



## EXPERIMENTAL RESEARCHES CONCERNING THE INFLUENCE OF THE OXYNITROCARBURIZING THERMOCHEMICAL TREATMENT OVER THE STRUCTURE AND CORROSION RESISTANCE OF C40 STEEL

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### ABSTRACT

*The aim of the paper is to present the results of the experimental researches regarding the corrosion behaviour of C40 steel, thermochemical treated by oxidant nitrocarburizing at 570°C. The thermochemical treatment was realized using an installation of oxidant nitrocarburizing. The characterization of the samples was done using laboratory equipments, in the following way: the composition of surface was determined using a RF GDS spectrometer (Jobin Yvon type), the structural analyses by X-ray diffraction (Rigaku Ultima IV) and scanning electron microscopy (MEB FEI). The evaluation of the corrosion resistance, which is the goal of this work, was done using the test method in salt fog, in the Laboratory of the Materials Engineering Department, Dacia plant.*

KEYWORDS: oxinitrocarburizing, C40 steel, corrosion

### 1. Introduction

The automotive industry is one of the largest consumers of materials, especially metallic materials, in the economy. The competition from this field, the technical progress and the requirements imposed to the automobiles, all these issues suppose the knowledge of the materials properties, the development of new materials, treatments and processing technologies, in order to ensure the operational requirements, the reliability or the aesthetic requirements, in terms of minimum costs. The choice and use of materials should always take into account the basic principle of engineering value, namely *achieving maximum performance with minimum costs*, this representing a very important and actual challenge, especially in the context of nowadays global economical crisis.

A solution to increase the reliability and durability in exploitation of different types of pieces is represented by the use of *superficial treatments* (heat and thermochemical treatments) or *thin films deposition techniques*. These treatments have as objective the substantial *improvement of the superficial properties*, as the *wear* resistance, the *corrosion* resistance, the *fatigue* resistance or the *aesthetic aspect*, [1, 2].

The goal of this paper is to present aspects regarding the influence of the *oxidant nitrocarburizing* thermochemical treatment (*oxinitrocarburizing*) over the superficial characteristics of *C40 steel*, especially aspects about the way this superficial treatment influences the *corrosion resistance* of the above-mentioned steel. An interesting point of interest is the characterization of the corrosion behavior in the case of an *application from automotive industry*.

*C40 steel* is a *non-alloyed special steel* which has a high purity, especially on non-metallic inclusions. It is suitable for the *treatment* of:

- *Quenching and tempering;*  
and/or
- *Superficial quenching or thermochemical treatment* (like *oxidant nitrocarburization*);

Particular conditions of elaboration and the control of the chemical composition may offer improved properties, allowing melting to severe exigencies.

These properties, associated with values of the *elasticity (Young) modulus* and of *hardenability*, high or in close ranges, lead to a good behaviour at cold deformation, to a good weldability and high toughness. *C40 grade* is defined by the European standard *EN 10083-2*.

## 2. Experimental details

The *oxinitrocarburizing treatment* was realized using an *installation of oxidant nitrocarburizing* (figure 1), with the following parameters: temperature -  $560 \pm 20^\circ\text{C}$ ; cycle time – 8 hours;  $\text{N}_2$  flow -  $0.4 \pm 0.1 \text{ Nm}^3/\text{h}$ ;  $\text{NH}_3$  flow –  $0.4 \pm 0.1 \text{ Nm}^3/\text{h}$ ;  $\text{CO}_2$  flow –  $0.3 \pm 0.01 \text{ Nm}^3/\text{h}$ .

The structure was evaluated using *X-ray diffraction*, *optical microscopy* (Leica DM-LM) and *scanning electron microscopy* (MEB FEI).

