

# Ionic Nitriding of Certain Parts Protected on Nonfunctional Surfaces With Special Paints. Electric Energy Consumed

Marius Bibu\*<sup>1</sup>

<sup>1</sup>Lucian Blaga University of Sibiu, Faculty of Engineering, Industrial Engineering and Management Department, Romania

**Abstract:** The experimental researches on the promotion of new technologies for the local protection of metallic parts against plasma nitriding, led to two types of special paints for protection in ionic nitriding, paints elaborated on the basis of copper lamellar powder in combination with magnesium oxide and carbon tetrachloride. In the created context, it was considered that the elaborated paints could be used not only for preventing the hardening during ionic nitriding of certain technological surfaces of the parts on which they are applied, but also for coating certain nonfunctional surfaces, their degasification taking such a long time. These nonfunctional areas could be: surfaces resulted from casting, fragments with macroirregularities, surfaces that contain slag, residues, soot, other oxides, impurities, etc. and are the cause for a very large number of transitions of the glow discharges in electric arcs. The use of special protecting paints for the plasma nitriding of the parts that present nonfunctional surfaces leads to a major reduction in the energy consumption. This paper presents the ways of determining the consumed electric energy on the basis of absorbed power in the case of ionic nitriding of certain parts protected on nonfunctional surfaces with special paints.

## 1 Introduction

When atoms of a foreign element are added to a solid state system, they cause an increase in the free energy. Since every system tends to reach its equilibrium state with the minimum energy, a transformation can occur and a new phase can be formed. One of the most fundamental processes by which a system attempts to reach a stable arrangement of its atoms is diffusion. These two phenomena, phase formation and diffusion, are of significant importance for the formation and growth of the nitride layer during ion nitriding.

The traditional methods of nitriding are based on deriving a source of nitrogen from a gas or compound. The two common sources of nitrogen are ammonia for gas nitriding, and cyanide to cyanate decomposition for salt bath nitriding. The gaseous method of nitriding is based on the simple decomposition of ammonia into its base components.

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\* Corresponding author: [marius.bibu@ulbsibiu.ro](mailto:marius.bibu@ulbsibiu.ro)

The elaboration of newer and more efficient technologies for the local protection of metallic parts against plasma nitriding laid the foundation for the production of two types of special protective paints based on sub-microscopical copper lamellas in combination with magnesium oxide. In the case of the first paint, (V-1), the aforementioned mixture is dispersed in a polystyrene varnish (polystyrene dissolved in carbon tetrachloride), and as concerns the second paint, (V-2), the mixture is dissolved only in carbon tetrachloride [1].

The experimental researches demonstrated that the behaviour of the special protective coats of paints was similar to that of the unprotected metallic surfaces. The glow discharge started a few seconds after the installation had been started, for the unprotected and protected surfaces simultaneously, the ionic bombardment being rapidly released (a few discharges in electric arc and minor scintillations occurred sporadically on both types of surfaces).

The special paints have been used as protection not only against nitrogen diffusion (i.e. hardening) during the ionic nitriding of certain surfaces of the parts, but also for coating certain unprocessed (nonfunctional) areas which require a long-term degassing.

These areas could be represented by: surfaces resulted from casting with hardened slag, residues, black oxide, areas with macroirregularities, oxidised areas, with impurities [1, 2].

## 2 Experimental Results

In order to be able to support more efficiently the aforementioned facts, three experiments have been conducted: EXP.1, EXP.2 and EXP.3, which consisted of running three ionic nitriding processes in a Klöckner-Ionon GmbH, S 750 x 4500 installation. The ionic nitriding operating conditions were:  $T = 550\text{ }^{\circ}\text{C}$ ,  $t = 12\text{ h}$ ,  $p = 2.5\text{ torr}$ , operating atmosphere 25%  $\text{N}_2 / 75\% \text{H}_2$ . These operating conditions were differently applied to three types of parts:

- completely grinded samples – EXP.1 –  $P_{m1}$  (metallic lustre surface);
- completely oxidised samples – EXP.2 –  $P_{t2}$  (surfaces resulted by casting with hardened slag and residues, black oxide, macroirregularities areas; oxidised areas, with impurities);
- completely paint-protected samples – EXP.3, –  $P_{p3}$  (initially surfaces resulted by casting with hardened slag and residues, black oxide, macroirregularities areas; oxidised areas, with impurities).

