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METHOD OF SIMULATION OF THREE-COMPONENT ATMOSPHERES IN THE CONTROL SYSTEM OF THE GAS NITRIDING PROCESSES

Key-words

Simulation of gas nitriding processes, nitrided layer, nitriding potential, heuristic model, analytical model, database, knowledge base, evolutionary algorithms.

Summary

The article presents the application of numerical methods for the simulation (prediction) of nitriding potential changes and a discharge of the three-component nitriding atmosphere. Models and algorithms used for simulations have been also described. Development trends of developed simulation methods in the context of their application in designing and controlling industrial gas nitriding systems have also been described.

Introduction

Gas nitriding plays an important role in the group of technologies of thermo-chemical treatment and hybrid technologies. In the case of complex processes, such as thermo-chemical processes, the following important research and technical problems are considered:

- The correlation of the dynamic characteristics of the process temperature and nitriding potential for various types of steel and different systems of heat capacities include charge mass-furnace-heating space between the furnace and heating sections.
- The selection of appropriate nitriding potential and atmosphere flow rate are important, because nitriding atmosphere composition is important for the kinetics of layer growth, particularly when the gas nitriding process is an element of duplex or hybrid treatment.
- The stabilisation of temperature within the process duration should be included.
- One should take into account the synergism of physico-chemical phenomena occurring in the process.
- Complicated technical conditions of in-situ measurements enabling to control on the basis of results of the process states;
- It is necessity to fulfil industrial requirements in the scope of remote control (and regulations when needed) of the course of the nitriding process and the diagnostics of emergency states.
- The optimisation of controlling, including technical and economical aspects should be considered. Efficient predictive models for controlling should be developed, which are essential because of thermal inertia in controlling of the retort temperature and delays in reaching the preset nitriding potential.

The work on solving above problems is now in progress, because of the significance of the gas nitriding. The importance of gas nitriding in industrial applications is proved by the fact that, for example, in Canada and the USA, which belong to the group of the most industrialised and innovative countries in the world, the gas nitriding technologies constituted 76%, 69%, 58% of the nitrogen-carbonising, carbon nitriding and nitriding technologies, respectively [2]. The popularity of the gas nitriding technology results from the following advantages:

- The relatively low temperature of the process in comparison with other methods of layer constitution;
- The possibility of nitriding elements ready to use while retaining sizes and shapes (no deformations and no dimensional modifications after the nitriding process);
- The possibility of the process automation;
- The material-efficiency of gas nitriding processes, and,
- The possibilities to obtain layers with the enhanced fatigue strength, corrosion resistance, and wear resistance.

The service sector of thermal treatment in Poland, parallel to other adaptation processes of Polish economy and technology to the economy of the European Union and the North America countries, should develop quickly. This statement is justified by predictions based on the increasing number of new constructions of furnaces and systems to control the gas nitriding processes, and the number of patents (Fig.1).

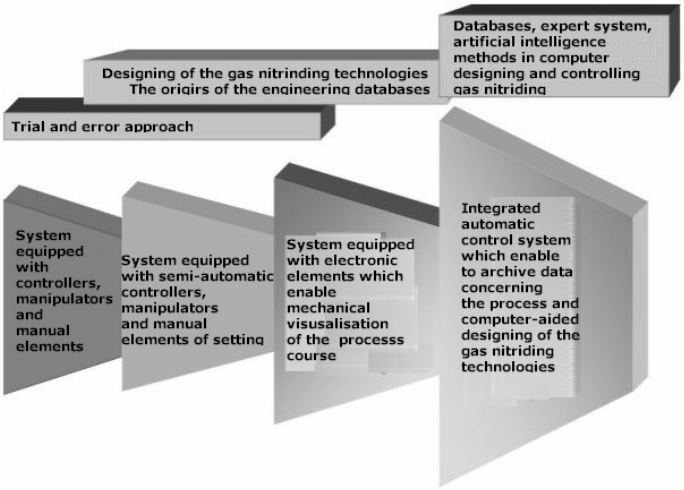

Fig. 1. The development of control systems for gas nitriding processes

Presented premises determined the origin of the research conducted by the authors. The main aim of the research was to develop and create the integrated and modular system of gas nitriding characterised by innovative solutions in the area of technology designing, and controlling and monitoring the realisation of the gas nitriding processes (Fig. 2).

