THE ANNALS OF “DUNAREA DE JOS” UNIVERSITY OF GALATI.
FASCICLE IX. METALLURGY AND MATERIALS SCIENCE
N^0. 4 – 2012, ISSN 1453 – 083X

SOME ASPECTS REGARDING THE INFLUENCE OF TRIBOLOGYCAL FACTORS ON THE SUPERFICIAL LAYERS OF STEELS TREATED WITH PLASMA NITRIDING

Carmen Penelopi PAPADATU, Elisabeta VASILESCU
“Dunarea de Jos” University of Galati
email: papadatu.carmen@yahoo.com

ABSTRACT

Some 38MoCrAl09 (SAE 4038) steel grade samples were subjected to plasma nitriding treatment regimes. The structural and diffractometric aspects of the superficial layers of the steel are studied after the wear tests by friction, using an Amsler type machine, taking two sliding degrees at different contact pressures and testing time. The tests were made to detect the sustainability to the material, the evolution of the superficial layer through different tests and to establish the influence of these tribological factors (operating parameters) on the superficial layers.

KEYWORDS: plasma nitriding process, wear process, tribological parameters

1. Introduction

Plasma nitriding, also known as *ion nitriding*, *plasma ion nitriding* or *glow-discharge nitriding*, is an industrial surface hardening treatment for steels and for other metallic materials.

Nitriding alloys are alloy steels with nitride-forming elements such as aluminum, chromium, molybdenum and titanium.

In plasma nitriding, the reactivity of the nitriding media is not due to the temperature but depends by the gas ionized state. In this technique intense electric fields are used to generate ionized molecules of the gas around the surface to be nitrided. The highly active gas with ionized molecules is called plasma.

The gas used for plasma nitriding is usually pure nitrogen.

Plasma nitriding modifies the strain limit, and the fatigue strength of the metals being treated. For instance, mechanical properties of austenitic stainless steel like wear can be significantly reduced and the hardness of tool steels can be double on the surface [1-3]. Plasma nitriding is often coupled with physical vapor deposition (PVD) process and labeled Duplex Treatment, with enhanced benefits. Many users prefer to have a plasma oxidation step combined at the last phase of processing to produce a smooth jetblack layer of oxides which is resistant to wear and corrosion.

The superficial layer is defined according to the type of interaction between the external action and materials.

In figure 1 given presented one of the first model of the superficial layer [2, 4, 6].

Introducing a surface treatment as nitriding process with plasma (ionic nitriding), increase the wear resistance and the resistance of corrosion increase too.

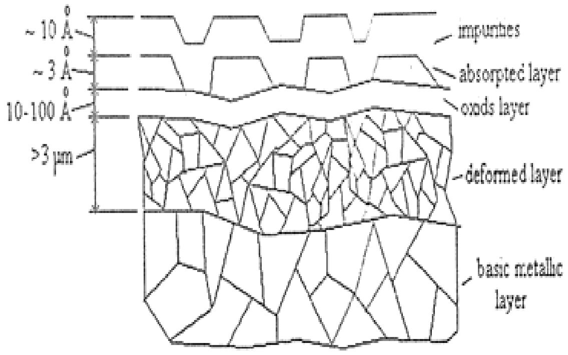

Fig. 1. *One model for the superficial layer*

The diffusion and interaction of the nitrogen with the basic material lead to structural constituents whose nature determines a major hardness of the nitrided layer.

The chemical combination area consists of two phases:

- *the* ε *phase* – a solid solution based on the chemical compound of Fe3N, rich in nitrogen (8.2% to 11.2%N) having a hexagonal compact (HC), crystal lattice, is highly resistant to wear, [1,4,5] and corrosion;

- *the* γ*’ phase* – a solid solution based on the Fe4N compound, presents a centred-face cube (C.F.C.) crystal lattice; has a lower nitrogen solubility (5.7% to 6.1%N), showing very high values of hardness and tenacity.

The diffusion area is composed of the following elements:

- *the* α *phase (the nitrided ferite)* – a nitrogen connate solid solution in Feα crystallizing in centre volume cube (C.V.C.) lattice, showing a maximum nitrogen solubility of 0.11% at a temperature of 590°C;

- *the* γ *phase (the nitrided austenite)* – a nitrogen connate solid solution in Feγ (C.F.C.), having a maximum nitrogen solubility of 2.8% at a temperature of 650°C(see Fig. 2) [2, 4].

