THE CASE DEPTH GROWING KINETICS OF THE AISI 9310 STEEL CARBON HARDENED PARTS FOR AERONAUTICAL APPPLICATION

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Lucrarea face parte dintr-un studiu de cercetare experimentală mai amplu privind influența principalilor parametri (termici, temporali şi chimici) ai tratamentului termochimic de carburare asupra nivelului valorilor principalelor caracteristici microstructurale, chimice şi fizico-mecanice ale stratului rezultat la carburarea pieselor realizate din oțelul AISI 9310. Prezenta lucrarea tratează numai aspectele referitoare la influența celor trei parametri principali ai tratamentui termochimic de carburare (temperatura procesului, T_K, durata procesului, t_K, şi potențialul de carbon, $\%C_{pot\ K}$) asupra principalei variabile geometrice a stratului carburat, adâncimea efectivă a stratului carburat ($\delta_{ef.}$). Originalitatea lucrării constă în modul de abordare al studiului experimental, respectiv prin utilizarea metodei experimentului programat activ, programarea fiind necompozițională de ordinul II.

The work is a part of a larger experimental research study on the influence of the main process parameters (thermal, temporal and chemical) of carburizing heat treatment on the level of the main carburizing microstructural features, chemical and physico-mechanical properties values resulting from the carbon hardening process of the AISI 9310 steel parts. This paper deals with only those aspects concerning the influence of the three key parameters of the carburizing process (process temperature, T_K , process time t_K, and the carbon potential ($\%\ C_{pot.K}$) on the main geometric variables of the case, namely the effective case depth ($\delta_{ef.}$). The work originality lies in the approach of the experimental study using the actively experiment scheduled method with the second order of noncompositional programming.

Keyword: Carburizing, effective case depth, carbon potential, the actively experiment scheduled method, programming matrix

1. Introduction

The complexity of the phenomena involved in the processes of heat treatments makes the study of these processes by means of the classical

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experimental research methods, inefficient leading in most cases, to unreliable results.

To solve the problems in the experimental researching processes which are based on diffusion phenomena using experimental method requires to apply the scheduled experimental method which permits to realize the empirical mathematical models which can be obtained either by passive or by active experiment methods [1].

The most information that are presented in the literature concerning the AISI 9310 steel characterize it in terms of the mechanical characteristics that can be obtained by carburizing chemical heat treatment without making reference on concrete ways in which these performances, often spectacular, can be obtained.

The majority of the works in the field of the chemical heat treatment presents the information concerning the relatively modest performance of the carbon hardened AISI 8620 steel, with a low content in alloying elements [2].

Although in the last time the AISI 9310 steel is commonly used to the manufacture of the carbon hardened parts for the aeronautical industry, in the literature few data are available concerning the performance, often spectacular [3], which can be obtained by applying the carburizing chemical heat treatment on the components made in this steel. This is why the present work, detached from a larger study on the carburizing treatment of components made from this steel, aims to fill some of these goals concerning the specific aspects of the carburizing process of the AISI 9310 steel parts, respectively how the main process parameters influence the actual effective case depth size and its growth kinetics.

2. The study of the growing case depth kinetics by active experimental sheduled method and the second order noncompositional programming

Existing knowledge about the processes that are based on diffusion phenomena generally require to resort to the use of the second order noncompositional programming [4], for explaining the interactions between process parameters (independent parameters) and their effects on the level of variation in structural features and the respective values of the physical and mechanical characteristics (dependent parameters).

The reason for using this type of programs consists on one hand in the fact that the processes underlying the formation of diffusion layers can not be mathematically defined using linear models [5, 6] and on the other hand, there are sufficient knowledge concerning the range where can be found the values of interest. In order to solve the problem it is sufficient to explain the experimental data using second-order nonlinear equations of the form:

$$Y = b_0 + \sum_{\substack{i=1 \\ 1 \le i \le k}}^{k} b_i x_i + \sum_{\substack{i=1, j=1, i \ne j \\ 1 \le i \prec j \le k}} b_{ij} x_i x_j + \sum_{i=1}^{k} b_{ii} x^2 + \cdots \tag{1}$$

where: Y is the dependent parameter investigated, and x_i, x_j independent parameters that influence the dependent parameter.

