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INFLUENCE OF SOME TECHNOLOGICAL PARAMETERS OF FLUIDIZED BED CARBONITRIDING ON THE SURFACE HARDNESS OF CARBONITRIDED PARTS FROM LOW ALLOY STEELS

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ABSTRACT

The paper describes the experimental research of thermos-chemical treatment of carbonitriding performed on samples of different brands of low alloy steels.

The technological parameters whose influence on the carbonitrided surfaces hardness was studied are thermo-chemical treatment temperature and the percentage of ammonia in the carbonitriding medium. Thermo-chemical treatments of carbonitriding have been carried out at low, medium and high temperatures.

The investigations were conducted with three values for the ammonia percentage, which lead to different results for the surface hardness of the samples subjected to the investigations.

The duration of treatment of two hours and thirty minutes, constant for all experiments performed, was chosen as the one commonly used in gas carbonitriding technologies.

KEYWORDS: thermo-chemical treatment, carbonitriding, temperature, hardness

1. Introduction

Ensuring sustainability of parts used in machine building is achieved through thermo-chemical treatment of carbonitriding.

The complex of properties most desired and most used in technological applications consists of [1]:

- high surface hardness of the piece;
- high toughness in the middle of the piece.

The thermochemical treatment of fluidized bed carburizing is used mainly in areas where heat transfer, mass transfer and heat or mass transfer are required [2, 3]. Among the most important applications of the fluidized bed are:

- chemical industry (catalytic reactions in which the catalyst is fluidized);
- oil industry (heavy oil catalytic cracking);
- thermal power (coal fluidized bed combustion).

The chemical activity of the medium is dependent on:

- proportion of gas at the furnace inlet
- the chemical composition at the working temperature because gases (methane and ammonia) undergo chemical reactions.

Thus, it is formed a gas mixture composed of hydrogen, nitrogen, ammonia and methane (in small portions), which ensures fluidity of sand grains at the working temperatures, in compliance with fluidization conditions. Practically, the properties specific to fluidized beds are achieved [4, 5].

The main chemical reactions in the case of fluidized bed carbonitriding which occur inside the furnace working atmosphere, which is a gas atmosphere, and between the latter and the surfaces of the parts, are:

$$2CO = C_{(\gamma)} + CO_2 \tag{1}$$

$$CO + H_2O = CO_2 + H_2 \tag{2}$$

$$CH_4 = C_{(\gamma)} + 2H_2 \tag{3}$$

$$NH_3 = N_{(\gamma)} + 3/2H_2 \tag{4}$$

Ammonia in atomic condition, necessary to the carbonitriding treatment, results from the phenomenon of dissociation of ammonia that takes place after an endothermic reaction [6] and, at temperatures of 500 °C, under the presence of dissociation energy, decomposition is complete:

$$2NH_3 \leftrightarrow N_2 + 3H_2 \quad (5)$$

After an endothermic dissociation reaction of methane gas active carbon atoms are obtained which enter the high temperature fluidized bed as a result of the contact with the hot sand grains [7].

$$CH_4 \leftrightarrow C + 2H_2 \quad (6)$$

In order to carry on the role for which the fluid bed is introduced into the system, the fluidization conditions must be fulfilled: the fluidization velocity, the properties of the grain solid, the fluidizing agent thermophysical properties, the characteristics of the fluidization chamber [5].

The technical and economic aspects offered by such facilities led to the development and expansion of the use of such heat treatment fluidized bed facilities [4, 8].

Due to the fact that at high temperatures the diffusion of carbon is higher than that of nitrogen, the carbonitrided layers structure at these temperatures is much close to that of the carbide layers [2, 8].

2. Materials and research method

The fluidized bed carbonitriding experiments were done on samples of 21TiMnCr12, 18MnCr10, 13CrNi40 steel whose chemical composition is shown in Table 1.

