

USE OF TAGUCHI METHODS FOR HIERARCHY OF INFLUENCE FACTORS IN THE APPLICATION OF CARBONITRATION IN A FLUIDIZED BED STEEL FOR 41Cr4 STEEL

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ABSTRACT

The work is based on carbonitriding in a fluidized layer with methane and ammonia gas applied to 41Cr4 steel samples. To achieve the best possible results, other factors must be taken into account, than those specific to carbonitriding. It was necessary to use a partially factorial working procedure due to the increase in the number of factors. An A18 matrix has been used, with 18 lines of experiments in which three levels have been modified for 6 factors. The objective function was fixed at the final hardness after carbonitriding, hardening and tempering. The carbonitriding was performed in fluidized bed with granular solid ground and sorted to 0.1- to 0.16mm, and brought to fluidization with a mixture of methane and ammonia. The hardening has been done directly after carbonitriding in the fluidized bed. The experiments have been performed according to Taguchi Methods and the results indicate an optimal regime and the influence of the factors considered on the hardness after the thermo-chemical treatment and the thermal treatment. Metallographic analysis has been performed on the carbonitrided layer.

KEYWORDS: fluidized bed, Taguchi method (T M), 41Cr4, carbonitriding

1. Introduction

The fluidized bed is used as a medium for heat treatments of small bulk parts providing high heat transfer and mass capacities. High heat and mass transfer coefficients lead to balancing treatment costs by drastically reducing the process time. The limitations brought by the fluidization are related to the dimensions of the fluidization spaces and implicitly to the dimensions of the parts, and last but not least to the treatment costs. The quality indicators are influenced by the positioning of the active surfaces in relation to the axis of the fluidization chamber [1].

41Cr4 steel is an alloy steel for improvement but to which subsequent surface treatments can be applied. Usually this steel is intended for: - machine parts and heat-treated parts with a guaranteed hardening depth according to the brand's quality curve such as: smooth shafts, grooved shafts, crankshafts, bandages.

The chemical composition of 41Cr4 steel is: C = (0.36÷0.44)%; P = max 0.035%; Mn = (0.50÷0.80)%; S = max 0.035%, Si = (0.17÷0.37)%; Cr = (0.8÷1.1)%.

Also, some residual elements are accepted: max. 0.3% Ni; max. 0.3% Cu; max. 0.02% Ti (Table 1).

Forms of delivery: hot rolled flat products; semi-finished products for forging; rolled and drawn wires
Recommended heat treatments:

Soaking annealing consisting of austenitization at T = (680 ÷ 720) °C followed by oven cooling.

Normalization for which austenitization is performed at T = (840 ÷ 870) °C followed by cooling in still air.

The most important treatment is martensitic volumetric hardening, which is done after austenitization at T = (820 ÷ 850) °C followed by cooling in water and with subsequent cooling in water or oil.

Afterwards, surface treatments can be made for nitriding, nitrocarburizing or local hardening by laser hardening.

Carbonitriding is considered the most widely used thermo-chemical treatment (surface heat treatment with diffusion) [1]. The presence of active nitrogen in the decomposition of ammonia makes it possible to increase the chemical activity of carbon at lower heating temperatures, which leads to a decrease in costs without affecting the structure/surface

properties complex. Carbonitriding leads to a controlled increase in surface carbon and it may lead to changes in surface properties relative to the base material if surface treatment is properly continued. The paper studies the situation in which, in order to increase the hardenability of the surface layer, a surface treatment is made prior to the hardening and recovery mentioned above. In this case, the fluidized bed is used as a carbonitriding medium, which brings new process control factors [2, 3]. The experimental procedure used is based on TM with orthogonal array [2] to determine the share of influencing factors on the overall heat treatment applied to 41Cr4 steel.

2. Experimental conditions

Samples of 41Cr4 steel have been used for experiments. The chemical composition is shown in Table 1 and it reveals the class of steels to which it belongs, namely steels with 1% Cr. The steel is intended for improvement, but the application prior to the improvement of a carbonitriding treatment versus the increase of the calcification of the surface layer

and thus, of the complex of superior surface properties, can be obtained [2-4].

It is a mass and energy transfer medium that can be very active which leads to high values of mass and energy transfer coefficients. For example, for an ITTSF furnace (5) with a fluidized layer with a diameter of 0.35 m and initial H/D ratio = 1.1 the temperature of the centre of a spherical sample with a diameter of 0.05 m introduced in the incubator with the temperature of 850 °C, shows that by increasing the fluidization speed $c =$ it occurs a significant decrease in equalization time to approx. 100 s of the sample temperature. For the same oven without a fluidized bed that has been transformed into an electric oven with a retort, the equalization time of the sample is 400 s. It is also found that the fluidization speed influences, within certain limits, the heat transfer process (Fig. 1).

Similarly, fluidization speed also influences the mass transfer coefficient, which becomes important in surface diffusion treatments such as carbonitriding (Table 2) [1].