Independent variables (x_i ...) taken in the analysis were those related to carbon enrichment phase: isothermal temperature T_K (x_1), the maintaining time t_K (x_2) at carburizing temperature and carbon potential $C_{pot\ k}$, (x_3). In the context of second order of noncompositional programming was imposed to vary the three independent variables at three levels of value, -1, 0, 1. The noncompositional second order programming plan is itself a selected segment of the factorial experiment 3^k (k is the number of factors). As the dependent variables of the process (Y_1 .... Y_n), were selected those featuress that allow a more complete characterization of the effects of surface enrichment in carbon due to maintenance of the AISI 9310 steel parts in the enriched gaseous hydrocarbon (methane) atmosphere.

This paper has taken in the analysis (as dependent variable) one of the most important dimensional variable of carbon hardened case, namely the effective case depth ($\delta_{ef.}$) expressed in [mm], which together with other characteristics, such as the carbon content ($C_{0.1mm}$), the retained austenite proportion ($\%RA._{0.1mm}$), the microhardness ($HV_{0.1mm}$), all of them measured at the 0.1 mm distance from the workpiece surface, the hardness of the workpiece surface expressed in Rockwell (HRC) or Vickers (HV) units and the case depth affected by the internal oxidation, all of them characterizing the quality of the carburizing process.

The samples for the evaluation of the actual effective hardened case depth ($\delta_{ef.}$) were taken from the final heat treated parts, respectively after annealing, hardening, tempering and subzero cooling, which were used for metallographic evaluation. Criteria for evaluating the effective case depth was the distance from the sample surface until the case reach a hardness value 513 HV or 50 HRC.

The second order programming noncompositional matrix, for k = 3 (number of independent parameters), the basic level, their range of variation and the results of case depth measurements are presented in Table 1.

To establish the algorithm for determining the particular forms of nonlinear models for the dependent variable taken in discussion, require the following steps for processing the experimental results obtained:

- Calculation of non linear model coefficients (b_0, b_i, b_{ij} ...);
- The statistical verification of nonlinear model coefficients;
- Calculation of the reproducibility results dispersion;
- Verification of the concordance of nonlinear model adopted.

Table 1

Noncompositional programming matrix of second order (k = 3)

-	F.V.	Parametru independent			δ_{ef}.
		1*	2*	3*	[mm]
Code	X_0	X_1	X_2	X_3	Y_1
Basic level (Z_{i0})		925	6	0,9	-
Range varation (ΔZ_i)		25	3	0,2	-
Higher level ($Z_{i0}+\Delta Z_i$)		950	9	1,1	-
Lower level ($Z_{i0}-\Delta Z_i$)		900	3	0,7	-
EXP.nr.1	+1	+1	+1	0	1.455
EXP.nr.2	+1	+1	-1	0	0.831
EXP.nr.3	+1	-1	+1	0	1.079
EXP.nr.4	+1	-1	-1	0	0.660
EXP.nr.5	+1	+1	0	+1	1.320
EXP.nr.6	+1	+1	0	-1	0.947
EXP.nr.7	+1	-1	0	+1	1.060
EXP.nr.8	+1	-1	0	-1	0.734
EXP.nr.9	+1	0	+1	+1	1.312
EXP.nr.10	+1	0	+1	-1	1.047
EXP.nr.11	+1	0	-1	+1	0.839
EXP.nr.12	+1	0	-1	-1	0.696
EXP.nr.13	+1	0	0	0	0.989
EXP.nr.14	+1	0	0	0	1.027
EXP.nr.15	+1	0	0	0	1.011

Note:
F. V. - a fictive variable;
*1 - the carburizing temperature, T_K [ºC] Z_1;
*2 - the maintaining time at carburizing temperature, t_K, [hours], Z_2;
*3 - the carbon potential for carburizing process $C_{pot.K}$, [%C] Z_3.

3. Experimental

3.1. Actual development of experimental batches and modalities for determining and evaluating the results of experimental research

The experiments were conducted in a batch furnace type using endogas atmosphere enriched with methane gas. The chemical composition of the samples used (AISI 9310 steel) is presented in Table 2.

Table 2

Chemical composition of AISI 9310 steel

Alloy (steel)	**Elements content, %**						
AISI 9310	**C**	**Si**	**Mn**	**Cr**	**Mo**	**Ni**	**Cu**
min.	0.07	0.15	0.4	1.00	0.08	3.0	max.
max.	0.13	0.35	0.7	1.40	0.15	3.5	0.35
actual	0.11	0.33	0.57	1.14	0.13	3.26	0.09

In order to study the influence of the independent parameters (temperature, maintaining time and carbon potential) on the microstructural and on the physical and mechanical characteristics of the effective case depth (parameter dependent) fifteen carburizing batches were carried out, of which the last three batches have been performed under the same conditions of temperature, time and carbon potential.