Table 1. *Chemical composition of grades of steel subject to research*

Nr. crt.	Mark	Chemical composition, % mass							
		C	Mn	Si	P	S	Cr	Ni	Ti
1	21TiMnCr12	0.2	0.95	0.28	0.014	0.016	1.05	-	0.06
2	18MnCr10	0.18	1.05	0.22	0.035	0.035	1.05	-	-
3	13CrNi40	0.13	0.45	0.22	0.035	0.035	1.45	4.00	-

The thermochemical treatment regimens studied at laboratory scale are shown in Table 2. In choosing these regimes, the recommendations from literature were taken into account and low, mid and high carbonitriding temperatures were covered for the same maintaining time of 2 hours and 30 minutes. The proportion of ammonia introduced into the system at a time was of 5%, 15%, 25%, in each variant of treatment carried out.

Table 2. *Thermochemical treatment regimes tested*

Nr. crt.	Proportion of ammonia [%]	Temperature [°C]	Duration of carbonitriding [min]
1	5	550	150
2		650	
3		750	
4		900	
5	15	550	
6		650	
7		750	
8		900	
9	25	550	
10		650	
11		750	
12		900	

In Figure 1 is shown in section the furnace in which the thermochemical treatment regimes described above were carried out.

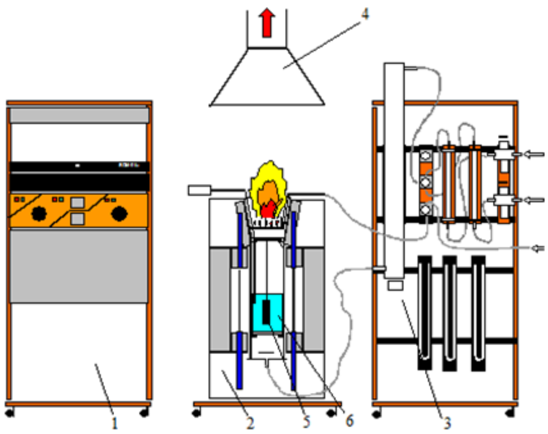

Fig. 1. *Laboratory installation for fluid bed thermo-chemical treatments: 1 - system of power supply and temperature control, 2 - the fluidized-bed furnace, 3 - technology gas supply rack, 4 - flue gas hood, 5 - sample, 6 - fluidized bed*

The thermochemical treatment technology is based on energy and mass transfer from piece to the environment and therefore a good circulation of the gaseous medium is very important to attain the objective of the thermochemical treatment.

The fluidized bed, which is made of granules of quartz sand, represents the carbonitriding environment, while the fluidizing agent is the atmosphere resulting from decomposition of methane and ammonia at the working temperature.

Changing the ratio between the two gases, ammonia and methane, lead to changes in the chemical composition of the fluidizing agent.

The contact between the gases introduced into the working chamber of the treatment furnace at the ambient temperature and the hot granular solids results in a decrease in the time of the dissociation reaction, which is an advantage of the treatment in the fluidized bed.

The fluidized bed is characterized by a high carbon potential, and the absence of the occurrence of deposits of carbon black which inhibits carburization. During the technological process, deposits of carbon black occur on the samples surface, but are removed by the solid granules from the fluidized bed, which are constantly moving and permanently gets in contact with the sample surface.

By conducting some preliminary experiments, the flow rates of methane and ammonia were established. Carbonitriding thermochemical processing regimens were made on the same plant, on the basis of a program in which variations of important technological parameters were established: the treatment temperature and the percentage of ammonia in the system. By means of a silica gel column, moisture was retained from methane and ammonia.

3. Research results

The experiments conducted have studied the influence of temperature and ammonia percentage on the surface hardness of the samples subjected to carbonitriding.

In order to determine the hardness of the carbonitrided samples surface obtained, after preliminary preparation, hardness test was performed by Vickers method with a load of 5 kg force.

Figure 2 shows the variation of the Vickers type hardness, HV5, for 21TiMnCr12 steel, depending on the temperature of treatment and the percentage of ammonia.

The highest value of hardness was obtained from samples that have been treated under the conditions: temperature of 550 °C and an ammonia concentration of 15%. The lowest values of surface hardness of this steel sample were registered in the processing regime characterized by temperature of 650 °C at a rate of 5% NH_3 and at the temperature of 750 °C with 25% NH_3.

