



<sup>1</sup>Iliia ZUMBILEV

## ABOUT WEAR RESISTANCE OF NITRIDED AND CARBONITRIDED LAYERS

<sup>1</sup>Technical University of Sofia – Branch Plovdiv, BULGARIA

**Abstract:** The aim of this work is to study the influence of nitriding and carbonitriding in a low-temperature plasma over steel 25CrMnSiNiMoN in an environment of ammonia and carburizing gases corgon and propane-butane on the wear resistance of the formed layers. The influence of the filling medium (ammonia corgon, propane-butane) in ion carbonitriding process and ion nitriding over the wear resistance of the layers formed is examined. The results indicate the co-relation between the use of the gases and the durability of the nitrided and carbonitrided layers. It has been found that steel 25CrMnSiNiMoN after ion carbonitriding in a medium of ammonia and corgon has the highest wear resistance of the three environments of saturation (ammonia, corgon, propane-butane).

**Keywords:** nitriding, carbonitriding, wear resistance, layers

### 1. INTRODUCTION

Supplying with machine-building products of high quality is directly connected with the increase of their reliability and durability which are defined from the durability of the products.

In the modern machine-building the ion nitriding and ion carbonitriding are widely used for the enhancement operational characteristics of the pieces and instruments.

The basic factors which influence on the durability can be divided into technological, constructive and operational [2, 4, 7]. Related to the technological are the structure, chemical, physical and mechanical properties and to constructive – a scheme of the contact, macro- and microgeometry of friction surfaces, lubrication diagram and more. Operational factors are relative speed of sliding, the relative load, temperature control and lubrication. Forming in the process of friction surface layer is characterized by high free energy and physical and chemical activity, but also with certain mechanical properties. It is this layer that depends on the mechanisms of contact reaction and the level of destruction by friction. During the friction of the materials occurs the so-called friction anisotropy, i.e. the friction coefficient depends on the orientation of the friction surface with respect to different crystallographic planes [6, 7]. It has been found that adhesion and friction coefficient depend on the nature of the crystal lattice. The materials with a hexagonal grid have a decreased adhesion, low coefficient of friction and higher wear resistance in comparison with metals having VCC or WCC lattice. It has been proven that by reducing of the coefficient of linear expansion and increasing of Young E's modulus of materials is increased their wear resistance [4, 5, 7].

It may be noted that the nitride and carbonitrided zones are characterized by a certain wear resistance, with which in most cases they exceed the cementated layers.

To define the wear of thin films are used different weight methods of PIN on Disc, Cylinder on Disc I Taber Test and a common drawback of the methods is that although the define of the export table of the coatings is accomplished using electronic scales with high accuracy (of the order of  $1 \times 10^{-5} g$ ) there is a measurement error that often exceed 10%. This applies particularly at smaller and harder RVD and SVD coatings with a thickness of less than 5 mm when combined with a small area of wear is obtained measurement error of up to 30%. When using an equipment of the existing companies BAQ-kaloMAX NT and CSM instruments - CALOTEST to define the abrasion resistance of thinner coatings, a major problem is the fixing of the angle between the sample and the solid roll in the scheme solid roll – surface.

To increase the wear resistance of the materials is necessary targeted improvement of the properties of their surface layer, which can be achieved by thermal, chemical-thermal treatment, and other types of a strengthening processes [1, 3, 10, 11].

The purpose of this work is to study the influence of nitriding and carbonitriding in a low-temperature plasma environment of ammonia and carburization Corgon gas and LPG on the wear resistance of the formed layers.

## 2. RESEARCH METHODS

### 2.1. Research materials and nitriding and carbonitriding modes

Research is put under complex alloyed steel 25CrMnSiNiMoN. Its chemical composition is studied with an instrument for automated analysis "Spektrotest" and is given in Table 1. From the steel are made samples with dimensions 15 x 15 x 10 mm.

Table 1. Chemical composition of the studied materials

Steel	Chemical elements, weight percentage					
	C	Si	Mn	Cr	Ni	Mo
25CrMnSiNiMoN	0.24	1.45	1.28	0.87	1.36	0.12

Table 2. Pre-heat treatment conditions

Steel	Hardening $t_{hard.}, ^\circ C$	Cooling medium	Tempering $t_{tem.}, ^\circ C$
25CrMnSiNiMoN	880	Oil	600

Samples are heat treated in a chamber furnace in an oxidizing environment by schemes according Table 2.

