4 Bruker (wt. %)

C	Mn	Si	Cr	Ni	Al	P	S
0.177	1.121	0.254	1.032	OES/Bulk 0.057	0.022	0.010	0.005
0.14 - 0.19	1.00 - 1.30	≤ 40	0.80 - 1.10	Datasheet -	0.015 - 0.05	< 0.035	< 0.035

It is evident that the chemical composition of the steel comply with the datasheet. Moreover, a relatively high amount of chromium (1.032 wt. %) promise an increase of the hardness by application of the nitriding due to an ability of chromium to create hard, stable nitrides.

2.3. Specimen preparation

The selected steel was obtained in a normalized state in a form of a steel rod. Two disc-shape specimens were cut-off from the rod and heat treated subsequently in accordance with conditions listed in (**Table 2**).

Table 2 Parameters of heat treatment

HARDENING			TEMPERING		
TEMPERATURE [°C]	TIME [min]	MEDIUM	TEMPERATURE [°C]	TIME [min]	MEDIUM
870	20	water	600	60	water

The heat treatment was followed by grinding finished by F-1000. Solely one specimen was plasma nitrided at 530 °C for 15 hours in PN60/60 RÜBIG appliance subsequently. The second specimen was retained in the heat-treated state. Subsequently, a segment of each specimen was cut-off for a reason to prepare metallographic cross-sectional specimens. The segments were molded to thermoplastic cylinders, grinded and polished by velvet with diamond paste with 0.5 µm sized grains subsequently.

2.4. Microstructure

Inverted Opto-digital microscope Olympus DSX500i was utilized for observation of the microstructure of the steel in original heat-treated state as well as for determination of the white layer thickness. The observed microstructure already published in [16] was in accordance with the parameters of the heat-treatment. After 15 hours of plasma nitriding in the nitriding appliance the white layer with thickness oscillating around 4 µm covered the specimen's surface. A significant porosity was not visible in the compound layer, hence the compound layer can also provide an enhancement of the surface's corrosion resistance [17,18].

2.5. Microhardness measurement

By utilizing a method listed in standard ISO 18203:2016(E) it is possible to determine so called nitriding hardness depth (NHD) from microhardness profiles [19]. Microhardness measurement was performed on the automated microhardness tester LM247 AT LECO. According to the results also already published in the paper [16] the NHD = 357 μm was determined. The maximum values of the hardness measured in a vicinity of the surface oscillated around 720 HV. In the literature, an increase of the microhardness in the surface layer is associated with an increase of the wear-resistance [20]. Thus, it is possible to assume the wear-resistance enhancement by application of the plasma nitriding.

2.6. Friction coefficient measurement

Kinetic friction coefficient was measured on the tribometer Bruker UMT-3 TriboLab by utilizing the Ball-on-Flat method which is described in the standard ASTM G133-05 [21]. The specimen is horizontally mounted to the linearly reciprocally movable support. A measurement parameters such as a load, frequency and also a duration must be preset. At first the ball indenter is vertically pressed to the specimen surface by an increasing force till the force reaches the predetermined value. After that, the support starts to move and the indenter slides the surface. Values of the indenter's load, friction force, acoustic emission and kinetic friction coefficient in dependence on time within the whole measurement duration are recorded. The parameters of measurements were set as follows: load = 10 N, duration = 1000 s, frequency = 5 Hz, stroke length = 10 mm, temperature = 23 °C, diameter of tungsten carbide ball indenter = 6.35 mm, medium – dry.

Kinetic friction coefficient is given by the formula 1 [21].

$$\mu_k = \frac{F}{P} \quad (1)$$

where:

μ_k – kinetic friction coefficient,

F – friction force (N),

P – normal force loading the indenter (N).

Results of the friction coefficient measurement are plotted as a curve into a graph in dependence on time. It is possible to distinguish four phases of tribology wear in the wear track within the measurement and that is also visible in the graph. Phases of initiation of wear and mutual adaptation of surface's roughness are called "running-in phase" [22]. Uneven values of the friction coefficient are typical for this phase. Due to an oxidation and due to a movement and entrapment of wear particles in the wear track, a protective tribology film is created hence the values of the friction coefficient become stable subsequently. This phase is called "Steady-state-wear" [22]. Although the last phase describing a total destruction of the surface also exists, measurements performed for a requirements of the paper were finished within the steady-state-wear phase.

According to experience from previous research, it is possible to assume, that the kinetic friction coefficient of nitrided surface in such measurement condition will be deteriorated (i.e. increased values) in comparison with the heat treated surface [23].

2.7. Wear-resistance assessment

For the assessment of the wear resistance of tested surfaces a cross-sectional profiles of the tribology wear tracks and scratches obtained by the Scratch Test method described in a following text were utilized. The cross-sectional profiles were measured by profilometer Talysurf CLI1000 with a contact sensor. Values of parameters of the cross-sectional profiles such as a profile area, a track width and a maximum depth were determined by the Talyprofile Platinum software.