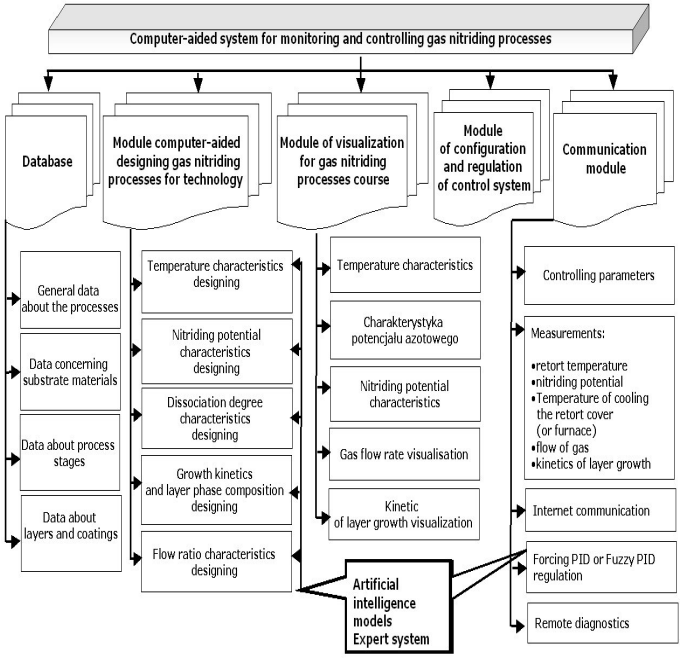

Fig. 2. The module structure of the gas nitriding system

An accurate control of parameters and characteristics of nitriding atmosphere is a necessary condition for using the gas nitriding in large-scale industrial applications. Such precise control is essential to obtain physico-chemical and maintenance properties of manufactured materials, and to ensure the repeatability and optimisation of the processes.

Fulfilling the mentioned condition, except for using up-to-date subassemblies of automation complying with ATEX directives, is also determined by efficient computer-aided designing of gas nitriding processes on the basis of the criterion set which is defined within the designing process [4,12], collected empirical data [9], and information provided by computer simulations.

1. The structure of system and the simulation concept

The system consists of the following modules:

- A database module, for collecting factual data concerning realised processes, and data acquired on-line within the process duration is needed [9] (Collected data is used to generate knowledge databases for system self-learning and for the inference about the properties of nitric layers or carbon nitride on the basis of data about the process milieu and substrate material.);
- A module for visualisation of the process milieu characteristics, enabling not only monitoring and analysing of the process course, but also requiring the determination of the values of parameters that control a process;
- A module of the system settings and definitions of global variables that determine the configuration of individual modules of the system;
- A module for communication among individual control systems, meter circuits and the computer software (Except for operational tasks concerning the data transmission among devices in the system, there have been implemented procedures enabling the assessment of correctness of system running, remote access to system database sources through the Internet and procedures that use data communications technologies for alarming about breakdowns.);
- A module is also developed to facilitate a computer-aided design of the technologies of the gas nitriding processes is equipped with, among other things, expert system, libraries of models and computer procedures for projecting (for instance ammonia expenditure) and the simulation of the following dynamic characteristics: temperature, nitride potential, dissociation, kinetics growth and phase composition of layers formed.

The conception of a modular and distributed structure of the system, using programmable logic controllers PLC, control desks, automatic protection against breakdowns, and computer software, allows the system to be adapted for various industrial and laboratory configurations of gas nitriding installation.

Super-ordinated computer software has been equipped with an application to simulate dynamic characteristics of the process milieu. The main aim of the

application is to increase the pace of designing, modifying and introducing the gas nitriding technologies as a result of computer analysis of many potentially possible variants of the process realisation. The-state-of-the-art achievements of virtual engineering have been applied, in particular triad conception [3]: theory-experiment-simulation (Fig. 3). A constructive utilisation of the simulation in case of many processes, especially multi-stage processes, where on the individual stage of the process the temperature is changed, enables one to avoid time-consuming and often costly tests necessary to develop an efficient technology of gas nitriding process.

Indirectly, the simulation, through coupling theory -> process, process -> simulation, modelling -> theory supplies information for the basic research, verifying the correctness of theories used, which is particularly essential in processes of thermo-chemical treatments. In the case of gas nitriding processes, a phenomenological model of the process is known, whereas mathematical equations which enable a quantitative description of phenomena occurring in the gas atmosphere on the boundary gas-metal and in the material nitrided are still being investigated [6].