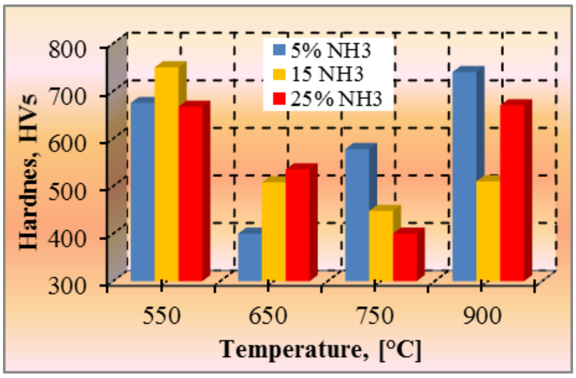

Fig. 2. *Variation of the surface hardness of 21MoMnCr12 steel samples, carbonitrided in fluidized bed for different proportions of ammonia*

For the samples of 18MnCr10 steel carbonitrided according to the regimens chosen, the change in surface hardness values is illustrated in Figure 3. At a temperature of 550 °C, the hardness corresponding to 15% NH_3 system is the highest for this temperature. Following the thermochemical processing at a temperature of 750 °C, the hardness values obtained when the percentage of NH_3 is 5 to 15% are very close. Increasing temperature to 900 °C makes the surface hardness to record hardness values of samples around 600 daN/mm^2 for all three concentrations of ammonia.

The lowest hardness value was obtained for the samples treated at 750 °C when the supply of NH_3 was of 25%.

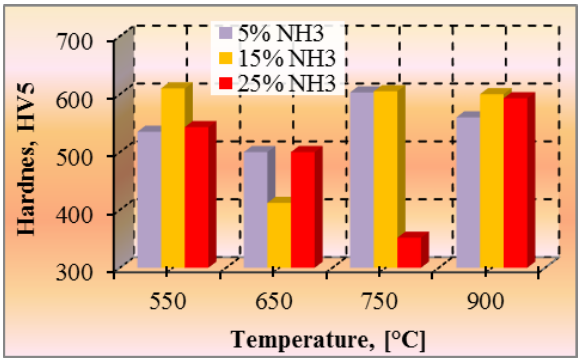

Fig. 3. *The surface hardness variation of the 18MnCr10 steel samples carbonitrided in fluidized bed for various proportions of ammonia*

Figure 4 shows for the samples of 13CrNi40 steel how hardness varies on the surface of the carbonitrided samples. In this case, it is noted that the hardness obtained is less than that for 18MnCr10 steel and also than that for 21TiMnCr12 steel.

The highest values of surface hardness of 13CrNi40 steel samples were obtained under the conditions of 750 °C treatment temperature and for an ammonia content of 5 to 25%.

For a content of 5% ammonia, an increase of the treatment temperature from 550 to 750 °C results in an increase in the hardness of the steel surfaces for these 13CrNi40 steel samples, and from 750 °C to 900 °C the hardness is slightly decreased. When the ammonia content is 15%, the highest hardness was recorded for 13CrNi40 steel samples treated at 750 °C.

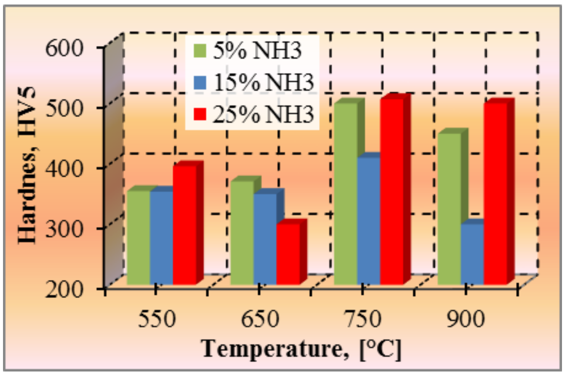

Fig. 4. *Variation of the surface hardness of the 13CrNi40 steel samples carbonitrided in fluidized bed for various proportions of ammonia*

Figures 5, 6 and 7 present the microstructures for one sample of each steel grade studied, Fig. 5 - 21TiMnCr12 steel, Fig. 6 - 18MnCr10 steel, Fig. 7 - 13Cr10Ni40 steel, for which the highest values of the surface hardness were obtained after processing according to one of the fluidized bed carbonitriding regimes experimented.

