Experimental studies regarding wear processes through dry friction of the superficial layer for an unconventional treated steel

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Abstract. The aim of the studies on tribomodels regarding the dry friction wear and the processes of the wear in this case is to determine the wear intensity for a certain type of tribomodel and for a certain type of material. An Amsler stand was used. The experimental study uses two distinct values of task of loading (Q). The rolls with different diameters were used in order to obtain different sliding degrees (ξ). Studies have been made for the improvement of the mechanical properties of a Chromium – Molybdenum alloyed steel, unconventional treated in a magnetic field. The Thermo-magnetic treatment was applied before a thermo-chemical treatment. The mechanical properties of the material have been improved, particularly in the case of a great content of aluminum and chromium. The hardness values of the superficial layers which have been obtained after a complex thermo-magnetic and thermo-chemical treatments, the superficial layers content and the behavior of the steels at the wear tests were used as criterion. Diffractometric analysis and a statistical modeling completed this study.

1. Introduction
An important quantity of the aluminum in the structure of the steel increases the thermo-magnetic treatment power and the results are the best. At the same time, the existence of aluminum content in the structure of the steel causes some hardening problems which are countered by the Chromium existence.

There were considered different thermo-magnetic treatments as improvement treatments with cooling in water in magnetic field applied before ionic nitriding treatment (plasma nitriding) at 530 °C. This temperature of the thermo-chemical treatment was considered at 530 °C, being specific for this kind of the improvement steel. The influence of such parameters affecting the nitriding layers’ thickness, hardness, composition and residual stress was evaluated [1]. In the literature, taking into account the nitriding process, at all temperature below 510-520 °C substantial quantity of the “S-phase” was found to be present, especially in the case of the austenitic stainless steel [2]. In case of low temperature (100 °C – 510 °C), plasma nitriding produces the expanded austenite (the S-phase) with good behavior at friction. Phase γ (Fe3N) appear at higher temperatures than 500 °C and reduces the thickness of the S-phase. In this paper it was considered an improvement steel grade alloyed with Aluminum and Chromium 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 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 stresses which were obtained by treatment of hardening/tempering. This process depends by the content of the carbon from the structure of the steel. In all the cases, 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
positive effect on the mechanical properties because the hardness of the steels and the wear resistance increase.

If Aluminum and Chromium contents increase in the structure of the steel the residual austenite quantity decreases more rapidly. The martensitic quantity and the hardness of the steel increases significantly, more than in the case of the steels 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 steels have an important influence. Because of these aspects, the tendency of breaking decreases and the probability of the fragile breakage no longer exists. Magnetostriction determines local oscillations resulting local plastic deformations [3, 5, 8, 14].

Magnetostriction determines a reduction of the quantity of the residual austenite. Furthermore, this situation implies a higher hardness of the material and for many applications - good endurance characteristics.

The control of the ion (plasma) nitrided layer using micro-hardness curves involves the determining of the micro-hardness from the superficial layer on the depth. Following this influence of the magnetic field can be seen the effects of the aluminum (1.18%) and chromium (1.38%) - as alloying elements of the steel - and the influence of the thermo-magnetic treatment on the micro-hardness of the superficial layers obtained through thermo-chemical treatment applied after thermo-magnetic treatments. This influence is determined by the increasing of the thickness of the superficial layer. This superficial layer thermo-magnetic and thermo-chemical treated has higher micro-hardness values than the superficial layer classical treated.

To complete this study, it was considered a statistical model. It is necessary to prove the causation between normal load (Q) and the worn-out depth of the layer (Uh). I resort to statistical methods for checking the next assumption: I considered that there is a causal relationship between the independent variable Q and dependent variable Uh. Between these variables there is a direct connection. Application of analysis models of regression and correlation implies some steps [10].

2. Experimental Procedure

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: 0.38 % 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 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. [2, 3, 4, 6].

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

The treatment t1 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, 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).

The second stage from the complex program of the treatments consists in applying the thermochemical treatment: a plasma (ion) nitriding at 530 °C, after thermo-magnetic treatment, applied at the different samples from the same steel grade considered. The treatments were considered such as: Tca = T3' = t3 + plasma nitriding; Tcc = T4' = t4 + plasma nitriding, T1' = Tclassic.

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.

