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EFFECT OF AUSTENITIZING PARAMETERS ON DISSOLUTION OF CARBIDES IN GAMMA SOLUTION AND CHEMICAL COMPOSITION OF MATRIX IN THE HARDENED STEEL OF 2% C AND 12% Cr TYPE WITH ADDITIVES OF W, Mo, V

ABSTRACT

The NCWV/D3 tool steel was the subject of the study. In the course of the investigation carried out it was found that during austenitizing of the steel the carbides contents decreased from 24.84 wt% in the annealed state down to 13.33 wt% after austenitizing at 1150 °C during 30 minutes. It was also stated that the carbides contents in the quenched steel after austenitizing in the temperature range from 900 to 1150 °C during 30 minutes may be determined from the relationship $cb[wt\%] = 28.60 - 0.46e^{0.003T}$

Dissolution of carbides during austenitizing brings about the increase of elements Cr, W, and V contents in austenite up to 7.27%, 1.06%, and 0.086%, respectively, after austenitizing at 1150 °C during 30 minutes. It was also noticed that there is a rectilinear correlation between the contents of dissolved carbides in austenite and the contents of Cr and W in matrix of the tool steel quenched after austenitizing in the temperature range from 900 to 1150 °C and constant time of 30 minutes.

Key words*: Tool steels, Austenitizing, Carbides contents, Matrix composition, Correlations*

INTRODUCTION

Fundamental research concerning the effect of austenitizing parameters on the chemical composition of matrix in quenched steels of type 2% C and 12% Cr was carried out by Sato et al. [1], Głowacki [2], and fragmentary studies by Kowalski [3], and Berns [4]. According to Sato et al. [1] as the result of dissolution of carbides $(Cr,Fe)_7C_3$ during austenitizing of steel containing 2.25% C and 12.03% Cr the content of chromium in matrix of the steel increases up to about 5.2% after the treatment at 1050 °C during 60 minutes. At the same time in the matrix of the steel the carbon contents increases up to 0.7%. A very interesting point is that Cr and C contents in the matrix of this steel during austenitizing at 850 to 1050 °C for 60 minutes are approaching a rectilinear dependence. According to Głowacki [2] for the steel containing 2.14% C and 11.50% Cr the chromium contents in the matrix increases successively up to 6.87% by dissolution of $(Cr,Fe)_7C_3$ carbides with the temperature increase after austenitizing at 1050 °C for 30 minutes. Głowacki finds also that prolongation of austenitizing time from 30 to 90 minutes at 950, 1050 and 1150 °C in practice does not introduce any changes in chromium content in matrix. According to Berns [3] during austenitizing steel of 2.05% C and 12.09% Cr at 960, 1050 and 1200 °C during 30 min the chromium contents in matrix are: 4.4, 5.2, and 7.6%, and carbon contents: 0.62, 0.77, and 1.10%, respectively. The studies concerned with the effect of austenitizing parameters in steel of 2% C and 12% Cr with the additives of W, Mo, and V were carried out by Berns [4], Haberling and Schruft [5], and with additives of W and V by Kałuża [6,7], Nykiel [8], and Nykiel and Hryniewicz [9]. It results from these works that changes of Cr

in matrix of quenched steels X210CrW12, X165CrMoV12, X155CrVMo121 [4] and NCWV [5-9] as the function of temperature and time of austenitizing are qualitatively similar to changes occurring in steels of this type without the additional alloying elements (W, Mo, V).
Contradictory results were obtained concerning the behaviour of tungsten in matrix. Berns [4] obtained a slight decrease of tungsten in matrix, whereas Kałuża [6,7] and Nykiel [8] found increase of tungsten in the matrix. Basic aim of the present studies is the analysis of relationships between the content of dissolved carbides during austenitizing and the contents of chromium, tungsten, and carbon in the matrix of NCWV/D3 steel. The complex studies to be carried out in this scope will allow us to perform a detailed analysis of adhesion of superhard layers/films coated over the working surfaces of tools made of about 2% C and 12% Cr type steels as well as hard chromium coating covered.

