

Influence of the Temperature of Vacuum Carburizing on the Thickness of the Carburized Layer and Properties of Steel Parts

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ABSTRACT

The properties of steel parts after carburizing in low-pressure acetylene are studied. The thickness of the carburized layer, its hardness, the microstructure, and the hardness of the core are determined after carburizing at 900 – 960°C with simultaneous shortening of the process from 104 to 44 min. The data obtained for the diffusion of carbon make it possible to predict the profiles of carbon distribution over the thickness with good enough accuracy.

Key words: vacuum carburizing; low-pressure gas carburizing, thickness of carburized layer; profile of carbon concentration

INTRODUCTION

Precision gears are important components determining the operating capacity and reliability of various parts and mechanisms of the aerospace industry. To provide the specified surface hardness, toughness of the core and dimensions of the part, the heat treatment is conducted in several stages. A typical heat treatment cycle of precision gears involves three stages, i.e., vacuum carburizing for raising the concentration of carbon in the surface layer, quenching for martensite, and tempering for improving the ductility and the toughness [1]. Carburizing is usually conducted at a temperature exceeding A_{c3} . Typical carburizing temperatures (800 – 1050°C) provide a high solubility of carbon in the austenite. As a rule, the carbon content in the surface layer is 0.8 – 1.0%. At higher carbon concentrations, the structure of the steels acquires an excess content of retained austenite or a grain boundary net of hypoeutectoid cementite. This results in flaking of the surface and early failure of the gear [2]. Quenching from the carburizing temperature causes formation of martensite both in the carburized layer and in the core.

Use of low-pressure gas in carburizing (as a rule, the process is called vacuum carburizing) has become quite popular in the last twenty years. This is explainable by the higher efficiency and improved ecological characteristics of vacuum carburizing as compared to the conventional process. The use of acetylene as a source of carbon for carburizing eliminates the problems of soot and tar formation typical for other hydrocarbon atmospheres, provides high quality and surface finish, structural homogeneity, reproducibility, rapid saturation of the surface layers with carbon, lowering of the gas consumption, and shortening of the duration of the process [3, 4]. Vacuum carburizing in an acetylene atmosphere is a diffusion-controlled process, because the dissociation of acetylene in the atmosphere of the furnace and the adsorption of the formed carbon on the surface occur

much more rapidly than carbon diffusion in the steel [5]. In vacuum carburizing, the decomposition of acetylene occurs under nonequilibrium conditions, which makes conventional methods of control of the furnace regime inapplicable. Computer simulation based on diffusion models is used to predict the parameters of the process which should provide the required thickness of the carburized layer and profile of carbon distribution over this thickness.

The aim of the present work was to study the possibilities of shortening of the total time of carburizing for precision gears by raising the temperature of the process without lowering the thickness of the carburized layer and its hardness and without worsening of the microstructure.

METHODS OF STUDY

Simulation. Rate of carbon diffusion in low-pressure gas carburizing. With allowance for the variation of the concentration profile with time, the steady stage of the diffusion is described by the equation

$$\frac{\delta C}{\delta t} = D \frac{\delta^2 C}{\delta x^2}, \quad (1)$$

where $\frac{\delta^2 C}{\delta x^2}$ is the rate of variation of the concentration gradient with distance $x = \frac{\delta C}{\delta t}$ is the variation of the volume concentration with time t , and D is the diffusivity of carbon at the carburizing temperature.

Equation (1) represents the second Fick's law [6]. The gradient of the carbon concentration varies with the absorption of carbon from the atmosphere by the surface and with carbon diffusion into the depth of the specimen.

The diffusion of carbon is described by the equation

$$\frac{C_s - C_x}{C_s - C_0} = \operatorname{erf}\left(\frac{x}{2\sqrt{Dt}}\right), \quad (2)$$

where C_0 is the initial carbon concentration in the steel, C_s is the carbon concentration on the surface of the steel, C_x is the carbon concentration at distance x from the surface, D is the diffusivity of carbon at the carburizing temperature, t is the carburizing time (in seconds), and $\operatorname{erf}(x)$ is the Gauss error function.

