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EXPERIMENTAL RESEARCHES CONCERNING THE PROTECTION AGAINST IONITRIDING OF ALLOYED STRUCTURAL STEELS

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Abstract: *The paper presents some experimental researches carried out by the authors for the purpose of determining the efficiency of the protection of a metallic surface against the effects of the thermochemical treatment of ionitriding (plasma nitriding), by means of layers of special paints based on lamellar copper powder. The efficiency was assessed by comparing the microhardness and the metallographical profile of protected, unprotected and untreated steel surfaces and was found to be very high.*
Key words: *thermochemical treatment, ionitriding, protective paint*

1. INTRODUCTION

In recent years, thermochemical treatments - and especially ionitriding (also known as plasma nitriding) have gained a wide usage in machines manufacturing processes, due especially to the advantages they offer in terms of improved hardness and strength of the superficial layer of parts and consequently this topic has been widely discussed by various authors (Ruset, 1991; Rie, 1999; Tracton, 2007). Ionitriding implies the bombardment of the metallic surface with active nitrogen ions.

However, in certain instances, some areas of the parts that are subjected to ionitriding must preserve their initial, lower hardness; in other instances, the ionitriding of the whole part would be non-economical if actually only a portion of it needs to increase its hardness. However, this topic has been less addressed by researchers (Tracton, 2007; Vermesan & Deac, 1992).

Therefore, a need arose also for the initiation of experimental researches regarding the conceiving of technologies for the local protection against the ionitriding of metallic surfaces.

The study presented in this paper started from the idea of elaborating protective mixtures (pastes or paints) to stop the adsorption, absorption and nitrogen diffusion phenomena at parts metallic surfaces, during the process.

Taking into account the ionic bombardment process' complexity, in a first stage of the experiments, there have been started a series of preliminary tests so as to exclude from the start on, based on specific criteria and real results, a large number of variants of protective mixtures that are not satisfying the goals set for them.

The subsequent tests were based on the idea that the protective layers should be of the same structural and chemical nature as the material that needs to be protected, in other words the protective layers need to be metallic (Bibu, 1998).

Since most of metals form, when heated in a nitrogen atmosphere, nitrides with different hardnesses and thermal stability, it was considered of interest to select those metals whose nitrides are not yet set at the temperature at which the ionitriding process takes place. This category includes the special metal from group I (Na, K, Cu) that form stable nitrides at the room temperature, nitrides that dissociate, however, in the presence of heat in atomic or molecular elements (Croitoru, 1988).

From this group of compounds, copper seems to be particularly favorable and therefore the experiments used the material in powder form.

The layers based on copper will “refuse” the nitrogen during the plasma nitriding process due to the impossibility of forming chemical combinations (copper nitride) at this working temperature (450…600°C). It was considered that in this way, there may be realised a chemical protection barrier on the metallic surfaces that are under the copper layer.

In this context, there have been elaborated isolating films against ionitriding, in other words there have been obtained protective paints that have as main objectives:
- stopping the development of ionitriding phenomena (adsorption, absorption and nitrogen diffusion) on the protected surfaces
- maintaining the base material's physical-chemical characteristics under the isolated areas;
- the normal development of the hardening process in the adjacent, unprotected areas;
- the easy removal of the layer after the process is finished;
- in general, to eliminate the deficiencies of actual protection technologies.

2. EXPERIMENTAL RESULTS

The realisation of protective paints based on copper powder, was generated by the idea of direct copper deposit on metallic parts that must be protected, avoiding this way the chemical and electro-chemical reactions.

The general experimental researches (Bibu, 1998) led to the elaboration of two original variants of special paints for local protection at plasma nitriding (ionitriding), paints made of lamellar copper powder (obtained by electrolysis or by physical dispersion in ball mills), having the shape of submicroscopic lamellae with metallic glitter and of extremely small dimensions (close to the semicolloidal-colloidal domain 5...50 μm). This powder in mixture with magnesium oxide was prepared as paste by adding polystyrene varnish (polystyrene dissolved in carbon tetrachloride, for the paint variant marked as P-1), or only by adding the carbon tetrachloride (for the paint marked as P-2).

The disperse colloidal system of copper lamellae (with dimensions between 10^{-9} and 10^{-4} m) avoids the forming of sediments and provides a high resistance to the film, an increase of the particle dimensions determining the decrease of kinetic stability.

The metallic samples subjected to the experiments have been elaborated from an alloy steel of wide use in mechanical engineering (39MoAlCr15), meant especially for nitriding and having the following chemical composition: 0,35...0,42%C; 0,35...0,60 %Mn; 1,35...1.65 %Cr; 0,15...0,25 %Mo; 0,20...0,45 %Si; 0,70...1,10 %Al; max. 0,035 %S and max. 0,035 %P.

The test samples, disks of ∅60x10 mm dimension, were divided into two different groups. The first group contained test samples in an annealed state, with a hardness of 220-250 HB. The second group consisted of samples in a hardened state (quenching followed by high tempering at 550°C), with hardness between 300-330 HB. Corresponding to the annealed state, the structure was formed of polyhedral ferrite and pearlite grains in approximately equal quantities, and in the case of the hardened state the structure consisted of tempering sorbite.

After the preliminary heat treatments, the test samples have been plane grinded (R_a=0.025 mm), and then degreased.

