OXIDIZING INFLUENCE OVER SURFACE STRUCTURES AND PROPERTIES FOR A3k STEEL NITROCARBURIZED IN FLUIDIZED BED

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ABSTRACT

Surface duplex thermochemical treatment (oxinitrocarburizing) has principal scope to obtaining superficial mechanical higher properties with increasing corrosion capacity. The paper is based by oxinitrocarburizing experiments made in laboratory conditions over steel samples (low carbon steels, A3k). Active media for treatments was obtaining by ammonia, methane and air. Effective treatment was made in endothermic atmosphere, methane and ammonia that produced smooth fluidization. In a second stage o postoxidation at different time was made. The results of duplex surface treatments were investigated by superficial hardness (HV_5) and by metallographic structure.

KEYWORDS: oxinitrocarburizing, fluidized bed

1. Introduction

Oxinitrocarburizing is a duplex thermochemical treatment that makes higher surface hardness, associated with good corrosion behavior and a superior surface aspect, thus the ulterior surface treatments is unnecessary. This technology is applied at the mechanical parts obtained by stamping, deep drawing, drilling and other. Examples of these parts are in automobile industry and these are made from wire and plate of steel with low carbon and high plastic cold deformation capability. An ulterior treatment by nitrocaburizing induces in first stage a substantial modification in surface structure and superior values of surface properties. Second stage is oxidizing at Fe_3O_4 and final impregnation of surface with emulsions that have a higher stability in time. Oxidizing at the high temperature steam conduced to increasing corrosion resistance by two mechanisms:

- bonding free atoms (Fe) at Fe_3O_4 (stabile combination)
- surface topology that have an irregular profile after intense oxidation (deep crevice) with high capacity of absorption for protection emulsion [2].

Table 1. *The chemical composition of the steel specimens used in experiments.*

C	Mn	Si	P	S	Al	Cr	Ni
max.0,11	max.0,45	max.0,5	max.0,035	max.0,04	0,02…0,10	max.0,08	max.0,1

2. Working methods

For experiments were used specimens from low carbon steel that having with good behavior at the cold plastic deformation.

A3k steel (STAS 9485-80) is non-alloyed steel with lower carbon content, for cold plastic deformation, in special for deep drawing, and ulterior protection painting.

Chemical composition is showed in Table 1. The specimens for experiments were cut by L profile (section 25mm x 25mm) with 25mm in length and ulterior grouped for fluidized bed charging.

The samples were arranged in fluidized bed furnace in central positions. An *air/gas=2,3* rapport was maintained, that represents a condition for economical gas (CH_4) consumption. In the fluidized bed an endothermic atmosphere was produced and a rest of methane is present for nitrocarburising processes.

Samples surface were successive polished with 80, 150, 320, 400, 600, 800 abrasive paper, and ulterior cleaning with alcohol.

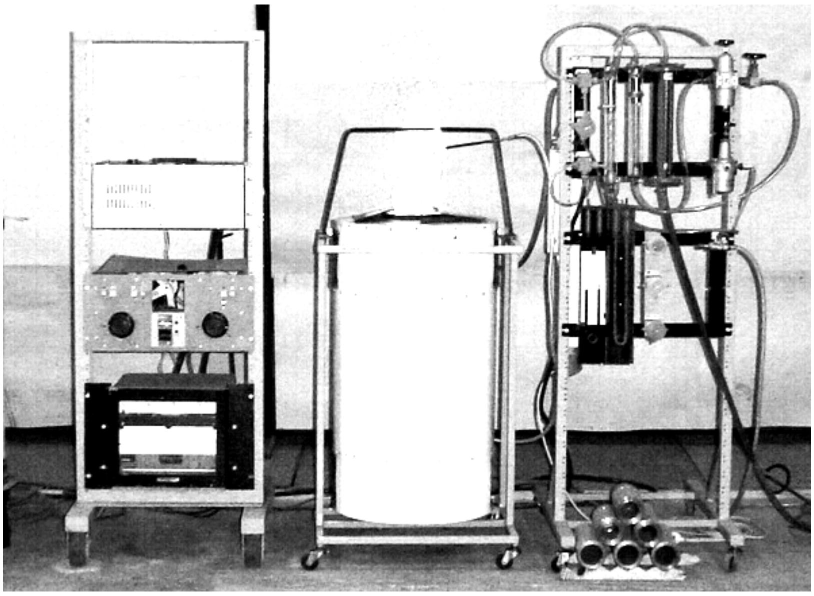

Fig. 1. *Laboratory fluidized bed plant for thermochemical treatments.*

Oxinitrocarburizing is a complex thermo-chemical treatment that is possible to realize on the same fluidized bed furnace. The classical technology offers three stages in furnaces with retort:

- nitrocarburising
- postoxidation in hot steam
- emulsion impregnation

At the nitrocarburizing process in fluidized bed following factors have important influences [4]:

- nitrocarburizing temperature (three valuees: 630°C, continuous cooling from 630 to 570°C, 570°C)
- nitrocarburizing time (1,5h; 2h; 2,5h)
- chemical activity of media (initial ammonia concentration or initial partial ammonia debit: aprox 23%)

Factors that have influence over post oxidation are:

- oxidation temperature (adopted 570°C, according to Figure 2)
- oxidation time (20,40 and 60min)
- oxidant gas concentation (hot steam, initial debit 1l/h)
- sample positions in furnace (central)

Thermochemical treatment media was realized in open furnace (Figure 1) that conduced to important decreasing of heating and cooling time and specimens were easy removal from furnace.

An open furnace has an important advantage: easy control processes because partial pressure of gasses is in direct correlation with initial debit participation.

Active nitrocarburizing media was obtaining by introduction in furnace a gas initial mixture from air, natural gases (>95% methane) and ammonia. Total initial debit was constant (500l/h air, 230 l/h methane, 520 l/h ammonia). In the furnace some initial gasses having thermal decomposing and some reactions:

- ammonia has a partial decomposition in mollecular nitrogen and hydrogen
- mathane has a partial decomposition in carbone and hydrogen
- methane has a interaction with the oxygen from air, thus the endothermic atmosphere is produced (air/gas=2,3 raport)

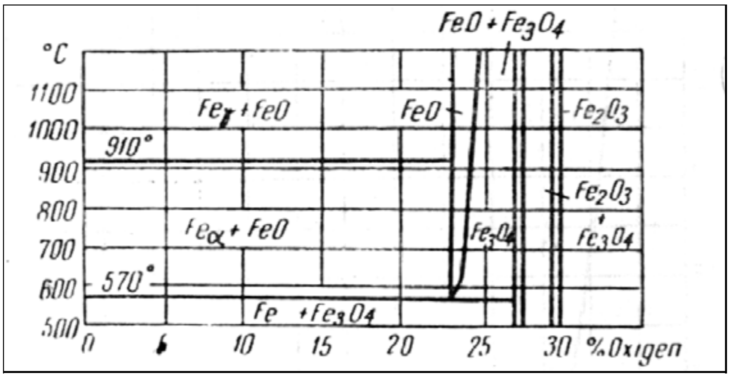

Fig. 2. *Fe-O equilibrium diagram, [3].*

Table 2. *Oxinitrocarburizing in fluidized bed regimes*

sample experiment	nitrocarburizing media	nitrocarburizing temperature	nitrocarburizing time	oxidizing media	oxidizing temperature	oxidizing time
U.M.	-	°C	h		°C	min
1	fluidized bed by solid granular (burned fire clay) and gas mixture (ammonia and nitrogen)	630	1,5	steam	570	20
2						40
3						60
4			2,0			20
5						40
6						60
7			2,5			20
8						40
9						60
10		630-570	1,5			20
11						40
12						60
13			2,0			20
14						40
15						60
16			2,5			20
17						40
18						60
19		570	1,5			20
20						40
21						60
22			2,0			20
23						40
24						60
25			2,5			20
26						40
27						60

The chemical composition of gas mixture that makes the fluidization and nitrocarburizing are hydrogen, nitrogen, carbon monoxide, ammonia (rest), methane (rest). Oxidizing is an ulterior operation that is made in concordance with Fe-O diagram (Figure 2), by water debit inlet (approx. 1l/h) to the base of bed.

After intense vaporization vapor continue up heating to regime temperature (570°C).

Water vapors make fluidization and will be realize favorable conditions for heat and mass transfer. Experimental matrix is presented in Table 3.

3. Results obtained

Results of oxinitrocarburizing in fluidized bed experiments were metallographic investigated for structure modifications (surface-core transition zone) and mechanical investigated (superficial hardness) for property modifications at surface.

Representative microstructures are presented in Figure 3…Figure 10. Nitrocarburizing time influence over surface hardness is showing in Figure 11 Postoxidation time influence over surface hardness is presented in Figure 12.

