Structural Changes in the Superficial Layers of a Non-conventional Treated Steel Subjected to a Wear Process

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It was considered a non - conventional treatment in magnetic field applied before the thermo-chemical treatment (nitro-carburizing in plasma), for one alloyed steel which is used in Machinery building industry. These researches have been made in order to improve the mechanical properties of Chromium-Molybdenum alloyed steel non-conventionally treated in a magnetic field. Through the thermo-magnetic treatment, applied before the plasma nitro-carburizing, the mechanical properties of this material have been improved, especially in this case of the steel with a considered content of Chromium (1.38%). These researches, through X-Ray diffractometry of the behavior of the superficial layers during the dry wear process, completed this work. The diffractometric characteristics of the superficial layers which have been obtained after a complex system of thermo-magnetic treatments followed by a thermo-chemical treatment, the superficial layers content and the behavior of the steel at the wear through dry friction tests were used as criterion. The paper is a review of the researches from the last few years.

Keywords: magnetic field, thermo-magnetic treatments, nitro-carburized layer, diffractometric characteristics

In the literature [1], the purpose of nitriding (N) and nitrocarburizing (CN) is to improve wear, corrosion and fatigue resistance of machinery parts from industry. These Improvement treatments can be understood if we study the superficial layers through microstructures and the hardness after treatment (fig. 1).

The superficial layer of a nitrided/nitrocarburized steel has 2–35 mm thickness and consists of the nitrides: ε – phase with variable composition of carbon and nitrogen, depending on the steel grade or the atmosphere type and it is unstable [2], or γ' - phase (Fe_4N), or a mixture which depends on the atmosphere and by the steel grade. This layer was called the *compound layer* or the *white layer*. Tribological properties (friction and wear) are determined by this compound layer. Under the compound layer exists a diffusion area, which goes deeper inside into the steel, until approximately 0.1–0.5 mm. In the case of the wear dry tests the Load (Q) bearing capacity and fatigue strength are largely determined by the micro-hardness (Vickers) and by the depth of the diffusion area.

For example, figure 1 shows the white layer and the underlying diffusion area (darker zone) in a nitrocarburized 1.38% Cr steel (7h/540°C). The white layer consists of only ε-phase with a thin, outer, porous area.

Nitrocarburizing started to be remarkably with the development of the salt bath process Tenifer (Tufftride) and the gaseous process Nitemper developed in 1965-1967. As compared to classical nitriding, for nitrocarburizing it is necessary a short time process, typically about 30 min to 6-7 h, performed at higher temperatures, about 540 – 580°C, compared to 500–540°C for gas/ion nitriding [1, 2].

During nitrocarburizing of medium or high carbon steels carbon is donated to the whiter layer by the steel. An important quantity of the aluminum in the structure of the steel increases the thermo-magnetic treatment power. Existence of aluminum content in the structure of the steel causes some hardening problems which are countered by the Chromium existence [3].

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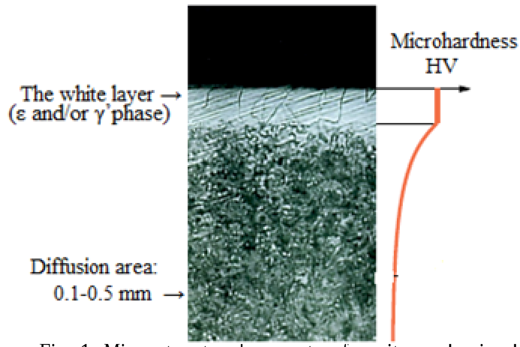

Fig. 1. Microstructural aspects of a nitrocarburized 1.38% Cr alloyed steel (7h/540°C)

In this paper, an improvement steel grade alloyed with Aluminum and Chromium was considered and the treatments temperatures have been chosen for this case. The increasing of the depth of the superficial layers in the case of unconventional treatment applied in magnetic field has been reported in accordance with the thickness of the superficial white layer for the same steel which suffered a classic improvement treatment before a thermo-chemical treatment. The magnetic field modifies the residual stresses which were obtained by treatment of hardening/ tempering. This process depends by the content of the carbon from the structure of the steel.