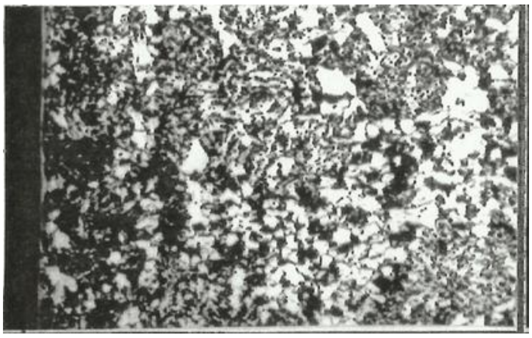

Fig. 5. *The microstructure of a 21TiMnCr12 steel sample carbonitrided at t = 550 °C, 5% NH_3, 100 X magnification, attack NITAL 2%*

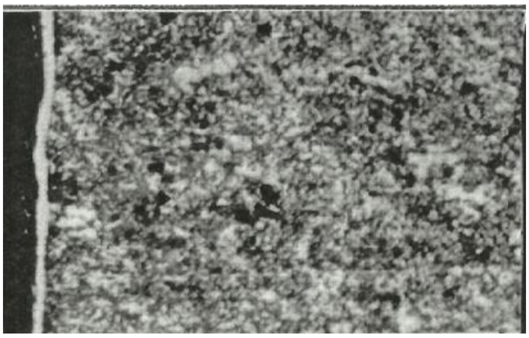

Fig. 6. *The microstructure of a18MnCr10 steel sample carbonitrided at t = 550 °C, 15% NH_3, 100 X magnification, attack NITAL 2%*

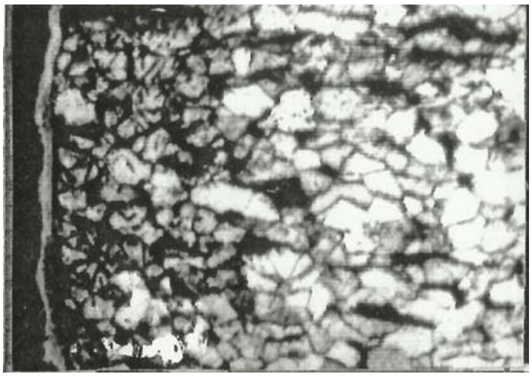

Fig. 7. *Microstructure of a 13CrNi40 steel sample carbonitrided at t = 750 °C, 25% NH3, 100 X magnification, attack NITAL 2%*

The structures present on the surface nitride layers and carbonitrides layers with various thicknesses.

4. Conclusion

Noting the variation of the HV5 type surface hardness values of the samples from the 3 studied low alloy steels, we can say that:

• decreasing the content of C resulted in the production of softer surfaces;

• for a content of 5% ammonia for 13CrNi40 steel surfaces are obtained with hardness increasing with the increase of the treatment temperature within the interval 550 °C – 750 °C, while for the interval between the 750 °C – 900 °C, the hardness is decreasing with increasing the temperature;

• at 650 °C, for samples of 13CrNi40 steel, hardness decreases as the values of the percentage of NH_3 are increasing from 5% to 25%, as opposed to what is recorded for the samples of 21TiMnCr12 steel, when at the same treatment temperature of 650 °C, the increase in the percentage of NH_3 increases hardness;

• for the samples of steel 18MnCr10 carbonitrided at 550 °C, the surface hardness increases when ammonia increases from 5 to 15% and decreases when ammonia increases from 15 to 25%, while for the samples annealed at 650 °C, the situation is exactly the opposite;

• a treatment temperature of 750 °C for steel 21TiMnCr12 highlights a decrease in hardness as the content of ammonia in the system is increasing;

• for 21TiMnCr12 steel samples, where the percentage of ammonia is 5%, the hardness increases proportionally with the increase of the treatment temperature between 650 °C - 900 °C;

• a concentration of 15% and 25% NH_3 during the carbonitriding of 21TiMnCr12 steel results in a decrease in hardness with increasing the treatment temperature in the temperature range 550 °C – 750 °C, after which the hardness increases in the range 750 °C – 900 °C;

The results of the experiments conducted to the confirmation of the special properties of fluidized beds (dispersed gas-solid medium) and the possible industrial application of the proposed technology and of the different variants derived from it.

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