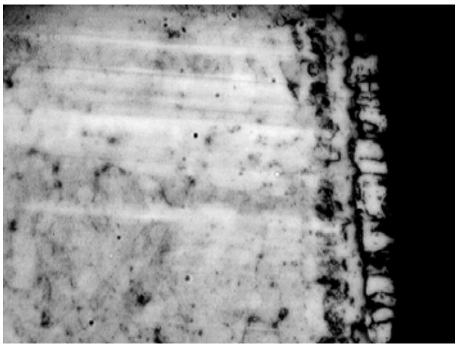

Fig. 3. *Microstructure of A3k sample 2 (350x).*

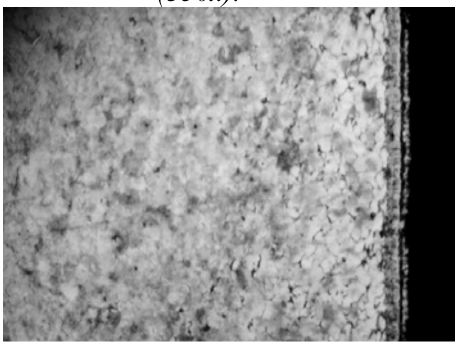

Fig. 4. *Microstructure of A3k sample 7 (170x)*

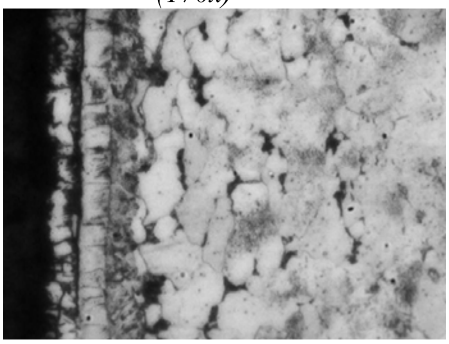

Fig. 5. *Microstructure of A3k sample 7 (350x)*

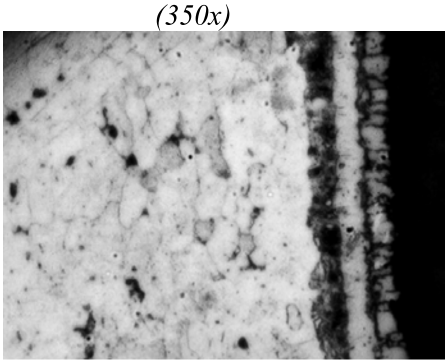

Fig. 6. *Microstructure of A3k sample 8 (350x)*

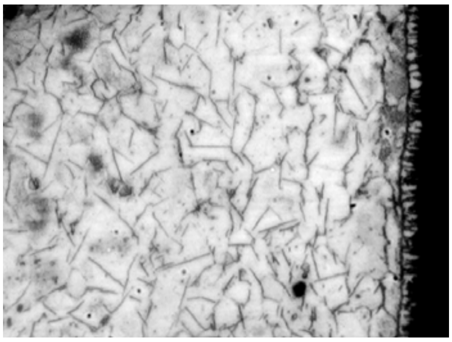

Fig. 7. *Microstructure of A3k sample 13 (350x)*

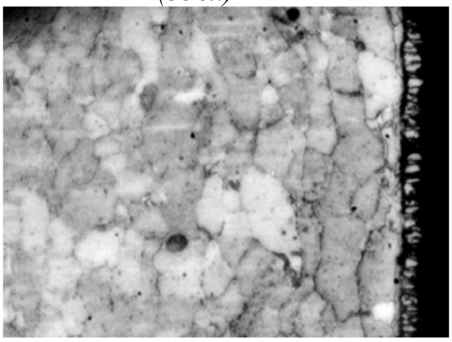

Fig. 8. *Microstructure of A3k sample 15 (170x)*

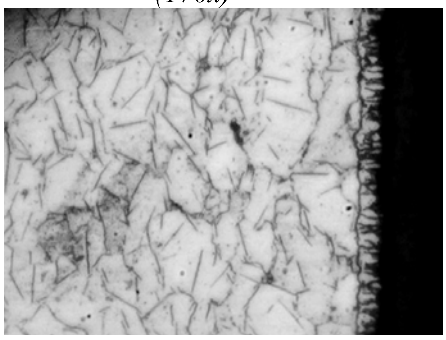

Fig. 9. *Microstructure of A3k sample 16 (350x)*

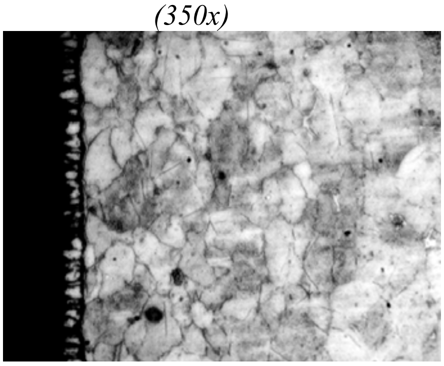

Fig. 10. *Microstructure of A3k sample 17 (350x)*

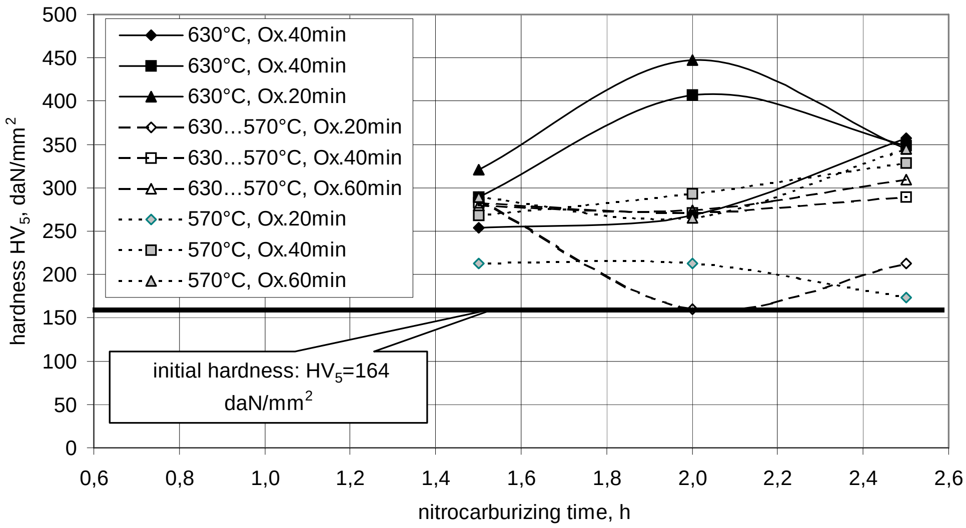

***Fig. 11.** Influence of nitrocarburizing time over surface hardness.*

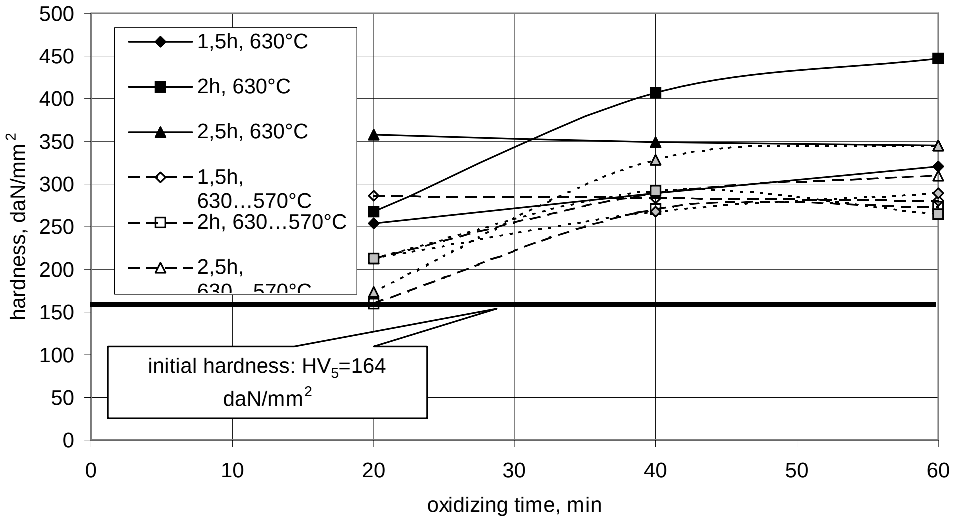

***Fig. 12.** Influence of post oxidation time over surface hardness.*

4. Conclusions

For all oxinitrocarburizing in fluidized bed experiments, surface hardness having a normal increasing with treatment time.

Microstructures showing diffusion zone for all experiments and combination layer for long nitrocarburizing times. After oxidation deep crevices were formed in combinations layer ($\varepsilon+\gamma'$) that having an important role for (emulsion accumulator) for increasing corrosion resistance,

For 40 and 60 min oxidizing time surface hardness decreasing because long oxidizing time produce nitrogen desorption, that affecting surface structure and superficial hardness.

Fluidized bed technology applications for oxinitrocarburizing treatment are useful for small series of parts with medium importance.

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