Equation (2) represents a well-known approximate solution of the second-order diffusion equation described by the second Fick's law. This solution is based on simplifying assumptions, and we should mention the following three associated complications [7].

1. The diffusivity D is not only a function of the temperature, but also of the concentration. For this reason, the actual concentration profiles differ from those calculated by the second Fick's law.

2. In many kinds of carburizing, the temperature and the concentration of carbon on the surface may vary with time. After the initial carburizing period, the surface concentration of carbon and the temperature commonly become lower because a part of the carbon has already diffused into the volume of the billet.

3. The alloying elements present in the steel (Mn, Cr, Si, Ni, Mo) may affect substantially the diffusion of carbon.

A mathematical model for predicting carbon concentration profiles under gas carburizing of steel has been suggested in [8]. The model allows for the temperature, the content of the alloying elements in the steel, and the variation of the carbon content in the billet. It is suggested to calculate the diffusivity of carbon by the equation

$$D = \frac{1.43 \exp\left(-\frac{19,700}{T}\right) \exp\left[0.0024 \exp\left(\frac{6790}{T}\right) (\%C)\right]}{1 - 0.232 (\%C)}, \quad (3)$$

where T is the absolute temperature in K. Equation (3) describes the dependence of the diffusivity of carbon in austenite as a function of the temperature and of the carbon content in the steel. However, it has been shown in [9] that the diffusivity of carbon is affected by the alloying elements present in the steel. The authors of [10] think that the activity of carbon in the steel a_s should be corrected with the help of coefficient q calculated from the equation

$$\begin{aligned} q = & 1 + [\%Si] (0.15 + 0.033 [\%Si]) + \\ & 0.0365 [\%Mn] - [\%Cr] (0.13 - 0.0055 [\%Cr]) + \\ & [\%Ni] (0.03 + 0.00365 [\%Ni]) - \\ & [\%Mo] (0.025 + 0.01 [\%Mo]) - \\ & [\%Al] (0.03 + 0.02 [\%Al]) - \\ & [\%Cu] (0.016 + 0.0014 [\%Cu]) - \\ & [\%V] (0.22 + 0.01 [\%V]), \end{aligned} \quad (4)$$

where the concentration of the alloying element is given in mass percent. The effect of the alloying components on carbon diffusion in equation (3) can be allowed for by multiplying the calculated diffusivity D by coefficient q for the given steel composition.

At the same conditions of carburizing, the carbon activity grows with decrease of parameter q . On the contrary, the lower the value of q the lower the carbon potential needed for maintaining a constant activity of carbon in the atmosphere. Consequently, the value of q may be used as a parameter of the efficiency of carburization of steel in a vacuum furnace.

In [7], the process of vacuum carburizing was simulated under the assumption that it occurs in two stages, i.e., carburization at a specific content of the gas filling the furnace chamber and subsequent diffusion that redistributes the initial concentration profile. The experimental concentration profiles plotted in this work match well the carbon concentrations calculated with the help of the equation

$$D = (0.07 + 0.06 (\%C)) \exp\left(-\frac{32,000}{RT}\right), \quad (5)$$

where $R = 1.99 \text{ cal} \cdot \text{mole}^{-1} \cdot \text{K}$ is the universal gas constant.

The diffusivity of carbon in iron and steel at the temperatures of 975 – 1075°C has been studied in [11]. The author suggests the following empirical equation for the diffusivity of carbon in austenite:

$$D = 0.47 \exp(-1.6\%C) \exp\left(-\frac{37,000 - 6600\%C}{RT}\right) q. \quad (6)$$

The value of q in this equation can be calculated by formula (5).

Later on, the diffusivity of carbon was calculated in [4] with the help of an equation

$$D = 0.47 \exp(-1.6\%C) \exp\left(-\frac{154.8 - 27.63\%C}{RT}\right) q. \quad (7)$$

The authors of [4] performed a comparative analysis of experimentally measured carbon concentration profiles and profiles calculated by the models suggested in [7, 8, 11]. The calculated data coincided well with the experimental results. It has also been reported that the carbon concentration profiles calculated in [8] with the help of Eq. (3) agree with the experimental profiles best of all.