Table 1. Chemical composition of 41Cr4 steel samples

C	Mn	Si	P	S	Cr	Ni	Cu	Mo	Al
0.4	0.6	0.3	0.019	0.02	0.97	0.2	0.27	0.065	0.015

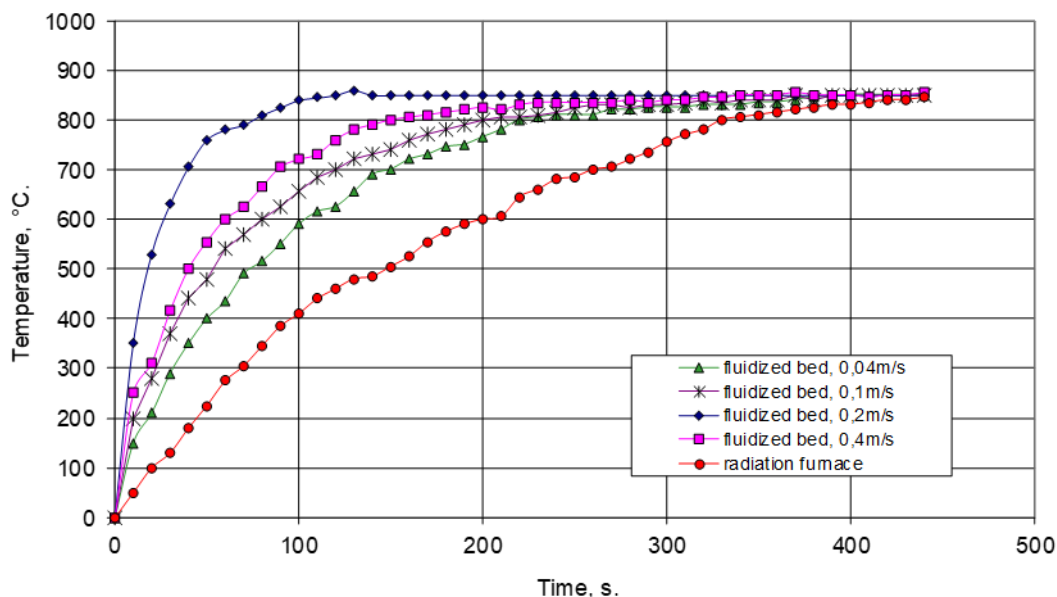


Fig. 1. Temperature variation of a stainless-steel spherical sample (2 inches) when introduced at an 85 °C in an electric furnace at and in a fluidized bed furnace (ITTSF (5), for different fluidization speed) [1]

Table 2. Fluidisation speed (levels for experiments)

no.	debit	under mesh speed	fluidisation speed
1	394 6.56	0.394	0.015 0.029
2	544 9.07	0.544	0.021 0.040
3	694 11.57	0.694	0.027 0.051

Table 3. Other factors that characterize used fluidized bed

parameter	symbol	value	m.u.
the average diameter of the solid	d_p	0.00024	m
form factor	ϕ	0.6	-
solid density	ρ_p	2650	kg/m ³
gas density	ρ_g	1.29	kg/m ³
kinematic viscosity	η	1.34E-05	m ² /s
gravitational acceleration	g	9.81	m/s ²
initial height of the bed	H	0.1	m
Inside diameter of the enclosure	D	0.096	m
Archimedes' criterion	Ar	1550.74	-
initial porosity	ϵ	0.52	-
fluidization speed	w	0.000154	m/s

Table 4. Factors considered to have significant influence over experiments

No.	factors	mu	range
1	carbonitriding temperature	°C	800...900
2	carbonitriding time	s	60...600
3	debit	l/h	433, 584, 734
4	tempering temperature	°C	100... 250
5	tempering time	min	5...15
6	position in furnace	-	low, medium, high
7	dimension of granular solid	m*10 ⁻³	0,10...0,16
8	fluidized bed volume	m ³	ct
9	steel chemical composition	%	ct.
10	fluidized bed shape rate D/H	-	1...1,5
11	ammonia rate	%	5...15
12	surface quality	-	small, medium, large

2.1. Experimental matrix

The multitude of factors influences the process of carbonitriding + hardening + tempering, to which are added the influencing factors of fluidization and carbonitriding in fluidized layer, making the factors in Table 5 to be selected from Tables 4. Previous experiments [3] for the carbonitriding of 41Cr4 steel in a fluidized layer with an initial gaseous mixture of methane and ammonia, showed the factors influencing the experiments and their fields of variation Table 4.

According to the procedure known as Taguchi Methods [6-8] when the number of factors is large, it is difficult to perform "full factorial" technological experiments due to increased costs, duration of experiments and their complexity, the use of experimental matrix being recommended. Using an appropriate mathematical model [6, 8], a matrix with a small number of experiments can be constructed.

For the proposed experiments we have the orthogonal matrix L18, with 18 lines, which has 8 factors considered and each three values. $3^8 = 6561$ experiments are replaced with 18 experiments.

Another property that results from the orthogonality of the matrix is that two columns can be omitted (column 1 and column 7) without influencing the result of the experiments, thus obtaining a matrix with 18 lines for 6 factors and each with three levels i.e., $3^6 = 729$ experiments are replaced with 18 experiments.

The results provide the weight of influence of the considered factors and they can be completed with a set of full-factorial experiments on a much smaller

number of factors and with smaller domains of variation [6, 8].

Orthogonal matrix L18 [6, 8] is presented in Table 6. Completing the L18 matrix with the values of the factor levels it will lead to the experiment matrix with 18 lines Table 7. The main difficulty of this experimental matrix is that the transition from one line to another is done by modifying the settings for several factors, which in the "full factorial" type experiment matrix means changing only one factor.