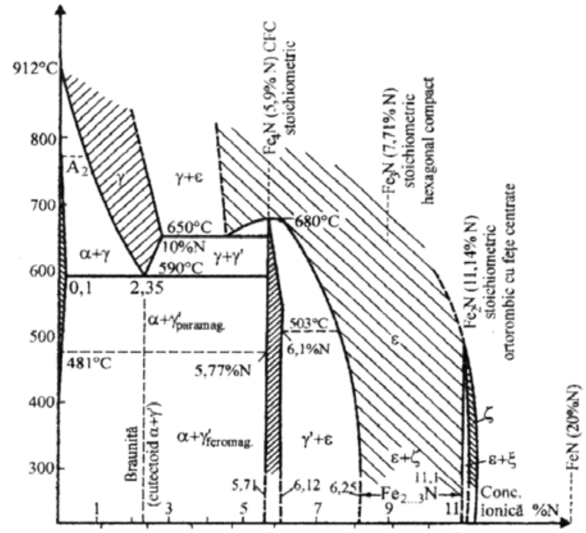

Fig. 2. *Fe-N diagram of equilibrum*

The transformation of γ→α’- martensite with nitrogen (a suprasatured solution of nitrogen in Feα), showing very high values of hardness, takes place from the nitriding temperature, in case of a rapid cooling of austenite with nitrogen (γ).

The hardness and the depth of nitrided layer, are determined both by the chemical composition of the chosen material and by the technological parameters of the thermo-chemical treatment [4].

In case of hard worn out parts [4], the layer structure selected has three areas corresponding to the rule as it is advised by the tribology studies:

a. – a thin layer of 0.02 to 0.04mm, not so rigid, showing a crystal lattice separate to the basic (hexagonal compact) material;

b. – a very tough and flexible layer, which is not breakable or deformable under very high stresses;

c. – a basic material showing adequate hardness, mechanical strength as well as a suitable tenacity in order to avoid material cracking under stress.

Figure 3 show the layer hardness in their sequence [2].

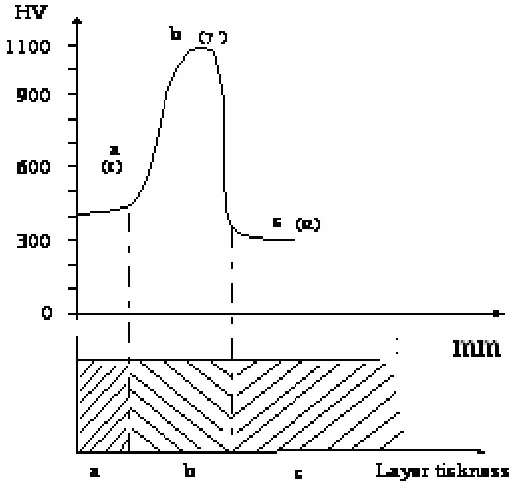

Fig. 3. *Hardness of the superficial layer according with the layer thickness evolution*

The wear behavior of coated surface is controlled by several factors such as the geometry of the contact, the topography of the surface, the material characteristics, mechanical properties of the material, microstructures of the material and the operating parameters (Q, ξ, Δt).

In this paper, are presented a series of researches regarding the structural and the tension state changes that appear and develop in the superficial layer of some materials subjected to damages processes as: running-in, friction, wear or fatigue.

For this aim, the tribomodeling operation has been used. The materials tested on tribomodels present some advantages versus in situ ones. Thus, the multifactorial planning of the experiments may be used and the testing optimal conditions, for a given material, can be found.

The friction and wear processes are complex, being of physical, chemical, mechanical or metallurgical nature[2, 3]. These processes appear during dynamic or static contact between surfaces of two solid bodies where may be or not be in a gaseous atmosphere, liquid or solid lubricant. The depth of the superficial layer varies between some atomic layers, in case of wear and chemical processes, and can attain up to 50-100 μm – in case of dry friction.

2. Experimental researches

For experiments were used 38MoCrAl09 steel grade samples. This material was subjected to the plasma nitriding treatment regime.