The total time of the carburizing process can be reduced by elevating the carburizing temperature. The final thickness of the carburized layer x can be calculated as a function of time for any temperature by the Einstein equation [12]

$$x = 1.414\sqrt{Dt}. \quad (8)$$

It should be noted that elevation of the treatment temperature with the aim to reduce its duration may raise the cost of operation of the furnace and cause inadequate grain growth in the carburized volumes. Consequently, it is necessary to find a compromise between the two parameters.

Experimental studies. We tested standard samples cut from carburizing steel Latrobe Lescalloy® 16NCD13 VAC-ARC (to be called 16NCD13 in what follows) from the same heat

[13]. Table 1 presents the chemical composition of the steel and the concentration ranges of its components.

Table 1. Chemical Composition and Permissible Range of Scattering of Component Concentrations in Steel 16NCD13 [13].

Value	Content of elements, wt.%								
	C	Mn	Si	S	P	Cr	Ni	Mo	Cu
Minimum	0.12	0.30	0.15	–	–	0.80	3.00	0.20	–
Maximum	0.17	0.60	0.40	0.010	0.015	1.10	3.50	0.30	0.35
Actual	0.15	0.45	0.23	0.010	0.015	1.03	3.48	0.25	0.13

To study the carburizing process, we used the following two types of test bars.

1. A standard V-type test bar (Fig. 1) imitating the profile of a gear, i.e., the tip, the flank and the root of the tooth.

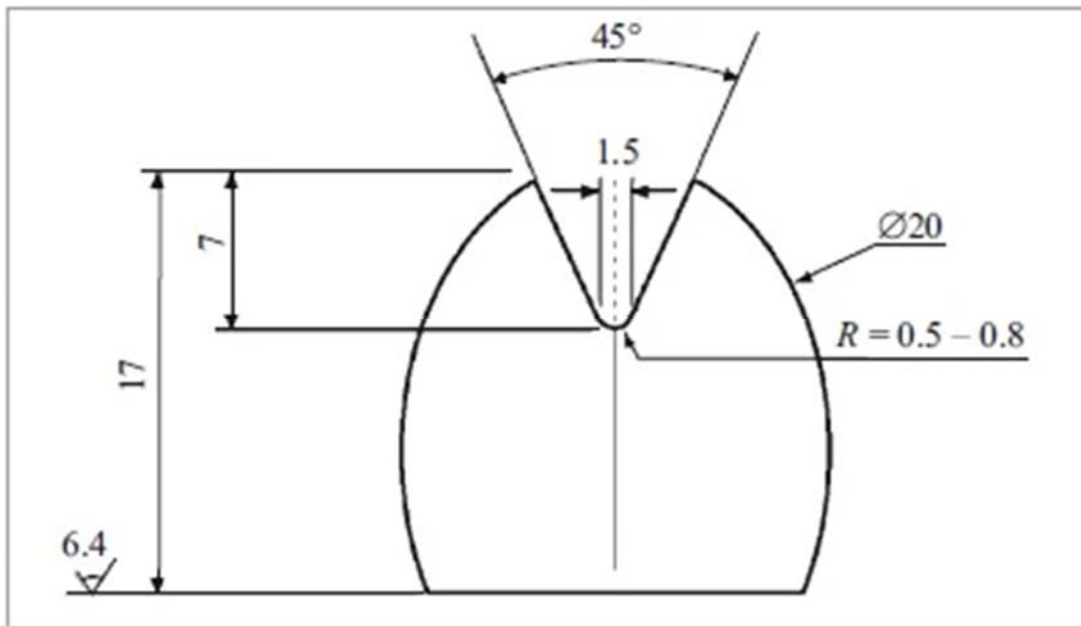


Fig. 1. Standard V-type test bar for simulating the carburization of gears.

2. A standard bar with diameter 20 mm and a length 120 mm for determining the carbon concentration profile.

We used three test pieces for carburizing in each test and one control test piece for determining the variation of the concentration of carbon over the thickness. The test pieces were placed into the furnace together with iron scrap imitating a standard charge with a mass of 30 – 50 kg.

