Study on the Influence of the Treatment in Magnetic Field on the Nitrided Layer in Plasma

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These researches have been made in order to improve the mechanical properties of a Chromium–Molybdenum alloyed steel non-conventionally treated in a magnetic field. Through the thermo-magnetic treatments, applied before thermo-chemical treatment, the mechanical properties of this material have been improved, especially in the case of a great content of chromium. The hardness values 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 behaviour 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, nitriding layer,

According to the literature [1-6], it was found that a large number of steels for bearings (RUL1, RUL2) and tools (CSOs and Rp sites) have an increased durability by increasing the resistances of corrosion and wear caused by the increasing of the hardness and the decreasing of the amount of the lost material (mass loss) in dry friction process as a consequence of the magnetic field applied during the improvement heat treatment of the steels.

Starting from the fact that the authors and researchers have not addressed the application of the thermo-chemical diffusion treatment after the thermo-magnetic treatment, the research novelty consists precisely in applying of the plasma nitriding after the thermo-magnetic treatment [6]. This unconventional treatment modifies the characteristics of the resistance of the steel, the hardness and the plasticity of the steels. Applying a thermo-chemical treatment with diffusion at the temperature under the temperature of the thermo-magnetic treatments, leads at high wear resistance. These researches were started in the doctoral thesis [6]. This thesis was finalized in 2005 but the researches continue.

The energy of the magnetic field may intervene in the balance of global power of the phase transformations of solid state altering the thermodynamics, kinetics and the mechanisms, the structures and properties of the steels.

The magnetic field leads to a decrease of the residual austenite content (Arez) during the annealing/hardening treatment of the tool steels, according to the literature [4]. For example, was calculated that 30% Arez in a hardened steel that it contains 1.1% C and 8% Ni, is transformed by returning to 360°C in 30 min, while the same steel, the same amount of the residual austenite was transformed by the treatment in magnetic field by returning to 360°C in 24 min.

Experimentally it was found that the magnetic field reduces the cooling capacity of the quenching medium , being reduced its influence over oil and water, while the aqueous salt solutions – accentuates the action of the magnetic field.

The cooling in magnetic field during thermo-magnetic treatment regimes modifies the characteristics of the steel, comparing with the classic treatment results. During the improvement treatment of the steel through thermo-magnetic treatment, the residual stresses realized by hardening decreases, the residual austenite quantity decreases too and the magnetic field has a positive effect on the mechanical and corrosion properties because the hardness of the steel and the corrosion resistance increase [5, 6, 9].

There were considered different thermo-magnetic treatments regimes as improvement treatments with cooling in magnetic field applied before ionic nitriding treatment (plasma nitriding) at 530°C. The modification of the white superficial layers depth in the cases of unconventional treatments applied has been reported to other microstructure of the same steels which suffered classic improvement treatment before thermo-chemical treatment [10-12].

The main cause of this phenomenon is the effect of magnetostriction which produces tensions in solid solution microvolumes – tensions that interact with their elastic stress field of dislocations. Magnetostriction is defined as a dimensional variation of ferrous-magnetic materials under the action of a magnetic field, also called Joule effect, which depends on the size and direction of the external magnetic field, the material and the heat (thermal) treatment previously applied to this material [1, 2, 6]. The effect of the magnetostriction decreases with higher temperatures and disappears at the Curie temperature. The mechanical oscillations produced by the alternative magnetic fields change the re-crystallization conditions, especially the germination velocity.

Experimental part

Materials and methods

For the experimental program, were considered the samples (rollers) from the material which is a steel grade of improvement for a machine part construction. This material has the following principal components: 0.42 % C, 0.02 % Al, 1.02 % Cr, 0.17 % Mo, 0.68 % Mn, 0.22 % Cu, 0.33 % Si, 0.32 %Ni, 0.03 % P, 0.026 % S – [AISI(SAE) 4142]. The existence of the Molybdenum content in the composition of the steel decreases the stiffening

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phenomenon. The outer diameter of the rollers has 40 mm and the inner diameter of the rollers has 16 mm. [6, 9].

The first stage from the complex program of treatments consists in thermo-magnetic treatments.

