"Boost-diffusion" vacuum carburising – Process optimisation

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ABSTRACT

The article presents the optimisation of the organisation of the vacuum carburising process in terms of the number and duration of "boost"/"diffusion" stages. The processes of two- and multi-segment carburising were examined with regard to the process duration and effectiveness of carbon transfer from the atmosphere to the charge surface. It was shown that division of the process into two segments (1 boost + 1 diffusion) results in extending the total process time, regardless of the carburising temperature. The total time and effectiveness of the carburising process depends on the minimum, momentary surface concentration of carbon, achieved in the diffusion stage.

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1. Introduction

Vacuum carburising has been gaining importance as a modern, environmentally-friendly and cost-effective method of surface hardening in the global machinery, automobile and aviation industry [1–3]. This technological expansion is founded on thorough exploration of the mechanisms of the phenomena, describing them with physical and mathematical models and solving them by numerical methods [4–7]. The process of vacuum carburising involves the supplying of carbon to the surface in a cycle of "boost" stages followed by "diffusion" segments [8–18]. Both the transfer and transport of carbon in "boost" and "diffusion" stages take place in transient state conditions. Carbon, used to build up the vacuum-carburised layers, comes from the carbon deposit which forms during the "boost" stage on the surface and in the thin layer near-the-surface as a result of the high carbon potential of the hydrocarbon-based atmosphere (Fig. 1). The deposit appears in two morphological phases – a thin, surface film of higher hydrocarbons and carbide formations on the surface and along the grain borders [5,6,19,20]. Decomposition of the hydrocarbons in the "diffusion" stage is fast – part of the deposit is dehydrated and adsorbed on the surface, whereas part is hydrated and leaves the chamber with off-gas. The process of carbon transfer to the surface is controlled by the carbides' dissolving rate rather than by the hydrocarbon's decomposition [5,6,20].

Key factors which affect the rate of build-up of vacuum-carburised layers include temperature and carbon concentration gradients in austenite. Carburization can be achieved by providing a high momentary concentration of carbon on the surface and in the thin layer near-the-surface, such a concentration being close to the maximum solubility of carbon in austenite. This in turn reduces the ability of austenite to absorb further carbon. It may result in formation in the process of microstructure with remnants of non-dissolved carbides – which is unacceptable in using the carburised objects [3,6,20].

The aims of the study are to develop recommendations for the optimum "boost/diffusion" segment arrangement for the effective growth of vacuum-carburised layers with the proper microstructure – layer without carbide precipitation.

The research problems dealt with in this article include:

- analysis of the organisation of a two- and multi-segment "boost/diffusion" process (Fig. 1);
- examination of the effectiveness of the formation of thick carburised layers by choosing the optimum momentary values of the minimum concentration of carbon in austenite at the surface – $C_{min} = 0.8\% \div 1.2\%$ C (Fig. 1);
- examination of the effect of the organisation of the "boost/diffusion" process on the effectiveness of carbon transfer from the processing atmosphere to steel.

2. Material and methods

2.1. Two-segment processes

The aim of the study was to compare the duration of two-segment and multi-segment processes. To this end, low-pressure carburising processes were conducted. For the carburizing trials

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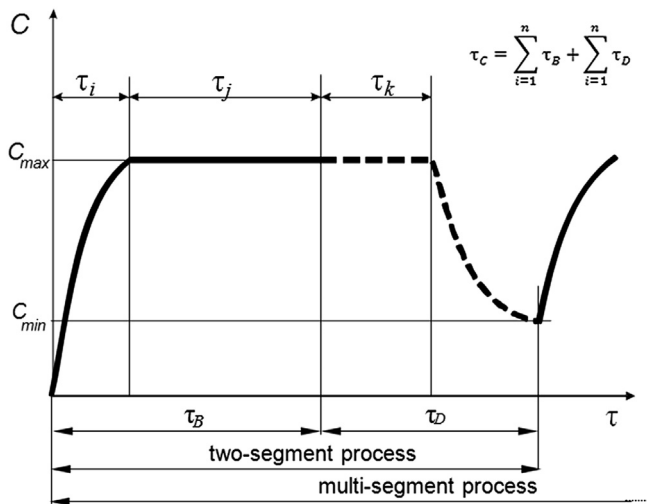
Fig. 1. Change of the surface concentration of carbon at different stages of steel vacuum carburising, where: τ_i – time before the maximum solubility of carbon in austenite is reached, τ_j – time of carbon deposit formation, τ_j – time of carbon deposit decomposition, τ_B – time of the "boost" segment, τ_D – time of the "diffusion" segment, τ_C – total time of the process, C_{max} – limit of carbon solubility in austenite at the process temperature, C_{min} – minimum concentration of carbon achieved at the "diffusion" stage.