Table 5. Selected factors over experiments

no.	factors	m.u.	selected levels		
			1	2	3
1	carbonitriding temperature	°C	800	850	900
2	carbonitriding time	min	1	5	10
3	debit	l/h	394	544	694
4	tempering temperature	°C	100	150	200
5	tempering time	min	5	10	15
6	ammonia rate	%	5	10	15

Table 6. Typical orthogonal array L18 for 6 factors and each 3 levels

Experiment	Factori specificati							
	1	2	3	4	5	6	7	8
	e	A	B	C	D	E	e	F
1	1	1	1	1	1	1	1	1
2	1	1	2	2	2	2	2	2
3	1	1	3	3	3	3	3	3
4	1	2	1	1	2	2	3	3
5	1	2	2	2	3	3	1	1
6	1	2	3	3	1	1	2	2
7	1	3	1	2	1	3	2	3
8	1	3	2	3	2	1	3	1
9	1	3	3	1	3	2	1	2
10	2	1	1	3	3	2	2	1
11	2	1	2	1	1	3	3	2
12	2	1	3	2	2	1	1	3
13	2	2	1	2	3	1	3	2
14	2	2	2	3	1	2	1	3
15	2	2	3	1	2	3	2	1
16	2	3	1	3	2	3	1	2
17	2	3	2	1	3	1	2	3
18	2	3	3	2	1	2	3	1

Table 7. Experimental matrix with 18 lines

experiment	specified factors					
	1	2	3	4	5	6
	carbonitriding temperature	carbonitriding time	debit	tempering temperature	tempering time	ammonia rate
	A	B	C	D	E	F
m.u.	°C	min	l/h	°C	min	%
1	800	1	394	100	5	5
2	800	5	544	150	10	10
3	800	10	694	200	15	15
4	850	1	394	150	10	15
5	850	5	544	200	15	5
6	850	10	694	100	5	10
7	900	1	544	100	15	15
8	900	5	694	150	5	5
9	900	10	394	200	10	10
10	800	1	694	200	10	5
11	800	5	394	100	15	10
12	800	10	544	150	5	15
13	850	1	544	200	5	10
14	850	5	694	100	10	15
15	850	10	394	150	15	5
16	900	1	694	150	15	10
17	900	5	394	200	5	15
18	900	10	544	100	10	5

Table 8. Hardness determination for each experiment and target function calculus

experiment	hardness		average hardness	target function
	HV1	HV2		
m.u	kgf/mm ²	kgf/mm ²	kgf/mm ²	dB
1	336	341	339	25.30
2	781	857	819	29.13
3	701	655	678	28.31
4	667	667	667	28.24
5	795	857	826	29.17
6	946	1027	987	29.94
7	825	825	825	29.16
8	781	781	781	28.93
9	810	891	851	29.30
10	494	509	502	27.00
11	874	810	842	29.25
12	857	825	841	29.25
13	713	689	701	28.46
14	825	857	841	29.25
15	891	874	883	29.46
16	752	781	767	28.85
17	726	701	714	28.53
18	739	766	753	28.77
			m=	28.68

Table 9. Calculus for average effect of each level for each factor over experiments

relation	value
mA1= $1/6(n1+n2+n3+n10+n11+n12)=$	28.04
mA2= $1/6(n4+n5+n6+n13+n14+n15)=$	29.09
mA3= $1/6(n7+n8+n9+n16+n17+n18)=$	28.92
mB1= $1/6(n1+n4+n7+n10+n13+n16)=$	27.83
mB2= $1/6(n2+n5+n8+n11+n14+n17)=$	29.04
mB3= $1/6(n3+n6+n9+n12+n15+n18)=$	29.17
mC1= $1/6(n1+n4+n9+n11+n15+n17)=$	28.35
mC2= $1/6(n2+n5+n7+n12+n13+n18)=$	28.99
mC3= $1/6(n3+n6+n8+n10+n14+n16)=$	28.71
mD1= $1/6(n1+n6+n7+n11+n14+n18)=$	28.61
mD2= $1/6(n2+n4+n8+n12+n15+n16)=$	28.98
mD3= $1/6(n3+n5+n9+n10+n13+n17)=$	28.46
mE1= $1/6(n1+n6+n8+n12+n13+n17)=$	28.40
mE2= $1/6(n2+n4+n9+n10+n14+n18)=$	28.61
mE3= $1/6(n3+n5+n7+n11+n15+n16)=$	29.03
mF1= $1/6(n1+n5+n8+n10+n15+n18)=$	28.10
mF2= $1/6(n2+n6+n9+n11+n13+n16)=$	29.15
mF3= $1/6(n3+n4+n7+n12+n14+n17)=$	28.79
m=	28.68

Table 10. Average effect of each level of each factor

factors	m.u.	levels		
		1	2	3
A carbonitriding temperature	dB	28.04	29.09	28.92
B carbonitriding time	dB	27.83	29.04	29.17
C debit	dB	28.35	28.99	28.71
D tempering temperature	dB	28.61	28.98	28.46
E tempering time	dB	28.40	28.61	29.03
F ammonia rate	dB	28.10	29.15	28.79
	average	m=	28.68	