For each batch of experiments the temperature and the carbon potential and their actual variation have been recorded. In Table 1 are presented the parameters (thermal, temporal and chemical) adopted in developing the fifteen experiments.

All actual cycles show the same general aspects of which the most significant features are the two following ones (see Fig. 1):

1) The batches loading was performed in the preheated furnace to the carburizing temperature process. During the batches loading the furnace temperature down to about 50÷60°C below to the initial point setting temperature. After about 12÷16 minutes, batch temperature reached the temperature programmed for carrying out the carburizing cycle and the record of maintaining time for process was done only after the carbon potential of atmosphere in the furnace has reached the prescribed value, specific to each experimental cycle.

For this reason the effective thermal cycles are easily moved to the right (recovery time) with a period of time which accounts for the time of batch loading, batch reheating to the process temperature and time for achieving in the furnace atmosphere the carbon potential prescribed.

2) Effective chemical cycles show a certain delay in the same initial period (recovery time) until achieving the carburizing temperature, whose duration varies according to the prescribed value of carbon potential.

Taking into consideration that chemical and thermal cycling displacements are systematic, uniform and proportional with the carbon potential and carburizing temperature in all experimental batches, the displacements have not altered the types and degrees of influence of independent variables (process parameters) on the dependent variables, respectively on the experimental results obtained.

3.2. Effective case depth ($\delta_{ef.}$) evaluation

To study the case depth growing kinetics it was necessary to determine and evaluate the main dependent geometrical variable of the case: the effective case depth hardened Y($\delta_{ef.}$).

The specimens (ø 18.5mm x 30mm) on which were made the measurements to determine the effective case depth were taken from the carbon final heat treated, hardened samples which were used for metallographic evaluations. Corresponding to each batch were evaluated the effective cases depth, ($\delta_{ef.}$), expressed in mm, based on the results measurements after the experimental HV hardness profiles curves tracing with a step of 0.1 mm (distance between two successive impressions). Fig. 2 presents the schedule for measurements of the effective case depth. For evaluation relationship (2) was used.

The criterion for evaluating the effective case depth was the distance from the surface of the sample until it reaches the 513 HV hardness. In Table 1 are presented the values obtained for effective case depth by processing the measurements from the hardness profile curves.

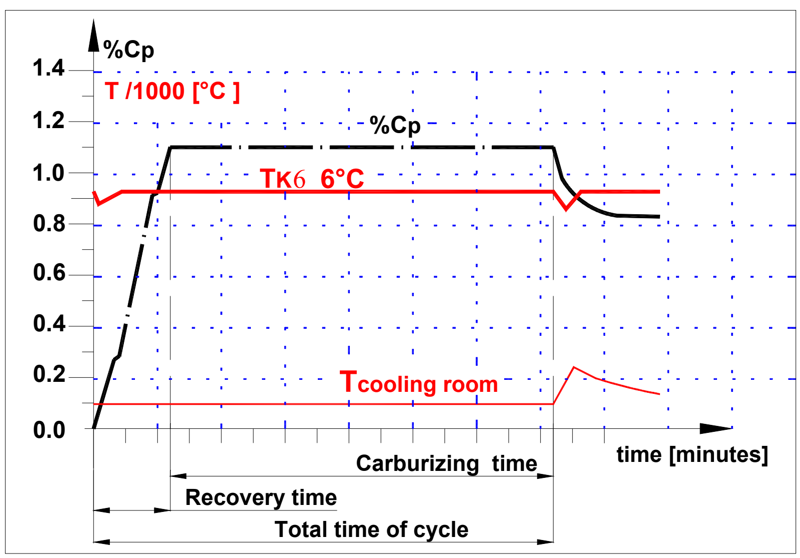

Fig. 1. Thermal and chemical cycles general characteristics used in the experimental study

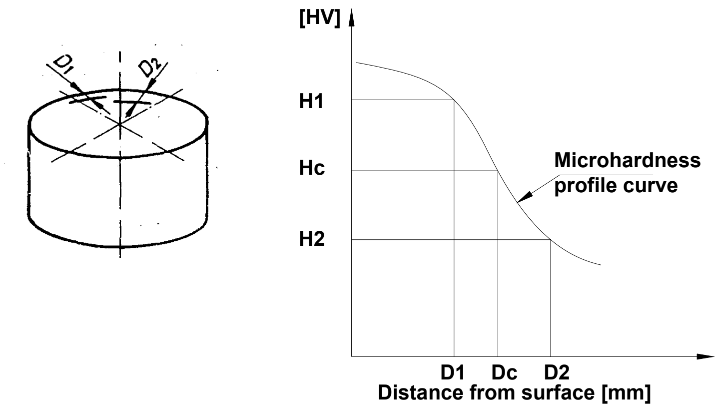

Fig. 2. Case depth evaluation schedule

$$D_C = D_1 + \frac{(D_2 - D_1)(\overline{H}_1 - H_C)}{\overline{H}_1 - \overline{H}_2} \tag{2}$$

where:

D_c - conventional case depth;

H_c - imposed micro hardness value;

$\overline{H}_1$, $\overline{H}_2$-micro hardness average values measurements at D_1 and D_2 distances.

4. Experimental research results

The mathematical models were accomplished by determining the regression equations, able to allow the prediction performance which can be obtained by choosing the conditions of the carburizing process on the AMS 6265 steel parts for the aeronautical industry.

After the stages of calculating of the coefficients and statistical verifications specific to the programming method chosen, the following particular forms of the regression equations specific to the steel taken into consideration in the analysis have resulted:

$$\begin{aligned} Y(\delta_{ef}) &= 1.009 + 0.1275X_1 + 0.2334X_2 + 0.1384X_3 \\ &+ 0.0512X_1X_2 + 0.0305X_2X_3 \end{aligned} \tag{3}$$

$$\begin{aligned} \delta_{ef.} &= -0.7309 + 0.007T_K - 0.5999t_K + 0.386C_{pot.K} \\ &+ 0.000681T_K \cdot t_K + 0.0508t_K \cdot C_{pot.K} \end{aligned} \tag{4}$$

The equations (3) and (4) represent the encoded form respectively the decoded of mathematical model of effective case depth.

5. Discussion

The comparative analysis of the two equations shows that the terms with the greatest influence are the terms of the first order degree (X_1, X_2, X_3, respectively T_K, T_K, $C_{pot\,K}$), and second-degree terms of the form X_1X_2 ($T_K \cdot t_K$) and X_2X_3, ($t_K \cdot C_{pot\,K}$) have significant influence the both equations.

Based on this finding it can be concluded that the proposed mathematical model is predominantly linear, fact also confirmed by the aspects of response surfaces and by the positions of certain isoproperties areas of the effective carburizing case depth (see Fig.3 - A, B, C, D, E and F)

The analysis of the encoded equation (3) shows that all independent coefficients for the variables (X_1, X_2, X_3, namely T_K, t_K, C_{potK}) are positive, both terms of degree, as well as the terms of the second degree, and X_1X_2, X_2X_3, (and T_Kt_K, t_KC_{potK}). This leads to the conclusion that the value of the dependent variable Y_1 (δ_{ef}.) increases with the increasing values corresponding to independent variables.

Regarding the numerical value and the sign of the influence coefficients which determine the degree and the direction of the influence of the independent variables, these may be more strongly evident in the case of decoded equation. Indeed the $b_i x_i$, $b_{ij} x_{ij}$, products, respectively $b_{ii} x_i^2$ are terms that by algebraic summing permit to obtain the dependent variable value. In the regression decoded equation, although the first order term (- $0.5999t_K$) has a negative value, in any combination of temperatures – time – carbon potential one obtains the positive and increasing value for the dependent variable Y_1 (δ_{ef}.) according to increasing values of the process parameters (T_K, t_K, $C_{pot.K}$). This is because for the any combination of the three independent variables (process parameters) namely T_K, t_K and C_{potK}, the sum of the positive terms is always greater than the sum of the negative terms of the decoded equation.

This behavior was expected for the following reasons:

1. carburizing temperature increases the growth rate of carbon transfer surface β according to the relationship $\beta = \beta_0 \cdot \exp(-Q/KT)$ and the diffusion coefficient D of carbon in austenite according to the Hirschheimer relationship [4] $D = 0.2\exp\left(-\dfrac{16608}{T_K}\right)$ This leads to positive influences in terms of growing case depth thickness;
2. increase the potential for carbon involves an increase in carbon monoxide concentration and consequently an increase in the product

CO x H_2 respectively the preexponential factor β_0 in the relationship mentioned at point 1°;

3. carburized case depth thickness dependence on the maintaining time agrees with the relationship $\delta = K \cdot \sqrt{t_K}$;
4. Combination of all these parameters accelerates more the growing of the case depth.