If Aluminum and Chromium contents increase in the structure of the steel the residual austenite quantity decreases more rapidly. The martensite (M) quantity and the hardness of the steel increase significantly, more than in the case of the steel with approx. the same content of Carbon but with lower quantity of Aluminum. As a consequence, the magnetic field intensity, the content of the Carbon and the content of the Aluminum from the steel have an important influence. The tendency of breaking decreases. Magnetostriction determines local oscillations resulting local plastic deformations [4-16]. Magneto-striction determines a reduction of the quantity of the residual austenite obtaining a higher hardness of the material and good endurance characteristics [3].

There were considered different thermo-magnetic treatments as improvement treatments with cooling in water in magnetic field applied before plasma nitrocarburizing at 540°C. This temperature of the thermo-chemical treatment was considered at 540°C, being specific for this kind of the improvement analyzed steel. This value of the nitrocarburizing temperature can't modify the properties obtained through the basic improvement treatments which consist of hardening and high tempering [4].

Experimental part

For the experimental program, were considered samples (rollers) from the material which is a steel grade good for a machine part construction, with the following composition: 0.38% C, 1.18% Al, 1.38% Cr, 0.17% Mo, 0.5% Mn, 0.058% Cu, 0.25% Si, 0.26% Ni, 0.026% P, 0.026% S. The existence of the Molybdenum (Mo) content in the composition of the steel decreases the stiffening phenomenon. The outer diameter of the rollers has 40 mm and the inner diameter of the rollers has 16 mm. [3, 7, 8].

The first stage from the complex program of treatments consists in thermo-magnetic treatments. The treatment t_1 represents a Martensitic hardening process (at 920°C) and high tempering (at 620°C), a classic treatment of improvement (Magnetic field intensity is H = 0).The other treatment, t_2, represents a hardening process at 920°C (just cooling in water in strong alternative current (A.C.) of magnetic field) and high tempering process at 620°C (just the cooling in water in strong A.C. magnetic field). The treatment t_3 represents a hardening process at 920°C (just cooling in water, in direct current of magnetic field) and high tempering process (just cooling in water, in D.C. magnetic field).

The second stage from the complex program of the treatments consists in applying the thermo-chemical treatment: a plasma (ion) nitrocarburizing at 540°C(7h), after thermo-magnetic treatment, applied at the different samples from the same steel grade considered. The treatments were considered such as:

$T_{ca} = T_2 = t_2$ + *plasma nitrocarburizing;*
$T_{cc} = T_3 = t_3$ + *plasma nitrocarburizing;*
$T_1 = T_{classic} = t_1$ + *plasma nitrocarburizing.*

The wear tests have been made using an Amsler machine, roller on roller, taking one sliding degrees (ξ = 10%), testing in time (3 h). The outer diameter of the rollers has 40 mm. The normal load is Q = 150 daN. The Moment of friction is Mf = 45 daN/mm^2. After each hour of wear tests the external diameter was measured and diffractometric aspects were studied. It were determined the wear resistance of the rollers through dry friction and the surface structure evolution for different parameters of testing regimes [3, 10, 11].

Results and discussions

Analyzing the presented diffractometric fragments, the variations of the quantities of the phases formed by alloying elements of the steel, are observed. These variations are determined by thermal/thermo-magnetic treatments applied before the thermo-chemical treatment.

Following the repartition degrees of the phases for this material, it can be observed, in the case of the application of the magnetic field-alternative current (H3 = 1300A/m, intensity) during the cooling at the hardening/tempering processes applied before of the plasma nitrocarburizing, the quantities of the hard phases (for example, chromium carbide, or Cr_2N, or Feα (M) - Martensite) obtained, are bigger than the quantities obtained in the case of classic treatment. This observation was proved by the existence of the highest heights of peak (respective, width of the diffraction lines) inside of the intervals: 41 ÷ 45^o and 81 ÷ 84^o.

In figure 2, diffractometric aspects in the case of classic treatment (T1) were presented.

Figure 3a shows diffractometric aspects in the case of non-conventional treatment T2 before the wear tests and the figures 3b, 3c and 3d show diffractometric aspects after one hour, or two hours, or three hours of wear tests on Amsler Machine.

Figure 3e shows diffractometric aspects in the case of non-conventional treatment T3 before the wear tests. Figures 3f, 3g and 3h show diffractometric aspects after one hour, or two hours, or three hours of wear tests on Amsler Machine.

In figure 4 are presented the distribution of the nitro carbides (CN): Fe_3(CN) and Fe_4(CN) phases after classic treatment and after nonconventional treatments (in magnetic field – alternative current respective, in magnetic field – direct current).