MATERIAL AND EXPERIMENTAL PROCEDURE

The material for study was NCWV/D3 tool steel [10] for cold working purpose of the composition given in Table 1.

Table 1. *Composition of material for the study, NCWV/D3 steel, in wt%*

C = 1.46	Mo = 0.05	Mn = 0.44
Cr = 11.56	Ni = 0.122	P = 0.024
W = 1.32	Cu = 0.073	S = 0.022
V = 0.31	Si = 0.27	N = 0.016

Heat treatment of samples of ϕ = 12 mm, l = 60 mm, assigned for determination of carbides contents using method of electrolytic extraction at 900 to 1150 °C and austenitizing times of 10, 30, and 90 minutes, was performed in a furnace with protective environment/atmosphere of technically clean nitrogen. The accuracy of temperature control was ±2 °C. After the austenitizing the samples were quenched in a hardening oil. To avoid eventual decarbonated layer the samples surface was ground on centreless grinder to remover the layer of thickness at least 0.5 mm.
Metallographic specimens were prepared mechanically using abrasive papers of the grit size from 100 to 2500 and afterwards they were polished using acqueous suspension of Al_2O_3 powder. Carbides in samples of annealed and quenched steel after austenitizing in the temperature range of 900 to 1150 °C were revealed by etching with Murakami's reagent (3 g potassium ferrocyanide + 100 g KOH + 100 cm^3 H_2O). Metallographic photographs were done by means of Epityp-2 microscope.
Measurements of carbide grains intetcepts' lengths were carried out on micrographs, done in different sites of samples, by drawing by chance secants 70 mm long. Then the intercepts' lengths of carbides cut by each secant were measured. Number of secants drawn on samples micrographs' sets of magnitude 1250 times of the annealed and quenched steel after austenitizing at 1000 °C was 84, and at 1150 °C was 160. Numbers of measured carbide grains' intercepts were 1124, 670, and 637, respectively.
The obtained carbide intercepts' lengths were gouped in proper distributive series of constant gradation equalling 0.2 µm. Carbides of the intercepts' lengths above 3.2 µm, due to their small number, were not taken into consideration in the distributions.
The electrolytic extraction of carbides was carried out in a 5%-HCl acquaeous solution of specific gravity of 1.19 g/cm^3, with the current density of 10 mA/cm^2. The extraction time was 18

to 20 hours. The carbides extraction method in details has been presented in [8, 9]. Percentages of Cr, W, and V in matrix of the quenched steel samples were determined by performing chemical analysis of electrolytes obtained after the carbides extraction. The percentage of chromium was determined by a potentiometric method whereas of tungsten and vanadium by a colorimetric method.

EXPERIMENTAL RESULTS AND DISCUSSION

According to the study results presented in [8] for the experimented steel, and by Geller [11] for the X12M steel of similar composition, apart from the chromium carbides of M_7C_3 type, the carbides of $M_{23}C_6$ type occur in very small amount which dissolve completely in austenite during austenitizing steel at 1050 °C during 30 minutes [8]. The occurrence of carbides of $M_{23}C_6$ in NCWV/D3 steel was also identified by Kałuża [6,7].

Big primary carbides of the investigated NCWV/D3 steel annealed and quenched after austenitizing in the temperature range from 900 to 1150 °C for 30 minutes usually possess irregular shape, whereas lesser secondary carbides possess spheroidal or close-to-spheroidal shape.

It results from histograms presented in Fig. 1 that in rods of diameter 13 mm of the investigated NCWV/D3 stee obtained by forging and then soft annealed the distribution of carbide grains intercepts' lengths is close to logarithic-normal with carbides of 0.4-mm intercepts' length being the most numerous fraction.