As it was already mentioned, the Scratch Test method was also utilized for the assessment of the wear resistance. The experimental method is similar to the Ball-on-Flat, hence the similar instrument (tribometer Bruker UMT-3) it is possible to use. Only three main differences between the performances of mentioned experimental methods exist. For the Scratch Test much slower support is utilized, solely one linear scratch is performed within one measurement and the ball indenter is substituted by the Rockwell's diamond HRC indenter. Generally, the Scratch Test is frequently utilized either for an assessment of Scratch hardness or mechanical failure modes of coatings such as adhesive or cohesive failures [24]. Due to the fact, that white layer behaves like coating, an observation of mechanical failure modes was utilized for the assessment of the wear-resistance. Two modes of Scratch Test can be performed. The first utilizes linearly increasing load of indenter within scratching, while in the second case, a constant load within scratching is maintained.

In the experimental measurements both modes of Scratch Test were used. At first, a scratch made with linearly increasing load was performed on the plasma nitrided surface. By microscopy a distance between the first cohesive ruptures and the initial point of the scratch was determined. Because of proportionality which exists between the distance and the linearly increasing indenter's load, the load typical for cohesive failure from the measurement can be deduced. The load can be considered as a critical load of the nitrided surface while using the Rockwell HRC indenter. The load was used for further measurements at constant indenter's load subsequently performed on surfaces of the plasma nitrided as well as on the heat-treated specimens. The parameters of measurements were set as follows: linearly increasing load of the indenter = 0 N to 50 N, constant load of the indenter = 28 N, duration = 60 s, scratch length = 10 mm, temperature = 23 °C, indenter – Rockwell's diamond HRC indenter, medium – dry.

3. RESULTS AND DISCUSSIONS

Experimental methods mentioned in the previous chapter were applied on the heat-treated and the plasma nitrided specimens for a reason to compare influence of the plasma nitriding on wear-resistance of the selected case-hardening steel. Such obtained results are presented below.

3.1. Friction coefficient measurement

Values of friction coefficient of the heat treated and the plasma nitrided surfaces, plotted together into a graph are visible in (Figure 1).

The expectation stated in the method's description related to the deterioration of friction coefficient by application of the plasma nitriding was confirmed. While running-in phase of heat-treated surface took about 50 seconds and transits straight to steady-state-wear phase, where friction coefficient values were moving between 0.4 and 0.5, in case of the plasma nitrided surface an increase of values is visible during 350 seconds and then stabilizes around value 0.65.

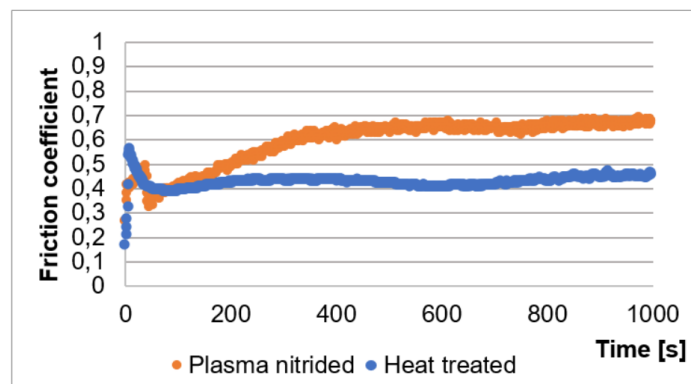


Figure 1 Friction coefficient measurement

The increase of friction coefficient values can be attributed to a movement of wear particles between contact surfaces, where in case of nitrided surfaces the mentioned particles also includes hard nitrides, which make an indenter's sliding more difficult.

3.2. Wear-resistance assessment

As it was stated already, the assessment of wear-resistance of heat treated as well as nitrided surface was based on cross-sectional profiles measurement performed by the profilometer Talysurf CLI1000. Average values of the measured parameters are listed in **Table 3**.

If solely the results listed in the table had been used for the wear-resistance assessment, a serious misinterpretation would have been done because the results seem to be very similar. For a better perception of differences between both wear tracks the results are supplemented by the (**Figure 2**) showing the profiles geometry.

Table 3 Average values of cross-sectional profiles' parameters of tribology wear tracks

	Maximum depth [μm]	Profile area [μm^2]	Track width [μm]
Heat treated	3.88	193.33	188.33
Plasma nitrided	1.42	181.00	206.50

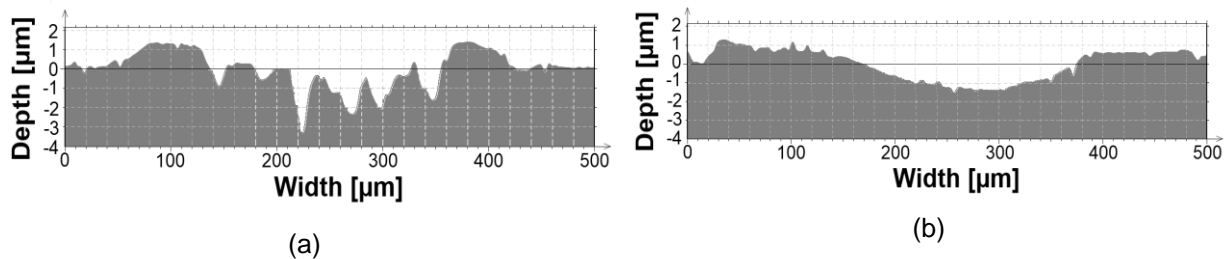


Figure 2 Examples of cross-sectional profiles of tribological wear tracks; a) heat treated; b) plasma nitrided

Despite the high similarity of the values listed in the (**Table 3**) a significant differences in profiles geometry exist and need to be included to the assessment. While the profile geometry of the wear track created on the heat treated surface could be described as very rough including sharp peaks and notches which could cause an initiation of ruptures within a service of components, the profile geometry of the wear track created on the nitrided surface fluently follows the geometry of the parabola. Thus, the plasma nitrided surface seems to be much suitable from a point of view of the wear-resistance.