Finally, the samples' isolation was done, using the paints specifically elaborated for protection at plasma nitriding, the films being applied on the horizontal surfaces as well on the cylindrical side surfaces. The provision of an optimal distance between the disks, in order to avoid the double cathode effect, has been realised by putting them in the loading device with the help of threading pins. Also, some test samples were manufactured from the same steel type, for comparison reasons and the efficiency of the protection was then assessed based on these.

The ionitriding process (15 hours / 530°C / 1.2 torr), took place in an INI-30 ionitriding installation. The subsequent cooling of the protected and unprotected (nitrided) samples was done in the work vessel until the temperature of 200ºC, in an NH_3 current, and afterwards continued in calm air.

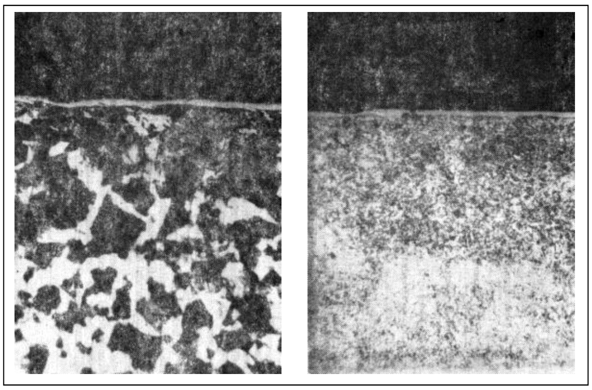
After the ionitriding, metallographical samples were extracted and prepared for macroscopic examinations, microscopic analyses and micro-hardness tests. Subsequently, the test samples have been carefully cleaned in order to not affect the metallic layers mechanically or thermally. Before the metallographic attack, the test samples were cut and the HV microhardness was measured on the surface layer and in the cross-section using a load of 2 N. These determinations had as purpose the hardness evaluation of the protected and unprotected metallic layers, as well as determining the depth of the ionitrided layer, to distinguish unprotected samples or an insufficient protection against the thermochemical treatment.

Corresponding to the unprotected metallic layers and ionic nitrided, the metallographic investigations emphasised a structure consisting of two distinctive areas (fig. 1.a and b), (consistent also with the results of (Ruset, 1991)):

- the white layer, with a thickness measured in microns and made of monophase nitride γ' of Fe_4N type (which has very good wear and strength properties);

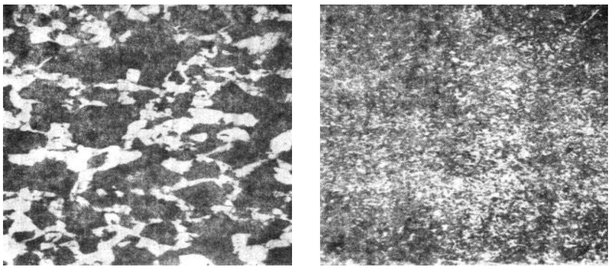
- the diffusion layer, after the white layer, has dimensions between 0.1-0.4 mm.

The samples microstructures that were protected against the diffusion process and subsequently subjected to plasma nitriding, are presented in figures 2.a and b. It can be noticed that the microstructures observed before the process are identical with those examined in the cross-section and on the surfaces that were protected and ionitrided: polyhedral ferrite and pearlite grains for the annealed state (fig. 2.a) and tempering sorbite for the hardened state (fig. 2.b).



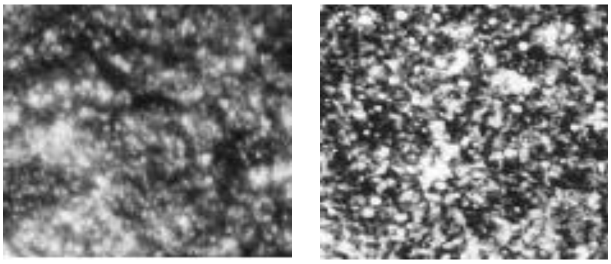
a b

Fig. 1. Microstructures (x500) in the cross-sections of two test samples made of 39MoAlCr15 steel, ionitrided (15h / 530° C):
a. initial annealing state; b. initial hardened state



a b

Fig. 2. Microstructures (x500) in the cross-sections of two steel test pieces 39MoAlCr15, protected with special paints and ionitrided (15h / 530° C):
a. initial annealing state; b. initial hardened state



a b

Fig. 3. The aspect of the superficial layer (x40) of the protective paints after ionitriding (15h / 530° C): a. paint P-1; b. paint P-2

3. CONCLUSIONS

Following the presented experimental researches, it was noticed that the surfaces covered with isolating layers made of special paints acted had the same characteristics as the untreated samples. The luminescent discharge ignites a few seconds after the process is started, at the same time for the unprotected and protected surfaces and the ion bombardment starts very quickly. On both types of surfaces, electric arc discharges and scintillations occurred intermittently in reduced numbers.

All types of investigations, analyses and attempts carried out with regard to the efficiency of the protection against ionitriding emphasised that the two special paints (P-1 and P-2) offer a very good protection against the superficial hardening induced by plasma nitriding.

Further researches will target the optimisation of the paints' composition and the behaviour of the paints on different types of surfaces and under various intensities of the thermochemical treatment.

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