Thus heat treated, test pieces were ground with a surface roughness  $Ra = 0.63 \mu m$ . Then, they are ion nitrided and carbonitrided in installation "Ion -20" by schemes according to Table 3.

As a saturating gas have been used two circles, one of ammonia ( $NH_3$ ) and Corgon (82% Ar and 18%  $CO_2$ ), and the other ammonia ( $NH_3$ ) and propane butane ( $C_3H_8 - C_4H_{10}$ ) in a different percentage. Temperature for processing both processes - nitriding and carbonitriding is  $550^\circ C$ .

### 2.2. Metallographic studies

To clarify the morphological features of nitrided and carbonitrided layers is made metallographic analysis. In determining the structure and thickness of the layers was used microscope - Axioskop, by which were made the metallographic photos.

The thickness of the nitrided and carbonitrided layer is determined by the depth to which the microhardness is obtained, equal to 50 plus the core. The measurement of the microhardness of the obtained layers is accomplished with hardmeter - "Leitz" with load 0.98 N (100 g), by the method of Vickers hardness. Grindings for examination were treated with 3% nitric acid solution in ethyl alcohol.

The wear rate is determined by the export volumes of layers  $V_{wear}$ . For this purpose, the diameter d of the formed in the layer footprint of the wear, measured using a measuring eyepiece of an metallographic microscope with an appropriate magnification x200. After that is calculated the worn out volume of coating on the dependency:

$$V_{wear} = \pi h^2 \left( R - \frac{h}{3} \right); \text{ where } V_{wear} \quad (1)$$

– worn out volume of the layer,  $mm^3$ , R – radius of the wearing sphere, mm, h – depth of the worn out area, mm.

For the worn out volume of the layer  $V_{wear}$ , expressed through the radius of the used sphere R and the radius of the worn out area r, the dependence is obtained:

$$V_{wear} = \pi \left[ R - \sqrt{R^2 - r^2} \right]^2 \left[ R - \frac{R - \sqrt{R^2 - r^2}}{3} \right], mm^3 \quad (2)$$

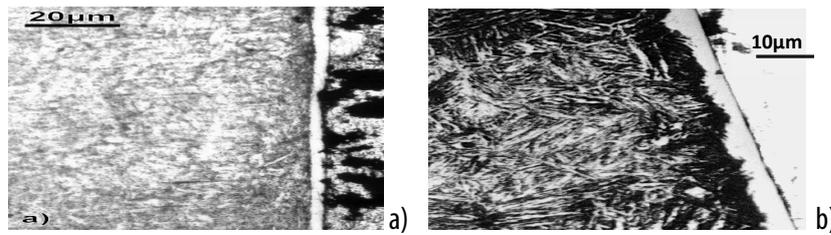
### 2.3. Wear resistance of layers

The formed layer of the surface was subjected to abrasive wear by friction in it by sliding rotatable on the ground and coated with an abrasive paste sphere made of heat-treated bearing steel 100Cr6, DIN17350, which is coated with a TiN with a thickness of  $2 \mu m$  and a hardness of  $HV = 21 GPa$ . For this purpose, the sample is rigidly secured to table of the device orientated to the layer above. Over the shaft is placed the coated with abrasive paste steel sphere. For this purpose, the sample is rigidly secured to table of the device orientated to the layer above. Is placed on the shaft coated with abrasive paste steel sphere. In determining the wear of the films formed was used designed and manufactured by us laboratory stand, which is shown in Figure 1.

All tests of wear of the base material and the layers (carbonitrided zone) were conducted under the same conditions: Revolutions of the shaft on the stand – 700 rev/min, Steel sphere with a diameter – 30mm, Revolutions of the sphere – 373 rev/min, Angle placement of the specimen -  $\alpha = 30^\circ$ . Time of testing – 3 min, Abrasive material - diamond paste with particle size of  $0,25 \mu m$ .

## 3. EXPERIMENTAL RESULTS AND ANALISYS

By measuring microhardness of ion nitrided and carbonitrided steel in depth, is defined a maximum surface hardness  $HV_{0,1}$  and the total thickness of the nitrided and carbonitrided layer  $\delta_{tot}$ - Figure 1. Using a metallographic microscope is set the thickness of the connected zone  $\delta_z$ . The results are given in Table 3. Depending on the mode of nitriding and carbonitriding, durability of test pieces vary throughout range. The results of Table 3 show the positive influence of the ion carbonitriding on the durability of the tested materials. Depending on the mode of carbonitriding, durability vary throughout range. The positive impact of ion carbonitriding over durability is associated with the presence of alloying elements (V, Mo, Cr, Mn) in the solid solution and its increased resistance after saturation with nitrogen, and carbon, as well as with slow coagulation of carbonitride particles. This in turn leads to an increase in the resistance of the carbonitrided layer against plastic deformation.



