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THE INFLUENCE OF THE CARBON CONTENT ON THE DEPTH OF THE CARBONITRURAT LAYER IN FLUIDIZED LAYER FOR 1C15, 1C25 AND 1C45 STEELS

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ABSTRACT

This paper presents the experimental research of carbonitriding thermochemical processing in fluidized layer performed on some samples of carbon steel with different carbon contents, the fluidized layer being made up of a mixture of quartz sand and gases (methane, ammonia and air).

The thermochemical treatments have been carried out over a time period of two hours and thirty minutes for all temperatures considered for the study. An important parameter of this type of thermochemical processing was also represented by the ratio of methane and ammonia that influenced the final results of the research.

The research results revealed the influence of the carbon content of the steels investigated on the thickness values of the carbonitrided layer for samples investigated.

KEYWORDS: carbo nitriding, fluidized bed, hardness, surface layer

1. Introduction

Carbonitriding is the thermochemical treatment that is normally applied on carburizing carbon steels, in a temperature range between 830...870 °C. The treatment environments used are various (solid, liquid, gaseous, plasma, vacuum) so that the concentration of carbon at the surface to be brought to 0.8...0.9% and that of the nitrogen to 0.3...0.4% [1].

The structure of the carbonitrided layer at these temperatures is much closer to that of the carburated layers because at high temperatures, carbon diffusion is more intense than that of nitrogen [2].

The durability of the carbonitrurate parts being one of the most important objectives in the construction of cars, by carbonitriding this objective is successfully accomplished.

The most desired and most used properties complex in technological applications consists in [3]:

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

Fluidized layer carbonitriding involves achieving an active carbonitriding environment inside a fluidized bed by overlapping two categories of phenomena:

- fluidization (transformation of a fixed bed of inert beads of firebrick with 0.10...0.16 mm average size in a fluidized bed gas mixture)
- carbonitriding, which involves choosing a gas mixture (methane and ammonia, in varying proportions) allowing the accelerated implementation of this treatment at the working temperature specific to carbonitriding.

Developing and extending the use of fluidized bed heat treatment facilities was and is determined by a number of advantages offered by such facilities. Technical and economic aspects related to the construction (acquisition) and the operation of such plants show that there are advantages over other types of equipment used in heat treatment [4].

The gas mixture for carburizing serves to generate at the treatment temperature the active carbon atoms which are adsorbed and then dispersed in the material. This mixture can be obtained from:

- natural gas,
- endogas purified by natural gas addition,
- exogas purified by natural gas addition,
- gaseous atmosphere obtained from the gasification of liquid hydrocarbons [3].

Active carbon atoms are obtained by a reaction of endothermic dissociation of methane gas as soon as it enters the high temperature fluidized bed as a result of contact with hot sand grains [5].

$$CH_4 \leftrightarrow C + 2H_2 \tag{1}$$

Methane easily dissociates at high temperatures and therefore the amount of methane that is placed in the oven must be very well controlled to avoid the production of the so-called hydrocarbon black. Active carbon excess that is not adsorbed becomes a layer of amorphous carbon and slows much the further process kinetics [6].

Ammonia is introduced into the workspace to generate nitrogen atoms that are active and also adsorbed and then diffused towards the interior of the material. Dissociation of ammonia occurs after an endothermic reaction [7] and at temperatures of 500 °C, under the presence of dissociation energy, the decomposition is total:

$$2NH_3 \leftrightarrow N_2 + 3H_2 \tag{2}$$

Also in the case of fluidized layer carbonitriding, the main chemical reactions that occur inside the furnace working atmosphere, which is a gaseous atmosphere (homogeneous systems) and between this and the parts surfaces (heterogeneous systems) are:

$$2CO = C_{(\gamma)} + CO_2 \tag{3}$$

$$CO + H_2O = CO_2 + H_2 \tag{4}$$

$$CH_4 = C_{(\gamma)} + 2H_2 \tag{5}$$

$$NH_3 = N_{(\gamma)} + 3/2H_2 \tag{6}$$

Carbonitriding in a fluidized bed is in fact a gas carbonitriding with conditions in the theoretical treatment, respectively the reactions of dissociation in the components of the medium, adsorption of the carbon and nitrogen atoms and finally their diffusion.

Carburized steels are carbon steels with a maximum carbon content of 0.25%, but may also be higher concentrations steels. After carburizing, the surface carbon concentration can reach 0.7...1.2%.

2. Materials and method of research

Samples of 3 carbon steel grades - OLC 15, OLC 25 and OLC 45, whose chemical composition is shown in Table 1, were subjected to experimental research.