The treatment t1 represents a Martensitic hardening process (at 850 °C) and high tempering (at 580°C), a classic treatment of improvement (Magnetic field intensity is H =0). The treatment t2 is the same with tx and represents a complete martensitic hardening process in weak alternative magnetic field (cooling in water) and high recovery process (just cooling in water, in strong Alternative current (A.C.) of magnetic field with H=1300 A/m). In this case, the sample was introduced in the centre of the electrical coil located in the walls of a cylindrical oven. The other treatment, t3, represents a hardening process (just cooling in water in strong alternative current (A.C.) of magnetic field) and high tempering process (just the cooling in water in strong A.C. magnetic field). The treatment t4 represents a hardening process (just cooling in water, in direct current of magnetic field) and high tempering process (just cooling in water, in D.C. magnetic field) [6, 9].

The second stage from the complex program of the treatments consists in applying the thermo-chemical treatment: a plasma (ion) nitriding at 530 °C, after thermo-magnetic treatment, applied at the different samples from the same steel grade considered. We noted: Tca = T3= t3 + plasma nitriding; Tcc = T4= t4 + plasma nitriding, T = Tclassic, T2 = Tx = t2 + plasma nitriding.

Micro-hardness (Vickers) was measured on the treated surface layer obtained by thermo-chemical treatment regimes shown above. Were performed a minimum eight determinations for each case [6, 9, 13, 14].

Experimental program continues with the wear tests, using the Amsler Stand. The tests of wear process estimate the material resistance. For this kind of tests there were used rings (rollers). The outer diameter of the conducting rollers has 40 mm or 44 mm and the inner diameter of the rollers has 16 mm. The rollers width (b) has 10 mm [6, 14]. It were determined the durability of the rollers and the surface structure evolution for different parameters of testing regimes. Every test of wear/friction had a duration by three hours, for each task loading value.

Experimental program continues with diffractometrical aspects of the superficial layer during three hours of wear tests. Diffractometry was study using a Dron 3 from the Laboratory of Dunarea de Jos University, Galati, Romania.

Results and discussions

In figures 1 (a, b) was presented the influence of the magnetic field applied on the hardness (HB) of the Steel [6, 8]. In this figure was presented the Brinell hardness values (HB) of the steel which was treated in magnetic field (t2, t3 and t4), before applying thermo-chemical treatment. The values presented were calculated as arithmetic average.

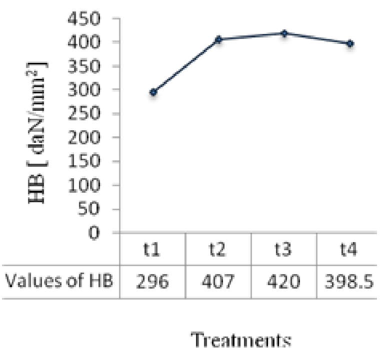


Fig. 1. The influence of the magnetic field on the hardness of the material [6, 9, 13]

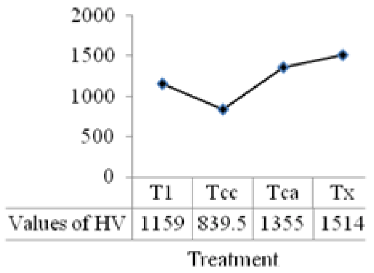


Fig. 2. The evolution of the micro-hardness values in the ion nitrided layer, depending on the treatments, at 0.02 mm distance from the surface of the samples [6, 9]

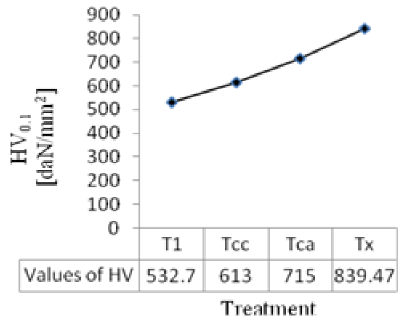


Fig. 3. The evolution of the micro-hardness values in the ion nitrided layer of material, depending on the treatments, at 0.25 mm distance from the surface of the samples [6, 9]

After plasma nitriding, the white superficial layers had a higher depth in the case of magnetic field.

