Intelligent control system for gaseous nitriding process

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In the present paper, the solutions of the following research issues have been presented:

- *dependencies between the process parameters and the layer structure have been defined, which serve to develop software for the control system of the process, with the view of obtaining a complex layer structure and optimal kinetics of its creation and growth,*
- *assumptions for the control system of the gaseous nitriding process have been developed on the basis of a complementary cooperation of the mathematical model and the indications of the magnetic sensor registering the nucleation and growth of the layer.*

These issues comprise two complementary ways towards the construction of intelligent control systems. The first one of them consists in developing innovative databases, expert systems to aid the operator in decision making regarding the choice of defined changes to the process parameters. This is connected with the knowledge of possibly all the factors and mechanisms which have an influence on the result of the process and which make it possible to design an algorithm of changes of process parameters that could guarantee optimal kinetics of the layer growth and its required structure. The other one additionally makes use of specially constructed sensors, which having been placed directly in the processes react to the growth of layers and their structure.

Key words: mathematical model, model of the nitriding process, system of visualization of nitriding process, control system

INTRODUCTION

A growing range of applications of the nitriding process, in which the layer nitrided should be characterized by better and better properties, requires the use of intelligent control systems. There are two complementary manners of behaviour towards the construction of such systems. The first one consists in developing modern databases and expert systems to support the operator in decision making concerning the selection of specific changes in the parameters of the process. It is connected with the knowledge of possibly all the factors and mechanisms which have an influence upon the result of the process, which make it possible to design algorithms of changes of process parameters, which guarantee an optimal kinetics of the layer growth and its required structure. The other one additionally uses specially constructed sensors, which placed directly in the process react on the growth of layers and their structure.

The issues discussed in this paper include these two manners of behaviour. In particular, they comprise the following:

1) a development of an experimental-theoretical model of the nitriding process to facilitate a simulation of the layer growth kinetics,
2) guidelines for assumptions for the construction of the following:
    - a system for the visualization of growth of the nitrided layer, whose principle of operation is based on a complementary interaction of a process result sensor (a magnetic sensor) and the experimental-theoretical model,
    - a system of an automatic control of the nitriding process, in which the block controlling the main process parameters, i.e. temperature, composition and the intensity of the flow of the nitriding atmosphere works on the basis of sensor signal-time courses registered by a magnetic sensor, mapping the growth of the nitrided layer.

The model was constructed on the basis of a mathematic description of the growth kinetics of the nitrided layer and experimental databases combining the process parameters with the structure of the nitrided layer. The mathematical description included the following:

- the growth kinetics of the nitrided layer in quasi-equilibrium conditions, depending on the process time and nitride potential,
- the growth kinetics of the diffusion layer.

The assumptions presented in this paper concerning the system of visualization of the nitrided layer growth and the control system of the layer growth include innovative solutions of a new generation. At present, an important issue in the gas nitriding technology is the possibility to control solely the process parameters, i.e. the process temperature and the composition of the gaseous atmosphere and the intensity of its flow. The creation and the growth of the layer is not directly controlled in the process. The process, due to the reasons we know about from practice, may proceed not in compliance with the assumptions. In such a case, we learn about the improper course of the layer creation only after its completion. The assumptions for innovative systems presented in the paper are based on the indications of the process result sensor: a magnetic sensor. In particular, the operating principle of the nitrided layer growth visualization system consists in a complementary interaction of the system of the sensor and the model of the process. The characteristic points of sensor signal-time courses registered by the sensor, which correspond to e.g. obtaining the maximum surface hardness or the creation of a continuous layer of iron (carbo)nitrides (of a few micrometers), constitute the initial conditions for the description by the model of further stages of the nitrided

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layer development. In the control system and, the software of the block controlling the main process parameters, i.e. temperature, the nitriding atmosphere composition and the intensity of the flow serves to assure that the kinetic sensor signal-time course registered by a magnetic sensor imaging the nitrided layer growth is compliant with the reference course.