the steel EN 18CrNi8 was selected. The chemical composition of the steel is shown in Table 1.

The steel was subjected to vacuum carburising at the temperatures of 1193 K and 1273 K. The processes were run in a multi-purpose high pressure gas quenching (HPGQ) vacuum furnace. The carburising atmosphere was produced by mixing acetylene, ethylene and hydrogen at the proportion of 2:2:1 as described in [8,9]. The quantity of gas introduced to the furnace chamber was proportional to the charge size and the process temperature, in accordance with the dispensing characteristics $V = f(A, T)$, where V – gas flow rate [m^3/h], A – charge surface [m^2], T – process temperature [K]. Two-segment carburizing processes were performed, consisting of one segment of the "boost" and one segment of the "diffusion". The times of individual segments in the process with the temperature of 1193 K were: $\tau_B = 18$ min $+ \tau_D = 40$ min and in the process with the temperature of 1273 K were: $\tau_B = 18$ min $+ \tau_D = 20$ min. The division into segments of 18/40 (in the two-segment process) was based on the assumption carburized layer thickness, that we wanted to get. We wanted to obtain a layer thickness $a = 0.6$ mm (for the criterion C: 0.4% and a surface concentration: 0.8% C) at a temperature of 1273 K. The duration of the segments boost/diffusion of the two-segment process was identified experimentally. In the next step we used the same time for segments with a temperature of 1193 K to give a layer thickness = 0.35 mm. The processing atmosphere was introduced to the furnace chamber at pressure 5 hPa. Quenching was done after carburising, under a nitrogen atmosphere at a pressure of 1.35 MPa, directly at the process temperature. Then the carbon concentration profiles in the surface layer were determined. Carbon distribution in the carburised layer was determined by glow discharge optical emission spectrometry. The carbon profile was determined as a function of the distance to the surface, by grinding down consecutive layers, approximately every 10 μm.

Table 1
Chemical composition of the steel used in the experiment.

Steel	%C	%Si	%Mn	%Cr	%Ni	%Mo	%Al	%V	%Cu
EN 18CrNi8	0.18	0.24	0.52	1.99	2.03	0.03	0.04	0.00	0.31

2.2. Multi-segment processes

The carbon profiles obtained in two-segment processes were used to perform simulations of multi-segment processes, so that the same carbon profiles were obtained. The issues of layer modelling in multi-segment processes were dealt with in the SimVacPlus®[21] program, based on a numerical solution of the physical model of cyclic formation and decomposition of carbon deposit in diffusion in a semi-infinite medium [3]. Both the model and the program have been verified in numerous experimental studies and industrial applications [22,23,24,25].

Simulations of the complete process of vacuum carburising of 18CrNi8 steel were also performed with the SimVaCPlus® program. In the first instance, the process was designed to achieve a layer thickness of $a = 0.35$ mm (for the criterion of carburized layer = 0.4% C) at the temperature of $T = 1193$ K. Multi-segment processes were designed so as to achieve different levels of the minimum surface carbon concentration in the "diffusion" stage – $C_{min} = 0.8\%$, 0.9% C, 1.0% C and they were compared with the experimental two-segment processes: $\tau_B = 18$ min and $\tau_D = 40$ min run at the same temperature as the simulations.