The structural and diffractometric aspects of the superficial layers of the steel are studied after the wear tests by friction. It was used an Amsler type machine, taking two sliding degrees at different contact pressures and testing time.

The tests were done to detect the sustainability to the material, the evolution of the superficial layer through different tests and was established the influence of these tribological factors (operating parameters) on the superficial layers.

Table 1 presents the 38MoCrAl09 steel grade composition. The steel analyzed reach a max score 4.5 for the inclusions and a fine grain (score 8-9).

Table 2 presents the mechanical characteristics of the steel 38MoCrAl09 (SAE 4038) [2, 4].

Table 1. *Chemical composition of the materials [%]*

Steel grade	C	Mn	Si	P	S	Cr	Cu	Mo	Al	Ni
38MoCrAl09 (SAE 4038)	0.38	0.50	0.25	0.026	0.020	1.38	0.058	0.17	1.18	0.26

Table 2. *Mechanical characteristics of the steel*

Steel grade	**$Rp_{0,2}$**	**R_m**	**A_5**	**Z**	**$KCU_{300/2}$**	**$KCU_{300/5}$**	**HB (State of annealing)**
	[daN/mm^2]		[%]		[daJ/cm^2]		
38MoCrAl09 AISI(SAE 4038)	85	100	15	50	9	6	229

The samples of the steel suffered the following treatments regimes: a martensitic hardening process at 920°C and high recovery at 620°C (classic improvement treatment noted with "t_1") followed by nitriding process at 530º C. We noted $T_1 = t_1$ + ionic nitriding (plasma nitriding) at 530°C.

The treated samples were used for wear tests on Amsler machine [7, 8, 9]. Were determined the durability of rollers and the surface structure evolution for different parameters of testing regimes. It could not be neglected other factors influencing the wearing process: the contact geometry of the friction couple (roller on roller, roller on ring etc.), the technological parameters (surface quality, heat treatments etc.) and the exploitation conditions (the thermal solicitation, for example).

Wear tests were carried out on an Amsler machine, using several couples of rollers, each couple corresponding to different sliding degrees ξ, defined as:

$$\xi = [\,(v_1 - v_2)\,/\,v_1\,]\ 100\ [\%] \qquad (1)$$

where v_1 and v_2 are the peripheral velocities of the rollers in contact, each one having their specific peripheral velocity due to a particular combination of angular speeds (n_1, n_2) and diameter sizes (d_1, d_2).

Index 1 or 2 are added for the roller 1 or 2, respectively, both of the same tested friction couple. For instance, ξ=10% is obtained for a pair of tested rollers having d_1=40mm, n_1=180rpm and d_2=40mm, n_2=162rpm; *18%*ξ= is obtained for a pair of tested rollers having d_1=44mm, n_1=180rpm and d_2=40 mm, n_2=162rpm; the level of the stress is corresponding to a specific load of 150daN (as normal load is *Q*=1.500 N) and the contact between roller is *b*=10 mm [2,4,5].

3. Experimental results

The phases and the characteristics identified at diffractometry analysis for 38MoCrAl09 steel grade after thermal treatment followed by plasma nitriding, are presented in table 3 [7, 10-*13*]. In this table we considered the following information: **Δθ** represents the angular displacement of a diffraction lines, Q represents the force applied, ξ represents the sliding degrees [%] and **c/a** represents the tetragonality degree of martensite [%].

By comparing the results obtained for code 122 samples (T1, Q=75 daN, ξ = 10%) and for code 222 samples (T1, Q = 150 daN, ξ = 10 %), we obtain the following results given in table 3:

Table 3. *Phases and their characteristics identified by diffractometry analysis*

Code sample	**I_{Fe3N}**	**B_{Fe3N}**	**I_{Fe4N}**	**B_{Fe4N}**	**c/a**	**Δθ**	**t**	**Q**	**ξ**
	[%]		[%]		[%]		[h]	[daN]	[%]
122	9	3.00	16	3.20	1.31	-0.04	0	-	-
122	22	4.40	17	2.98	1.31	-0.04	1	75	10
122	22	4.88	16	3.20	1.29	-0.05	2	75	10
122	19	4.75	16	3.07	1.29	-0.10	3	75	10
222	10	3.33	12	3.00	-	-	0	-	-
222	5	2.94	6	2.444	2.526	-0.10	3	150	10
422	6	4.00	8	3.2	-	-	0	-	-
422	10	3.33	6	2.4	2.00	-0.15	3	190	20
322	4	4	20	1.81	2.526	-	0	-	-
322	6	4.40	26	2.4	2.526	-	1	150	20
322	4	4	20	1.81	2.26	-	2	150	20
322	20	5	26	3.33	2.260	-0.10	3	150	20