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

Nr. crt.	Mark	Chemical composition, % mass								
		C	**Mn**	**Si**	**P**	**S**	**Cu**	**Cr**	**Ni**	**Ti**
1	1C 15	0.14	0.53	0.22	0.017	0.023				
2	1C25	0.22	0.48	0.32	0.025	0.450	0.21	0.17	0.10	
3	1C45	0.201	0.951	0.281	0.014	0.016	-	1.05	-	0.06

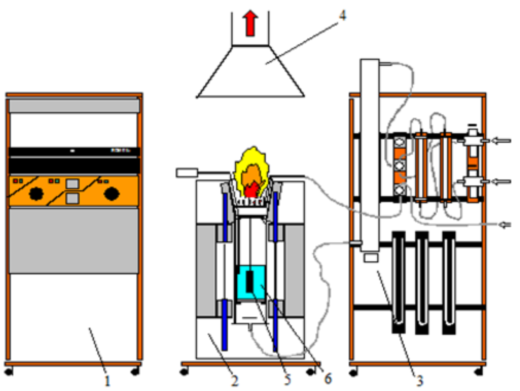

Fig. 1. *Laboratory installation for fluidized bed thermochemical treatments: 1 - system of power supply and temperature control, 2 - fluidized-bed furnace, 3 - technological gas supply rack, 4 - flue gas hood, 5 - sample, 6 - fluidized bed*

The laboratory scale installation for the thermochemical treatment of carbonitriding in a fluidized bed with the aid of which experiments have been performed is shown schematically in Figure 1.

The carbonitriding regimes that have been experienced at laboratory scale are shown in Table 2. The tests carried out had in view the influence of carbon content of the three steel grades considered in research, on the size of the carbonitrided layer depth, depending on process parameters.

With the chosen experimental regimes, the low, medium and high carbonitriding temperatures were covered, at the same value of maintaining duration of 2 hours and 30 minutes for three values of the ammonia percentage (5%, 15%, 25%) introduced in the system.

Table 2. *Thermochemical treatment regimes tested*

Nr. crt.	Proportion of ammonia [%]	Temperature [°C]	Duration of carbonitriding h, min
1	25	900	2h 30min
2		750	
3		650	
4		550	
5	15	900	2h 30min
6		750	
7		650	
8		550	
9	5	950	2h 30min
10		750	
11		650	
12		550	

The technology is based on mass and energy transfer from medium to pieces. This is why a good circulation of the gaseous medium is particularly important for a successful treatment.

The carbonitriding medium is a fluidized bed made of quartz sand grains, the fluidizing agent being an atmosphere resulting from decomposition of methane and ammonia at the working temperature.

The chemical composition of the fluidizing agent is amended by the proportions of gas at the entrance. Dissociation reactions take place quickly due to the contact of the gases introduced into the working chamber of the treatment furnace at ambient temperature and the hot granular solids. The fluidized bed is characterized by a high carbon potential and the absence of the occurrence of deposits of carbon black which inhibits carburization. Deposition of carbon black on the surface of the samples is removed from the fluidized bed of solid granules which come into contact with the surface of the sample permanently.

Methane and ammonia flow rates, the working temperature and other technological parameters were determined by preliminary experiments. Carbonitriding experiments were done on the same plant on the basis of a program in which variations of the important technological parameters were established (Table 2). The humidity of methane and ammonia was retained by a silica gel column.

3. Experimental results

After conducting experimental research on the samples, there were conducted investigations of the carbonitrided layers made without subsequent tempering (at the balance) in order to highlight the carbonitrided layer thickness variation according to the parameters of the thermochemical treatment regime for the three types of steel studied. The carbonitrided layer measurement was made in metallographic format.

Figure 2 shows the variation of the carbonitrided layer thickness at a temperature of 550 °C depending on the percentage of ammonia for the three steel types studied. The highest value of the carbonitrided layer thickness in these conditions was obtained for samples of 1C25 at a rate of 5% ammonia. The shallowest carbonitriding was recorded on samples of 1C45 at a rate of 5% ammonia. For the value of 15% ammonia, the carbonitrided layer depth decreases with the decrease in carbon content for the three grades of steel undergoing thermochemical processing. At 25% ammonia, the layer depth is increases in the following order: 1C45, 1C15, 1C25.