MATHEMATICAL MODEL

The course of the kinetics of the process depends on the speed of particular partial stages. An equation describing the kinetics of its slowest stage is an equation which describes the kinetics of the whole process. The speed of particular stages of the creation and growth of the nitrided layer changes together with the time of the process. A chemical reaction of ammonia dissociation on the surface of the nitrided element is the slowest stage in the initial phase of the layer creation. While the layer is growing, the diffusion of nitrogen in steel is becoming the slowest stage. This results in the fact that the kinetics of the whole process is a complex time function. However, through a proper selection of the following parameters: temperature, the intensity of the flow of the nitriding atmosphere and its composition, one can make the transport of nitrogen in steel [1] play the main role in the whole process. Experimental data collected for such cases concerning the growth of particular mono-phase zones in the nitrided layer [2], as well as the growth of such zones in other diffusion systems, e.g. metal-metal [3], indicate a parabolic low of the growth of phase zones:

$$\Delta x_i = k_i \sqrt{t} \qquad (1)$$

where:

Δx_i – thickness of ith phase in n-phase layer after process time t (for nitrided layer, the maximum value n=3),

k_i – kinetic parameter of the growth of ith phase, the so-called constant of parabolic growth of ith phase,

t – process time.

Equation (1), with experimentally designated constant k_i, serves to describe the growth kinetics of phase zones in a given temperature. For practical reasons, this is enough in most cases, as the knowledge of k_i value in different temperatures for all the phases of the diffusing system makes it possible, in accordance with equation (1), to determine the change of the thickness of a given Δx_i phase in the process time function. However, this is an oversimplified description of the growth kinetics, which makes it impossible to determine, in the case of a nitrided layer, e.g. nitrogen diffusion coefficients in individual layer phases or nitrogen concentrations on phase borders. One cannot foresee on the basis of this equation the influence of the remaining phases creating the layer upon the speed of the growth of a given phase, either. For this reason, it became necessary to develop general mathematical equations describing dependencies between growth parameters (k_i) and diffusion parameters, which facilitated the determination of diffusion coefficients in mono-phase layer zones, as well as forecasting of the phase growth in the function of the process time. In the model developed, whose particulars were given in paper [4], the final result are the equations given below (2), which in a direct manner connect the kinetic parameter of a given phase k_i with the difference of concentrations on the phase borders and an effective diffusion coefficient in the phase:

$$\sum_{j=1}^{n} \alpha_{i,j} k_i k_j = \left(D_i^c\right)_{ef} \Delta c_i \qquad \text{i=1,2,3} \qquad (2)$$

where:

$$\alpha_{i,j} = \left\{ \begin{matrix} \bar{c}_i, i > j \\ \frac{1}{4}\left(3\bar{c}_i + c_{i,i+1}\right), i = j \\ \bar{c}_j, i < j \end{matrix} \right\}$$

k_i – kinetic parameters of the growth of ith and jth phase,

Δc_i – difference of concentrations of the diffusing element on the border of i^{th} phase.

This is a system of non-linear equations, which can be solved with e.g. a method of successive approximations (iterations).

$$k_i = \frac{2\left(D_i^c\right)_{ef} \Delta c_i}{\sum_{j=1}^{n} \alpha_{ij} k_j} \qquad i = 1,2,3 \quad 1-\varepsilon,\ 2-\gamma',\ 3-\alpha \qquad (3)$$

In the method of successive approximations, e.g. for zone ε, the following dependencies are obtained:

$$k_\varepsilon^{(1)} = \frac{2\left(D_\varepsilon^c\right)_{ef} \Delta c_\varepsilon}{\sum_{j=1}^{3} \alpha_{ij} k_j^{(0)}}, \qquad k_\varepsilon^{(2)} = \frac{2\left(D_\varepsilon^c\right)_{ef} \Delta c_\varepsilon}{\sum_{j=1}^{3} \alpha_{ij} k_j^{(1)}}, \qquad (4)$$

From the system of equations obtained it is evident first of all that the growth parameters are the greater the greater the difference of concentration of the borders of phases Δc_i is, in accordance with the diagram of the phase equilibrium, as well as the greater the effective diffusion coefficient in a given phase – $(D^c_i)_{ef}$ is.