For code 122 sample, after each hour of wear process, we couldn’t saw sesizable modification of Phases quantities inside in the superficial layer. But, it was an exception, for Feα quantity, which increase simultaneous with the increase of wear process time. The width of the Fe_4N (40˚ - 42˚) phase increases after three hours of wear process. By comparing the code 222 sample with the code 122 sample, we can observe the following aspects:

- at code122 sample -after three hours of wear process, the Fe_3N phase quantity increased. It was visible the increase of Feα (M) phase quantity too.

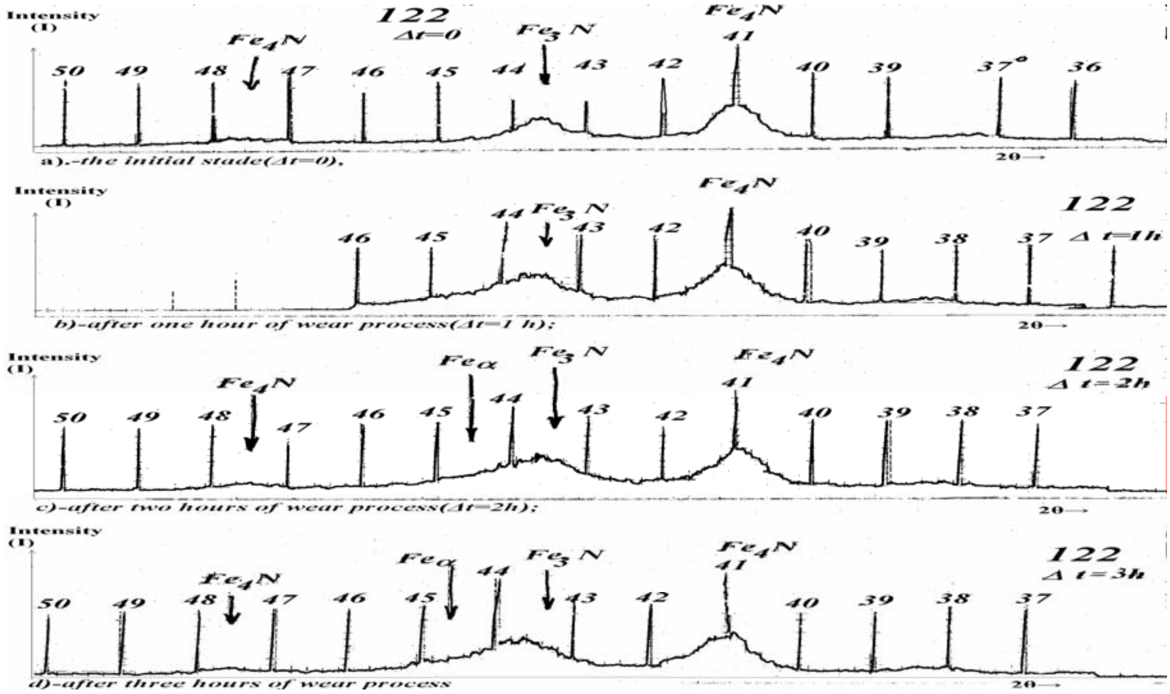

Fig.4. *Diffractometry aspects for code 122 samples (T_1,Q=75 daN, ξ=10%)*: a).-*the initial stade (Δt=0), b)-after one hour of wear process (Δt=1 h); c)-after two hours of wear process (Δt=2h); d)-after three hours of wear process (Δt=3h), at the same conditions*

Results that in the case of classic treatment (T_1), when the sliding degrees (ξ) increase (because the increase of the specific load), the quantities of Feα and Fe_3N phases (hard phases) decrease.

Finally, we can obtain a decrease of hardness value inside in the superficial layers, after wear process through friction. This variation was correlated and verified with the mass loss evolution during and after wear process.