*XRD patterns* have been recorded using a Rigaku Ultima IV diffractometer with a  $\theta$ - $\theta$  goniometer, working with a copper X-ray tube, parallel beam optics and a flat secondary graphite monochromator. The operating voltage and the tube current were 40 kV and 30 mA. The  $2\theta$  scan region was 200-1400, with a step of 0.050 and the counting time of 2 s. Grazing incidence x-ray diffraction measurements were performed in the same angular range, with an incidence angle of  $20^\circ$ , in order to minimize the substrate contribution [4].

The *evaluation of the corrosion resistance*, which is the goal of this paper, was done using the *test*

*method in salt fog*. This consists of an accelerated corrosive attack with salt fog artificially created, of different composition, in the conditions of determined temperature and pressure. The degradation of the surface is appreciated visually by the appearance of the corrosion points or the appreciation of the relative size of the corroded surface. The test was done in the Laboratory of the *Materials Engineering Department*, inside a chamber (figure 2) where it salt fog was realized in the following conditions:

- the concentration of the sodium chloride in water:  $50 \pm 5 \text{ g/l}$ ;
- the density of the salt solution at  $20 \pm 2^\circ\text{C}$ :  $1,025\text{-}1,040 \text{ g/cm}^3$ ;
- pH salt solution: 6,5-7,2;
- the working temperature:  $35 \pm 2^\circ\text{C}$ ;
- the pressure and the pulverization are maintained constant and are adjusted so that it has to be realized a quantity of 1-2 ml/h of recovered solution on a surface of  $80 \text{ cm}^2$ . It was followed the *appearance of corrosion* and the *evolution of the corrosion degree*, at different periods (96 and 200 hours).



Fig. 1. Installation of oxidant nitrocarburizing Fig. 2. The salt fog chamber used for the experiments

## 3. Results and discussions

### 3.1 The chemical composition

The chemical composition of *C40 grade*, according with *EN 10083-2*, is presented in *table 1* (the content of chemical elements and the admitted tolerances), while *table 2* shows the results of the chemical analysis in the case of the samples (*C40 grade*) used for the experiments from this work.

### 3.2 Structure

#### *The structure after annealing*

The structure of the experimental samples, after annealing, was composed of *grains of pearlite and*

*ferrite* (figure 3), uniform distributed. The percentage of pearlite was 55-60%. This structure is associated to hardness values of *211-222 HB*.

#### *The structure of the oxinitrocarburized layer*

Images of the *oxinitrocarburized layer* are shown in *figures 4* (using *optical microscopy*) and *5.b* (*scanning electron microscopy analysis*). *EDX analysis* (figure 5.a) of the oxinitrocarburized layer revealed the presence of the diffusion elements, oxygen, nitrogen and carbon.

The metallographic micro-sections containing the oxinitrocarburized samples were attacked with 2-4% nital for three times, in order to put in evidence better the *white layer*. The *white layer*, which is uniform, has a medium thickness of 12-15  $\mu\text{m}$ . This is

composed of *two phases (zones): the first phase (zone)*, of about 2-4  $\mu\text{m}$ , where the presence of *oxygen, carbon and nitrogen* is being signaled, followed by the *second phase (zone)*, up to 10-14  $\mu\text{m}$ , where a *layer of combinations*, composed of *nitrogen and carbon*, is noticed.

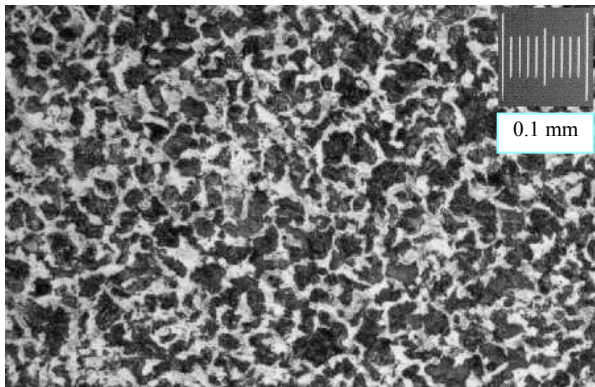
As an important remark, samples' surface before treatment has to be clean, without oxides, because the presence of an oxide layer can stop the diffusion process and, therefore, the generation of the white layer.