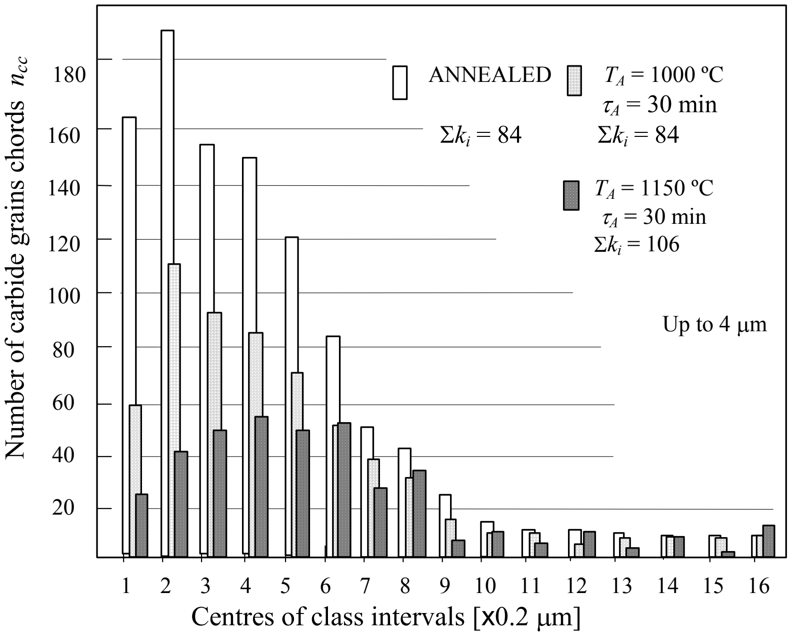


Fig. 1. Histogram of carbide grains intercepts' lengths distributions in NCWV/D3 steel: ▯ – annealed and quenched after austenitizing for 30 minutes at: ▯ – 1000 °C, ▯ – 1150 °C

Qualitatively similar distributions were obtained in samples of the quenched steel after austenitizing at 1000 and 1150 °C, with decreasing number of carbides of the least dimensions, that is of intercepts' lengths down to about 1 μm, occurring.

In the quenched steel after austenitizing at 1000 °C during 30 minutes still the most numerous fraction are the carbides of intercepts' lengths of 0.4 μm whereas after austenitizing at 1150 °C the carbides fractions of intercepts of 0.6 to 1.2 μm. Micrographs of carbides of the investigated NCWV/D3 steel annealed and quenched after austenitizing at 950, 1050, and 1150 °C respectively, for 30 minutes are presented in Fig. 2.