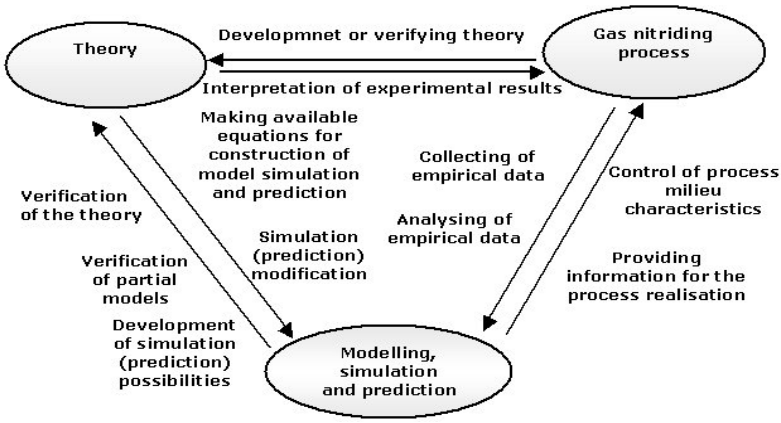

Fig. 3. The conception of computer simulations adapted to design the gas nitriding processes [3]

Difficulties concerning the development adequate models of gas nitriding processes result from synergistic interactions among simultaneously occurring physical phenomena, for instance, ammonia transport in the gaseous phase, chemisorption by a surface of a solid body, the desorption of ammonia dissociation products, the transport of nitrogen atoms through the surface of a metal, and their diffusion to the inside of nitrided material. Therefore, it is necessary to apply simplified models [5], and the characteristic simulation of the process milieu is used mainly for technological requirements, but together with deduction, it enables one to understand comprehensively and epistemologically the course of the gas nitriding process.

The designed system uses models to simulate the following characteristics of the process milieu as follows:
- The process temperature in the time function,
- The nitriding potential in the time function,
- The dissociation degree in the time function, and
- The growth kinetics of the layer and changes of concentrations and nitrogen concentration profiles on the phase boundaries.

The nitriding atmosphere, one of the main elements of gas nitriding process, has a significant influence, and temperature, on the kinetics of nitriding process and obtaining physico-chemical and maintenance properties of nitrided layers (particularly their thickness and phase composition) is programmed by a technologist. Concerning the importance of precise controlling of nitriding atmosphere in nitriding process, which is necessary to obtain programmed properties of substrate, the application enables one to simulate a nitriding potential N_p, degree of ammonia dissociation α, and atmospheric composition and expenditure at furnace chamber inlet, but for atmospheric composition simulations, models for three-component atmospheres containing nitrogen, ammonia and dissociated ammonia have been applied. This choice is justified by the fact that this kind of atmosphere combines features of two-component atmospheres nitrogen + ammonia, ammonia + dissociated ammonia, and it might be a reference to atmospheres containing only ammonia [10].

2. Assumptions for models to simulate characteristics of three-component process atmospheres

To develop simulation models the following simplified assumptions has been formulated:

1. The nitriding process depends on thermodynamics and kinetics of chemical reactions that are present while nitrided layer is constituted. Thermodynamic relationships enable one to determine a local equilibrium between nitrogen on the layer surface and nitriding atmosphere, and between nitrogen in α, γ', ε phases and on phase boundaries α/γ', γ'/ε. Equilibrium system *Fe-N* provides information concerning nitrogen equilibrium concentrations in α, γ' and ε phases on boundaries: $\alpha/\alpha+\gamma'$, $\alpha+\gamma'/\gamma'$, $\gamma'/\gamma'+\varepsilon$ and $\gamma'+\varepsilon/\varepsilon$ [7,8,11].
2. The results of the simulation are as follows: characteristics of nitriding potential Np, atmosphere flow rate and volume fractions:
   [1]a - ammonia NH_3,
   [2]b - nitrogen N_2,
   [3]c - dissociated NH_{3zd}.
3. Characteristics of nitriding potential play a significant part in gas nitriding processes. The way that changes of the potential in time and temperature functions of the process are performed determines the structure and growth

kinetics of nitrided layer [6,8]. With appropriately high value of the nitriding potential in the nitriding:

- iron – nitrides layer, is constituted through nucleation in the first γ' phase sequence, and then ε phase on the surface of γ' phase. In such a layer in which the structure is consistent with the Lehrer model and *Fe-N* equilibrium diagram, quasi equilibrium of ammonia concentrations, dependent only on the process temperature, is established on its intrinsic phase boundary. As a result, nitriding potential after exceeding a threshold value influences only mutual relations between the thickness zones of nitrides layer;
- steel – between diffusion nitrided layer and near surface layer of iron nitride, there is quasi equilibrium of nitride concentration, after the near surface, the growth kinetics of diffusion nitrided layer depends only on the process temperature;

The growth kinetics of diffusion nitrided layer depends on the following:

- The concentration of alloy elements and their affinity of nitrogen;
- The process temperature influencing the value of nitrogen concentration interstitially dissolved in the iron lattice.

As a result, for a particular grade of steel, when applying the appropriately high value of the nitriding potential that guarantees the formulation of near surface nitrides layer, the growth kinetics of diffusion layer should be dependent only on the process temperature [5,6].

Therefore, we can use a simplified a phenomenological model of the process of nitrides layer constitution that enables the application of equations to calculate equilibrium concentration of nitrogen, formulated for iron both on the basis of Lehrer model, and modified Lehrer model, for which data has been derived from empirical research [6]. The simplifying assumption concerning omitting the influence of the active surface of the retort and charge on the speed of dissociation has been made.

4. Information concerning the model[1] $T_i=f(t_i)$ or the values of parameters that characterise layer properties are defined by a technologist or are automatically acquired from the expert system designed for inferring the properties of the nitrided layer on the basis of the substrate material parameters and parameters of nitriding atmosphere.
5. For mono- and multi-stage processes temperature change range in the model $T_i=f(t_i)$ is from 350°C to 590C°..
6. The diluting atmosphere has the following initial stage a_0NH_3-b_0N_2-c_0NH_{3zd}, and after diluting a_1NH_3-b_1N_2-c_1NH_{3zd}. Whereas, in a general case, a diluting atmosphere is a three-component atmosphere a_rNH_3-b_rN_2-c_rNH_{3zd}. Within diluting the atmosphere takes values $a(t_i)NH_3$-$b(t_i)N_2$-$c(t_i)NH_{3zd}$ while the following equations are fulfilled:

[1] temperature model: value of temperature T_i in t_i moments.

– $a_0 + b_0 + c_0 = 1$,
– $a_1 + b_1 + c_1 = 1$,
– $a(t_i)+b(t_i)+c(t_i)=1$.

7. Characteristics of dissociation degree $\alpha_i=f_\alpha(t_i)$ might be considered in simulations that expresses the proportion of volume fraction of a part of the ammonia in the diluting atmosphere, which has been dissociated to the volume of diluting atmosphere.
8. A evolutionary algorithm will be used to optimise the changes in atmospheric composition.

An application of the discrete temperature model in the time function $T_i=f(t_i)$ as a key input information was justified by the following premises:

– Small temperature changes within the nitriding process might have a significant influence on changes of nitriding potential and atmospheric composition.
– The optimisation of the total process duration, especially in industrial conditions, should take into account not only nitriding time at a fix temperature, but also the time of heating and cooling the charge.
– The possibility of applying thermo-dynamic relationships to determine the values of nitriding potential[2] and ammonia fraction in nitriding atmosphere is from the temperature with the various values of nitrogen C_N equilibrium concentrations.

The knowledge of dynamic (kinetic) characteristics of changes in the nitriding atmosphere is significant for projecting systems to control nitriding processes. It enables the determination of time-constants necessary to control the process and to predict the values of various types of technological parameters, e.g. flow rate and inlet atmospheric composition, which influences the properties of nitriding atmosphere. Dynamic characteristics might also be used to develop atmosphere characteristics for a steady state, i.e. when the lack of changes in temperature or degree of dissociation occurs.