The carburizing process was conducted in a single-chamber vacuum furnace with working space 600 × 900 × 600 mm. The furnace provided convective heating at 150 – 850°C and vacuum heating at 500 – 1320°C. The modes of vacuum carburizing in an acetylene atmosphere (C₂H₂) were as follows: 900°C for 104 min, 920°C for 95 min, 940°C for 64 min, and 960°C for 44 min. The subsequent heat treatment involved annealing at 670°C for 3 h, quenching in a high-pressure gas (10 bar N₂) from 820°C (45 min) and tempering at 140°C for 3 h.

The carbon potential in the furnace atmosphere was maintained at a maximum level of 1.4% to provide the surface carbon concentration of 0.8 – 0.9%. This prevented formation of a cementite net over grain boundaries. Annealing was applied to imitate the conventional process of gear production. The mode of the annealing provided the possibility of machining and grinding of the carburized gears before the quenching and tempering cycle.

The carburizing temperature was raised by 20°C for each test in the range from 900 to 960°C. The duration of the carburization at each temperature was chosen so as to provide a constant thickness of the carburized layer equal to 0.5 mm. The other parameters of the process were kept constant.

The commercial process for carburizing precision gears in low-pressure gas consists of several stages. At the start of the process the billets after the mechanical treatment are charged into the furnace. Then the working chamber of the furnace is evacuated to a pressure below 0.7 mbar. After the removal of excess oxygen, the temperature in the furnace is first increased to 650°C and then (when the temperature is levelled) to 850°C at a nitrogen pressure of 2 bar. Then the billets are heated by convection. After the hold at 850°C, nitrogen is evacuated from the chamber, the pressure in the furnace is lowered to less than 1 mbar, the temperature is raised to the specified value, and acetylene is fed into the furnace. The pressure in the stage of carburization is usually maintained at a level of 0.1 mbar. In this stage, carbon is absorbed by the austenite and hydrogen is liberated. As soon as the austenite is saturated with carbon, the feeding of acetylene is stopped after the designed hold, and carbon diffuses into the volume of the metal. In the stage of diffusion, acetylene is evacuated from the furnace.

When the carburization cycle is completed, the furnace temperature is lowered to 850°C. The charge is cooled rapidly in an argon atmosphere with a programmed rate of 60 – 65 K/min. When cooled, the billets are withdrawn from the furnace and placed into the second vacuum chamber, where they are annealed before grinding, which is conducted for obtaining the designed tooth shape with the required tolerances. The billets are annealed at 670°C for 3 h and cooled in a gas flow or under a fan in a nitrogen atmosphere at a pressure of 1.2 bar. After the finishing mechanical treatment, the billets are quenched from 820°C in nitrogen at a pressure of 10 bar and then tempered at 140°C for 3 h.

Having done all these operations, we studied the microstructure of the carburized layer and of the core of the test bars. To uncover the grains, we used the Marshall etchant [14]. The etching was conducted until the appearance of black deposit on the surface. The deposit was removed by polishing, and the process was repeated several times to obtain a

satisfactory image. The grain size was determined by the intercept method according to ASTM E112–13 [15]. We calculated the average grain size for each treatment temperature. The microstructure of the carburized samples was studied under an optical microscope at a magnification of $\times 500$. For this purpose the samples were covered with tar, polished with a diamond paste and etched in 4% nital (No. 74 in Standard [16]).

We measured the Vickers microhardness and determined the mechanical properties. The specimens for the tensile tests were cut from cylindrical test bars according to ASTM A370–14 [7].

RESULTS AND DISCUSSION

Figure 2 presents the experimental and calculated carbon concentration profiles for all the carburizing temperatures used. The curves have a form typical for carburizing and virtually similar for the different temperatures of the process. This shows that elevation of the carburizing temperature at the corresponding shortening of its duration does not change the depth of carbon penetration.