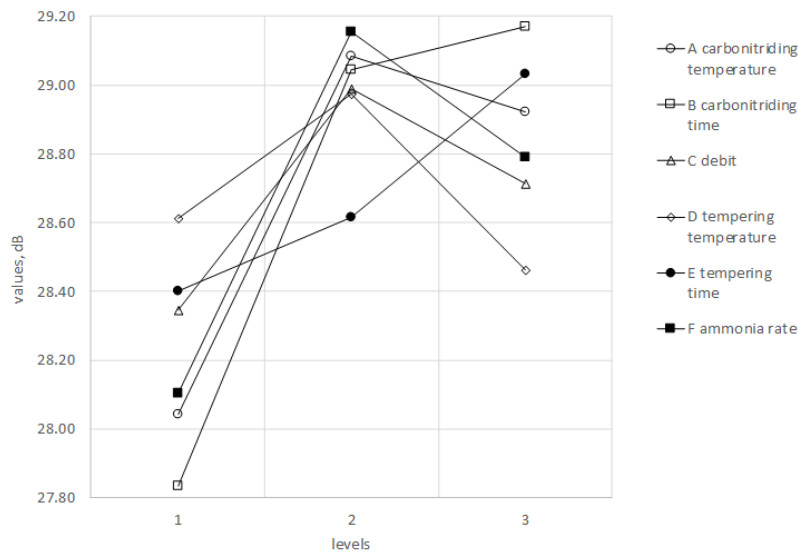


Fig. 2. Graphical representation for average effect of each level of each factor

Table 11. ANOVA Analysis

	factor	SSDTF	weight of factor
	m.u.	(dB) ²	%
A	carbonitriding temperature	3.79	22.06
B	carbonitriding time	6.53	37.98
C	debit	1.25	7.27
D	tempering temperature	0.84	4.86
E	tempering time	1.25	7.25
F	ammonia rate	3.53	20.57
	Total	17.18	

3. Experiments

The experiments have been performed following the experimental matrix from Table 7. After completing the 18 experiments on steel sample cabs thus treated, hardness measurements were made on the flat surface, far from the edges (Table 8). For each experiment, the objective function was calculated with the relationship specific to the dependence "the greater is the better" (it is desired to achieve the highest hardness):

$$n = 10 \log (HV_m)$$

The average objective function was calculated for all 18 experiments (Table 8). An important step in the procedure is to calculate the average effect of the level of each factor for all 18 levels (6 factors x3 levels). This is shown in Table 9. It should be noted that the properties of orthogonal matrices also include the frequency of identical occurrence in the matrix for each level of each factor, which means that simplifications of the mathematical model do not influence the achievement of the technological objective by more than a few percent [6, 8].

The reorganisation of the values of the mean effect of each factor as in Table 10, allows an intuitive graphical representation (Fig. 2). This graphical representation shows how each factor influences the selected range of variation, respectively the allure of the influence (linear ascending, linear decreasing, with maximum, with minimum, without influence, etc.).

4. Results and discussion

The metallographic analysis of the surface layers after carbonitriding is shown from Fig. 5 to Fig. 20.

The microstructures show the combined influence of factors A, B, C and F that influence the

carbonitriding process in the fluidized layer. An analysis of the micrographs shows that:

- for carbonitriding temperatures of 800 °C (experiments 1, 2, 3, 10, 11 and 12);
- for carbonitriding temperatures of 850 °C experiments 4, 5, 6, 13, 14 and 15;
- for carbonitriding temperatures of 900 °C experiments 7, 8, 9, 16, 17 and 18.

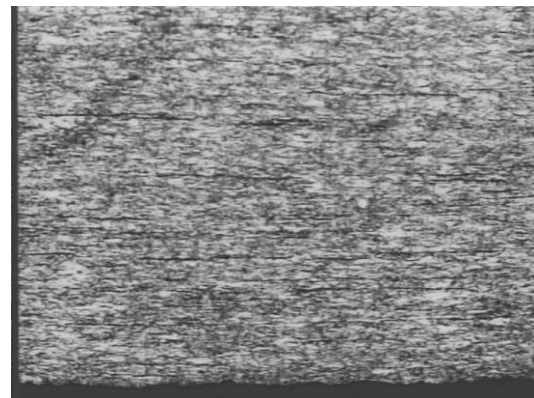


Fig. 3. Heat and thermochemical treatment according to regime 1 (magnification 50x, attack: nital 2%)

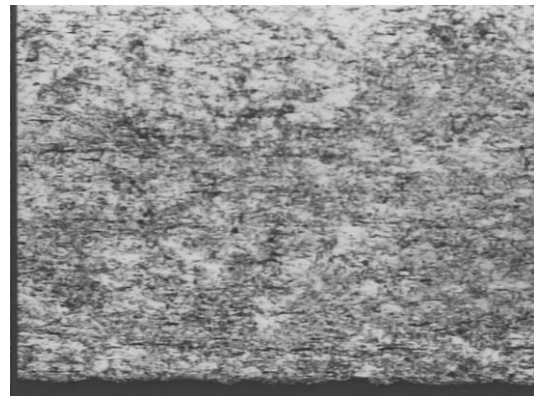


Fig. 4. Heat and thermochemical treatment according to regime 2 (magnification 50x, attack: nital 2%)

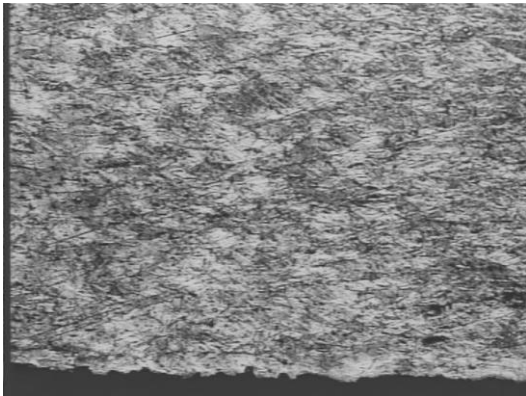


Fig. 5. Heat and thermochemical treatment according to regime 3 (magnification 50x, attack: nital 2%)