3. Models applied in the simulation of characteristics of three-component atmospheres

In the simulation of characteristics of three-component atmospheres the following mathematical models have been used:

1. **Models** to calculate the equilibrium concentrations C_N formulated on the basis of thermodynamics and kinetics of chemical reactions [11]:
   – Equilibrium potentials in the temperature function describe the following equations:

[2] including boundary potentials $Np_{\alpha/\gamma'}$ i $Np_{\gamma'/\varepsilon}$

$$N_p^{\alpha/\gamma'} = \exp\left(\frac{4556}{T} - 12{,}88\right)\left[Pa^{-0.5}\right] \tag{1}$$

$$N_p^{\gamma'/\varepsilon} = \exp\left(\left(\frac{60536}{T} - 56{,}85\right)^{0.5} - 9{,}63\right)\left[Pa^{-0.5}\right] \tag{2}$$

– Relations among nitrogen concentration in α, γ' phases and ε and potential N_p, and temperature T, and boundary courses α/γ' and γ'/ε in the N_p and T function have been calculated with a modified equilibrium system $NH_3/H_2^{1,5}$ – (Fe,N) (the Lehrer model) [11]. It should be emphasised that the knowledge of nitrogen concentration dependencies in separate phases in the range of their stability in the Lehrer model is particularly useful in projecting of nitriding processes that enable manufacturing of precise nitrided layers.

2. **Model** of nitride potential in the ammonia dissociation degree function (α), ammonia content (a), nitrogen (b) and dissociated ammonia (c) in the three-component inlet atmosphere has a composition aNH_3-bN_2-cNH_{3zd}:

$$N_p(\alpha,a,b,c) = \frac{(0{,}75\cdot\alpha - 0{,}375\cdot c\cdot\alpha + 0{,}75\cdot c)\cdot(1+a) - 1{,}5\cdot(1-b-c) - 0{,}75\cdot c}{(0{,}75\cdot c - 1{,}5)\cdot(0{,}75\cdot\alpha - 0{,}375\cdot c\cdot\alpha + 0{,}75\cdot c)^{1.5}} \tag{3}$$

$a+b+c=1;\ 0<a\le 1;\ \alpha>0$

where: α – ammonia dissociation degree in the nitriding atmosphere expressed as a proportion of a doubled volume of a part of the ammonia s in the inlet atmosphere, which was dissociated within nitriding process, to the volume of the outlet atmosphere $\alpha=2s/(1+s)$.

In the general case, a, b, c and α are the time function.

3. **Models** of change in the volume fraction of ammonia, nitrogen and dissociated ammonia in the temperature, volume of furnace retort, atmosphere flow rate and time function are described by the following equation:

$$a(Q,t) = a_o\cdot\exp\left(\frac{-e\cdot Q\cdot t}{V_p}\right) + a_1\cdot\left(1-\exp\left(\frac{-e\cdot Q\cdot t}{V_p}\right)\right) \tag{4}$$

$$b(Q,t) = b_o\cdot\exp\left(\frac{-e\cdot Q\cdot t}{V_p}\right) + b_1\cdot\left(1-\exp\left(\frac{-e\cdot Q\cdot t}{V_p}\right)\right) \tag{5}$$

$$c(Q,t) = c_o\cdot\exp\left(\frac{-e\cdot Q\cdot t}{V_p}\right) + c_1\cdot\left(1-\exp\left(\frac{-e\cdot Q\cdot t}{V_p}\right)\right) \tag{6}$$

where: $a(Q,t)$, $b(Q,t)$, $c(Q,t)$ – correspondingly, volume fraction of ammonia, nitrogen, dissociated ammonia after t time,
a_o, b_o, c_o – correspondingly volume fraction of ammonia, nitrogen, dissociated ammonia in the initial atmosphere,
a_1, b_1, c_1 – correspondingly volume fraction of ammonia, nitrogen, dissociated ammonia in the diluting atmosphere,
Q – a total flow rate of the diluting atmosphere,
V_P – furnace volume,
t – time,
e – constant 2,71828 (determined experimentally).

4. **Evolutionary** model [1] for optimising the diluting atmosphere. The model uses [1]:
   - A floating-point representation of chromosomes, which are vectors containing the volume fraction of nitrogen, ammonia, dissociated ammonia and the degree of dissociation is used;
   - A selection operator that works in the following way: After the assessment of individuals (chromosomes) is completed, sorting the population is done. In addition, the individuals with the highest value of fitness function are placed at the beginning. Half of the best-suited individuals are chosen from the population; whereas, the other half is generated with mutation and recombination operators;
   - A non-uniform mutation operator is used, and the probability of a gene mutation equals 0.5, while the value of the mutation is situated between 0 and 0.999;
   - A recombination operator (crossing-over) – the probability of recombination is determined on the basis of average value and standard deviation, and standard deviation of the population. Two individuals take part in recombination; however, only one element of the chromosome undergoes changes, for example, the volume fraction of nitrogen in the diluting atmosphere; and,
   - An original function of individual assessment is created, formulated on the ground of relation (3) – (6).