In fig. 4 were presented the diffractometry aspects for code 122 samples, after each hour of wear process.

In figure 5 are presented some difractometry aspects for code 222 samples, code 322 samples and code 422 samples, after three hours of wear process.

The Fe_4N phase quantity is approximately equal in all the three situations. At code 322 sample we can observe the greatest quantity of martensite. All the samples suffered the same treatment: T1 (Classic treatment), the difference between them is the different value of each sliding degrees (ξ). For example code 322 sample was concerned at three hours with Q=150 daN (as the code 222 sample), but the sliding degrees was double then 222 sample case. Between code 422 sample and code 322 sample, the only difference is the increase of the effort value (Q).

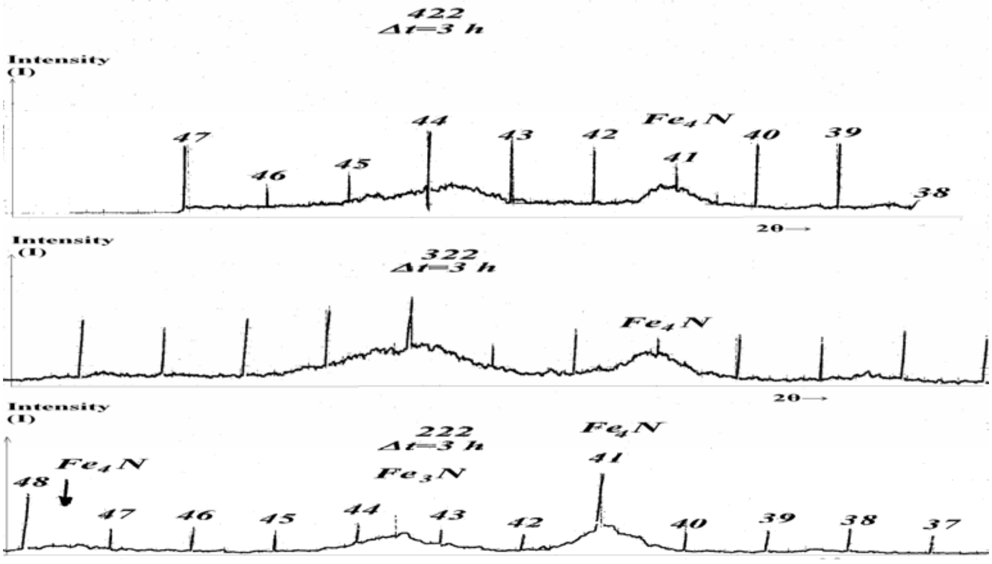

Fig. 5. *Diffractometry aspects for:222 samples (T_1,Q=150 daN, ξ=10%),*
322 samples *(T_1,Q=150 daN, ξ=20%), 422 samples(T_1,Q=190 daN, ξ=20%)*

In figures 6...11 are presented graphic evolutions for layer characteristics (parameters) during the wear process. For example, these parameters as: internal tensions of second order vs. test time or, the evolution of the thetragonality degree of martensite (c/a) vs. test time, discover the influence of the tribological aspects on the superficial treated layers. The tests were done to detect the sustainability to the material, the evolution of the superficial layer through different tests and to establish the influence of these tribological factors (operating parameters) on the superficial layers.

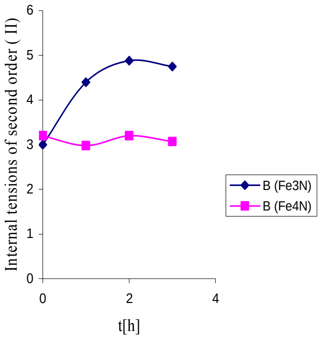

Fig. 6. *Evolution of the internal tensions of second order (B211~σ_{II}) vs.wear test time; (122 sample, Q=75 daN ξ=10%).*

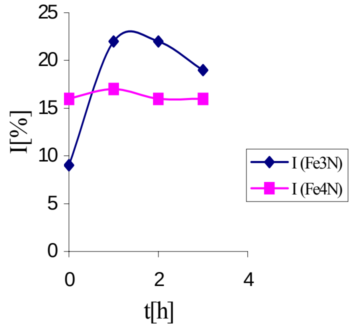

Fig. 7. *Fe_4CN and Fe_3CN phase distribution vs.wear test time (122 sample, Q=75 daN, ξ=10%).*