In figures 2 and 3 are presented the evolution of the micro-hardness values (Vickers) in the ion (plasma) nitrided layer, versus the treatments, at 0.02 mm respectivelly, 0.25 mm distance from the surface of the samples, in depth of the samples (of the leading rolls of wear).

In figure 4, was represented the microhardness ($HV_{0.1}$) evolution versus the thickness of the nitrided layer (GT1), corresponding to the treatments T1 and T2.

After plasma (ion) nitriding, the white superficial layer had a higher depth in the case of the applying the magnetic field.

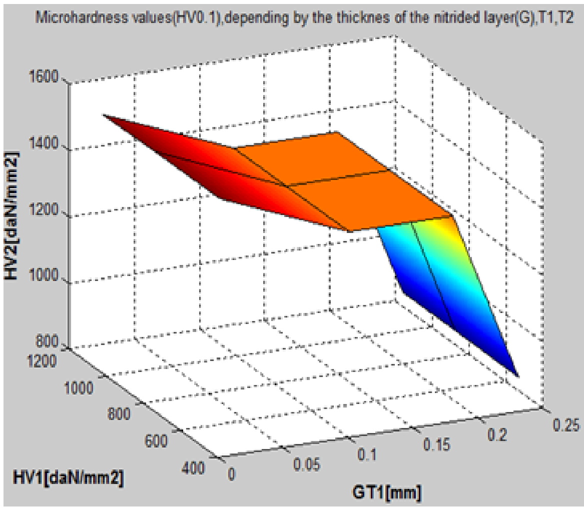


Fig. 4. Microhardness evolution versus the thickness of the nitrided layer (GT1), corresponding to the treatments T1 and Tx.

In figure 5 was represented the evolution of the nitrided layer thickness versus the applied treatment.

Experimental program continues with the wear dry tests, using the Amsler Stand. The tests of wear process are designed to estimate the material resistance [9, 15-18]. For this kind of tests there were used rings (rollers).

The evolutions of the thickness of the worn-out layer depth (Uh) as a function of the task loading (Q) which is equivalent with a normal force (Q) were represented in figures 6 and 7 using Matlab Program. In these figures were represented the evolutions of the worn-out layer depth v.s. sliding degrees (ξ) and the normal load (Q = 75 daN), for the treatments T1(classic) and Tx (Tx = T2) – a non-conventional treatment.

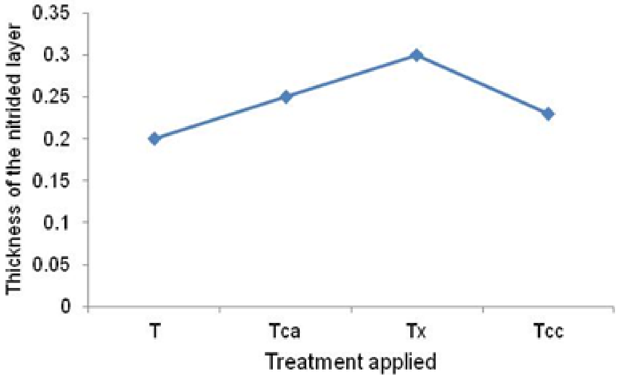


Fig. 5. The evolution of the layer' thickness v.s. the applied treatments

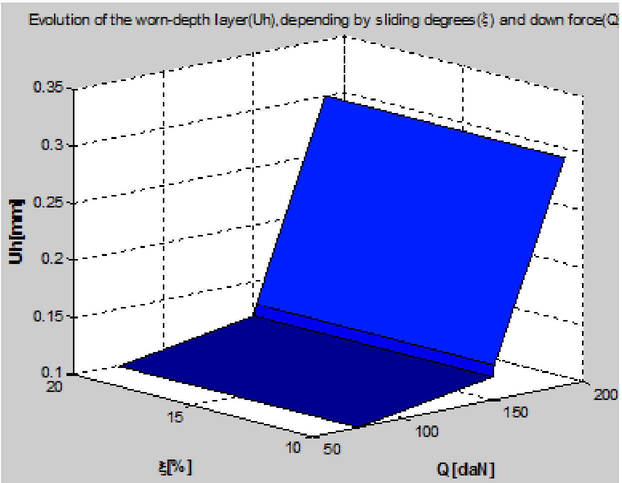


Fig. 6. Evolution of the worn-out layer depth v.s. sliding degrees (ξ) and the normal load considered for Q = 75 daN value, after T1