Formulated assumptions, mathematical models and evolutionary model constitute the base for building the application to simulate characteristics of three-component process atmospheres.

Required data to simulate characteristics of three-component process atmospheres are as follows: the temperature model, the volume fraction of ammonia and dissociated ammonia in inlet atmosphere, the value of equilibrium concentration or value of nitrogen potential of nitriding atmosphere, the degree of ammonia dissociation, the volume of furnace retort and limitations concerning minimal and maximal gas flow rate in the inlet atmosphere. Depending on the simulation variant, additional information may contain a model of ammonia dissociation and a model of nitrogen potential.

The first stage of the simulation consists in searching the database on account of criterion that is a model of the process temperature. The temperature or similar[3] model found in the database enables the usage of other process atmosphere characteristic, out of available and obtained information concerning the characteristics of three-component atmospheres for the process. An alternative to database search is to define the model in analytical form or as a result of interpolation after defining the sequence of value pairs: time, temperature.

In the next stage of the simulation, the temperature value T_i for each time value t_i is determined. These values are input data for calculating the dynamic characteristics of nitrogen potential in the temperature and time function on the basis of relations resulted from the Lehrer model or linear approximation in the case, when nitrogen potential model has been defined. Applying relations (3) – (6), for each t_i values of volume friction ammonia, dissociated ammonia and nitrogen of inlet diluting atmosphere, in nitrogen potential function and degree of ammonia dissociation are calculated.

The last simulation stage consists in determining the characteristics of inlet atmosphere flow rate. There are certain limitations concerning minimal and maximal values of the flow rate characteristics while determining the characteristics.

The evolutionary model might also be used for the simulation; mean square error is a criterion to stop evolutionary calculations; the error is calculated on the basis of data taken from the temperature model and temperature values determined by the (1) – (6) relations.

The evolutionary algorithm enables the optimisation of changes in diluting atmosphere, considering the criteria of minimal change in volume fraction ammonia or dissociated ammonia in the inlet atmosphere.

4. The application to the characteristics simulation of three-component process atmospheres

The computer software is an exemplification of the application to the characteristic simulation of three-component process atmospheres (Fig. 4). Additional option (Fig. 5) implemented in the software is the possibility of an initial and final selection (after the dilution) of the atmospheric composition and composition of diluting atmosphere.

[3] resemblance is determined on the basis of mean square error and basic statistical parameters: mean and standard deviation.

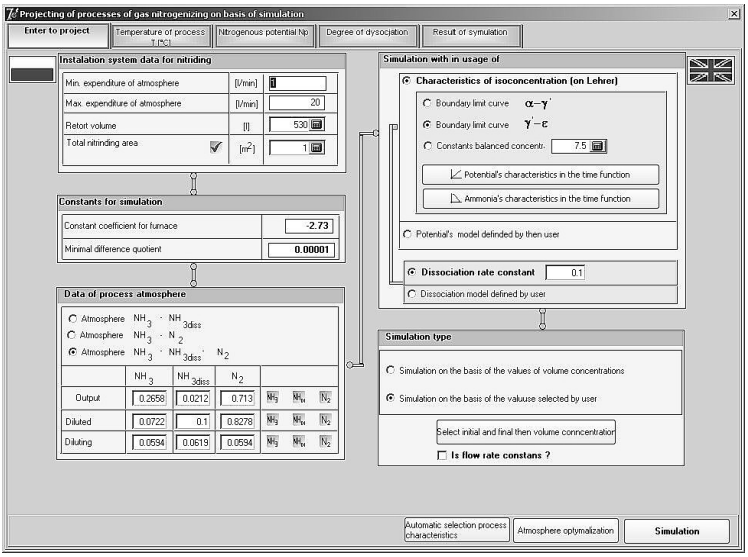

Fig. 4. User interface of the application to the characteristics simulation of three-component atmospheres