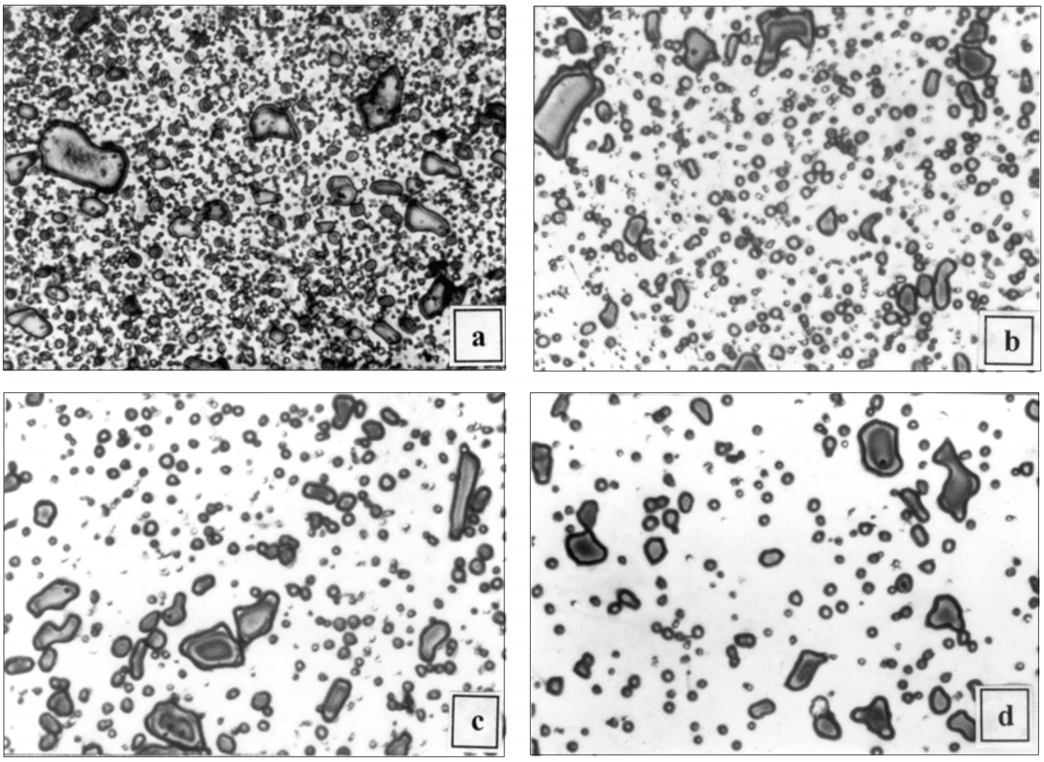


Fig. 2. Picture of carbide grains in NCWV/D3 steel: (a) in the soft annealed state, (b,c,d) quenched after austenitizing for 30 minutes at: (b) 950 °C, (c) 1050 °C, and (d) 1150 °C. Murakami's reagent was used for etching. Magnification 1000 ×

The weight percentage of carbides in the experimented steel after soft annealing determined by electrolytic extraction method equals 24.84% within an accuracy of ± 0.295%. The determination results of the weight percentage of carbides as the function of temperature and time of austenitizing are presented graphically in Fig. 3.

It results from the Fig. 3 that during austenitizing of NCWV/D3 steel at 900 to 1050 °C the carbides dissolve the most intensively during first 10 minutes of treatment. Extending the time of austenitizing from 30 to 90 minutes results in decreasing of carbides content in a very small degree, that is at 1100 °C of 0.2%, and at others of about 0.1%. Regarding this one may assume that after 30 minutes of austenitizing at 900 to 1150 °C in the studied steel practically an equilibrium between austenite and carbides occurs.

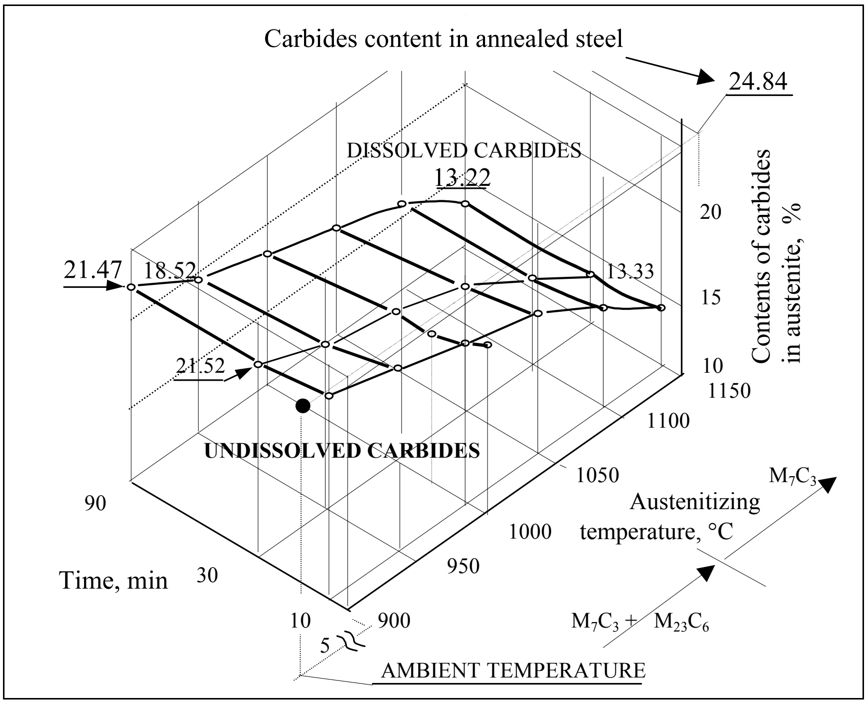


Fig. 3. Effect of temperature and time of austenitizing on the contents of non-dissolved and dissolved carbides in austenite of NCWV/D3 steel

Based on the experimental results obtained it was found that the percentage of carbides in the NCWV/D3 steel after austenitizing at 900 to 1150 °C during constant time equalling 30 minutes the following empirical equation may be determined:

$$cb[wt\%] = 28.60 - 0.46e^{0.003T} \qquad (1)$$

where:

cb – carbides content in steel, in wt%

T – temperature, in °C.