In the second instance – to achieve the layer thickness of $a = 0.60$ mm (for the same criterion) at the temperature of $T = 1273$ K. Multi-segment processes were designed so as to achieve different levels of the minimum surface carbon concentration in the "diffusion" stage – $C_{min} = 0.8\%$, 1.0% C, 1.2% C and they were compared with the experimental two-segment processes: $\tau_B = 18$ min and $\tau_D = 20$ min run at the same temperature as the simulations.

In each case, the last diffusion segment C min was chosen so as to achieve a specific surface content of carbon and the layer thickness corresponding to the carbon profiles obtained in two-segment processes.

The results of the experiments and the simulation calculations are shown in Table 2 and Table 3.

2.3. Thick carburised layers

Then, simulations of multi-segment processes were performed in the SimVaCPlus programme to achieve thick carburised layers. Multisegment processes were designed to achieve different levels of C min from 0.8% to 1.2% C. The process temperature was set at 1273 K and a thickness = 2.0 mm and 4.0 mm (for the criterion carburized layer = 0.4% C).

The aim of the study was to determine the effectiveness of carburisation as a method of achieving layers of great thickness.

Table 2
The segments of vacuum carburising processes obtained for steel 18CrNi8 at 1193 K for different variants of a minimum surface concentration of carbon (C min) in stage of diffusion. Effective case depth $a = 0.35$ mm (for criterion 0.4% C).

C min	Process1 (simulation)	Process2 (simulation)	Process3 (simulation)	Process4 (real)
	0.80%	0.90%	1.00%	2-Segment process
Temperature	1193 K	1193 K	1193 K	1193 K
Number of cycles	6	7	8	1
Segments duration [mm:ss]				
Boost	11:30	13:20	15:20	18:00
Diffusion	45:35	37:44	32:55	40:00
Eff. case depth (crit 0.4%). [mm]	0.35	0.35	0.35	0.35
Surface [%]	0.88	0.88	0.88	0.88
Total duration [mm:ss]	**57:05**	**51:04**	**48:15**	**58:00**

Table 3
The segments of vacuum carburising processes obtained for steel 18CrNi8 at 1273 K for different variants of a minimum surface concentration of carbon (C min) in the stage of diffusion. Effective case depth $a = 0.58$ mm (for criterion 0.4% C).

C min	Process1 (simulation)	Process2 (simulation)	Process3 (simulation)	Process4 (real)
	0.80%	1.00%	1.20%	2-Segment process
Temperature	1273 K	1273 K	1273 K	1273 K
Number of cycles	4	5	7	1
Segments duration [mm:ss]				
Boost	11:30	13:00	16:00	18:00
Diffusion	41:20	30:15	25:49	20:00
Eff. case depth (crit 0.4%). [mm]	0.58	0.58	0.60	0.60
Surface [%]	1.04	1.04	1.04	1.04
Total duration [mm:ss]	**52:50**	**43:15**	**41:49**	**38:00**

Table 5
The segments of vacuum carburising processes obtained for steel 18CrNi8 at 1273 K for different variants of a minimum surface concentration of carbon (C min) in the stage of diffusion. Effective case depth $a = 4.0$ mm (for criterion 0.4% C).

C min	Process1 (simulation)	Process2 (simulation)	Process3 (simulation)
	0.80%	1.00%	1.20%
Temperature	1273 K	1273 K	1273 K
Number of cycles	40	54	72
Segment duration [mm:ss]			
Boost	01:02:00	01:21:31	02:25:00
Diffusion	2.10:41:36	1.17:08:51	1.08:48:00
Eff. case depth (crit 0.4%). [mm]	4.00	4.03	4.00
Surface [%]	0.80	0.81	0.81
Total duration [d.hh:mm:ss]	**2.11:43:36**	**1.18:30:22**	**1.11:13:00**

The "effectiveness" applies both to the process duration and to the balance of carbon transported from the atmosphere to the charge surface.