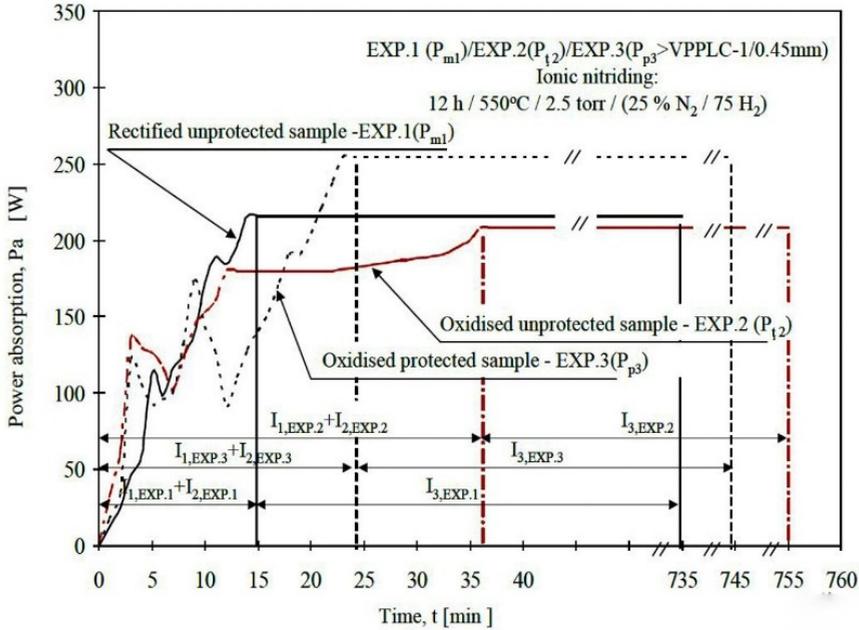
The specimens were made of steel 39CrAl6, of  $\varnothing 60 \times 10\text{ mm}$ , improved beforehand at 28...30 HRC. Regardless of the surface was finely grinded ( $R_a = 0.025\text{ mm}$ ) or oxidised, the unprotected samples were degreased before the process. The paint coats were brushed on the degreased surfaces, with drying intermediary times ( $\approx 10\text{ minutes}$ ).

During the first interval  $I_1$ , parts are degassed, cleaned and preheated, the gas ions bombard the surfaces of the parts removing impurities, oxides, etc., and preparing them for the ionic nitriding proper. Thus, by cathode disintegration, the ionic bombardment cleans the charge and its suspension attachment, phase in which the transitions of glow discharges in electric arcs are, usually, frequent. The gaseous fluid pressure within the working chamber slowly builds up, the glow discharge becomes slowly stable, the current voltage and strength step up, and therefore the temperature value increases, too. The third interval –  $I_3$ , is that of the ionic nitriding proper, which is running at constant temperature and pressure. In the last interval –  $I_4$ , the charge is cooling within the tank down to  $\approx 200^{\circ}\text{C}$ , and then is taken out and it cools at the ambient temperature [3, 4].

In figure 1 the variations of the consumed power time are comparatively presented - ( $P_{am}$ ,  $P_{at}$ ,  $P_{ap}$ ) function of temperatures ( $T_m$ ,  $T_t$ ,  $T_p$ ), discharge voltages ( $U_{dm}$ ,  $U_{at}$ ,  $U_{ap}$ ) and corresponding current densities ( $j_m$ ,  $j_t$ ,  $j_p$ ), for the three types of determinations EXP.1,

EXP.2 and EXP.3, regarding the intervals  $I_1$ ,  $I_2$  and  $I_3$  of the ionic nitriding cycles corresponding to the considered experiments [5].

By integrating the curves representing the power consumption time variation on the first two intervals  $I_1$  and  $I_2$  (for the heating up to the ionic nitriding steady running temperature, 550 °C, of the samples used in experiments EXP.1...EXP.3), the energies necessary for reaching the specified temperature were obtained.