**Table 1.** The chemical composition of C40 grade, according with EN 10083-2

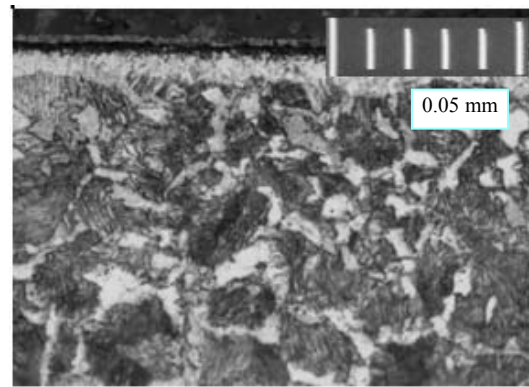
| [%] | C    | Si   | Mn   | P     | S    | Cr   | Mo   | Ni   | Cr+Mo+Ni |
|-----|------|------|------|-------|------|------|------|------|----------|
| Min | 0,37 | -    | 0,50 | -     | 0,02 | -    | -    | -    | -        |
| Max | 0,44 | 0,40 | 0,80 | 0,035 | 0,04 | 0,40 | 0,10 | 0,40 | 0,63     |

**Table 2.** The chemical composition of the material (C40 grade) used for the samples

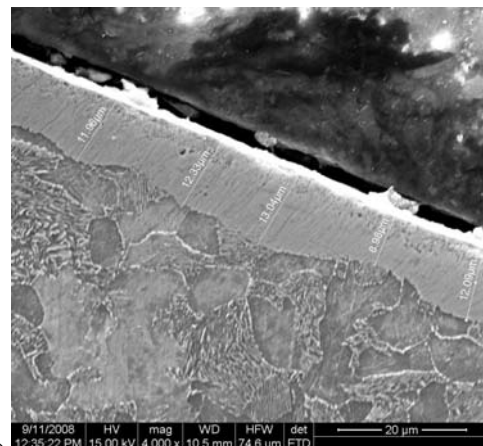
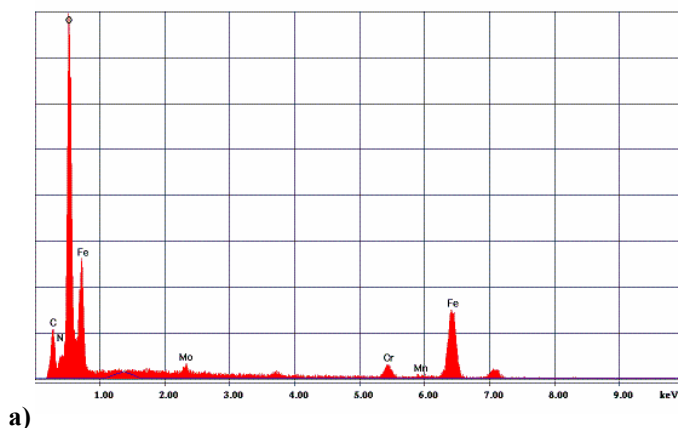
| [%]      | C    | Si   | Mn   | P     | S     | Cr   | Mo   | Ni   | Cr+Mo+Ni |
|----------|------|------|------|-------|-------|------|------|------|----------|
| Obtained | 0,42 | 0,23 | 0,60 | 0,009 | 0,027 | 0,13 | 0,08 | 0,04 | 0,25     |



**Fig. 3.** The structure of the samples, annealed (normalized), attack nital, x200



**Fig. 4.** The structure of the oxinitrocarburized layer, attack nital, x1000



**Fig. 5. a)** EDX analysis of the oxinitrocarburized layer  
**b)** The structure of the oxinitrocarburized layer, x4000, without attack, SEM analysis