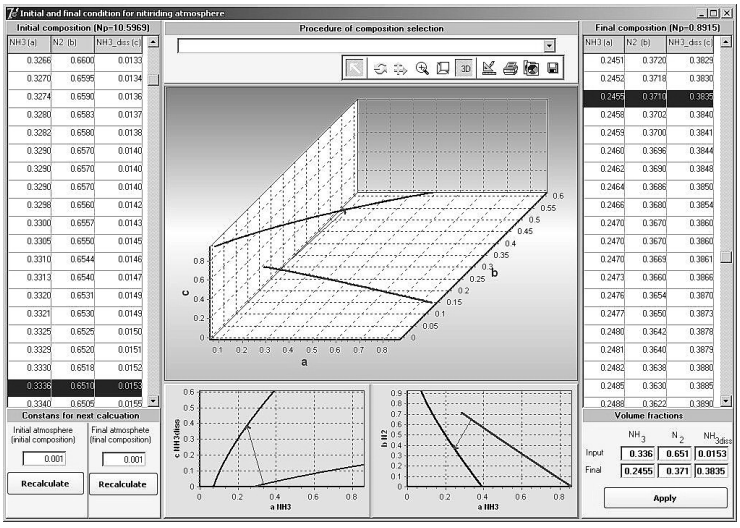

Fig. 5. User interface for a choice of composition of initial and final atmosphere

Results of the simulation with parameters presented in Figure 7 have been shown in Figures 6, 7, and 8.

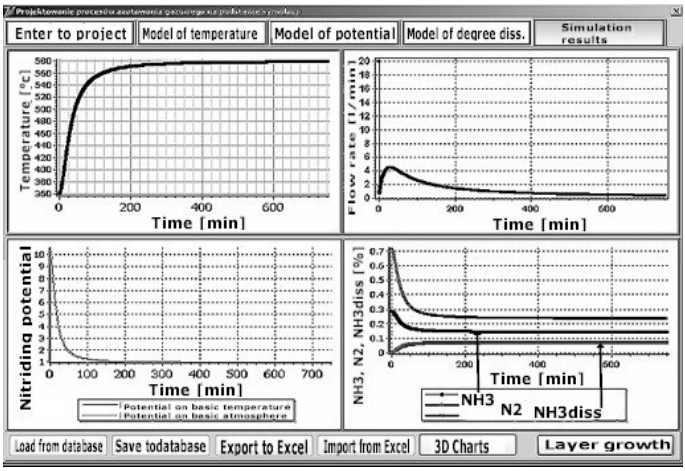

Fig. 6. Simulation results of the characteristic of three-component atmosphere and atmosphere flow rate

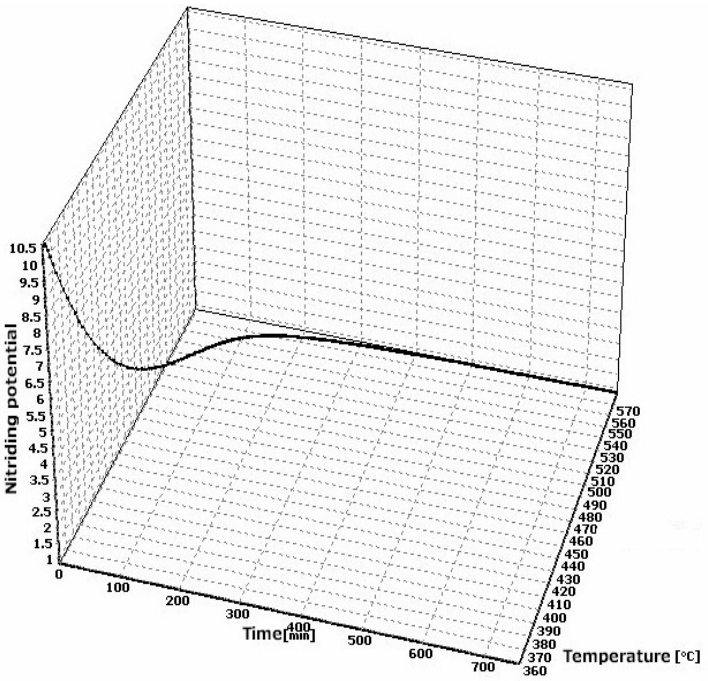

Fig. 7. 3D visualisation of the simulation result of three-component atmosphere: time, temperature, nitriding potential