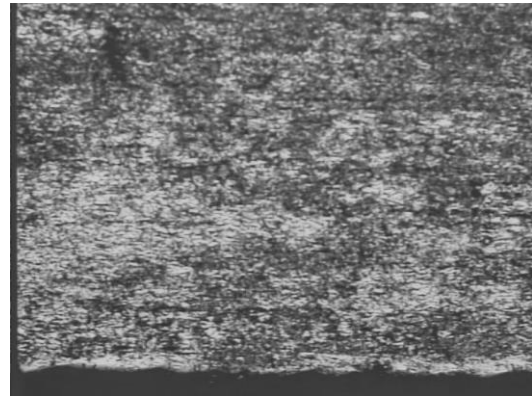


Fig. 8. Heat and thermochemical treatment according to regime 6 (magnification 50x, attack: nital 2%)

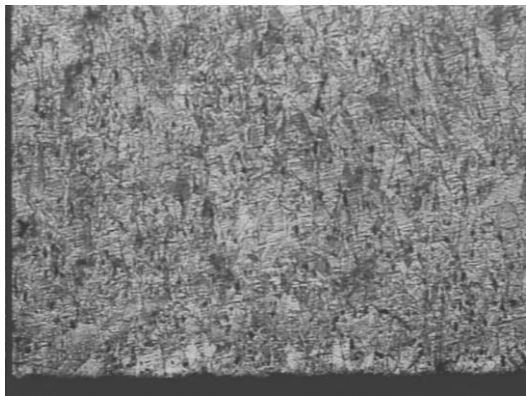


Fig. 6. Heat and thermochemical treatment according to regime 4 (magnification 50x, attack: nital 2%)

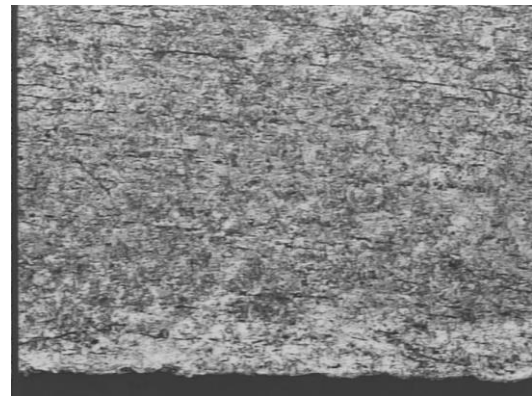


Fig. 9. Heat and thermochemical treatment according to regime 7 (magnification 50x, attack: nital 2%)

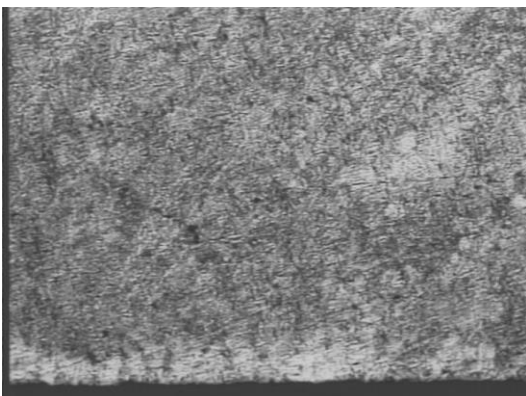


Fig. 7. Heat and thermochemical treatment according to regime 5 (magnification 50x, attack: nital 2%)

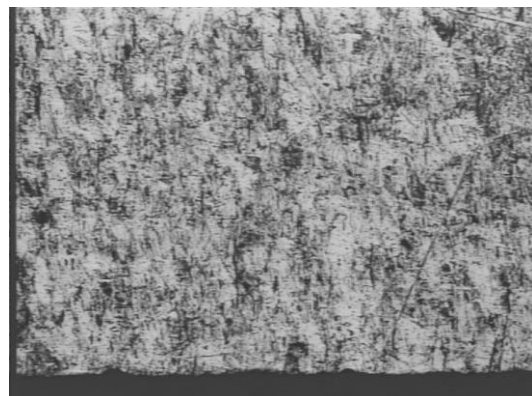


Fig. 10. Heat and thermochemical treatment according to regime 8 (magnification 50x, attack: nital 2%)

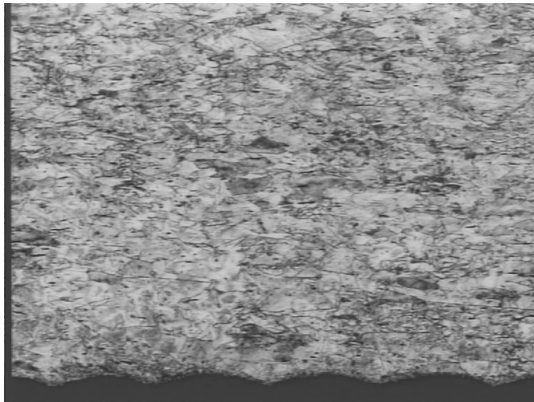


Fig. 11. Heat and thermochemical treatment according to regime 9 (magnification 50x, attack: nital 2%)