6. Graphical processing of experimental results

The graphical processing of the experimental research results (Fig. 3) creates a more suggestive picture of the manner in which the three independent variables of the carburizing process, namely the temperature at which that process is carried out, T_K, the maintaining time at this temperature, t_K, and the carbon potential, C_{potK}, of the furnace atmosphere influence the value of the effective dimension of the case depth.

The charts presented below were drawn using one proper regression equations (coded or uncoded) of the mathematical model established and by assigning values (-1, 0, 1) to the one of the process parameters corresponding to the three levels of variation specified in the noncompositional second order for k = 3 programming matrix of the experiment.

6. Conclusions

The analysis of the results obtained after performing experimental leads to the following conclusions:

1. The effective case depth size is influenced by the temperature, the process time and by the carbon potential of furnace atmosphere according to the mathematical model, calculated and expressed by both equations (3 and 4).

2. The changes (increasing or decreasing) the value of any of the three process parameters will cause the increasing or decreasing the case depth thickness value accordingly to his mathematical model.

3. The mathematical model calculated in this work can be applied to AMS 6265 carburizing steel parts in order to estimate the actual size of the effective case depth in a wide range of temperatures (900 ºC $\le T_K \le$ 950 ºC), maintaining time (3 hours $\le t_K \le$ 9 hours), carbon potentials (0.7 %C $\le T_K \le$ 1.1 %C) and can be applied to optimize the carburization process.

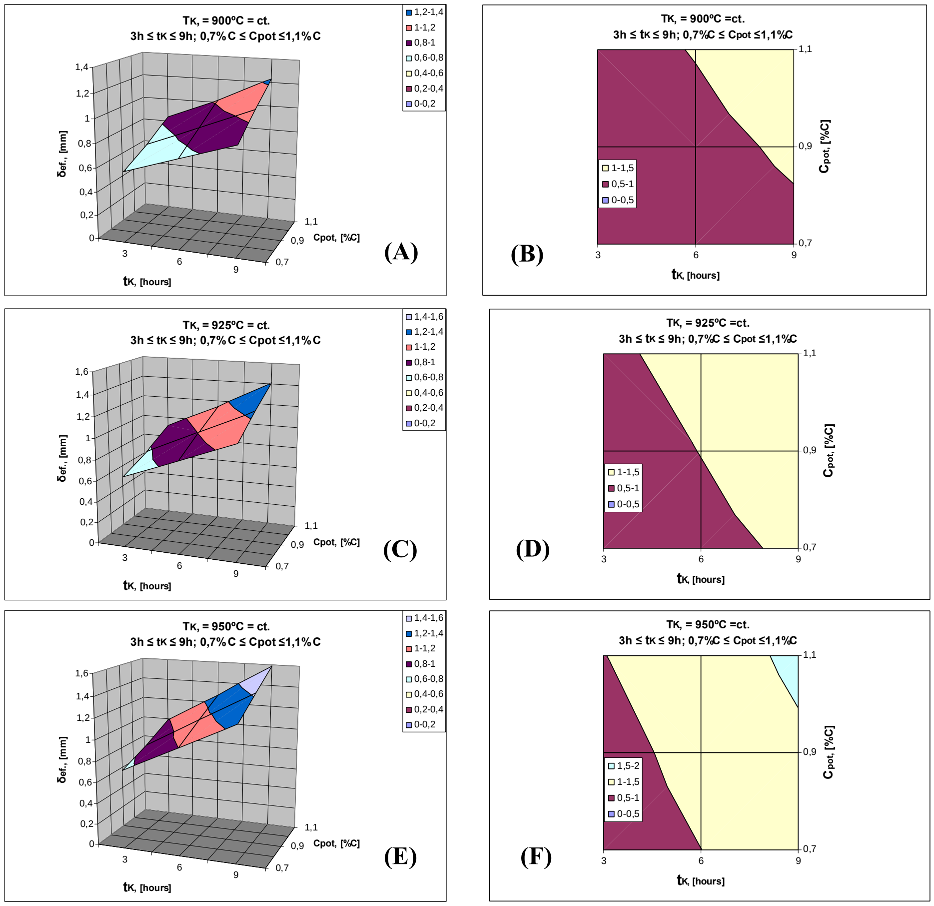

Fig. 3 The surfaces response of the mathematical model that describes the variation of dimensionals depth case at the temperatures: (A) T_K = 900 °C, (C) T_K = 925 °C and (E) T_K = 950 °C and the delimitation areas of isoproperties (B) (D) (F), depending on the temporal and chemical parameters of the process in carburizing AMS 6265 steel parts.

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