To this end, the value describing the balance has been defined as an effective coefficient of carbon transfer (coeff C_{eff}). The value of C_{eff} was calculated as the ratio of the amount of carbon introduced to steel and the total amount of carbon introduced to the furnace chamber during the "boost" stages. The values both of the dividend and the divisor are referred to the unit charge area (1 m^2).

$$C_{eff} = C_C/C_A$$

where: $C_A = C_C + C_E + C_B C_A$ — total amount of carbon introduced to the furnace chamber in the "boost" stages, C_C — the amount of carbon absorbed by the charge for the build-up of the carburised layer, C_E — total amount of carbon released from the chamber with off-gas, C_B — the amount of carbon in by-products (soot, tar), deposited on cold chamber walls and on the pumping system. The results of calculations conducted for thick layers are shown in Table 4 and 5.

3. Results and discussion

3.1. A two-segment vs. a multi-segment process

The division into "boost" and "diffusion" segments is of key importance in the optimisation of the duration of a carburising process for obtaining the assumed distribution of carbon and the required layer structure. Decreasing the surface concentration of

Table 4
The segments of vacuum carburising processes obtained for steel 18CrNi8 at 1273 K for different variants of a minimum surface concentration of carbon (C min) in stage of diffusion. Effective case depth $a = 2.0$ mm (for criterion 0.4% C).

C min	Process1 (simulation)	Process2 (simulation)	Process3 (simulation)
	0.80%	1.00%	1.20%
Temperature	1273 K	1273 K	1273 K
Number of cycles	19	25	39
Segment duration [mm:ss]			
Boost	00:31:00	00:38:00	01:01:00
Diffusion	/13:06:13	/09:20:33	/07:33:11
Eff. case depth (crit 0.4%). [mm]	2.0	2.0	2.0
Surface [%]	0.8	0.8	0.8
Total duration [hh:mm:ss]	**13:37:13**	**09:58:33**	**08:34:11**

carbon in the "diffusion" stage (C_{min} in Fig. 1), to a level that is too low, extends the process time, but it also ensures full decomposition of the carbides formed. A high concentration of carbon at the end of the "diffusion" stage, should theoretically reduce the process time. However, it also increases the risk for carbides left undissolved.

The distribution of carbon in the surface layer of 18CrNi8 steel following two-segment vacuum carburising, $\tau_B = 18$ min $+ \tau_D = 40$ min at the temp. 1193 K and 1273 K was shown in Fig. 2. The illustrations Fig. 3a and b present a comparison of the duration of multi-segment and two-segment processes to examine the effectiveness of such a division.

As is shown by Fig. 3, increasing the threshold of minimum carbon surface concentration results in a reduction of the whole process duration, both at low and high temperatures. However, using a two-segment division — one "boost" and one "diffusion" segment — extends the total process time τ_C.

This is especially apparent for lower carburising temperatures. It is because large amounts of carbides, formed in one long "boost" segment, must be dissolved during a single "diffusion" segment.

The surface carbon concentration at the end of the "diffusion" stage affects not only the duration of the carburising process, but it can also affect the carbon profile in the surface layer of the carburised steel (Fig. 4. This is especially true for low-temperature processes, when dissolving of carbides is slower than the carbon diffusion stream in austenite.
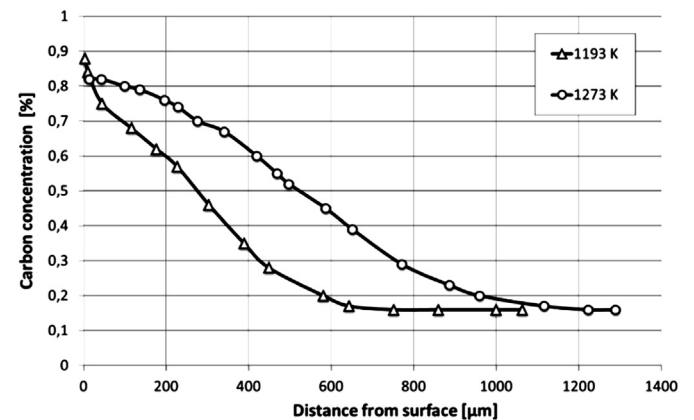