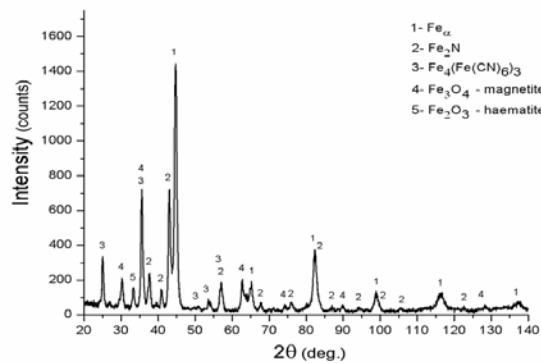
Figure 6 presents the x-ray diffraction pattern of the oxinitrocarburized sample. It shows the composition of the *surface layer (white layer)*, after the oxidant nitrocarburizing treatment.

The analysis of the white layer put in evidence that this is composed of *iron nitride ( $\text{Fe}_2\text{N}$ )*, *cabonitride  $\text{Fe}_4(\text{Fe}(\text{CN})_6)_3$* , and *magnetite ( $\text{Fe}_3\text{O}_4$ )*.

### 3.3 Corrosion resistance

For the *corrosion test*, there were used oxinitrocarburized *shafts*, parts that are used in the automotive industry. The oxinitrocarburized samples and the ones without treatment were exposed to the salt fog for 96 and 200 hours. In the case of the treated samples, there were find no corrosion points after 96 hours (*figure 7, oxinitrocarburized shaft*).

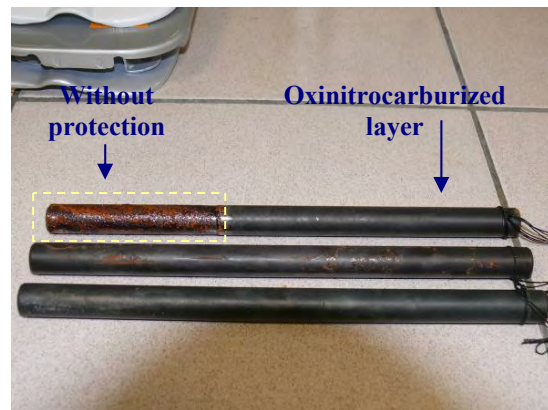
On the other hand, after 200 hours, it was remarked a corrosion of 2-5% in the case of oxinitrocarburized samples, while for the untreated pieces the corrosion level was about 80%. An interesting comparison between the treated and untreated samples (*shafts*) can be seen in *figure 8*. It is obvious the rust on the untreated half of the shaft, while the another oxinitrocarburized half presented a good resistance.



**Fig. 6.** Qualitative phase analysis of C40 steel after oxidant nitrocarburizing treatment



**Fig. 7.** Shaft – C40 steel; Oxinitrocarburized surface; Exposure at salt fog 96 h; without corrosion



**Fig. 8.** Shaft – C40 steel; Oxinitrocarburized surface; Exposure 200 h; corrosion 2-5%

### 4. Conclusions

The oxidant nitrocarburizing (oxinitrocarburizing) thermochemical treatment has a *positive action* over the *corrosion resistance* of C40 steel. The improvement of the anti-corrosive properties is especially due to the *white layer*, which is generated during the thermochemical treatment. This is composed of *iron nitride* ( $Fe_2N$ ), *carbonitride*  $Fe_4(Fe(CN)_6)_3$ , and *iron oxide* ( $Fe_3O_4$ ).

It was noticed that the *corrosion resistance* increases proportionally with the *thickness of the oxinitrocarburized layer*. The results are comparable with the resistance of the pieces coated by *zinc coating*

*process* (8-10  $\mu m$ ). Moreover, the oxinitrocarburized layers combine the *corrosion resistance* with *excellent tribological characteristics*, as previous studies of the authors revealed [3].

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