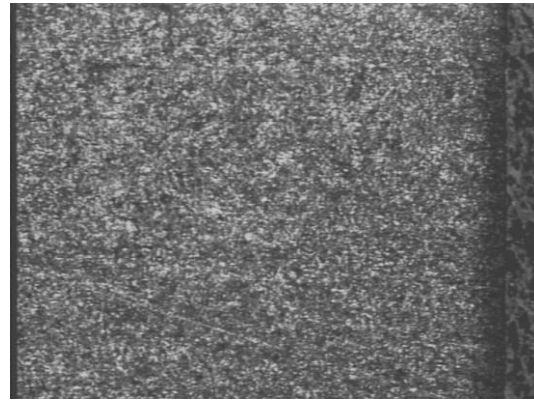


Fig. 14. Heat and thermochemical treatment according to regime 12 (magnification 50x, attack: nital 2%)

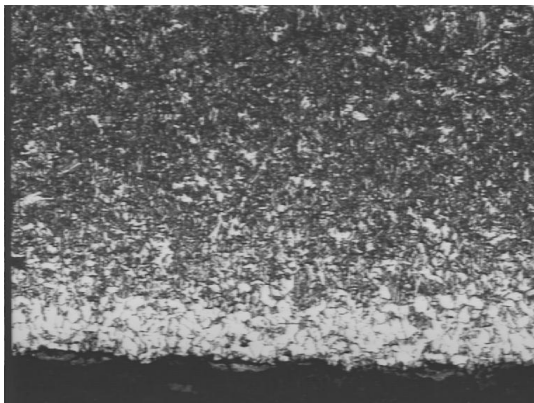


Fig. 12. Heat and thermochemical treatment according to regime 10 (100x magnification, attack: nital 2%)

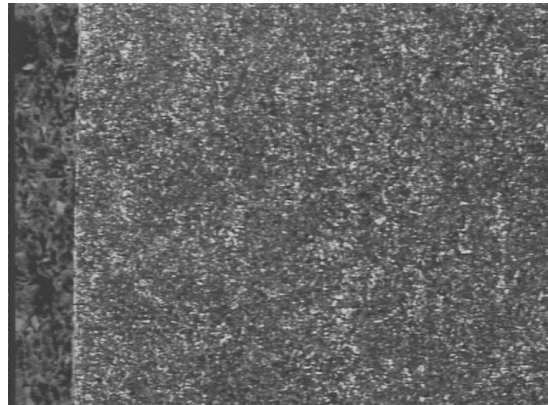


Fig. 15. Heat and thermochemical treatment according to regime 13 (100x magnification, attack: nital 2%)

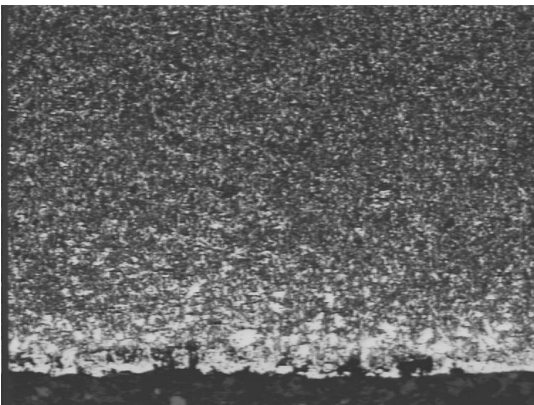


Fig. 13. Heat and thermochemical treatment according to regime 11 (magnification 100x, attack: nital 2%)

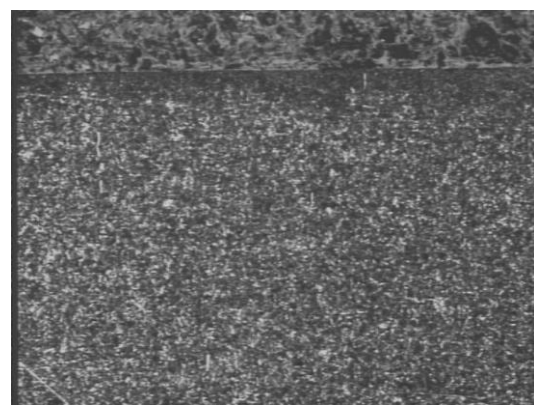


Fig. 16. Heat and thermochemical treatment according to regime 14 (100x magnification, attack: nital 2%)

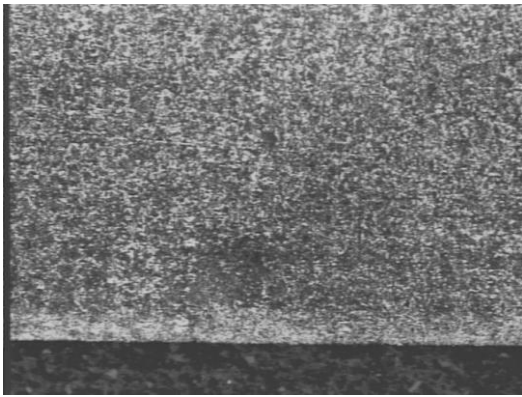


Fig. 17. Heat and thermochemical treatment according to regime 15 (magnification 100x, attack: nital 2%)