Fig. 2. Distribution of carbon in the surface layer of 18CrNi8 steel following two-segment vacuum carburising, $\tau_B = 18$ min $+ \tau_D = 40$ min at the temp. 1193 K and 1273 K.
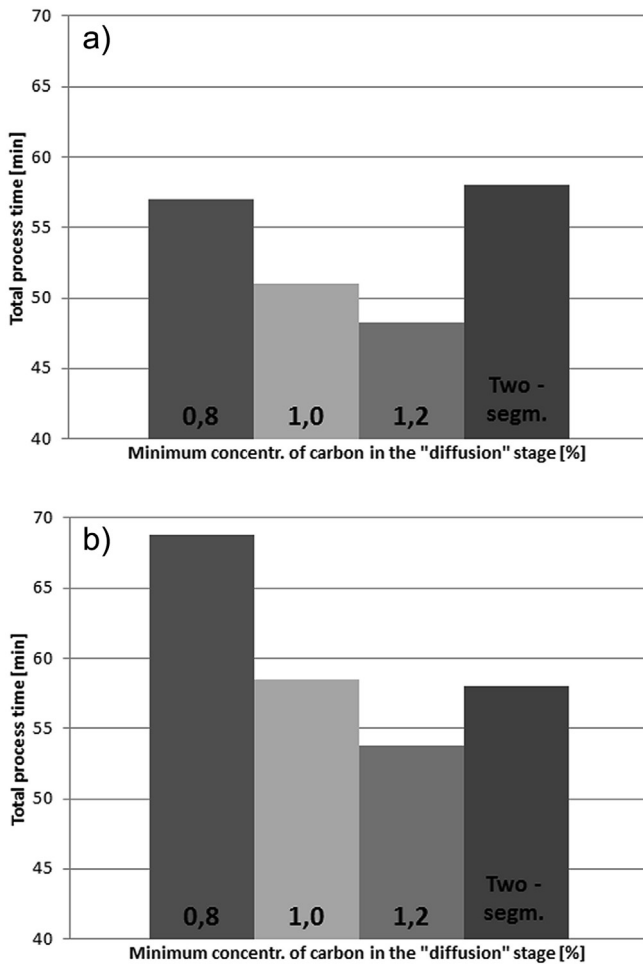
Fig. 3. Comparison of the total time of multi-segment carburising of 18CrNi8 steel for different variants of the minimum concentration of carbon and a two-segment process, a) Process temperature 1193 K, thickness of the carburised layer 0.35 mm b) process temperature 1273 K, thickness of the carburised layer 0.65 mm.

The essence of reduction of the time for a carburising process is its division into segments and maintaining the right carbon surface concentration in the "diffusion" stage. The research results have shown that using a two-segment division leads, most of the time, to unjustified extension of the process time.
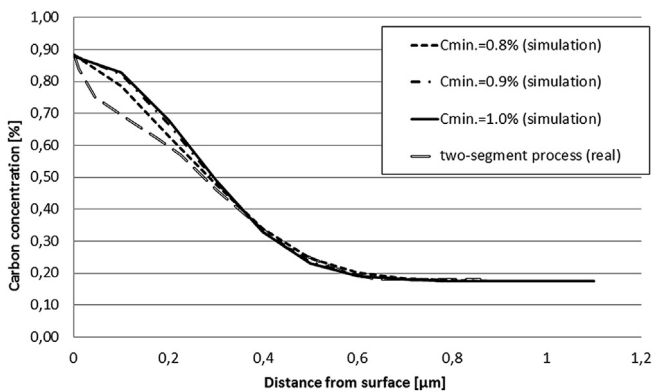

Fig. 4. Carbon profiles obtained in two- and multi-segment process of vacuum carburising of 18CrNi8 steel at the temp. of 1193 K, differing by the value of the minimum surface concentration of carbon, achieved in the "diffusion" stage.

Achieving a specific value of C_{min} in the "diffusion" segment enables modelling the carbon profile while maintaining the same layer criterion (thickness, surface concentration) (Fig. 4). After thermal treatment, it results in specific technological properties of the surface layer.

3.2. Thick carburised layers

Differences in the process duration in carburising thin layers (as shown in Fig. 3) for each variant C_{min} are relatively small (not more than a dozen minutes). However, the differences are significant for the variants of treatment performed to obtain thick layers (over 2 mm). Considering this, it was justifiable to examine the effectiveness of carburising in terms of the momentary, minimum surface concentration, obtained in the "diffusion" stage. A comparison of the carburising process duration times is shown in Fig. 5.