There were considered the following notations:

IM_1 = the existence of the highest peak (respective, width of the diffraction lines) inside of the interval: 41 ÷ 45^o;

IM_2 = the existence of the highest peak (respective, width of the diffraction lines) inside of the interval: 81 ÷ 84^o.

The evolutions of IM1 and IM2 for each kind of treatment and depending on the wear tests duration were presented in the following figure 5.

Feα (*M*) quantity increases in the case of nonconventional treatments T_2 and T_3. The nonconventional treatment in magnetic field – alternative current has the better results, comparing with the classic treatment T_1 (without magnetic field).

Figure 6a, b and c show the evolution of CrN and Cr_2N distribution in superficial layer for each kind of treatment and depending on the wear tests duration. In the case of the nonconventional treatment T2 (A. Current) the higher quantity of the nitride Cr_2N was obtained.

The nitride *Cr_2N* particles have a hexagonal *crystal structure* with lattice parameters a=0.481 nm and c = 0.448 nm [17]. Chromium increases and nickel decreases the nitrogen solubility in Fe-Cr-Ni-austenite. It is well known that Cr and Mn content decrease when Ni increases the thermodynamical activity of nitrogen and carbon in ferrous austenite [18]. This situation means that Cr and Mn atoms attract C and N ones and Ni pushes them out from solution. Vanderschaeve et al. [19] investigated the mechanism of

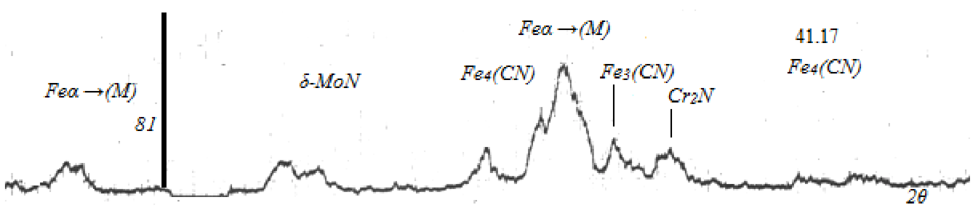

Fig. 2. Fragment of diffractometric specimen (samples code R2) for treatment T_1 (classic treatment)

Fig. 3. Fragment of diffractometric specimen (samples code R3 and R5) for different treatments: a) Samples code R3 - unconventional treatment T_2; b) Samples code R3 - unconventional treatment T_2, after one hour of wear process *(Δt = 1h)*; c) Samples code R3 - unconventional treatment T_2, after two hours of wear process*(Δt = 2h)*; d) Samples code R3 - unconventional treatment T_2, after three hours of wear process *(Δt = 3h)*; e) Samples code R5 - unconventional treatment T_3, before the wear tests (Δ*t = 0h)*; f) Samples code R5 - unconventional treatment T_3, after one hour of wear tests *(Δt = 1h)*; g) Samples code R5 - unconventional treatment T_3, after two hours of wear tests *(Δt = 2h)*; h) Samples code R5 - unconventional treatment T_3, after three hours of wear tests (Δt = 3h)

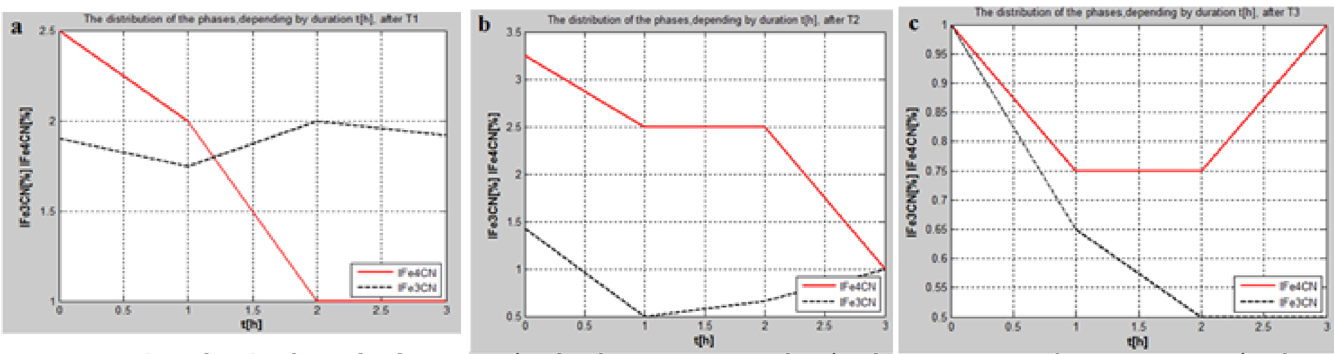

Fig. 4. Fe_3CN and Fe_4CN phases distribution: a - after the classic treatment T_1; b - after the nonconventional treatment T_2, c - after the nonconventional treatment T_3, depending on the wear tests duration (Δt)