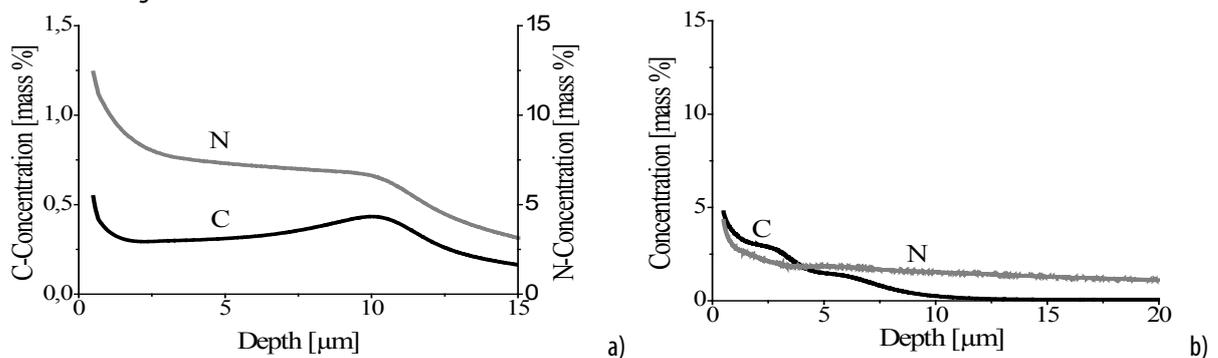
**Figure 1** - Microstructure of steel 25CrMnSiNiMoN after carbonitriding with  $t = 550^{\circ}\text{C}$ ,  
 a)  $P_{\text{NH}_3} = 280\text{ Pa}$ ,  $P_{82\% \text{ Ar} + 18\% \text{ CO}_2} = 120\text{ Pa}$ ,  $\tau = 4\text{ h}$ ; b)  $P_{\text{NH}_3} = 360\text{ Pa}$ ,  $P_{2\text{C}_3\text{H}_8 - 4\text{H}_{10}} = 40\text{ Pa}$

Table 3 shows that the steel 25CrMnSiNiMoN has high durability in two modes of treatment. After ion carbonitriding with temperature  $550^{\circ}\text{C}$ , duration of treatment 4h, a pressure of the ammonia chamber  $P_1 = 280\text{ Pa}$ , Corgon pressure in the chamber  $P_2 = 120\text{ Pa}$  is established the largest increase in the abrasion resistance of up to 54%.

**Table 3.** Results of the wear of nitrided and carbonitrided materials

Nº of mode	Process CTP	$P_1, \text{NH}_3, \text{ Pa}$	$P_2, \text{Pa propane-butane}$	$P_2, \text{ Pa Corgon}$	$P_{\text{tot}}, \text{ Pa}$	$\tau_{\text{tot}} \text{ h}$	$\text{HV}_{0.1}$	$\delta z \text{ } \mu\text{m}$	$r \text{ mm}$	$h \text{ } \mu\text{m}$	$V_{\text{wear}} \text{ mm}^3$	$V_{\text{wear}} \text{ \%}$
1	nitriding	400	-		400	4	1072	9.7	0.315	3,3	0,000515	39
2	carbonitriding Corgon	280		120	400	4	930	6	0.293	3.0	0.000385	54
3	carbonitriding propane-butane	180	20	-	200	4	841	4.2	0.305	3,1	0,000452	46
4	carbonitriding Corgon	200	-	200	400	2	890	5	0.293	3,0	0,000385	54
5	carbonitriding Corgon	350	-	350	700	2	740	5	0,320	3,3	0,000515	39
6	carbonitriding propane-butane	360	40	-	400	4	985	4.5	0.315	3,4	0,000548	35
7	without CTP						35 HRC		0,354		0,000844	

The amount of wear depth  $h = 3\mu\text{m}$ , and the worn out volume of the layer  $V_{\text{wear}} = 0.0003857\text{ mm}^3$  - Mode 2. Similar data (the amount of wear depth  $h = 3.0\mu\text{m}$ , and the worn out volume of the layer  $V_{\text{wear}} = 0.0003857\text{ mm}^3$ ) were observed in mode 4 ( $550^{\circ}\text{C}$  temperature, duration of treatment 2h, ammonia pressure  $P_1 = 200\text{ Pa}$ , pressure Corgon  $P_2 = 200\text{ Pa}$ ). This is due to the presence of a major amount of  $\epsilon$  phase to carbonitrided zone (Figure 3), as well as uniform distribution of the carbon and nitrogen in the connected zone – Figure 2.