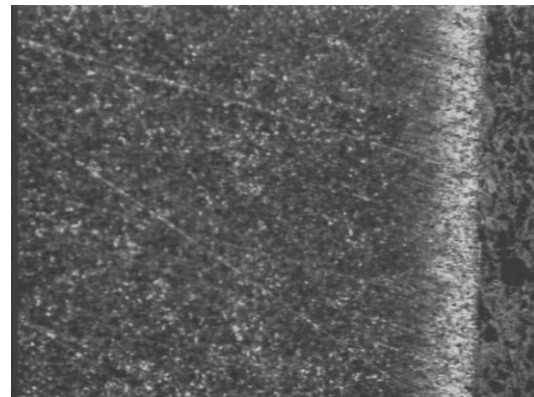


Fig. 20. Heat and thermochemical treatment according to regime 18 (100x magnification, attack: nital 2%)

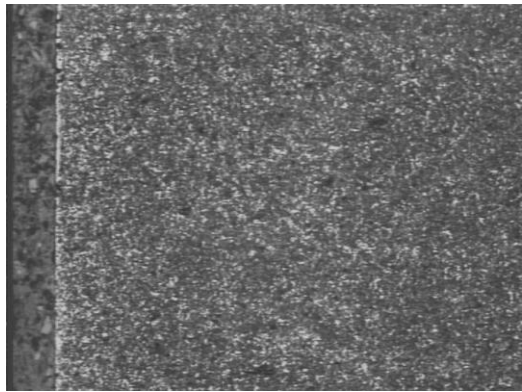


Fig. 18. Heat and thermochemical treatment according to regime 16 (magnification 100x, attack: nital 2%)

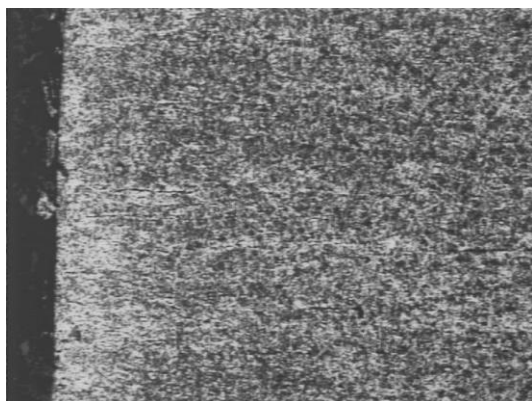


Fig. 19. Heat and thermochemical treatment according to regime 17 (magnification 100x, attack: nital 2%)