**Fig. 1.** Power absorption time variation on intervals  $I_1$ ,  $I_2$ , and  $I_3$  during the plasma nitriding process of the protected and unprotected parts, for EXP.1, EXP.2 and EXP.3

The energy values were calculated as follows:

- unprotected surface (grinded,  $R_a = 0.025$  mm),  $P_{m1}$ :  $E_{EXP.1}^{I_1+I_2} = 12.5$  KJ;
- unprotected surface (surfaces with: macroirregularities, hardened slag, residues, black oxide, oxides, impurities etc.),  $P_{12}$ :  $E_{EXP.2}^{I_1+I_2} = 344.7$  KJ;
- protected surface (initially surfaces with: macroirregularities, hardened slag, residues, black oxide, oxides, impurities etc.),  $P_{p3}$ :  $E_{EXP.3}^{I_1+I_2} = 206.8$  KJ.

One can notice that in order to heat in glow discharge, at the ionic nitriding operating temperature (550 °C) the part with macroirregularities, residues, oxides or impurities ( $P_{12}$ ), although formerly degreased, the necessary quantity of energy is 2.6 times higher than that consumed for the same type of part but with grinded surface ( $P_{m1}$ ). Thus, the quantity of energy consumed for heating part  $P_{12}$  is 164% higher than that consumed for the grinded part  $P_{m1}$ , or:

$$E_{EXP.1}^{I_1+I_2} = 0.38 E_{EXP.2}^{I_1+I_2} \quad (1)$$

If the surface of the sample with macroirregularities, residues, oxides or impurities is protected by the special paints V-1 or V-2 against ionic nitriding (sample  $P_{p3}$ ), the quantity of energy necessary for heating it at the same plasma nitriding steady running temperature is 40% lower compared to the case in which it is unprotected (the energy consumed for heating sample  $P_{12}$  is 1.7 times higher than that consumed when the same part is protected -  $P_{p3}$ ):

$$E_{EXP.3}^{I_1+I_2} = 0.60E_{EXP.2}^{I_1+I_2} \quad (2)$$

The quantity of heating energy under operating conditions of sample P<sub>p3</sub> is 1.6 times higher (an increase of 37%) than that of the grinded sample (P<sub>m1</sub>) or:

$$E_{EXP.1}^{I_1+I_2} = 0.63E_{EXP.3}^{I_1+I_2} \quad (3)$$

Taking into account only the period of keeping it in the process (interval I<sub>3</sub> with a duration of 12 hours), the energies consumed in this phase in the case of samples P<sub>12</sub> and P<sub>p3</sub> are:

$$E_{EXP.2}^{I_3} = 9331 \text{ KJ} \quad (4)$$

$$E_{EXP.3}^{I_3} = 10973 \text{ KJ} \quad (5)$$

The quantity of energy necessary for maintaining the protected sample P<sub>p3</sub> at the steady running temperature (550 °C) for 12 hours is 17.6% higher (an increase of 1.18 times) compared to the energy corresponding to the unprotected sample P<sub>12</sub>, or:

$$E_{EXP.2}^{I_3} = 0.85E_{EXP.3}^{I_3} \quad (6)$$

The following values are obtained as a result of an estimate calculation of the total consumed energies in intervals I<sub>1</sub>+I<sub>2</sub> (36 min. for P<sub>12</sub> and 24 min. for P<sub>p3</sub>) and I<sub>3</sub> (12 hours for P<sub>12</sub> and 12 hours for P<sub>p3</sub>), respectively:

$$E_{EXP.2}^{tot} = E_{EXP.2}^{I_1+I_2} + E_{EXP.2}^{I_3} = 9676 \text{ KJ} \quad (7)$$

$$E_{EXP.3}^{tot} = E_{EXP.3}^{I_1+I_2} + E_{EXP.3}^{I_3} = 11180 \text{ KJ} \quad (8)$$

In this context, the total consumed energy during EXP.3 is higher by 15.5% (1.15 higher) compared to that consumed during EXP.2:

$$E_{EXP.2}^{tot} = 0.87E_{EXP.3}^{tot} \quad (9)$$

The results demonstrate that the energy savings made on intervals I<sub>1</sub> and I<sub>2</sub> during the heating of sample P<sub>p3</sub> compared to sample P<sub>12</sub>, generates a reduction by ≈ 2...5 % of the total consumed energy during a ionic nitriding process for a part with macroirregularities, residues, oxides, etc., and protected (P<sub>p3</sub>), compared to the similar case of an identical, yet unprotected part (P<sub>12</sub>). An analysis regarding the input energy on discharge, consumed during the ionic nitriding process, highlights the fact that the value E<sub>tot</sub> is mainly determined by E<sup>(I<sub>3</sub>)</sup>, and then in decreasing order by E<sup>(I<sub>1</sub>)</sup> and E<sup>(I<sub>2</sub>)</sup>. As resulted from the conducted experimental determinations, the value of the energy consumption during the nitriding phase proper I<sub>3</sub> represents a substantial quantity of the total energy consumption:

$$E^{(I_3)} \cong (0.90 \dots 0.92)E^{tot} \quad (10)$$

Similarly, the value of the energy consumption corresponding to the cleaning-preheating (I<sub>1</sub>) and heating (I<sub>2</sub>) phases was calculated:

$$E^{I_1+I_2} \cong (0.08 \dots 0.10)E^{tot} \quad (11)$$

Because intervals I<sub>1</sub> and I<sub>2</sub> are interconnected by the phenomena occurring within the process, an energy consumption differentiation would be useless. The energy balance of ionic nitriding installations can be represented by means of the following general equation, which takes into consideration the main processes that occur within the working tank and on the cathode surface, [1, 7, 8]:

$$E_{abs} = mc\Delta T + E_c + E_{cv} + E_r + E_{ig} + E_{dm} + E_{es} + E_{pc} + E_{aux} \quad (12)$$

The signification of the terms in the equation is: E<sub>abs</sub> – the input power [kJ]; mcΔT – the potential energy in the charge; E<sub>c</sub>, E<sub>cv</sub>, E<sub>r</sub> – energy wasted by conduction, convection and radiation [kJ]; E<sub>ig</sub> – ionising energy of the gas [kJ]; E<sub>dm</sub> – molecular dissociation energy [kJ]; E<sub>es</sub> – re-emission energy [kJ]; E<sub>pc</sub> – cathode disintegration energy [kJ]; E<sub>aux</sub> – energy consumed by the adjacent equipment [kJ]. The energies corresponding to the first three

operating phases of a ionic nitriding cycle  $E^{(1)}$ ,  $E^{(2)}$  and  $E^{(3)}$  contain terms from the right member of the equation (12), except for the auxiliary energy, but with different weights according to the phase and the parameters of the plasma nitriding process [1, 9].

### 3 Conclusions

The use of the process of ion nitriding has opened the way to a more precisely controlled surface metallurgy on nitrided gears. The present use pulsed plasma nitriding method now allows metallurgists the ability to prevent arc discharge and over heating experienced with the traditional methods of continuous dc plasma generation. The ability to manipulate the plasma glow by partial pressure adjustment to follow the tooth profile reduces the risk of non-uniform case formation in the tooth root.

The nonfunctional (unprocessed) surfaces of the parts to be nitrided, can be insulated by means of the protective paints V-1 or V-2, in order to reduce the time for the degasification-cleaning-preheating phases  $I_1$ , and avoid the electric arc glow discharges. By protecting the nonfunctional surfaces of the parts, one makes them comparable to that of the processed areas (their behaviour during the ionic conditions is similar), the recommended type of insulation also reducing the times from hours to minutes.

In practice, the advantages are mainly energy related, because: real parts contain unprocessed areas of certain variable proportions (20...70 %). Even in the case of parts with small unprocessed areas, these areas can cause a lot of trouble during the heating phase, thus prolonging the degasification time by several hours. If this delay of reaching the operating temperature point takes place at high temperatures, where energy loss is significant, then the energy consumption during the degasification-cleaning-preheating and heating phase  $I_1$  and  $I_2$  be estimated, comparatively with the total consumption at:

$$E^{I_1+I_2} = (15...20\%)E^{tot} \quad (13)$$

The value of energy consumption during the ionic nitriding phase proper  $I_3$  would be more significant:

$$E^{I_3} = (80...85\%)E^{tot} \quad (14)$$

As concerns the energy consumed by the adjacent equipment ( $E_{aux}$ ), it was estimated at the value given by relation 12, which reflects a low consumption of this energy user category:

$$E_{aux} = (0.02...0.06)E^{(I_3)} \quad (15)$$

In this case, the special protective paints (V-1 and V-2) insulation of the nonfunctional areas (unprocessed) causes a significant energy consumption decrease during the degasification-cleaning-preheating and heating phases, without affecting the energy consumption increase during the nitriding phase proper, because the paint protected surfaces are not very large compared to the total surface of the parts [1].

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