**Figure 2** - Distribution of carbon (C) and nitrogen (N) in the depth after carbonitriding of steel 25CrMnSiNiMoN at:  $t = 550^{\circ}\text{C}$ ,  $\tau = 4\text{ h}$ ;  
 a)  $P_{\text{NH}_3} = 200\text{ Pa}$ ,  $P_{82\% \text{ Ar} + 18\% \text{ CO}_2} = 200\text{ Pa}$ , b)  $P_{\text{NH}_3} = 360\text{ Pa}$ ,  $P_{2\text{C}_3\text{H}_8 - 4\text{H}_{10}} = 40\text{ Pa}$

It may be noted that of all the modes of processing steel 25CrMnSiNiMoN after carbonitriding with ion in a medium of ammonia and propane-butane is observed the largest amount of worn out material when:  $550^{\circ}\text{C}$  temperature, duration of treatment 2h, ammonia pressure  $P_1 = 360\text{ Pa}$ , pressure of the LPG  $P_2 = 40\text{ Pa}$ . The amount of wear depth  $h = 3.4\mu\text{m}$ , and the worn out volume of carbonitrided layer  $V_{\text{wear}} = 0.0005488\text{ mm}^3$  (mode 6 - Table 3). The resulting layer has a carbonitriding microhardness  $985\text{HV}_{0.1}$  and a thickness of the connected zone  $4.5\mu\text{m}$ . It is found an increase in the wear resistance of carbonitrided area by 35%. When testing carbonitrided steel 25CrMnSiNiMoN, the sphere has penetrated at a depth of  $3.4\mu\text{m}$  in the carbonitrided zone, which is less than its thickness. Despite the big hardness of the layer, less durability can be attributed to the increased concentration of a carbon (Figure 2) in the zone and carbonitrided presence of a large amount of cementite therein, Figure 3 b. This leads to an increase of the coefficient of the friction.

Test results of wear resistance of the ion nitrided steel 25CrMnSiNiMoN at 550°C, pressure of ammonia 400 and time of processing 4h, shows lower wear resistance from the ion-carbonitride in a medium of ammonia and Corgon and propane butane (Mode 1 - Table 3). It is found an increase in the wear resistance of the carbonitrided area by 39%. This is explained by the difference in the morphology of the phase formation and composition of the carbonitrided and nitrided zone in the ion nitriding and carbonitriding.