In the case of alloy ferrite, which includes alloy elements with a smaller enthalpy of creation of nitrides than iron, an important objective for mathematical modelling is, apart from formulae describing a change of the layer thickness, the determination of the profiles of nitrogen dissolved in the set of matrix and nitrogen bound in nitrides of alloy elements. Both these profiles form the distribution of hardnesses in the nitrided layer. As it is well-known, alloy elements which form nitrides are characterized by a different chemical affinity to nitrogen. If there is a strong affinity between these elements (Ti, V and Cr above 2.5 wt.-%) and nitrogen, then the front of the reaction of the creation of nitrides is ahead of the front of nitrogen diffusion [5-8]. Consequently, a sharp inter-phase border is created between the layer and the core. In this case, the analysis of the kinetics of the growth of the diffusion layer amounts to calculations of the growth of its thickness, while making use of the model developed for the processes of internal oxidation [9-11]. In this model, the diffusion of alloy elements is neglected and it is assumed that there is a constant concentration of nitrogen on the surface, in equilibrium with the nitriding atmosphere. With these assumptions, the thickness of the diffusion layer may be described with the following relationship:

$$\xi^2 = \frac{2}{r} \frac{[N]}{[X]} Dt \qquad (5)$$

where:

ξ – thickness of diffusion layer,

[N] – surface concentration of nitrogen in at. %,

[X] – concentration of alloy element in at. %,

r – ratio of number of nitrogen atoms to the atoms of alloy element in nitride,

D – nitrogen diffusion coefficient in ferrite,

t – process time.

In the presence of alloy elements, which are characterised by a less chemical affinity to nitrogen (e.g. Mn, Mo, Cr<2.5 wt.-%) as compared to those mentioned above, the interphase boundary the layer – the core becomes less sharp [12]. This is due to the advance of the front of the reaction of the creation of nitrides by the nitrogen diffusion front. This excludes the possibility of the application of the abovementioned model. In such cases, the growth kinetics for the diffusion layer is determined by the calculation of the profiles of nitrogen dissolved in alloy ferrite and nitrogen bound in the nitrides of nitride-creating elements. These profiles are obtained by solving standard Fick's laws, which include additionally terms which determine the order and invertibility of the reaction of a new phase creation. With the assumption that the concentration of the diffusing element (nitrogen) on the metal surface is constant and is in equilibrium with the surrounding medium, and while assuming additionally an independence of the nitrogen diffusion coefficient from its concentration, equations to describe the nitrogen concentration change and the change of the substitutional alloy element concentration, with which nitrogen reacts, can be presented in the following form [13]:

$$\frac{\partial c_1}{\partial t} = D_1 \frac{\partial^2 c_1}{\partial x^2} - k c_1 c_2 \qquad (6)$$

and

$$\frac{\partial c_2}{\partial t} = -k c_1 c_2 \qquad (7)$$

where:

c_1 – nitrogen concentration,

c_2 – concentration of the substitutional element in steel, with which diffusing nitrogen enters into a second order reaction with a constant k speed,

D_1 – nitrogen diffusion coefficient.

With the solution of the abovementioned system of equations it is assumed that a local thermodynamic balance is obtained very quickly. Accepting a solution with the use of Crank-Nicholoson's method [14], at each time-step, the profile of nitrogen bound in the nitrides of alloy elements is calculated (with a determined affinity of alloy elements to nitrogen being taken into account), as well as the profile of nitrogen dissolved in alloy ferrite, which takes part in further diffusion and the creation of new nitrides of alloy elements.