The differences in the duration of the multi-segment carburising for different variants of C_{min} are significant (Fig. 5). The difference between the shortest and the longest process, needed to obtain a carburised layer of $a = 2.0$ mm is equal to 5 h. However, the process can be longer by as much as 24.5 h to obtain a 4.0 mm thick layer. As in short carburising processes (Fig. 3a and b), it is the optimum option to maintain the surface carbon concentration at the end of the "diffusion" stage at a level of around 1.2%.

Apart from the process duration, the effectiveness of carburising should also be considered in terms of the balance of carbon transported from the atmosphere to the charge surface C_{eff}. The value of the coefficient, as a function of the carbon concentration gradient in the "diffusion" stage, calculated for different layer thickness values, is shown in Fig. 6. The coefficient C_{eff} is the highest for the minimum surface concentration of 0.8% C. Nevertheless, carbon is effectively transferred when $C_{min} = 1.2\%$ C. Therefore, it is the reverse relationship to the process duration. The above shows that in order to optimise the carburising process in terms of the process duration, as well as efficiency of carbon transfer, the coefficient of carbon transfer per unit process duration should be determined ($\psi = C_{eff}/\tau_c$).

Fig. 7a and b shows the change of the coefficient of carbon transfer efficiency per unit process duration for different layers of thickness obtained. The functions which describe the coefficient of carbon transfer per process time unit (Fig. 7a and b) reach their maximum for C_{min} equal to about 0.95% C. Therefore, the optimum multi-segment process of thick layer carburising should be run such as to maintain $C_{min} \approx 0.95\%$ C.

'Formation of thick layers requires using high temperatures in the carburising process and the choice of the optimum C_{min} in the
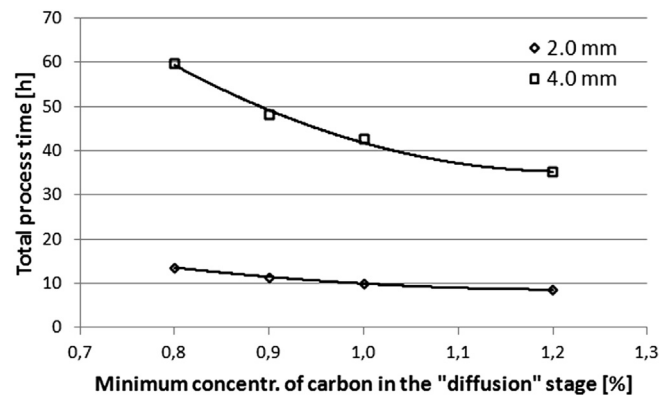

Fig. 5. Comparison of the total time of multi-segment vacuum carburising for different variants of the minimum surface concentration of carbon for the layer thickness of 2.0 and 4.0 mm 18CrNi8 steel, process temperature 1273 K.
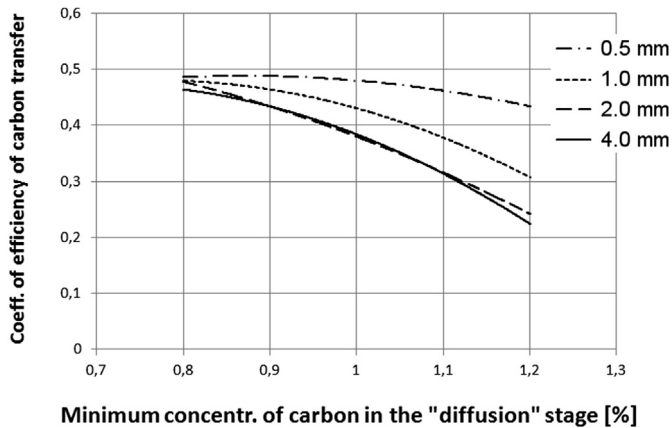
Fig. 6. The relationship between the carbon transfer efficiency (C_{eff}) and different variants of the minimum surface concentrations (C_{min}) achievable in the "diffusion" stage for different thicknesses of the carburised layers. 18CrNi8 steel, process temperature 1273 K.