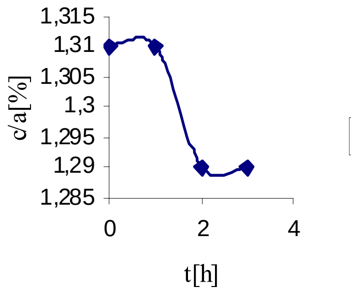

Fig. 8. *Evolution of the martensite thetragonality grade (c/a) vs wear. test time; (122 sample, Q=75 daN, ξ=10%)*

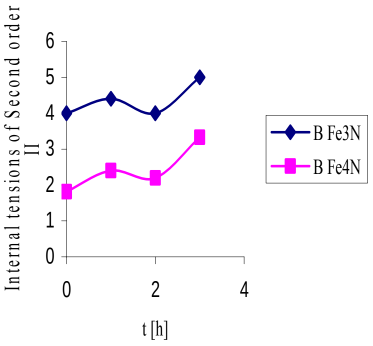

Fig. 9. *Evolution of the internal tensions of second order (B211~σ_{II}) vs. wear test time; (Code 322 sample,Q=150daN ξ=20%).*

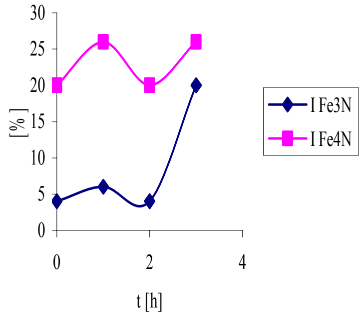

Fig. 10. *Fe_4CN and Fe_3CN phase distribution vs.wear test time (Code 322 sample, Q=150daN, ξ=20%).*

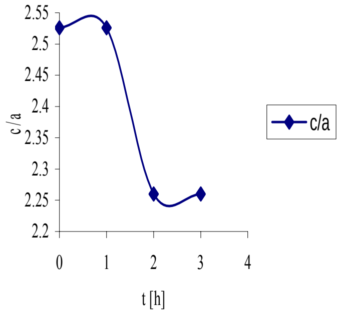

Fig. 11. *Evolution of the martensite thetragonality grade (c/a) vs wear. test time; (Code 322 sample, Q=150daN, ξ=20%)*

Diffractometric analysis were performed by a Dron 3 system [12,13]. The curves of variation for the phases distribution and other characteristics within superficial layers show the evolution of the superficial layer transformation during the wear process.

4. Conclusions

The diffusion and the interaction of the nitrogen with the basic material lead to structural constituents whose nature determines a major hardness of the nitrided layer.

The friction and wear processes are complex, being of physical, chemical, mechanical or metallurgical nature. These processes appear during dynamic or static contact between surfaces of two solid bodies where may be or not be in a gaseous atmosphere, liquid or solid lubricant. The depth of the superficial layer varies between some of atomic layers, in case of wear or chemical processes, and can attain up to 50-100 µm – in case of dry friction.

Analyzing the diffractometries presented in fig.4 (a,b,c,d), it were observed the following aspects : the evolution of residual austenite –$A_{rez.}$- (38°÷47°), the evolution of marthensite -M- (81°÷92° and 44°÷45°), the evolutions of Fe_3N(43°÷44°) and Fe_4N (41°). In the case of code 122 sample, the applied force was constant (Q=75 daN) in all three hours at tests of wear and the tribological factor (ξ= sliding degrees = 10%) was constant, too. It was studied the variation of the phases quantities after each hour of wear process. In the case of code 122 sample, after each hour of wear process, it couldn’t be observed sesizable modifications corresponding to the quantities of the phases from superficial layer. Exists one exception: the quantity of Fe_α(M) which increase simultaneous with the wear process length. The width of the dimension for the quantity of phase Fe3N increase constantly following three hours of wear process.