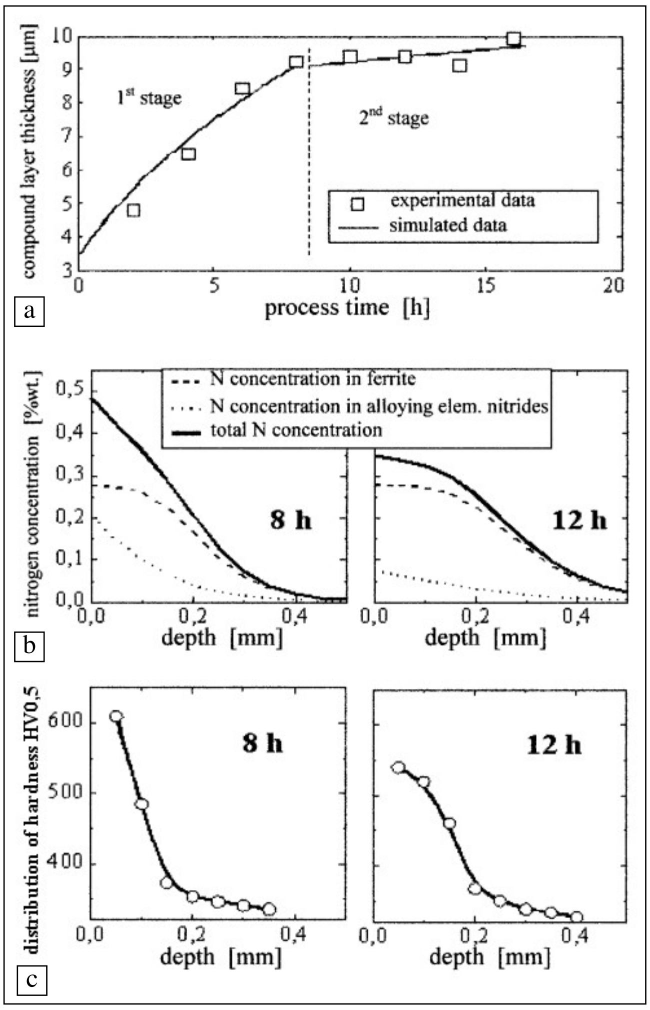

Fig. 1 – The thickness of compound layer as a function of nitriding time (a), profiles of nitrogen concentration in diffusion layer (b) and profiles of hardnesses (c). Parameters of the nitriding process – 1 stage: T = 753K, K_N = 20, t = 8h, 2-stage: T = 803K, K_N = 0,45, t = 8h.

Fig. 1 – Spessore dello strato composto in funzione di tempo dil nitrtrurazione (a), profili di concentrazione dell'azoto nello strato di diffusione (b) e profili della durezza (c). Parametri del processo di nitrurazione – 1a fase: T = 753K, K_N = 20, t = 8h, 2a fase: T = 803K, K_N = 0.45, t = 8h.

MODEL OF THE PROCESS

In order to precisely design the structure of the layer with the use of the experimental-theoretical model, it is necessary to consider in mathematical descriptions possible all the mechanisms which play a part in the growth of the nitrided layer in the subsequent stages of its growth. For this reason, a required element for the creation of models is a properly developed database, which comprises larger and larger sets of features which are characteristic of the layers, and which determine their various utility features.

In the case of the nitriding process, which has been applied in industry for many years now, there already exist vast sets of experimental data which connect the layer structure with their properties. However, it is necessary to conduct further complementary research to include both issues from the field of metal physics and materials engineering, and to indicate new mechanisms which form the growth kinetics of layers.

In the nitriding process model developed, the thesis [15,16] proven by the authors of the article was taken into consideration, i.e. that the phase composition of a layer of (carbo)nitrides has an influence upon the value of nitrogen concentration on the boundary *(carbo)nitrides layer / the diffusion layer*. In other words, in the model developed, it was considered that in the case of steel there are cases of departure from the quasi-equilibrium of nitrogen concentrations on the boundaries of the layer zones, contrary to the nitrided layer on iron. Moreover, it was considered that the evolution of the phase composition of the layer of (carbo)nitrides also has an influence on growth kinetics of the diffusion layer.

With the help of the model developed, simulations of the growth kinetics for the layer of iron (carbo)nitrides were conducted. Also, simulations of the distributions of the concentrations of nitrogen bound in the nitrides of alloy elements and nitrogen dissolved in the alloy ferrite were carried out. The profile constituting the total of these concentrations (cf. Fig. 1) was analysed, as well. The simulations covered a two-stage process, whose algorithm of parameter changes is provided in Fig. 1a.