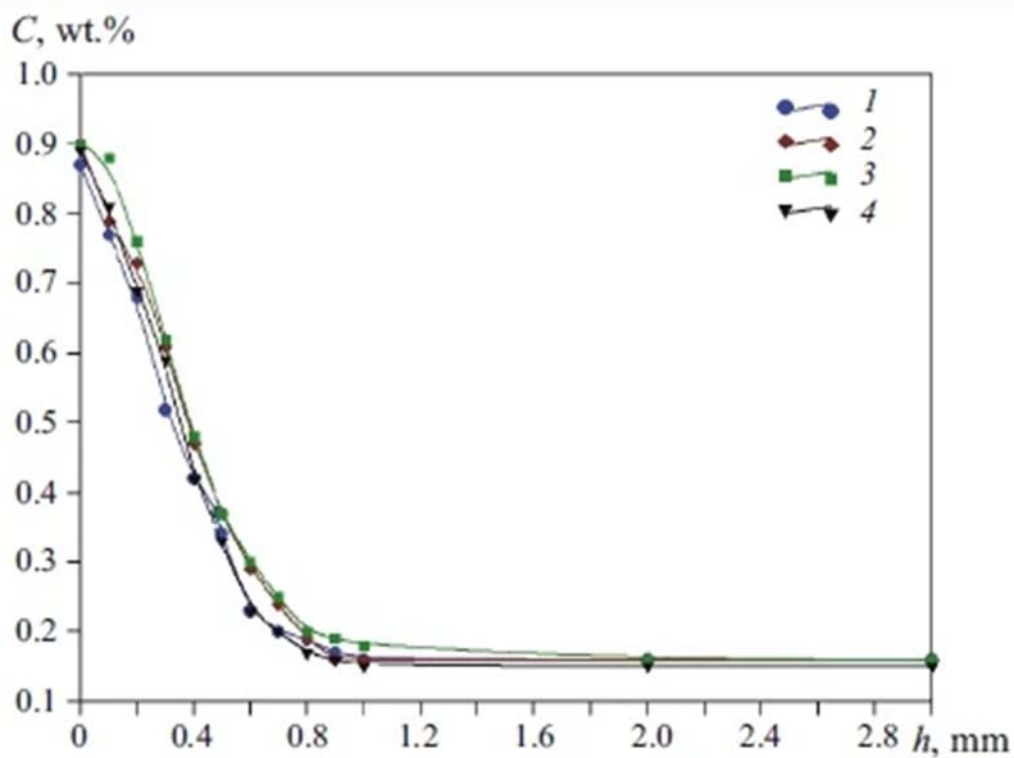


Fig. 2. Carbon concentration profiles (h is the distance from the surface) obtained after carburizing of steel 16NCD13 in the following modes: 1) 900°C, 104 min; 2) 920°C, 95 min; 3) 940°C, 64 min; 4) 960°C, 44 min.

Figure 3 presents the curves describing the variation of the average hardness over the thickness of carburized layer. It can be seen that the hardness profiles, like the carbon concentration profiles, do not vary with the temperature of carburizing of steel 16NCD13.