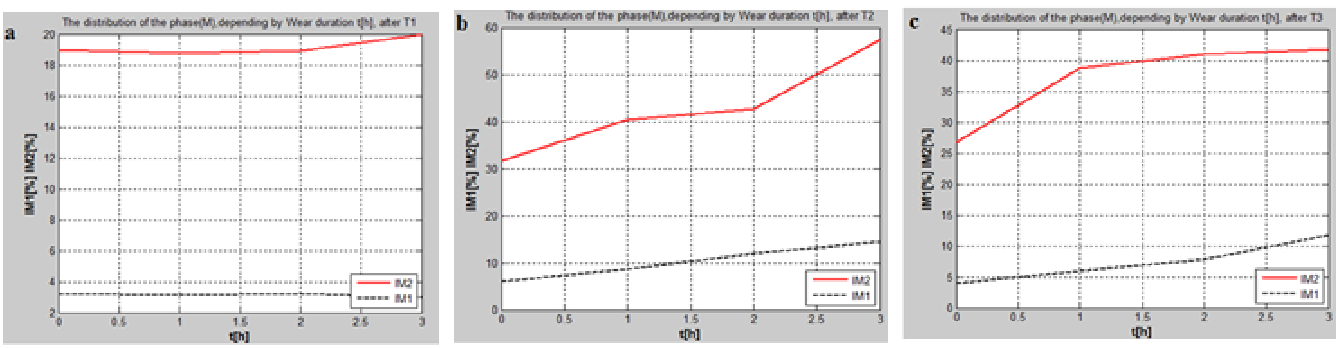

Fig. 5. Fea (M) distribution in superficial layer: a - after the classic treatment T_1, b - after the nonconventional treatment T_2 , c - after the nonconventional treatment T_3, depending on the wear tests duration (Δt)

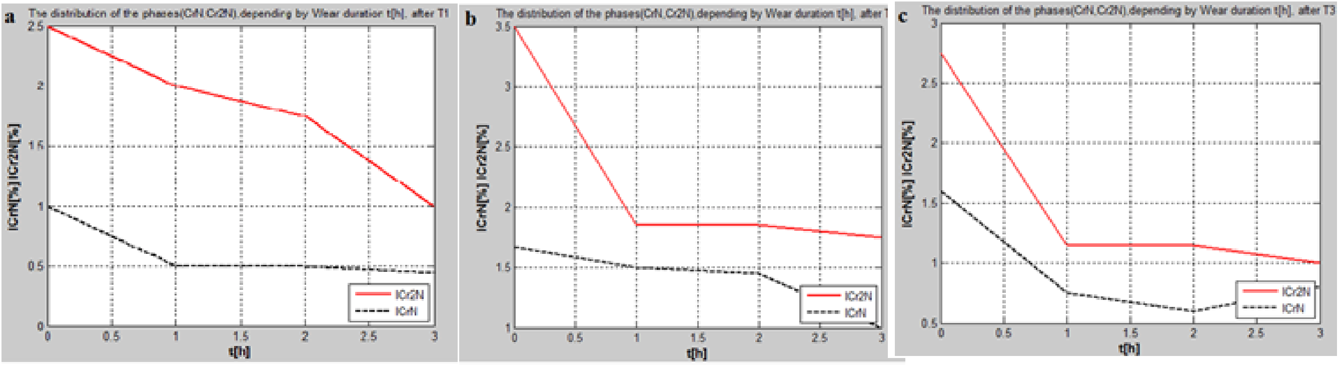

Fig. 6. CrN and Cr_2N distribution in superficial layer a - after the classic treatment T_1,; b - after the treatment T_2,; - after the treatment T_3, depending on the wear tests duration (Δt)

Cr_2N precipitation in high nitrogen CrMn alloyed steel and they found that the reaction of discontinuous Cr_2N precipitation is governed by two processes: at first by the intergranular diffusion of Chromium and at the second by its bulk diffusion.