"diffusion" stages, with a view to reduction of the total process time and achieving a high efficiency of carbon transfer from the atmosphere to the charge surface. The effectiveness is associated with the process cost-effectiveness, but it also has a positive effect on environmental protection by reducing the total amount of carbon emitted from the chamber with off-gas. Moreover, the amount of

Fig. 7. Relationship between carbon transfer efficiency per process time unit (C_{eff}/τ_C) and the minimum surface concentration (C_{min}) obtainable in the "diffusion" stage for the layer thickness: a) 2.0 mm and b) 4.0 mm 18CrNi8 steel, process temperature 1273 K.

by-products (soot, tar), deposited on the cold furnace chamber walls and the pump system will also decrease, which has an effect on the process cleanness and the reliability of devices.

The experiment leads one to the conclusion that − considering all the factors − the optimum option involves a multi-segment division in which the minimum carbon concentration in the "diffusion" segments should be about 0.95% C at the process temperature of 1273 K (Fig. 7). It is the best compromise between reducing the duration time of the process and making the optimum use of the carbon potential of the carburising atmosphere.

4. Conclusions

1. The optimum option involves a multi-segment division in which the minimum carbon concentration in the "diffusion" segments should be about 0.95% C at the process temperature of 1273 K
2. The process division into two segments (1 boost + 1 diffusion) results in an extension of the total process time, regardless of the carburising temperature.
3. The total time and effectiveness of the carburising process depend on the minimum, momentary surface concentration of carbon, achieved in the diffusion stage.
4. The minimum concentration of carbon achieved in the "diffusion" stage affects the profile of the carbon produced during the carburising process − this can be used to control the surface layer properties. Maintaining a high surface concentration during the diffusion phase may result in incomplete dissolving of carbides in the layer despite the total concentration of carbon being below the solubility boundary of carbon in austenite.

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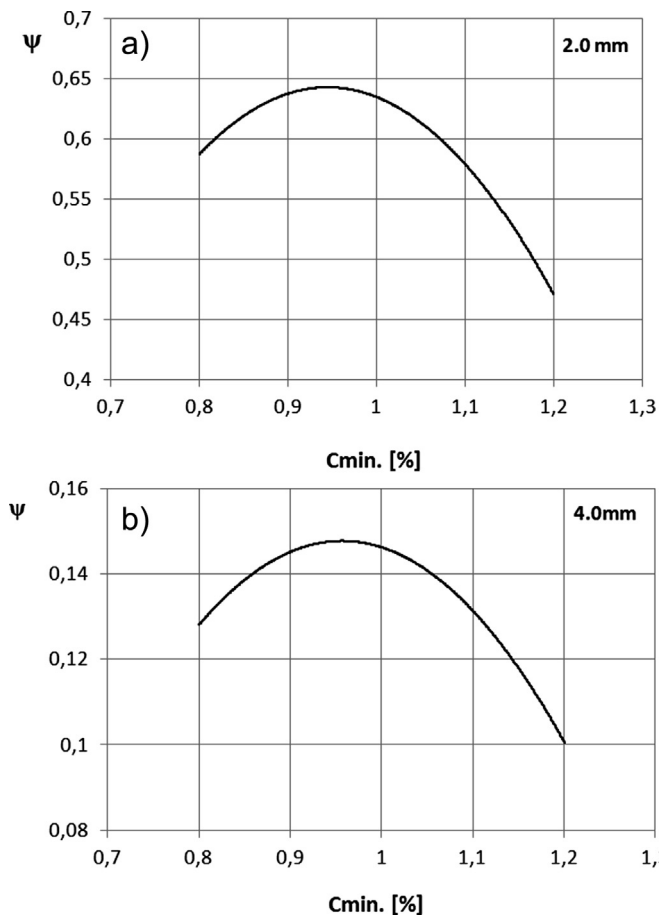[25] Kula P, Pietrasik R, Dybowski K, Paweta S, Wolowiec E. Adv Mater Res 2012;452−453:401−6.