The wear tests have been made using an Amsler machine, roller on roller, taking two sliding degrees (ξ = 10% or, 20%), testing in time (3 hours). After each hour of wear tests the external diameter was measured. It were determined the wear resistance of the rollers through dry friction and the surface structure evolution for different parameters of testing regimes. Some factors which influence this kind of wear process are: the contact geometry of the friction couple (roller on roller), the technological parameters (surface quality, thermo-chemical treatment) and the exploitation conditions (for example: the thermal solicitation). Wear tests were carried out on an Amsler machine, using several couples of rollers, each couple corresponding to different sliding degrees ξ, defined as [3, 4, 7, 14]:

\[ \text{conditions (for example: the thermal solicitation). Wear tests were carried out on an Amsler machine, using several couples of rollers, each couple corresponding to different sliding degrees } \xi, \text{ defined as } \{3, 4, 7, 14\}. \]
where \(v_1\) and \(v_2\) are the peripheral velocities of the rollers in contact, each one having their specific peripheral velocity. The rollers are in a direct contact (a linear contact) and the friction process created through a particular combination of angular speeds \((n_1, n_2)\) and diameter sizes \((d_1, d_2)\) exists. Index 1 or 2 are added for the roller 1 or 2, respectively, both of the same tested friction couple.

For instance, \(\xi = 10\%\) is obtained for a pair of tested rollers having \(d_1= 40\) mm, \(n_1=180\) rpm and \(d_2= 40\) mm, \(n_2=162\) rpm; \(\xi = 20\%\) is obtained for a pair of tested rollers having \(d_1= 44\) mm, \(n_2=180\) rpm and \(d_2=40\) mm, \(n_2=62\) rpm; the level of the stress is corresponding to a normal load \((Q)\) of 150 daN and the linear contact between rollers is \(b=10\) mm (represents the width of the rolls) [5,8].

3. Results and discussion
After plasma (ion) nitriding, the white superficial layers had a higher depth in the case of applying the magnetic field. In figure 1 was presented the evolution of the micro-hardness values (Vickers, \(HV_{0.1}\)) in the plasma nitrided layer, depending on the treatments, measured in depth of the samples (of the leading rolls of wear). It has been noted that: \(HV_a\) – microhardness of the samples treated in magnetic field (alternative current); \(HV_c\) - microhardness of the samples treated in magnetic field (direct current); \(DGR\) – The thickness of the white layer (the superficial layer).

**Figure 1.** The evolution of the microhardness \((HV_{0.1})\) depending by the treatment and by the thickness of the layer.

**Figure 2.** A comparison between the microhardness of the layer \((HV_1)\) obtained by classic treatment and the microhardness of the layer \((HV_4)\) obtained through the unconventional treatment T4.
In the figures 2 and 3 were presented a comparison between the microhardness of the layer obtained through the classic treatment and the microhardness of the layer obtained by unconventional treatments (T3 and T4). The study was continued with diffractometric analysis.

In the figures: 4, 5,...,until 11, were presented the variation of the diffractometric characteristics, the distribution of the phases from the superficial layers and the internal tensions evolution depending by duration of the wear process through the dry friction.

It was observed an improvement of the mechanical properties (hardness) in the superficial layer because of the distribution of the γ'-Fe₄N phase, especially in the case of the increasing of the normal load (Q) and for the increasing of the sliding degrees (ξ). 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 [8], and it was explained why this phase is so important. According with [7], the magnetization properties described are so far are consistent with a single phase of epitaxial film, having a cubic structure and a positive anisotropy constant, i.e., the [100] directions are easy magnetization axes. All the magnetic characteristics presented so far are then dictated by the value of the anisotropy constant. This value can be estimated from the hysteresis loops obtained applying the field along a hard magnetization axis, if the value of the saturation magnetization is known [8].

![Figure 3](image-url)  
**Figure 3.** A comparison between the microhardness of the layer obtained by classic treatment (HV1) and the microhardness of the layer obtained by unconventional treatment T3 (HV3), taking into account the thickness of the superficial layer (GT1).

![Figure 4](image-url)  
**Figure 4.** Distribution of I₁Fe₄N and I₁Fe₃N in the superficial nitrided layer depending by the duration of the wear process, for Q = 75 daN and ξ = 10 % (T1).

![Figure 5](image-url)  
**Figure 5.** Distribution of I₁Fe₄N and I₁Fe₃N in the superficial nitrided layer depending by the duration of the wear process, for Q = 150 daN, ξ = 20 % (T1).
Figure 6. Distribution of $I_{Fe_{4N}}$ and $I_{Fe_{3N}}$ in the superficial nitrided layer depending by the duration of the wear process, for $Q = 150$ daN, $\xi = 20\%$ (T3).