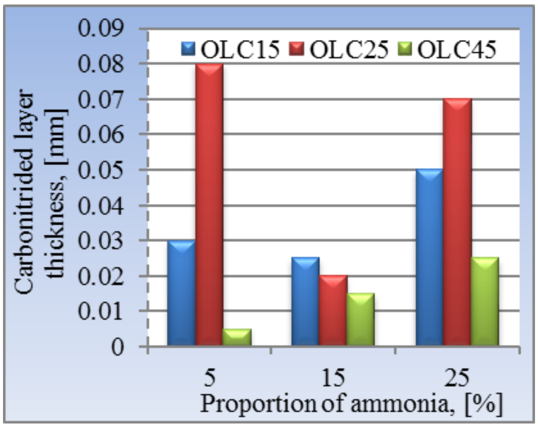

Fig. 2. *Variation of the carbonitrided layer thickness at 550 °C according to the proportion of ammonia for the three steel types studied*

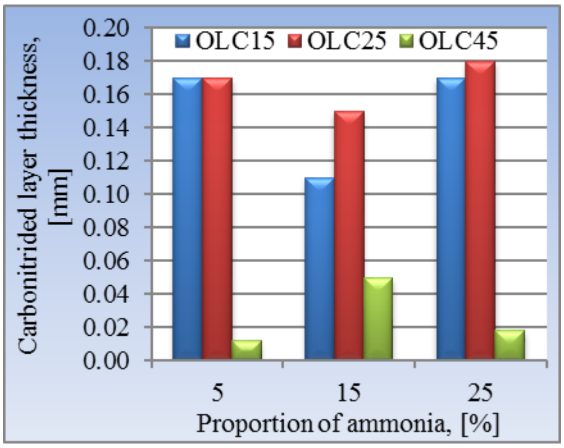

Fig. 3. *Variation of the carbonitrided layer thickness at 650 °C according to the proportion of ammonia for the three steel types studied*

The variation of the carbonitrided layer thickness at 650 °C according to the percentage of ammonia for the three steel types studied is shown in Figure 3.

At this thermo-chemical treatment temperature, for samples of 1C15 and 1C25 it has been observed that the depth of the carbonitrided layer has the same value when the ratio of ammonia is 5%. For both steels, the layer depth decreases when the ammonia percentage increases to 15%, but with a greater decrease for 1C15. At a rate of 25% ammonia, an increase in the thickness of the carbonitrided layer can be observed in both steels, at levels higher than those recorded when the percentage of ammonia is 5%.

The samples of 45 1C reach a maximum depth of the carbonitrided layer in the conditions shown in Figure 3, where the percentage of ammonia is 15%.

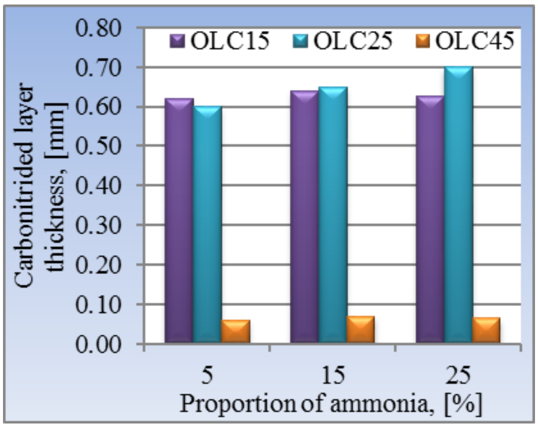

Fig. 4. *Variation of carbonitrided layer thickness at 750 °C, according to the proportion of ammonia for the three steel types studied*

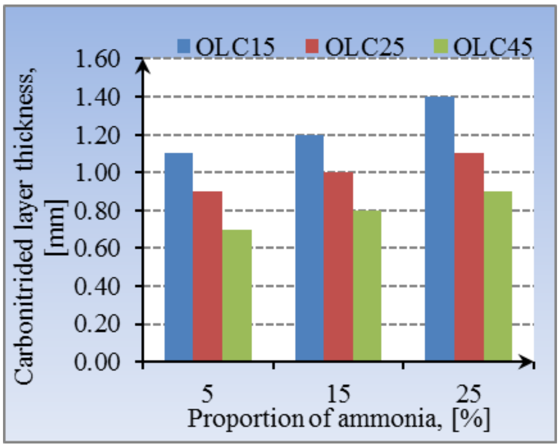

Fig. 5. *Variation of the carbonitrided layer thickness at 900 °C, according to the proportion of ammonia for the three steel types studied*

The results obtained by processing the samples at treatment temperature of 750 °C is shown in Figure 4. At this temperature of thermochemical treatment, for the samples of 1C25 it was observed that the carbonitrided layer depth increases with increasing the proportion of ammonia in the system. For the other two steels, a maximum in the depth of the layer can be noticed when the percentage of ammonia is 15%. Layer depth values for the samples of 1C15 are superior to those obtained for the samples of 1C45 steel.