A comparison of the experimental data concerning the changes of the thickness of the layer of iron (carbo)nitrides with the calculated courses (Fig. 1a) serves to indicate the fact that the model developed maps in the correct manner, the change of the thickness of this layer in function of the process parameters. Also, a comparison of the distributions of the total of nitrogen concentrations (Fig.1b), with the experimental data of hardness (Fig. 1c) serves to indicate an explicit similarity in their courses for the first and second stages of the process. This testifies that with the help of the model developed, one can also simulate in the correct manner the growth kinetics of the diffusion layer.

THE CONTROL SYSTEM TO THE NITRIDING PROCESS

In the new generation control system to the nitriding process it was foreseen, independently of the solutions obtained so far concerning the control of the nitriding atmosphere, a complementary interaction of the magnetic sensor and the model of the process. The magnetic sensor constitutes an element of the system mapping, directly in the process, the creation and growth of the layer. This system monitors the nucleation and the growth of the layer nitrided as a result of a change of the value of the magnetic permeability of the ferromagnetic diffusion layer, and the creation of iron nitrides being paramagnetic in the temperatures of the process.

In accordance with the idea of the functioning of the system, the model of the process plays a double role. As priority, on the basis of a simulative analysis of the layer growth with the assumption being made concerning its specific structure, this model serves to determine the most advantageous algorithm of the changes of the process parameters. The second important purpose of the process model for the automatics system is obtaining information, in the course of the process, concerning the development of the nitrided layer. In particular, the model facilitates a current presentation of the following:

- profiles of nitrogen dissolved in the alloy ferrite and the profiles of nitrogen bound in nitrides of alloy elements,
- a total profile of nitrogen in the alloy ferrite, mapping qualitatively the hardness profile, and
- concurrently with the magnetic sensor, it informs about the thickness of the layer of (carbo)nitrides.

These are very important data which characterise the layer nitrided, and which condition its usable properties. The model is the only tool with which one can obtain these. Only the thickness of the compound layer is concurrently determined by the system of the magnetic sensor. However, it is a well know fact that already at the mathematical description, which constitutes the basis of the experimental-theoretical model of the process, if the growth kinetics for the nitrided layer on iron, difficulties are met which are the result of e.g. lack of possibilities for an accurate and experimental determination of the values of coefficients of nitrogen transfer over the inter-phase boundary of gas – metal. The knowledge of the values of these coefficients is especially important on the initial stage of the process, in which the reaction of ammonia dissociation on this boundary constitutes a factor which determines the speed of the whole process. For this reason, mathematical descriptions usually do not cover the initial stage of the layer creation, as it is assumed that its duration is so short that the description of the kinetics may be limited to the layer which has already been formed, and which has constant and equilibrium concentrations on its boundaries. In the case of iron, neglecting this stage has practically speaking no impact upon the differences between the calculated and experimentally determined growth of the thickness of the diffusion layer, and only introduces a constant error between the real and calculated thicknesses of the zones of the nitrided layer, while on steel the conditions for the process on the stage of the layer creation are most often decisive for its further correct growth. In the light of the above, such a solution of the analysis of the layer growth kinetics, in which the initial stadium is neglected, results in discrepancies between the actual course of the layer creation and the one forecasted in accordance with the model.

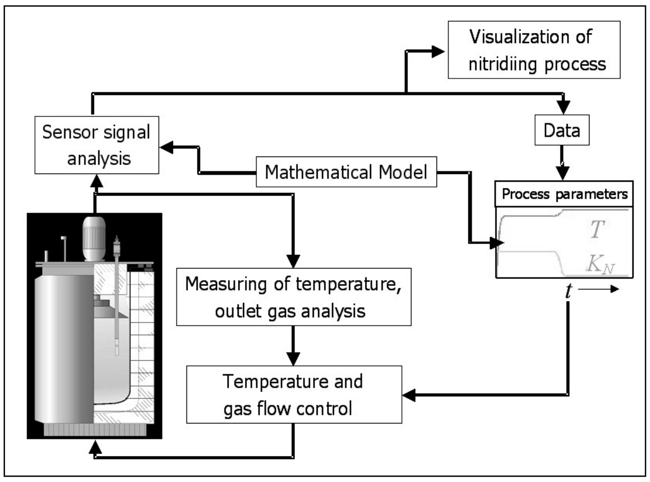

Fig. 2 – Block diagram of the automatics system of the nitriding process with the visualization system for the course of the layer growth.