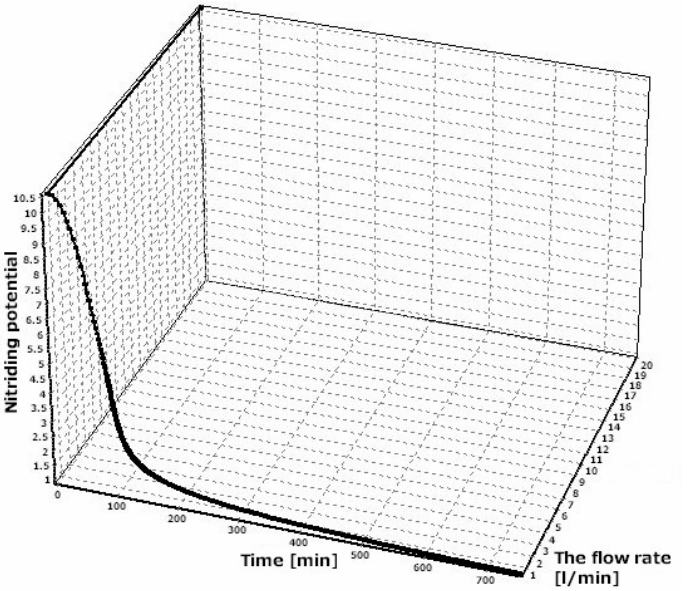

Fig. 8. 3D visualisation of the simulation result of three-component atmosphere: time, nitriding potential, flow rate atmosphere

The software was developed with the Delphi package. Characteristics obtained within the simulation were applied for gas nitriding processes of tools and machine parts.

Conclusion

1. The application of models and developed simulation methods of gas nitriding processes enable to track efficiently the various variants of process realisation.
2. The use of evolutionary algorithm enabled the determination of the optimal changes of three-component atmosphere.
3. The use of developed methods integrated with databases and the export system ensures efficient designing of gas nitriding processes.
4. The applied user interface for developed simulation methods makes available the presentation of input information and simulation results in the form of diagrams, in particular.
5. Drawings of limited curves of the course of dissociation degree can be created, which correspond to boundary nitriding potentials of nitriding atmosphere $Np_{\alpha/\gamma'}$ and $Np_{\gamma'/\varepsilon}$ and dissociation for values of potentials, referring to specific concentrations of nitrogen in phases of nitrides with chosen atmospheric composition in the temperature function.

6. Relationships can be determined of ammonia volume fraction in the atmosphere NH_3-N_2-NH_{3zd} for the constant value or assumed model of ammonia dissociation in the time function, volume fractions of ammonia, dissociated ammonia and nitrogen corresponding to the following boundary potentials: $Np_{\alpha/\gamma'}$ and $Np_{\gamma'/\varepsilon}$ or equilibrium concentrations of phases α, γ', ε.
7. Three-dimensional presentations can be made of characteristics in a co-ordinate system: t-T-N_p, t-T-Q and volume fractions of ammonia, ammonia, dissociated ammonia in the function time and temperature function.
8. The simulation results provide an input information for simulation of the layer growth kinetics and nitrogen concentration profiles on phase boundaries α, γ', ε.
9. The simulation results may be exported to files accepted by Excel, which enables one to use them in other programmes or databases.
10. An experimental verification of developed simulation methods for characteristics of three-component atmospheres of gas nitriding process atmospheres confirmed their practical suitability.

To sum up, the possibilities of simulation constitute an efficient tool for computer-aided designing of the gas nitriding processes.

Within the continuation of research concerning the simulation of three-component process atmospheres, it has been planned to take into account in developed models the influence of the active surface (the internal surface of retort and the surface of charge) on the dissociation speed.

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Recenzent:
Leszek MAŁDZIŃSKI

Symulacja charakterystyk trójskładnikowych atmosfer procesowych w systemie sterowania procesami azotowania gazowego

Słowa kluczowe

Symulacja procesów, regulowane azotowanie gazowe, warstwa azotowana, potencjał azotowy, model heurystyczny, model analityczny, baza danych, baza wiedzy, logika rozmyta.

Streszczenie

W artykule przedstawiono zastosowanie metod numerycznych do symulacji zmian potencjału azotowego i wydatku trójskładnikowej atmosfery azotującej. Zaprezentowano wybrane modele i algorytmy oraz uzyskiwane z ich użyciem wyniki. Wskazano kierunki rozwoju opracowanych metod symulacyjnych w kontekście ich zastosowań w projektowaniu i sterowaniu przemysłowymi instalacjami azotowania gazowego.