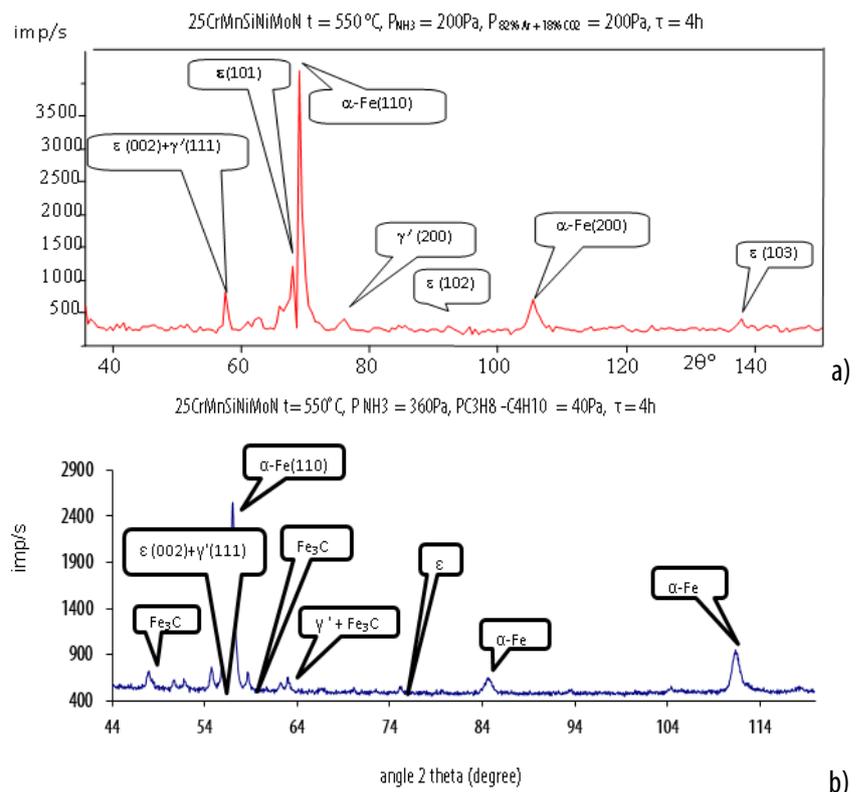
#### 4. CONCLUSION

□ 25CrMnSiNiMoN steel after ion carbonitriding at:  $t = 550^\circ\text{C}$ ,  $P_{\text{NH}_3} = 200\text{Pa}$  (280Pa),  $P_{82\% \text{Ar} + 18\% \text{CO}_2} = 200\text{Pa}$  (120Pa),  $\tau = 4\text{h}$  has the highest wear resistance (wear depth  $h = 3.0\ \mu\text{m}$ , the worn out volume of layers  $V_{\text{worn}} = 0.0003857\text{mm}^3$ ). Lowest wear resistance (wear depth  $h = 3.4\ \mu\text{m}$ , the worn out volume of layers  $V_{\text{worn}} = 0.0005488\text{mm}^3$ ) was observed after carbonitriding in a medium of ammonia and propane-butane.

□ It was found that after ion carbonitriding of the steel a higher wear resistance than after ion nitriding is given.

#### REFERENCES

- [1.] Toshkov V., Theoretical and practical aspects of the nitriding of iron and iron-carbon alloys in low-temperature plasma. Dissertation, Sofia, 1997.
- [2.] Toshkov V., Nitriding in a low-temperature plasma, King, 2004.
- [3.] Rusev R., Theoretically-experimental analysis of the system Fe-N-C and its application in carbonitriding technologies. Dissertation, Varna, 1998.
- [4.] Rusev R., Characteristics, mechanism of the formation and morphology of the carbonitrided layers, obtained in an environment of ammonia and carbon oxide. Dissertation, Varna, 1979.
- [5.] Vurhoshkov E., Carbonitriding of steel in a glowing discharge. Dissertation, Sofia, 1985.
- [6.] Fisher-Chatterjee P., W. Eysell, u.a., Nitrieren und Nitrocarburieren, Sinterfingen, Expert Verlag, 1994
- [7.] Lampe Thomas, Plasmawärmebehandlung von Eisenwerkstoffen in stickstoff- und kohlenstoffhaltigen Gasgemischen, VDI-Verlag GmbH, Düsseldorf, 1985
- [8.] Nitriding and Nitrocarburising, Contract Heat Treatment Association, Aston University, Birmingham, 1996.
- [9.] Toshkov V., A. Zjumbilev. Thermal and Thermochemical Treatment of Tool Steels in Vacuum. Proceedings of the 1-st International Conference on Heat Treatment and Surface Engineering of Tools and Steels, 08-11 June, 2005, Pula- Croatia, pp. 229-234.
- [10.] Toshkov V., A. Zjumbilev, N. Tonchev. Influence of nitrided layers quantitative parameters on wear resistance of tool steels, Proceedings, of the 1-st International Conference - The Coatings in Manufacturing Engineering, 1999, Thessalonki- Greece, pp. 433-439.
- [11.] Zjumbilev A, I. Zjumbilev, About the Wear Resistance of steels, nitrided in high temperature plasma. Proceedings of the 47-th Conference Metallurgists, August 24-27, 2008, Winnipeg, Manitoba, Canada, pp.103-113



**Figure 3** - X-ray diagram of steel 25CrMnSiNiMoN after carbonitriding at:  $t = 550^\circ\text{C}$ , a)  $P_{\text{NH}_3} = 200\text{Pa}$ ,  $P_{82\% \text{Ar} + 18\% \text{CO}_2} = 200\text{Pa}$ ,  $\tau = 4\text{h}$ ; b)  $P_{\text{NH}_3} = 360\text{Pa}$ ,  $P_{\text{C}_3\text{H}_8\text{-C}_4\text{H}_{10}} = 40\text{Pa}$ ,  $\tau = 4\text{h}$