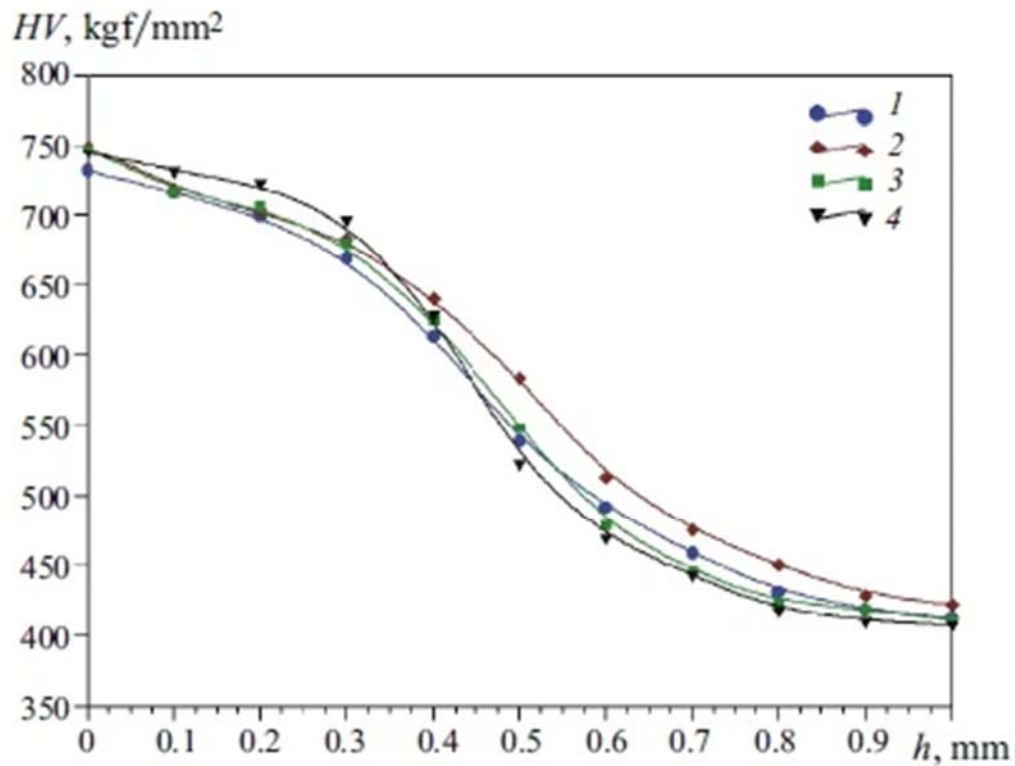


Fig. 3. Variation of hardness over the thickness of hardened layer (h is the distance from the surface) obtained after carburizing of steel 16NCD13 in the following modes: 1) 900°C, 104 min; 2) 920°C, 95 min; 3) 940°C, 64 min; 4) 960°C, 44 min.

Increase of the temperature of carburizing may change the grain sizes and hence the mechanical properties of the steel. The results of the tensile tests of samples carburized at different temperatures are presented in Table 2. The mechanical properties of the steel satisfy the specifications after carburizing at all the temperatures tested except for 960°C, though the elongation at this temperature remains high enough. The strength characteristics decrease with growth of the temperature of carburizing. Table 2 also presents the results of the measurement of grain sizes. Some growth of the grains with the carburizing temperature is not statistically significant. The expected growth of the grain size in this case is compensated by decrease of the time of the carburizing process at higher temperatures.

Table 2. Mechanical Properties and Grain Sizes in Steel 16NCD13 after Carburizing

Carburizing mode	σ_r , MPa	$\sigma_{0.2}$, MPa	δ , %	d_{av} , μm	d_{con} , μm
900°C, 104 min	1297 (0.7)	1037 (0.5)	16 (0.3)	7.19	10.9
920°C, 95 min	1227 (0.7)	986 (0.5)	14 (0.1)	8.36	10.5
940°C, 64 min	1219 (2.2)	983 (0.7)	14 (0.6)	10.00	10.0
960°C, 44 min	1149 (1.3)	916 (1.2)	14 (0.3)	11.53	9.6
Required properties	1180 – 1380	≥ 980	≥ 8	–	–

Note. The standard deviations are given in parentheses.

Notations: d_{av}) average grain size; d_{con}) grain size converted according to ASTM E112 [15].

The diagram of austenite transformations under continuous cooling of the steel shows that in cooling from the temperature Ac_3 to $\leq 100^\circ\text{C}$ during less than 40 sec (i.e., at the cooling rate of about 1080 K/min) the structure contains a mixture of martensite and bainite with hardness about 44 HRC; according to [18], this corresponds to the hardness of the core (about 430 HV). Steel 16NCD13 has such hardness after oil quenching. This value is somewhat higher than after quenching in a high-pressure gas. It can be assumed that the reduced values of the strength characteristics after carburizing at 960°C are a consequence of incomplete occurrence of martensitic transformation in the core of the sample. The elevated hardness of the carburized layer indicates that its structure is fully martensitic after the cooling in the high-pressure gas.

The microstructure of the carburized layer of all the samples is represented by tempered high-carbon martensite, while the microstructure of the core consists of tempered low-carbon martensite. Bainite has been observed in the core of the sample carburized at 960°C, which agrees with its reduced ultimate strength.

The thickness of the carburized layer was calculated with the use of different diffusion models. The results of the calculations are presented in Table 3 and Fig. 4.