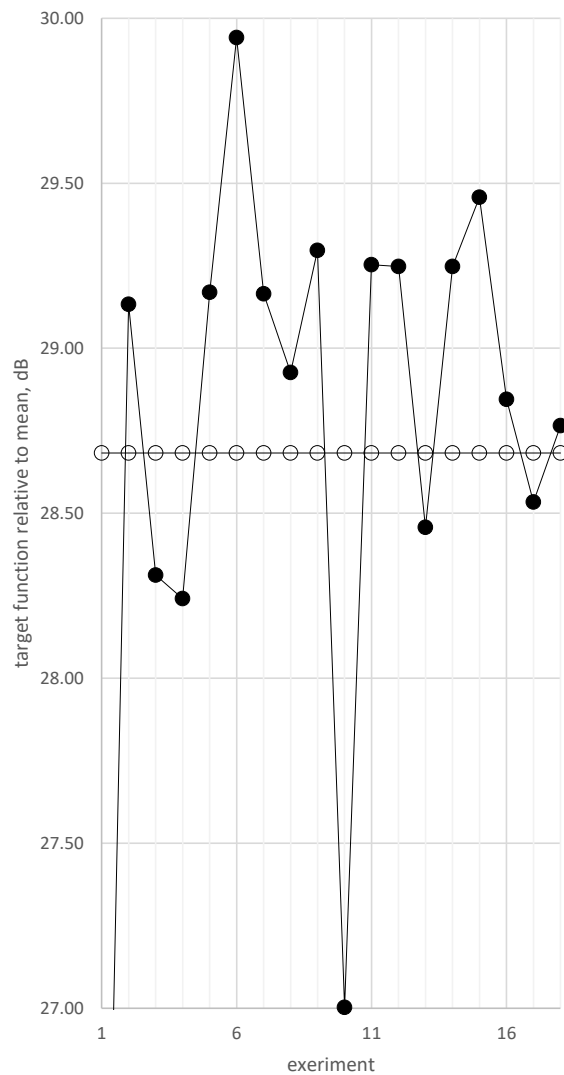


Fig. 21. Target function variations over experiments and mean

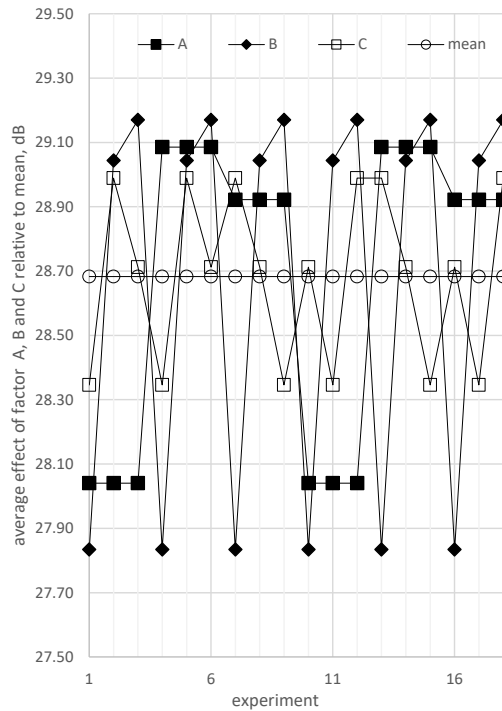


Fig. 22. Average effect of factor A, B and C over experiments relative to mean

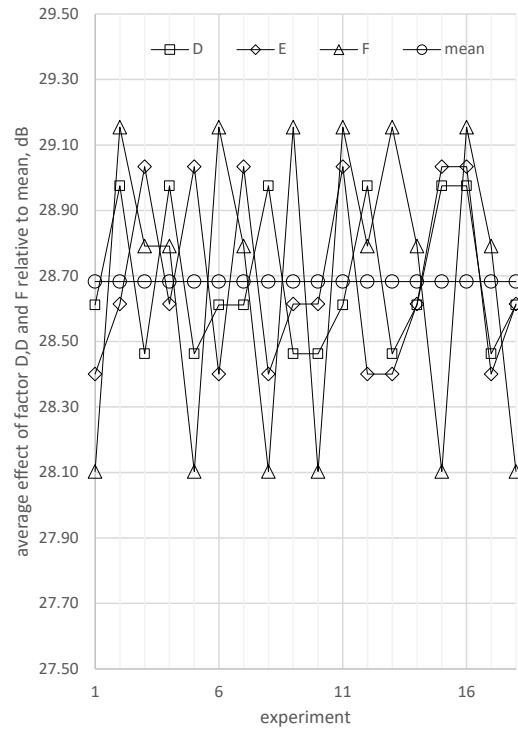


Fig. 23. Average effect of factor E, F and G over experiments relative to mean

Table 13. Optimal setting for each level of each factor

factors		levels			
		m.u.	1	2	3
A	carbonitriding temperature	dB		29.09	
B	carbonitriding time	dB			29.17
C	debit	dB		28.99	
D	tempering temperature	dB		28.98	
E	tempering time	dB			29.03
F	ammonia rate	dB		29.15	
		average	m=	9.69	

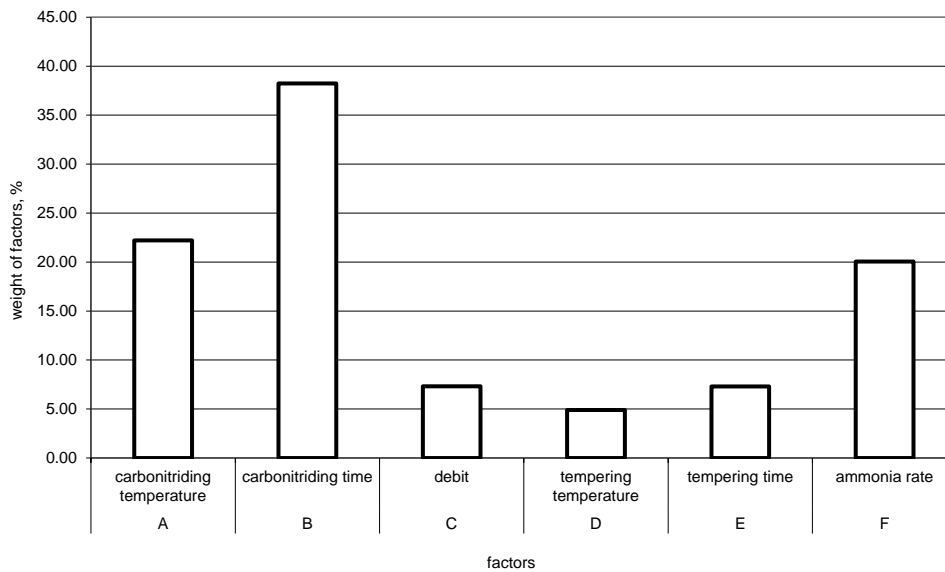


Fig. 24. Weight of factors over experiments

4. Conclusions

The carbonitride layer at high temperatures has a structure similar to the carburized layer (microstructures made before the final hardening) (Fig. 5-20) nitrogen increases the diffusion coefficient of carbon simultaneously with the decrease of the treatment temperature.

Carbonitriding temperatures allow a direct hardening from that temperature, with the advantage of the open chamber used for fluidization.

The mathematical experimentation procedure in orthogonal matrix with its afferent mathematical model offers the different importance of the factors on the experiment, the character of the influence (linear, logarithmic, with maximum / minimum) (Fig. 2).

The microstructures confirm the realization of the thermal and thermochemical treatment processes.

The highest hardness is obtained at temperatures of 850 °C and a holding time of 10 min (regime 6) (Table 7, Fig. 8). The lowest hardness has been obtained at temperatures of 800 °C and a holding time of 1min (Table 7, Fig. 3). The influence of temperature and time on carbonitriding is most obvious and is in line with recognized technologies [9, 10].

The greatest influence on a carburized layer has carbonitriding temperatures, carbonitriding time and the proportion of ammonia. These factors can be considered centroid factors, for which the measurement accuracy is significantly increased.

The least influence on the carburized layer has the inlet gas flow, tempering temperature and return time (Fig. 22).

Experiments show once again that the use of fluidized layers in heat and thermochemical treatments reduces the duration of treatments directly (through specific heat and mass transfer phenomena)

and indirectly by reducing loading / unloading times due to working with the enclosure open (Table 7).

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