Figure 5 shows the variation in thickness of the carbonitrided layer at 900 °C, according to the proportion of ammonia for the three steel types studied. The carbonitrided layer thickness increases with the increasing of ammonia percentage, for all three types of steel studied. The highest values were obtained for 1C15 in all cases, while the lowest values were obtained for steel 1C 45. In this case, it can be concluded that the carbonitrided layer thickness decreases as the percentage of carbon in the samples studied increases. At this temperature of 900°C, the nitriding process prevails and, normally, the increase of the proportion of ammonia makes its pervading to take place deeper in the samples.

Figure 6 shows the microstructure of a OLC 15 steel sample with the highest carbonitrided layer (1.4 mm) obtained at a temperature of 900 °C for a percentage of 25% ammonia.

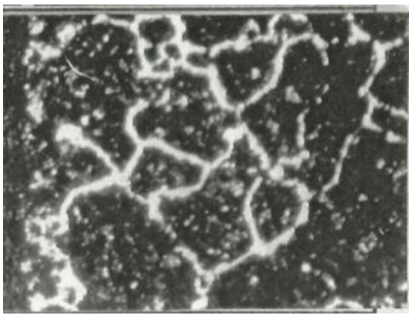

Fig. 6. *1C15 steel microstructure carbonitrided in fluidized layer at 900 °C / 2 h, 30min, 100 X magnification, attack NITAL 2%*

Figure 7 shows the microstructure of a sample of OLC 25 thermo-processed at a temperature of 750 °C with a hold time of two hours and 30 minutes, and with an intake of ammonia 25% in the carbonitriding. At 750 °C, for this sample it was obtained the highest carbonitrided layer of all samples from the three steels studied.

Figure 8 shows the microstructure of the OLC 45 sample with the greatest thickness of the carbonitrided layer, which was obtained by processing at a temperature of 900 °C / 2h, 30min with a content of 25% ammonia.

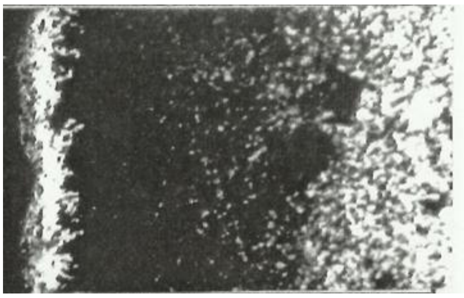

Fig. 7. *1C25 steel microstructure carbonitrided in fluidized layer at 750 °C / 2h, 30min, 100 X magnification, attack NITAL 2%*

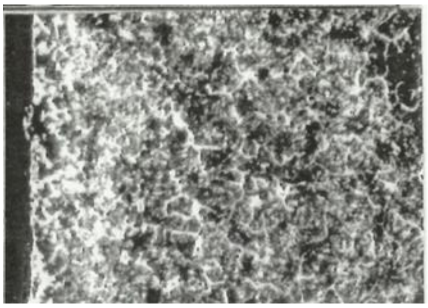

Fig. 8. *Microstructure of 1C45 steel carbonitrided in fluidized layer at 900 °C / 2h, 30 min, 100 X magnification, attack NITAL 2%*

4. Conclusions

Experimental research conducted revealed that at a fluidized bed carbonitriding temperature of 900 °C, for a processing time of 2 hours and 30 minutes and with a percentage of ammonia of 25%, there were obtained the highest depths of carbonitrided layer for the samples of three steel grades studied.

For the regimes at 900 °C, the carbonitrided layer thickness increases as the carbon content decreases in the samples investigated. At this temperature, the highest layer thickness is recorded for the samples of 1C 15. Also at 900 °C it is observed that with increasing the proportion of ammonia in the system, there occurs an increased thickness of the carbonitrided layer, too.

At 750 °C for IC 25 samples, it can be noticed that with the increase of the percentage of ammonia, there is an increase in the thickness of the carbonitrided layer. However, the difference between the maximum thickness of the carbonitrided layer in similar conditions of time and percentage of ammonia, at 900 °C and 750 °C is of 0.5 mm, which is not a neglectable difference.

An important advantage owned by heat treatment and fluidized bed thermochemical facilities is the large range of operating temperatures. Another advantage is the ability to vary within wide limits the initial composition of the gas, respectively the actual composition of the fluidized bed. Therefore, a single installation can make a large number of thermochemical treatments simply by altering the composition of gases entering the fluidized bed.

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