In the case of the nonconventional treatment T_2 (alternative current) the higher quantity of the Cr_2N was obtained. Analyzing the up-figures, it can be observed that the non-conventional treatment in magnetic field increases the quantity of the nitride Cr_2N phase. For example, before the wear tests on Amsler Machine, the quantity of the phase Cr_2N in the case of T2 and T3 treatments is higher than in the case of the classic treatment (T1). In the cases of nonconventional treatments (T2 and T3), after one hour of wear tests the nitride Cr_2N quantity decreases suddenly and deeply, under 2 respectively 1.5%, maintaining these values almost constantly during an hour of wear tests after. In the case of classic treatment, the quantity of Cr_2N decreases continuously for three hours of wear tests. If the quantity of Cr_2N decreases, the brittleness of tempering decreases too. It can be proved that the behavior of the steel during the wear tests is better in the cases of non conventional treatments in magnetic field.

In figures 7 and 8 were represented the Feα(M) distributions, depending on treatments, using Matlab Program.

There were considered the following notations:

B_1= internal tensions of second order obtained in the case of the classic treatment T_1;

B_2= internal tensions of second order obtained in the case of the nonconventional treatment T_2;

B_3= internal tensions of second order obtained in the case of the nonconventional treatment T_3

The evolution of the internal tensions of second order is presented in figure 9.

There were considered the following notations:

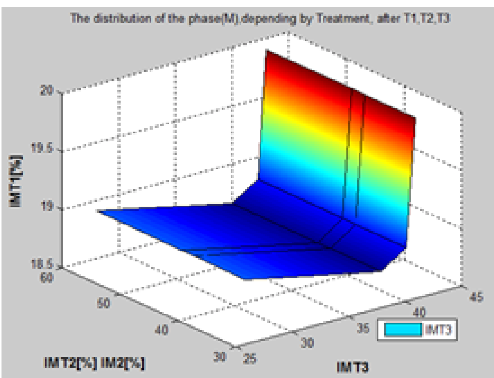

Fig. 7. Fea(M) distribution, depending on treatments: The increases of the Feα(M) quantity in the case of the nonconventional treatment T_2, considering the classic treatment T_1.

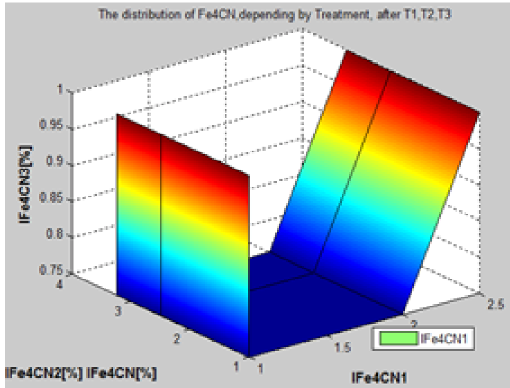

Fig. 8. Fe_4(C,N) distribution depending on treatments: the increases of the Fe_4(C,N) quantity in the case of the nonconventional treatment in magnetic field - alternative current (T_2)

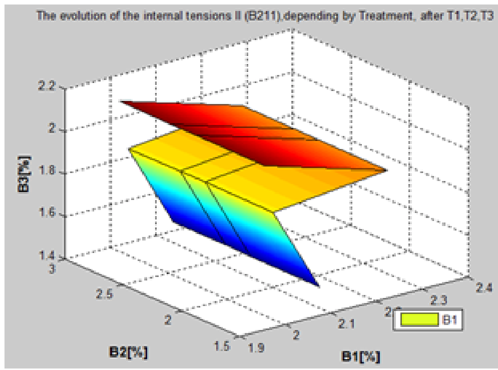

Fig. 9. Internal tensions of second order (B211) evolution, depending on the treatments: T_1, T_2, T_3.

c_1 = tetragonality grades (c/a) evolution, corresponding to classic treatment (T_1);

c_2 = tetragonality grades (c/a) evolution, corresponding to a non-conventional treatment in magnetic field (alternative current, T_2);

c_3 = tetragonality grades (c/a) evolution, corresponding to a non-conventional treatment in magnetic field (direct current, T_3).