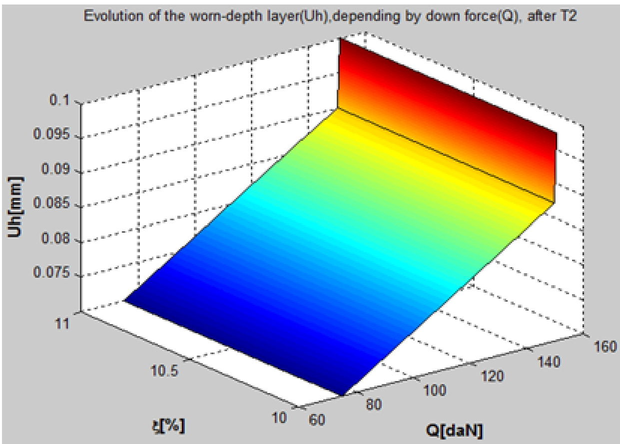


Fig. 7. Evolution of the worn-out layer depth v.s. sliding degrees (ξ) and the normal load considered for Q = 75 daN value, after Tx (T2)

Diffractometry aspects

In figures 8 and 9, are presented the researches possibilities at X-Ray Diffractometry using a Dron 3, from Dunarea de Jos\ University. The angular velocity of the sample is ω = 1°/ min.

In the analysed superficial layers, the Fe_4N phase has a higher hardness than the Fe_3N phase.

Thus, in the case of thermo-magnetic treatments applied before the thermo-chemical treatments, it was observed a higher quantity of Fe_4N phase as compared to the quantity of the Fe_3N phase. In this case, the nitrided layer depth increases and the resistance to wear increases, too, with more than 25%. The resistance to corrosion increases (more than 34%), too [6].

It was observed an improvement of the mechanical properties (hardness) in the superficial layer because of the distribution of the γ'-Fe_4N phase, especially in the case of the normal load (Q) and for the increasing of the sliding degrees (ξ) [6]. The structural and magnetic properties of epitaxial γ'-Fe4N iron nitrides films have been investigated by Costa – Krämer J L, Borsa D M, and others, in [19], and it was explained why this phase is so important [13].

Fe_4N is a phase with a higher hardness value, being a *hard* phase. If the quantity of this phase increases in the superficial layers the micro-hardness and the wear resistance increase too.

Fe_3N is a phase which promotes a fatigue phenomenon in the superficial layers because of the cyclical distribution of this phase.

Cyclical distribution of the phase Fe_3N which corresponding to the samples treated with the classic treatment, T1, determines a special fatigue of the solicitation, with the danger of the exfoliation process. In the case of the samples treated in magnetic field (Alternative current: T3 = Tca), this cyclical distribution of the phase Fe_3N decreases. As a consequence, the danger of the exfoliation process decreases in the superficial layer [6].

The properties of the nitrided superficial layer depend on the nature of nitrides phases. With the increasing of the temperature of the nitriding, the nitriding duration is reduced, but decreases the hardness of the superficial layer because of the coalescence of the nitrides of alloying elements.

The phase Fe_2N (ξ)- has a rombic structure (the deformed structure of the phase ε) and small values of the hardness. This phase not appeared in the structure of the steel.

Explanation: Phase Fe_2N (ξ) is stabile in equilibrum with ammonia until to maxim 450°C [13]. The nitriding treatments have been made at 530°C, a temperature higher than 450°C. This phase was decomposed and disapeared from the superficial layer. The micro-hardness and the wear