Table 3. Calculated (h_{calc}) and Experimental (h_{exp}) Values of Thickness of Carburized Layer in Steel 16NCD13

Carburizing mode	h_{calc} , mm				h_{exp} , mm
	By Fick's law [12]	By Goldstein's model [7]	by Collin's model [8]	by Tibbetts' model [11]	
900°C, 104 min	0.36	0.33	0.45	0.28	0.50
920°C, 95 min	0.39	0.36	0.49	0.31	0.55
940°C, 64 min	0.36	0.33	0.46	0.29	0.49
960°C, 44 min	0.34	0.30	0.43	0.27	0.48

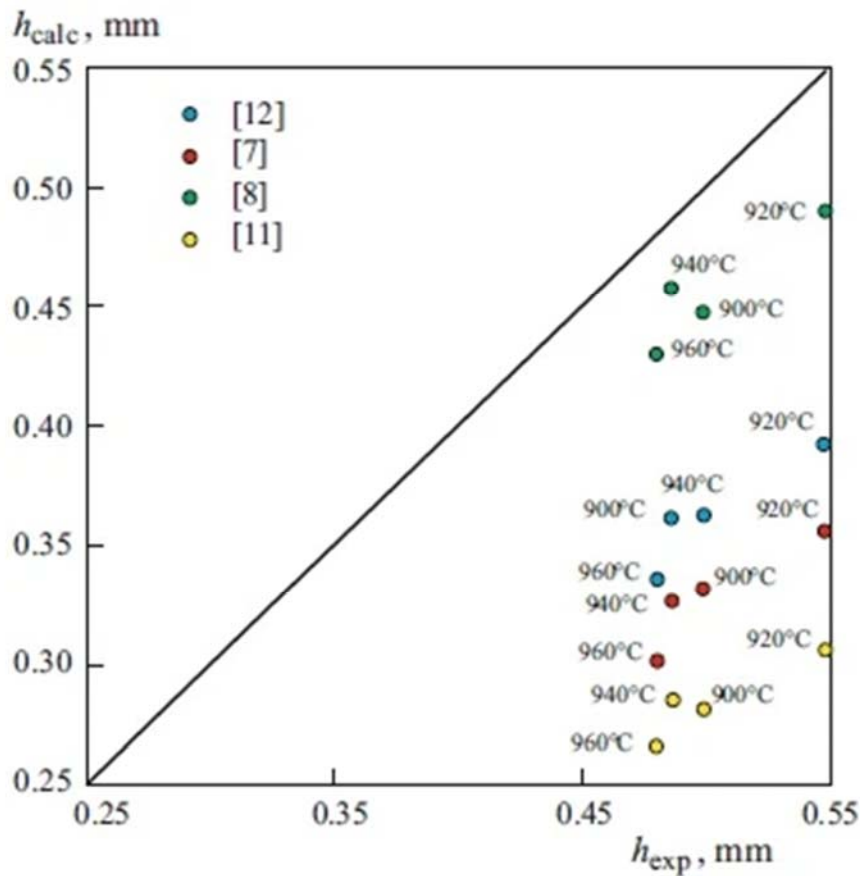


Fig. 4. Correlation between the depth of diffusion in carburizing of steel 16NCD13 at 900 – 960°C as calculated by the models of [7, 8, 11, 12] (h_{calc}) and determined experimentally (h_{exp}) (the straight line gives an ideal coincidence).

The thickness of the diffusion layer is described the best by the model of Collin [8].

The carbon concentration profiles were calculated by the models of [7, 8, 11, 12] and compared to the experimental profiles (Fig. 2). Model [11] modified in [4] describes the measured carbon concentration profiles the best. In [4], the carburizing was conducted in an acetylene atmosphere, while in [8] the atmosphere was composed of 31.8% H₂, 23.5% N₂ and 0.6% CH₄. Methane decomposes much slower than acetylene [19].

Figure 5 presents the results of comparison of experimental carbon concentrations at various distances from the surface and of the calculated data obtained by different models. It can be seen that on the whole the calculations give lower carbon concentrations in the carburized samples. The model of Tibbetts [11] seems to be the most accurate, especially at some depths and combinations of the temperature and the time of carburization and at some distance from the surface.