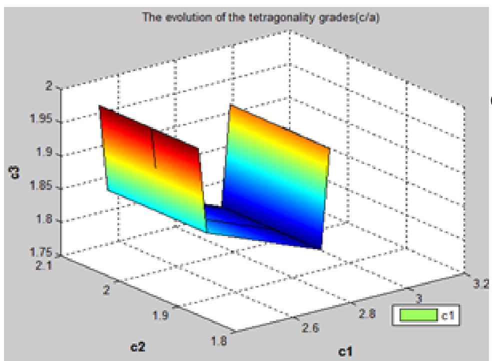

Fig. 10. The evolution of the tetragonality grades (c/a), depending on wear tests duration and treatments

In figure 10 are presented the evolutions of the tetragonality grades (c/a), depending on treatments and by wear tests duration (Δt).

Microstructural aspects were presented in figure 11 [4].

In figure 11 were presented Microstructural aspects of a nitrocarburized 1.38% Cr alloyed steel (7h/540°C) after the classic improvement treatment (T_1), thermo-magnetic treatment – alternative current (T_2) and respectively after the thermo-magnetic treatment - direct current (T_3).

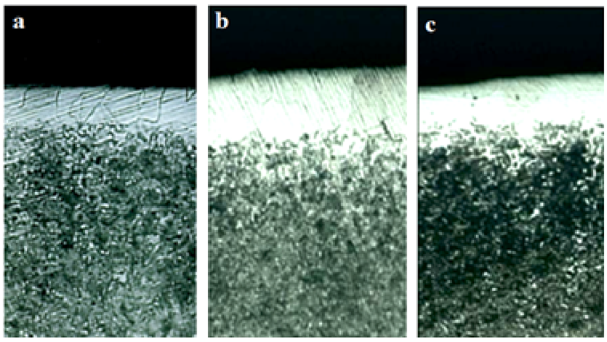

Fig. 11. Nitrocarburized superficial layer (samples code R2), before the wear tests (100X) Nittal Attack 2%: a - treatment T_1; b - treatment T_2; c - treatment T_3

Following the distribution of the phases inside the thermo-chemical superficial layer treated and the evolution of the microstructural aspects, was obtained that the thickness of the white layer is higher in the case of the nonconventional treatment in magnetic field - T_2, comparing with case of the classic treatment T_1. This fact was explained through the mechanical oscillations which appear with the application of the magnetic field – alternative current on the improvement treatment.

The role of these mechanical oscillations, during the diffusion process, is very important because of the tensions produced by magnetostriction and because of the volumetric dimensional changes which provoke the increases of diffusion coefficient. In ferromagnetic border area, some tensions appear. Here we can observe the gradients of tensions because of the magnetostriction. These gradients of tensions determine the increases of the diffusion coefficient of the atoms.

This process continues through the interaction of these gradients of the tensions and the internal tensions redistributed during the diffusion process. Finally, a new factor of influence on the diffusion process appears.

Mechanical oscilations produced by the magnetic field – alternative current modify the recrystallization conditions. Through this situation it can modifies the germination speed. That is why, through the application of the magnetic field - alternative current during the improvement treatment of the steel, it creates a finer granulation, favoring the diffusion.

Conclusions

In the diffractometric fragments, the variations of the quantities of the phases formed with alloying elements of the steel, are observed. These variations were determined by thermal/thermo-magnetic treatments applied before the thermo-chemical treatment.

If it is applied a magnetic field (a thermo-magnetic treatment), the magnetostriction appear. The increasing of the depth of the superficial layers in the case of unconventional treatment applied has been reported in accordance with the depth of the superficial layer for the same steel which suffered a classic improvement treatment before a thermo-chemical treatment. The magnetic field modifies the residual stress. This process depends by the content of the carbon from the structure of the steel. The cooling in magnetic field has been made during the improvement treatment of these steels, the residual stresses by hardening decreases, the residual austenite quantity decreases too and – as a result - the magnetic field has a positive effect on the mechanical properties because the hardness of the steel and the wear resistance increase.

The originality consists of applying the thermo-chemical diffusion treatment after thermo-magnetic basic treatment, with the mention that the thermo-chemical treatment temperature is lower than the temperature of thermo-magnetic treatment. This condition has been mentioned in order to not modify the properties of the material during the thermo-chemical treatment.

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