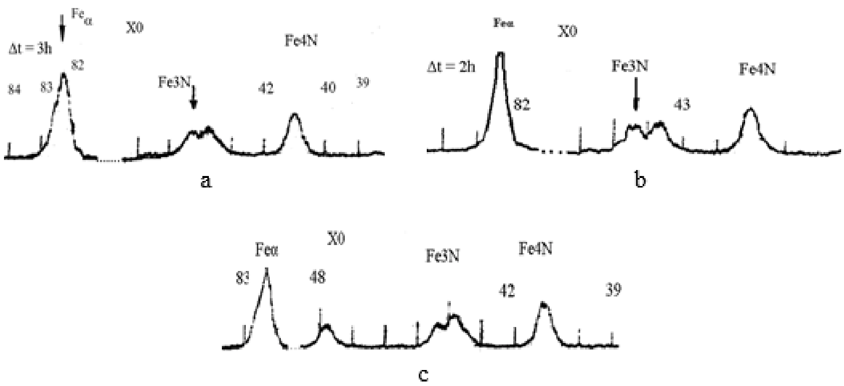
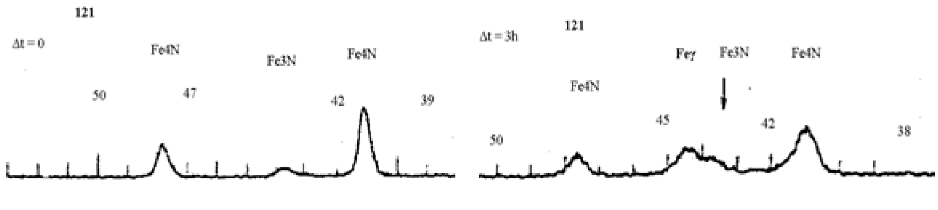
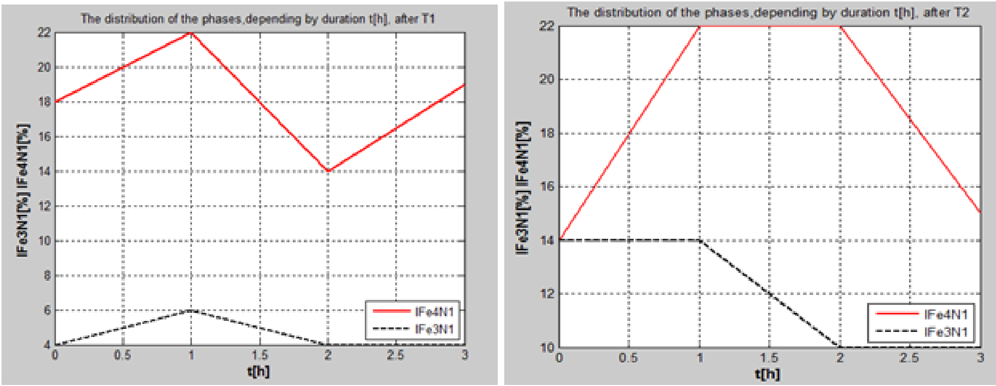


Fig. 8 X-Ray analysis corresponding to X_0 samples (T2, Q=75daN, ξ=10%) [6] after three hours of wear tests (a), two hours of wear tests (b), respectively at the initial moment (c)



a b

Fig. 9. X-Ray analysis corresponding to 121 samples (T1), conditions of work: Q=75daN, ξ=10% [6] before of wear tests (a) and after three hours of wear tests (b)



a b

Fig. 10. Distribution of the phases: Fe_3N and Fe_4N in the superficial layer versus wear process duration, for Q=75 daN and ξ=10%, for the treatment T1 (classic) (a) and Tx (non-conventional) (b)

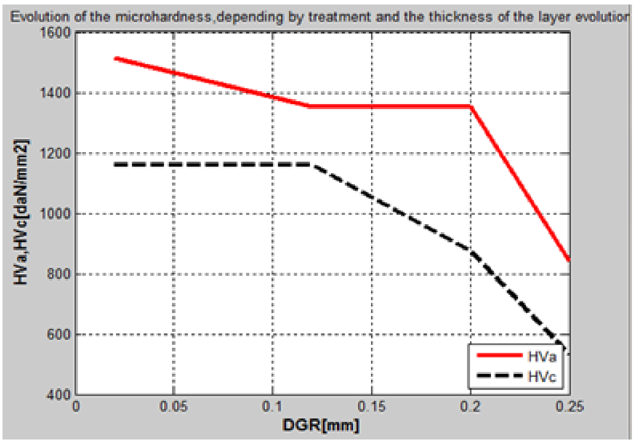
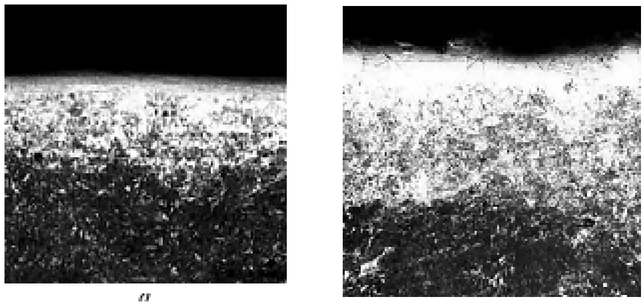
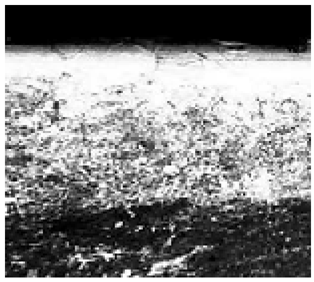