Figure 7. Distribution of $I_{Fe_{4N}}$ and $I_{Fe_{3N}}$ in the superficial nitrided layer depending by the duration of the wear process, for $Q = 150$ daN, $\xi = 20\%$ (T4).

Figure 8. Distribution of $B_{Fe_{4N}}$ and $B_{Fe_{3N}}$ in the superficial nitrided layer depending by the duration of the wear process, for $Q = 75$ daN, $\xi = 10\%$ (T1).

Figure 9. Distribution of $B_{Fe_{4N}}$ and $B_{Fe_{3N}}$ in the superficial nitrided layer depending by the duration of the wear process, for $Q = 150$ daN, $\xi = 20\%$ (T1).

Figure 10. Distribution of $B_{Fe_{4N}}$ and $B_{Fe_{3N}}$ in the superficial nitrided layer depending by the duration of the wear process, for $Q = 150$ daN, $\xi = 20\%$ (T3).

Figure 11. Distribution of $B_{Fe_{4N}}$ and $B_{Fe_{3N}}$ in the superficial nitrided layer depending by the duration of the wear process, for $Q = 150$ daN, $\xi = 20\%$ (T4).
In figures 8 - 11, one could observe that the internal tensions of the phases determine a lower resistance of the Fe₃N phase during the cyclical fatigue. This fact was determined by mechanical oscillations created by the magnetic field (A.C. current) through the permanently changes of the field lines directions, because of the magnetostriction existence. This magnetostriction from thermo-magnetic treatment changes the re-crystallization conditions, the speed of germination. That’s why the hardness of the superficial nitrided layers increases. The degree of the Martensitic Tetragonalitate (c/a) is higher in the case of γ’-Fe₄N (see figure 12).

Figure 12. The evolution of the c/a characteristic, depending by duration, in case of the treatments: T1 (classic) and T3 (unconventional treatment).

In figures 13 - 15, were presented some micro-structural aspects of the superficial nitrided layer, in case of T1 (classic treatment) and in cases of T3 and T4 (unconventional treatment regimes), considering a Nital attack 2%. From the structural point of view, applying a magnetic field (A.C. or, D.C.), there is a noticeable finishing grain size, from 9 value until 7-8 value, This situation will enable a better diffusion all along the grain boundary, when applying a thermo-chemical diffusion treatment such as ion nitriding.

Figure 13. Superficial layer thickness, in the case of T1 treatment (x100).

Figure 14. Superficial layer thickness, in the case of T3 treatment (cooling in A.C. Current –magnetic field), (x100).

Figure 15. Superficial layer thickness, in the case of T4 treatment (cooling in D.C. Current –magnetic field), (x200).

It was considered the evolution of the worn-out layer depth (Uh) during three hours of the wear process through dry friction process and the values are presented in Table 1.

Table 1. The worn-out layer depth evolution

| Q [daN] | 75  | 150 | 190 |
|---------|-----|-----|-----|
| Uh [mm] | 0.09| 0.14| 0.18|

Figure 16. Worn-out depth of the superficial layer (Uh) evolution, depending by sliding degrees (ξ =10% or 20%) and normal force (Q), for the treatment T1 case.
In figure 16, the worn-out depth (Uh) of the superficial layer increases simultaneously with the increase of the normal load (the down force, Q).

4. Statistical model

It can be said that the linear model of uni-factorial regression can describe with success the relationship between the two indicators analyzed.

The main problem of any regression model is the determination of the model parameters, operation which can be performed using the method of least squares. It starts from the regression equation of a simple linear model:

\[ y = a + bx + u \]

\[ t = 1 \]  \hspace{1cm} (3)

where: \( t, u \) are theoretical values of variable \( y \) obtained only function by the values of essential factor \( x \) and by the estimators values of the parameters \( "a" \) and \( "b" \), respectively, \( "\hat{a}" \) and \( "\hat{b}" \).

To prove the causation between a normal load (Q) and the worn-out depth of the superficial layer (Uh), resort to statistical methods for checking the next assumption: it is assumed that there is a causal relationship between the independent variable (Q) and a dependent variable (Uh). Between these variables there is a direct connection. Application of analysis models of regression and correlation implies the following steps.