Fig. 2 – Schema a blocchi del sistema automatico del processo di nitrurazione con il sistema di visualizzazione per il corso della crescita di strato.

In the automatics system discussed, the imperfections of the model are eliminated by means of a complementary interaction of the magnetic sensor system, which registers *sensor signal-time* courses mapping in a direct manner the growth process of the nitrided layer. The characteristic points of these courses correspond with e.g. obtaining the maximum surface hardness or the creation of a continuous (a few micrometers') layer of iron (carbo)nitrides. They constitute the initial conditions for the description by the model of further stages of the development of the layer nitrided. Fig. 2 presents a block diagram of the system, in which the magnetic sensor together with the process model constitute an independent module which facilitates a visualization of the course of the layer growth. This block diagram also includes a system of an automatic control of the nitriding process, in which the software of the block controlling the main process parameters, i.e. the temperature, the composition and intensity of the flow of the nitriding atmosphere serves to ensure that the kinetic *sensor signal-time* course is registered with a magnetic sensor, mapping the nitrided layer growth, is compliant with the reference course.

In order to present the visualisation system in a detailed manner (Fig. 3), a single-stage process of 4340 (AISI) steel nitriding was selected and conducted in the temperature of 833K and at the nitriding potential $K_N = 1.19$.

Article [17] presents an analysis of the course of a layer creation on the example of iron nitriding, which was registered with a magnetic sensor. The kinetic *sensor signal-time* courses obtained in this case should be treated as model courses, which constitute a reference point for the interpretation of courses registered during nitriding of steel. Every steel owing to its specific chemical composition and phase structure is characterized by an individual susceptibility of magnetic properties to changes being the result of the creation of the nitrided layer. Also, processes accompanying the layer development, such as e.g. the dispersion of the nitrides of alloy elements being created or their coagulation, which have an influence, among other things, on the stress state in the diffusion layer. Consequently, the form of the *sensor signal-time* courses depend on the features characteristic of a given steel. Of course, the mapping proven:

increase /decrease of compressive stresses ⇒ decrease / increase of magnetic permeability ⇒ decrease / increase of the sensor signal