Fig. 11. Microhardness values in the nitrided layer versus the time of the wear process, for Q=75 daN, ξ=10%,(Hva corresponding to the treatment T1 and HVc corresponding to Tx)



a c



b

Fig. 12. Microstructure after T1, (x100) (a), after T3 –Alternative Current, before the wear tests (b) and after T3 –Alternative Current, after one hour of dry wear tests (c) Nital Atack 2%

resistance of the steels increase because of this situation [14].

During the wear process it was made a study regarding the distribution of the phases Fe_3N and Fe_4N in the superficial layer depth during every hour of wear process.

These distributions in the depth of the nitrided superficial layers is uneven. That's why the nitriding process is not uniform.

It has been noted that: Hva – microhardness of the samples treated in magnetic field (alternative current); HVc – microhardness of the samples treated in magnetic field (direct current); DGR – The thikness of the white layer (the superficial layer).

In figure 11 was presented the evolution of the micro-hardness values (Vickers, HV0.1) in the plasma realize layer, depending on the treatments**,** measured in depth of the samples (of the leading rolls of wear)**.**

Microstructural aspects were presented in figure 12 [6]. From point of view microstructural, after applying a magnetic field (A.C. or D.C.), we have to observe a finish of granulation and a decreasing of the crystalline grains size, from 9 to 7-8. This situation allows a good diffusion in along the boundaries – in the case of plasma nitriding, a thermo-chemical treatment with diffusion.

This modification is a consequence of the Magneto-striction – which represents a dimensional volumic variation of the ferromagnetic material (the steel) under the magnetic field action (Joule effect). It creates internal tensions which have interactions with the tensions created by dislocations. The $A \rightarrow M$ transformation is accelerated. The Martensite grains increase and they pressed the grains of rezidual Austenite.

Needles of martensite have preferential orientation in according with magnetic field application. After thermo-chemical treatments the depth of the superficial layers varies in depth, the maximum value will be in the case of the non-conventional treatment (Alternativ current-magnetic field). The diffusion coefficient increases because of the finish of the grains because of the magnetic field applied.

In the case of low temperature (100-500°C), plasma nitriding produces the expanded austenite (the S-phase) with good behavior at friction. Phase γ (Fe_4N) appear at higher temperatures than 500°C and reduces the thickness of the S-phase.

Conclusions

The present paper involves the application of the diffusion thermo-chemical treatment after the thermo-magnetic one. The temperature of the former being lower than that of the latter, except that the thermo-chemical treatment applied after the thermo-magnetic treatment should not modify (due to the high temperature) the improvements of the mechanical properties by the thermo-magnetic treatment. It was made a short comparison between classic treatment and unconventional (thermo-magnetic) treatment.

It has been shown that, when applying an alternative current (A.C.) of magnetic field during the treatment (*for example, H=1300 A/m*), the thickness of the thermo-chemical treated layer increases up to 25%.

It was observed that for the treatment T1 applied to steel grade (classic treatment), the martensite quantity and the nitrides are maintained constants after the friction-wear process. In the case of alternative or continuous magnetic field applied to the steel (Tca, Tcc), it was observed a higher initial quantity of martensite and nitrides. During the wear process, the martensite quantity increase and the Fe_3N quantity decrease. A good influence of the thermo-magnetic treatment on the surface layer resulted in a higher hardness [4] and a good wear resistance.

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