First step is represented by a correct identification of the variables \( X \) and \( Y \). In this case, Q will be considered the factorial variable \( X \) (the cause) and Uh will be the dependent variable \( y \) (the efficient variable).

The second step is represented by a verification of the existence of the connection between \( X \) and \( Y \). For this method, the graph of the correlation called the correlogram will be used. Based on the graph of correlation we can say that between the two variables, there is a direct connection. Because of the tendency towards the concentration is around a straight line, the graph suggests a linear connection.

The third step is the establishment of the mathematical form of the connection: the development of the regression equation corresponding to this regression model. In the case of regression unifactorial, it is considered that on a resulting characteristic \( "y" \) acts the only variable factor \( "x" \) and the others can have a constant and negligible action: \( y = f(x) \). Considering that the graphic of the function \( f(x) \) is a straight line, results a linear model of the simple regression [10]. The function of estimation is given by the expression:

\[ \hat{y} = a + bx \]

\[ (4) \]

Modeling function is given by the following expression:

\[ y_{xi} = a + bx_i, \]

\[ (5) \]

for the linearity of connection and it means an additive interaction scheme of variables, in conditions by uniform changing with constant amounts of characteristic “\( x \)” [11].

The parameter “\( a \)” is the value of the regression function at the point \( x = 0 \). This parameter “\( a \)” is the ordinate by origin, meaning the point where the regression right intersects the 0y axis.

The parameter “\( b \)” called the regression coefficient, shows the amount with which you can modify the variable “\( y \)”, to change the variable with one unit.

The Method of least squares assumes the minimization of the following function:

\[ F(\hat{a}, \hat{b}) = \min_{\hat{a}} \sum_{i=1}^{n} (y_i - \hat{y})^2 = \min_{\hat{a}} \sum_{i=1}^{n} (y_i - \hat{a} - \hat{b}x_i)^2 \]

\[ (6) \]

The minimum condition of this function implies the formation of the system of two equations with two unknowns:

\[ n\hat{a} + \hat{b} \sum_{i=1}^{n} x_i = \sum_{i=1}^{n} y_i, \]

\[ (7) \]

\[ \hat{a} \sum_{i=1}^{n} x_i + \hat{b} \sum_{i=1}^{n} x_i^2 = \sum_{i=1}^{n} x_i y_i, \]

\[ (8) \]
For this case, \( n = 3 \).

One can write the equations from the following system of equations:

\[ 3a + b (75 + 150 + 190) = 0.09 + 0.14 + 0.18 \]
\[ a (75 + 150 + 190) + b (75^2 + 150^2 + 190^2) = 75 \cdot 0.09 + 150 \cdot 0.14 + 190 \cdot 0.18 \]

Final results are:

\[ b = 0.00076 > 0; \quad a = 0.0315 \]

Because the coefficient “\( b \)”, also called *regression coefficient*, has a positive value, it shows a direct relation between the two variables. In the graph of correlation the regression coefficient (b) represents the straight line slope. Replacing the values of “\( a \)” and “\( b \)” in the regression equation, it was obtained the new regression equation:

\[ \hat{y} = 0.00076 x + 0.0315 \quad \text{(9)} \]

or, \( y_i = 0.00076 x_i + 0.0315 \quad \text{(10)} \)

It is estimated that the connection between the two variables (Q and Uh) is the straight line equation. Using the coefficients “\( a \)” and “\( b \)” it is calculated the regression equation value of each ‘\( x \)” characteristic size. These values of the regression equation are also called theoretical values of \( y \) according to \( x \). The operation of replacing of the real terms \( y \) with the values of regression equations (theoretical values) is called adjustment.

To check the accuracy of parameters calculation of a regression function it is used the following relation:

\[ \sum \hat{y} = \sum y \quad \text{(11)} \]

This relation is based on the fact that by calculating the regression equation it is obtained a redistribution of the degree of factors influence. To emphasize the logical connection between phenomena is necessary to investigate a large number of individual cases in which deviations (in either direction) is offset each other [13].