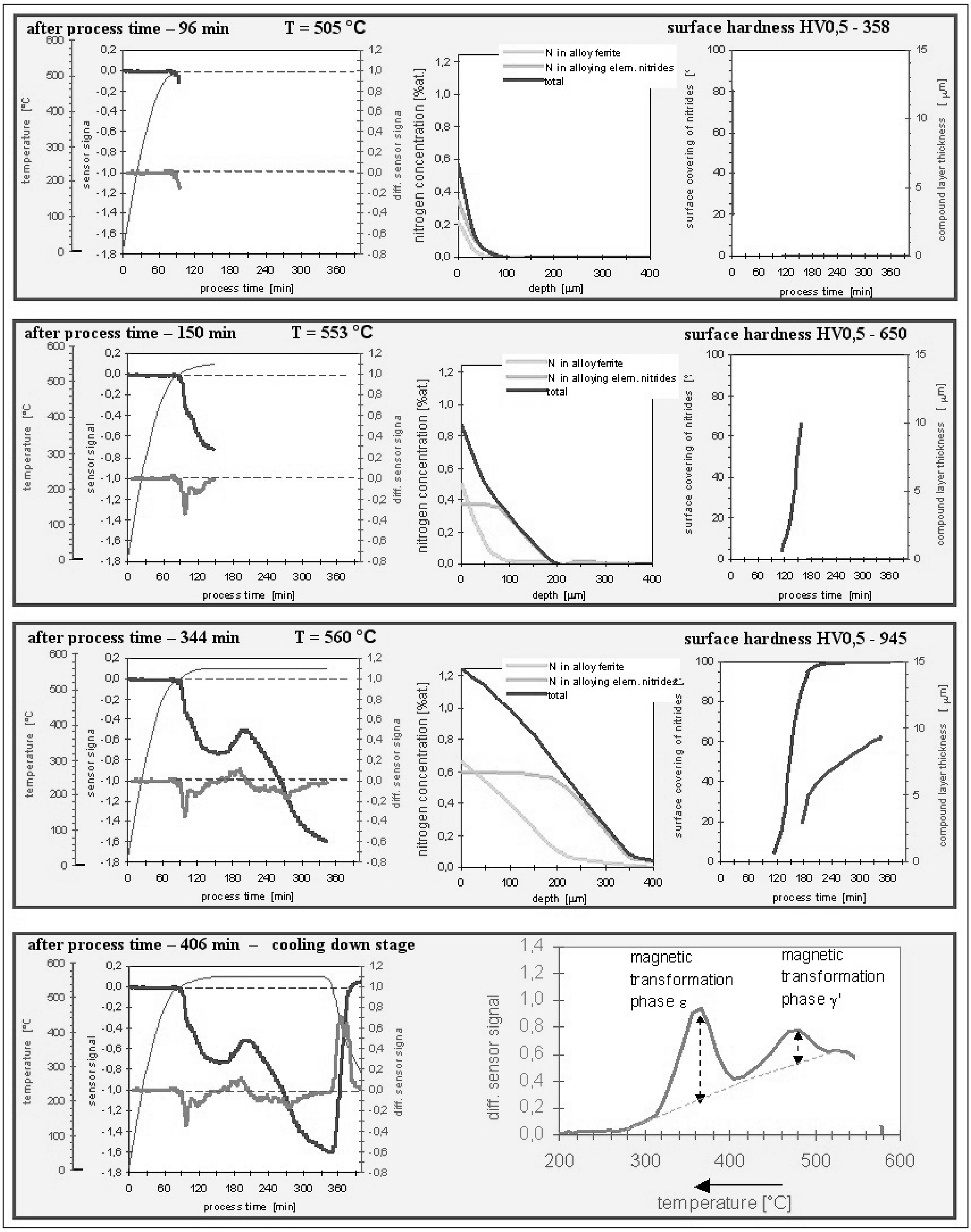

Fig. 3 – Presentation of the work of the visualizing system for the nitrided layer growth on the example of nitriding 4340 (AISI) steel.

Fig. 3 – Presentazione del lavoro del sistema di visualizzazione dello sviluppo dello strato nitrurato sull'esempio di nitrurazione dell'acciaio 4340 (AISI).

is correct for every ferromagnetic steel, yet in every case it exerts a different influence on the form of the course registered by the magnetic sensor.

One of the problems which occur during the control of industrial nitriding processes with a magnetic sensor is the so-called temperature background. It is an influence of changes of the electromagnetic properties of a steel sample on the registered signal of the sensor, produced by temperature changes, which occurs while the furnace is being heated to the process temperature, and during the execution of a few-stage process the moment one process stage goes to another. The temperature background is an individual feature of every steel and should be subtracted from the indications of the sensor by the proper software of the system. As a result, while the furnace is being heated till the moment the layer starts to create, which initiates the start of ammonia dissociation (ca. 623K), the signal registered by the sensor does not change (Fig. 3). Then, atom nitrogen generated as a result of ammonia dissociation, diffuses to the interior of the sample and by generating stresses in it, causes a reduction of the sensor signal. Together with the registration of the *sensor signal-time* courses, the software of the sensor system facilitates a presentation of its differential signal, whose extreme points indicate the occurrence of characteristic layer creation stages, like e.g. the beginning of the creation of surface iron nitride phases (Fig. 3). This is documented by an X-ray analysis of nitrided samples together with the sample located in the sensor and samples individually taken out from the retord, in characteristic moments of the process evident from the indications of the sensor [17]. On the basis of the differential course, one can also obtain information on the phase composition and the nitride layer created in the process and located by the surface. As it is know, [18,19] ε and γ' phases, which are paramagnetic in the temperatures of the process, are distinguished by a different Curie temperature, i.e. the magnetic transformation point - γ' phase goes into a ferromagnetic state in the temperature of 763K, and ε phase in the range of temperatures of 303-593K. For this reason, the course of the sensor signal, registered during the cooling down stage, has two maximum values being the result of magnetic transformations of the abovementioned phases. The area limited by these maximum values is proportional to the volume fraction of ε and γ' phases in the layer of nitrides.