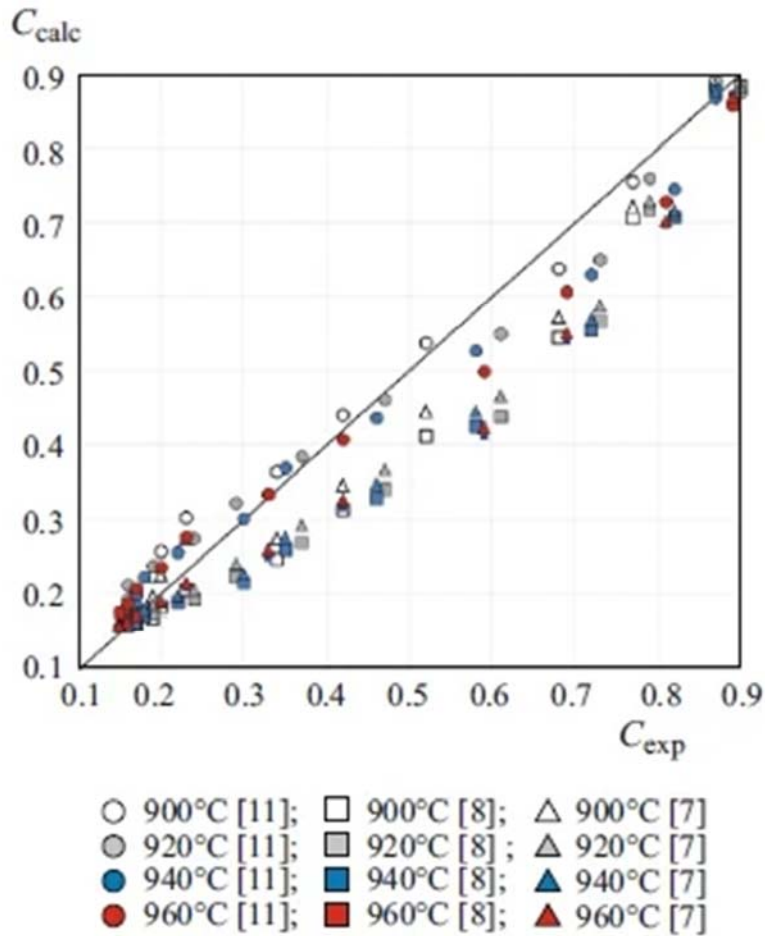


Fig. 5. Correlation between carbon concentrations calculated by different diffusion models (C_{calc}) and determined experimentally (C_{exp}) at different distances from the surface after carburizing of steel 18NCD13 at 900 – 960°C (the straight line gives an ideal coincidence).

CONCLUSIONS

Elevation of the carburizing temperature and simultaneous reduction of the time of the process give similar carbon concentration profiles and thicknesses of the carburized layer. The hardness profiles at such changes in the carburizing temperature and time are also similar except for the substantially lower hardness after the carburizing at 960°C.

The measured carbon concentration profiles are approximated well by the four diffusion models used. The model of [8] in combination with Einstein's law describes the best the thickness of the carburized layer, the model of [11] modified in [4] gives the best coincidence with the measured carbon concentration profiles. This model, which allows for the effect of the temperature and of the growth of the concentrations of carbon and of the alloying components on the diffusivity of carbon in austenite, has been developed for

carburizing in an acetylene atmosphere and approximates the best the carburization conditions used in the present work.

The microstructure in the carburized layer after the final heat treatment is represented by high-carbon tempered martensite. In the core of the test bars the structure consists chiefly of low-carbon tempered martensite. Formation of bainite has been suppressed in all the samples except for the sample carburized at 960°C. The values of the ultimate tensile strength and of the yield strength after carburizing at 960°C do not satisfy the specifications, though the elongation remains high enough.

The results presented show that the carburizing process used for hardening precision gears does provide the required properties in the hardened layer and in the core at the carburizing temperatures of up to 940°C. Such increase of the carburizing temperature is possible at considerable shortening of the carburizing time.

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