Comparing the real values of \( y \) with the adjusted values for resulting feature \( \hat{y} \), there are small differences, presented in table 2. It is estimated that the bond between these two variables (Q and Uh) is represented by the equation of straight line (Figure 17). Using Matlab software, it was obtained the correlation graphic corresponding to the equation (9), for the values of correlation from table 2 (figure 17).

| Table 2. Adjusted values |
|--------------------------|
| \( x \) | \( \hat{y} \) |
| 75   | 0.0885 |
| 150  | 0.1455 |
| 190  | 0.1759 |

| Table 3. Values compared |
|---------------------------|
| \( \hat{y} \) | \( y - \hat{y} \) |
| 0.0824 | 0.09 - 0.0885 = 0.0015 |
| 0.170 | -0.0055 |
| 0.216 | 0.0041 |

**Figure 17.** Representation of the equation: \( \hat{y} = 0.00076 x + 0.0315 \)

The fourth Step represents the characterization of the connection intensity between these two variables using the correlation coefficient "\( r_{xy} \)” and the correlation report [10, 11, 13].

\[ \hat{y} = \frac{1}{n} \sum_{i=1}^{n} x_i y_i \quad \text{(12)} \]
The above relation, for covariance coefficient, can be written [10, 11, 13]:

\[
M(x \cdot y) = n^{-1} \sum_{i=1}^{n} x_i y_i, \tag{13}
\]

For \( n = 3 \), \( \bar{y} = M(x \cdot y) = 61.95/3 = 20.65 \).

The simple correlation coefficient “\( r \)” can be written [13]:

\[
r = \frac{n \sum x_i y_i - \left( \sum x_i \right) \left( \sum y_i \right)}{\sqrt{n \sum x_i^2 - \left( \sum x_i \right)^2} \sqrt{n \sum y_i^2 - \left( \sum y_i \right)^2}} \tag{14}
\]

\[r = \left( 3 \cdot 61.95 - 415 \cdot 0.41 \right) / 20450^{1/2} \cdot 0.0123^{1/2} = 15.70/15.85 = 0.99 > 0\]

In this case, \( r_{x,y} = 0.99 > 0 \)

This result indicates the correlation, a direct connection between these two variables (Q and Uh). Theoretically, if the value is close by 1, we have a very strong connection between the variables. In this case, it can be talk about a relative deterministic connection because \( r_{x,y} = 0.99 \) and the following relation: \( 0.95 \leq r \leq 1 \) was demonstrated. For the interpretation of nonzero values for coefficients of correlation, an explanation graphics is much more suggestive in mathematical statistics. The value of the correlation coefficient is in the dependency pairs \((x_i, y_i)\) with the distribution of values in a rectangular XOY references.

Regarding the geometric configuration of the corresponding distribution points, the distinction is made between the following cases:

a). The points are alignment along a line: it can be a right ascending line \( (r_{x,y}=1) \), or it can be a right line downward \( (r_{x,y} = -1) \). These situations indicate a dependence relation between the two variables.

b) The points are dispersed random, the cloud of the points hasn’t orientation. The two variables are independent or uncorrelated \( (r_{x,y} = 0) \).

It is calculated the simple ratio of correlation \( (R_{x,y}) \) taking into account the multiple ratio of correlation according to [10,11,12,13].

It is find that \( R_{x,y} \) is approximately equal with the simple coefficient of the correlation “\( r \)”. To confirm the linearity of the connection, the following relation must be met:

\[
| r_{x,y} | = R_{x,y} \tag{15}
\]

In this case,

\[
| r_{x,y} | = R_{x,y} = 0.99
\]

It was confirmed statistically the direct linear connection between these two variables: Q and Uh.

The correlation is positive because the graph of the correlation is linear ascending and is strong enough because the points are close. It is a linear correlation. Testing suitability of the model is the formula for the correlation coefficient obtained \( (0.95 \leq r \leq 1) \).

5. Conclusions

The first research direction was to increase the mechanical characteristics (hardness) of the steel through thermo-magnetic treatment in order to applying the thermo-chemical diffusion treatment under thermo-magnetic treatment temperature. Another line of the research was to study the influence of the thermo-magnetic treatment on the superficial layer thermo-chemically treated.

The originality consists of applying the thermochemical 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 after the thermo-magnetic treatment during the thermo-chemical one.
A mathematical model has been made to confirm the connection between some characteristics of the material according with the parameters of treatments.

A statistical model from Economics which demonstrated the causal relation between the normal loading (Q) and the depth of the worn-out nitrided layer (Uh) after three hours of wearing process, has been proposed in this study.

During this work, a correlation, a regression line through the linear model of uni-factorial regression using the least squares approximation method has been proposed. It was demonstrated that a direct connection exists between these two variable.

In the cases presented up, it can be talk about a relative deterministic connection because $r_{x,y} = 0.99$ and the following relation: $0.95 \leq r \leq 1$ was demonstrated.

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