More distinct differences were found in case of cross-sectional profiles of scratches obtained by the Scratch Test. A scratch obtained on the plasma nitrided surface with linearly increasing load of indenter in range from 0 N to 50 N and the detail of the first cohesion ruptures at the distance 5.68 mm (28.4 N) are shown in (**Figure 3**).



Figure 3 Scratch Test wear track with detail of the first cohesive ruptures at 5.68 mm (28.4 N); magnification: 200x and 1000x respectively.

According to the scratch observation the load 28 N was established as critical one. The load was utilized for subsequent measurements with constant load of the indenter performed on the surfaces of both specimens. **Table 4** includes average values of cross-sectional profiles parameters of the scratches.

Contrary to the previous case, a positive influence of the plasma nitriding on the wear-resistance is obvious even from the table. The positive influence of the plasma nitriding is also confirmed by the profiles geometry. Examples of cross-sectional profiles of scratches obtained at the constant load of the indenter are shown in (Figure 4).

Table 4 Parameters of cross-sectional profiles of the Scratch Test wear tracks

	Maximum depth [μm]	Profile area [μm^2]	Track width [μm]
Heat treated	6.10	491.00	110.67
Plasma nitrided	1.33	64.53	76.53

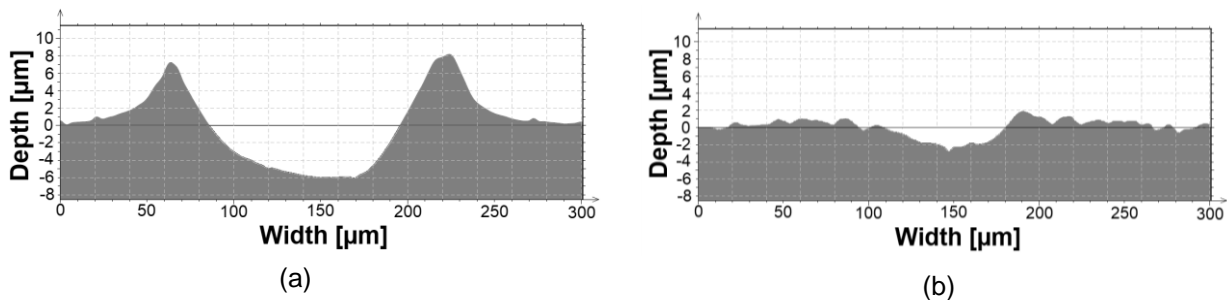


Figure 3 cross-sectional profiles of Scratch Test wear tracks; a) heat treated; b) plasma nitrided

4. CONCLUSION

The aim of the paper was to ascertain a possibility of wear-resistance enhancement of the surface of case-hardening steel 16MnCr5 by application of selected plasma nitriding technology. The plasma nitriding was chosen as an alternative surface technology for carburizing for a reason to reduce possibility of geometric distortion occurring during quenching as a subsequent process of carburizing. By the research the following results were found.

The nitrided layer obtained after 15 hours in the nitriding appliance was composed of 4 μm thick compound layer and the diffusion layer of nitriding hardness depth equal to 357 μm . The maximum values of microhardness about 720 HV near the surface was measured.

By the friction coefficient measurement a prolongation of the running-in phase as well as significant increase of the friction coefficient values after application of the plasma nitriding was found. The phenomenon could be attributed to a movement of hard nitrides along a contact area in the wear track causing the surface more resistive to a movement of indenter.

The wear-resistance was assessed by utilizing parameters of the cross-sectional profiles of the wear tracks obtained by Ball-on-Flat as well as Scratch Test methods. While in case of the tribology wear-tracks obtained by the Ball-on-Flat the values of parameters of both tested surfaces were similar, in case of Scratch Test a significant enhancement after plasma nitriding was found. By comparison of all cross-sectional profiles geometry, the positive benefit of the plasma nitriding in all cases was confirmed.

In the paper the results proving the surface wear-resistance enhancement by application of plasma nitriding on the steel 16MnCr5 are presented. Thus, in those cases, where very deep surface case is not demanded, a substitution of carburizing by the plasma nitriding should be promoted for a reason to avoid possible geometric distortion and cracks occurrence in consequence of quenching after carburizing, while the surface hardness and the wear resistance is remained. Moreover, in many applications where components are subjected to a corrosive environment especially in military applications, a surface corrosion resistance by a presence of nitrided layers could be enhanced.

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