In figure 5, in the case of sample code 122 and in the case of sample code 322, were applied: the same force (Q=150daN), the same treatment (T_1) but, the tribological factor (ξ) was modified (it was double, through comparing with the fig. 4 case). It was observed that, the increase of the tribological factor (ξ) and the quantity of the phase Fe3N decreases, after three hours of wear process. The quantity of the Fe_4N phase it is approximately equal, in the presented cases. Following the evolution of the characteristics from the figures: 6÷8, it was observed that the internal tensions of second order (B211~σ_{II}) increases uniform, until at maximum (corresponding to Δt=2h) and after that, these tensions decrease until Δt=3h. For the case of B_{Fe4N} , the evolution is cyclic (increase-decrease) for the each 0,5 hour length. This evolution is constantly for all the wear process length (Δt =3h).

I_{Fe3N} decrease, although the force applied (Q=150 daN) is constant and the tribological factor (ξ = 20%) is constant, too.

In the case of code 322 sample (see figs. 9 ÷ 11), simultaneous with the double values for the applied force (Q= 150 daN) and for triblological factor ξ = 20% (it was double, too), it was observed a cyclic evolution, constantly, for B_{Fe3N} and I_{Fe3N}, which lead at the material tiredness, inside in the superficial layer.

References

[1]. Gheorghies, C., Papadatu, C.P., Ştefănescu, I. - “Study on the model of behaviour of some non-conventional treated steels, during friction process”, CENTIMFE, Marinha Grande, Portugal, 2004;

[2]. Preda,A., Levcovici,S., Papadatu,C. - “*Research works regarding the influence of the nitrided layer structural properties on the wear behaviour of the turbo-blowers pinions and gear wheels*”, ROTRIB 2003, Galati.

[3]. Popescu, N. et al. - *Tratamente termice neconvenţionale*, Ed. Tehnica, Bucureşti, 1990.

[4]. Papadatu, C.P. - *Cercetări privind ameliorarea proprietăţilor şi creşterea fiabilităţii unor oţeluri folosite în construcţia utilajelor metalurgice*, PhD Thesis, University “Dunarea de Jos” of Galati, 2005;.

[5]. Papadatu, C.P. - *Aspects regarding tribological behaviour of nitrocarburized superficial layer applied after thermo-magnetic treatments, using X-ray diffractometry*, Buletinul Institutului Politehnic din IASI, Tomul L, Fasc. 6A **,** 2008;

[6]. Levcovici, **S**, s.a.- *Ingineria suprafetelor*, Note de curs.

[7]. Crudu, I., Palaghian, L., Gheorghies, C. -“*Structural modifications in the superficial layer of metallic material in wear and fatigue process*”, Japan International Tribology Conference, Nagoya, 2004;

[8]. Bursuc I., Gheorghies C. - “*Influence of the internal stresses on the physico-mechanical properties of metals”*, Analele Stiintifice ale Universitatii Al .I. Cuza din Iasi, Fizica Solidelor, Tomul XXXVIII-XXXIX, 2000;

[9]. Crudu I., Gheorghies C. - *Incercarea materialelor*, Editura Tehnică, Bucuresti, 1986;

[10]. Crudu I. et al. - “*Structural modification in the superficial layer of the material in wear and fatigue processes*”, Nagoya, Japan**.,** 1990.

[11]. Gheorghieş C., Crudu I. - “*A Cybernetic Structural Model Applied to the Study of Friction Process*”, NORDTRIB 2000, Porvoo, Finland.

[12]. Gheorghies C. - *Modificari structurale in procese de uzura si oboseala*, Editura Tehnica, 1999, Bucuresti.

[13]. Gheorghieş C. - *Metode fizice de control nedistructiv*, Editura Porto-Franco, Galati, 1999.

[14]. Bucşă M., Crudu I., Gheorghies C. - Experimental study of the failure processes within some metallic and ceramic coatings”,Thesaloniki, Greece**.,** 1999;

[15]. **Papadatu, C.-P**., **Bordei, M**. - “*Changes in Tribological Behaviour of Thermo-Chemical Treated Steels after Thermo-Magnetic Treatments*”, Metalurgia International Revue, vol. XV, Special Issue no. 8, ISSN 1582-2214 , pag..41 – 49, 2010.

[16]. Ştefănescu I., Gheorghies C. - *Difractometric Study of the Structural Instabilities in Rolling-Bearing Steels*, Buletinul Institutului Politehnic din Iasi, Tomul XLVI (L), Fasc.3-4**.,** 2000.