Together with the indications of the sensor, which map the growth of the nitrided layer, calculations are conducted with the help of the model of the process, which represent the development of profiles of nitrogen dissolved in the alloy ferrite, and of nitrogen bound in the nitrides of alloy elements, the degree to which the surface is covered with iron (carbo)nitrides, and in the further part of the process, informing on the changes of the thickness of the (carbo)nitride layer depending of the process time.

SUMMARY

The model of the nitriding process presented in this paper will serve to determine the procedures of a comprehensive simulation of the process. It covers the growth kinetics of mono-phase zones in the nitrided layer on iron and low-carbon steels, as well as the calculation of nitrogen profiles in the diffusion layer on alloy steels. In particular, the model proposed, describing the growth of individual zones of the nitrided layer in the conditions of a quasi-equilibrium, is a simple and convenient tool facilitating both the determination of the nitrogen diffusion coefficients in mono-phase layer zones, as well as forecasting their growth in the function of the process time and the nitrogen potential. In the case of a steel including alloy elements with a smaller enthalpy of the generation of nitrides than iron, the model facilitates a calculation of nitrogen concentrations, which constitute a starting point for the development of procedures enabling forecasting of hardness profiles on the basis of the process parameters assumed and the chemical composition of steel.

The system of an automatic control of the process presented in the paper constitutes an innovative and unique solution. In the new control system, independently of the so-far existing solutions concerning the control of the nitriding atmosphere, interactions of the magnetic sensor system and the process model have been foreseen. The magnetic sensor is a component of the system which maps directly in the process the creation and growth of the layer. In turn, the model serves to determine the most advantageous algorithm of the changes of the process parameters, and to provide complementary to the indications of the magnetic sensor, information concerning the growth of the nitrided layer.

The issues analysed in this paper and the results obtained constitute elements of the construction of modern and effective control systems, in which artificial intelligence elements may be applied. These systems make it possible to face new challenges connected with the applications of the nitriding process.

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ABSTRACT

CONTROLLO DI SISTEMA INTELLIGENTE PER IL PROCESSO DI NITRURAZIONE A GAS

Parole chiave:
modellazione, processi, trattamenti termici, controlli

Nel presente lavoro vengono presentate le soluzioni dei seguenti oggetti di ricerca:

- *sono state definite dipendenze fra i parametri di processo e struttura degli strati, al lo scopo di sviluppare il software per il sistema di controllo di processo, con l'intento di ottenere una struttura con strato complessa e una cinetica ottimale di creazione e crescita,*
- *sono stati sviluppati i presupposti per il sistema di controllo di processo della nitrurazione a gas sulla base di una cooperazione complementare del modello matematico e delle indicazioni del sensore magnetico che registra la nucleazione e la crescita dello strato.*

Questi aspetti contengono due direzioni complementari verso la costruzione di sistemi di controllo intelligenti. Il primo consiste nello sviluppo di database innovativi, sistemi specializzati per aiutare l'operatore nelle decisioni frelative alla scelta di cambiamenti definiti dei parametri di processo. Ciò è collegato possibilmente alla conoscenza di tutti i fattori e meccanismi che influiscono sui risultati del processo e che permettono di progettare un algoritmo di cambiamenti dei parametri di processo che potrebbero garantire la cinetica ottimale della crescita dello strato e della relativa struttura necessaria. L'altro utilizza sensori specialmente costruiti, posti direttamente all'interno dei processi che reagiscono alla crescita degli strati e della loro struttura.