It was found that the highest hardness of the NCWV/D3 steel in the quenched state, equalling 66 HRC keeps steady after austenitizing at 1000 °C during 30 minutes. The increase in hardness up to 67.3 HRC of the steel may be reached by applying sub-zero treatment at minus 196 °C. The effect of austenitizing time at 1000 °C upon the hardness of NCWV/D3 steel is presented in Fig. 4.

The effect of austenitizing time during 30 minutes on the carbides contents in the studied NCWV/D3 steel may be described by empirical equation:

$$cb[wt\%] = 18.33 + 4.25e^{-0.706\tau} + 2.26e^{-0.072\tau} \qquad (2)$$

where:

cb – carbides content in steel, in wt%

τ – austenitizing time, in min.

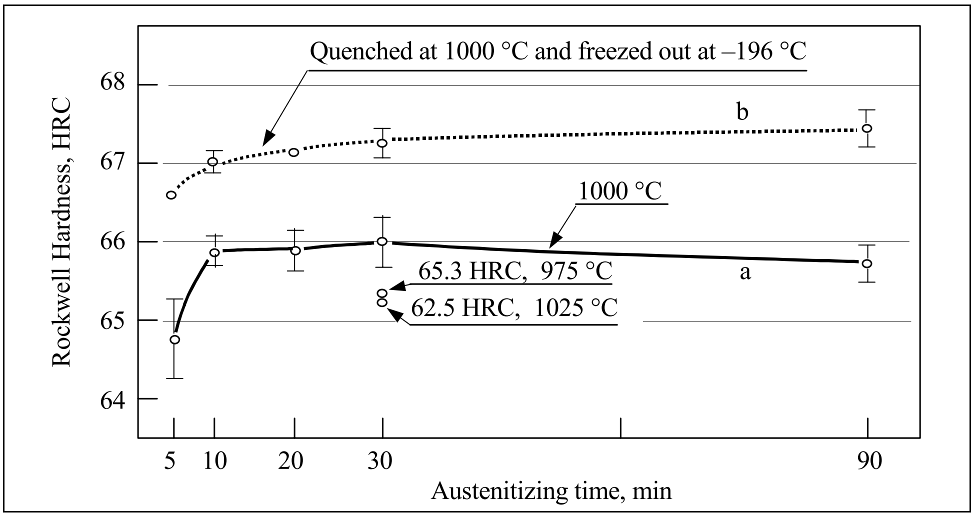


Fig. 4. Effect of austenitizing time at 1100 °C on the hardness of NCWV/D3 steel: (a) quenched, (b) quenched and sub-zero treated at minus 196 °C

Comparison of carbides contents calculated according to formula (2) with the results obtained from the experiment, that is obtained using electrolytic extraction method, is presented in Table 2.

***Table 2.** Comparison of calculations and experimental data*

τ_A [min]	Calculations	cb[%] experimental
5	cb[%] = $18.33 + 4.25e^{-0.706 \bullet 5} + 2.26e^{-0.706 \bullet 5} = 20.04$	19.81
10	cb[%] = $18.33 + 4.25e^{-0.706 \bullet 10} + 2.26e^{-0.706 \bullet 10} = 19.46$	19.46
20	cb[%] = $18.33 + 4.25e^{-0.706 \bullet 20} + 2.26e^{-0.706 \bullet 20} = 18.89$	18.87
30	cb[%] = $18.33 + 4.25e^{-0.706 \bullet 30} + 2.26e^{-0.706 \bullet 30} = 18.60$	18.62

Knowing the contents of carbides in the annealed steel and the contents in quenched steel after austenitizing at given temperature and time one may simply determine the amount of carbides dissolved in austenite. The contents of dissolved carbides is essential because it affects the degree of austenite saturation with carbon and alloying elements, and in case of the NCWV/D3 steel, apart from carbon, also with chromium, tungsten and vanadium. It is well known that the degree of austenite saturation with the higher mentioned elements affects the mechanical properties of martensite and the amount of retained austenite after quenching operation. The determinations of chemical compositions in matrix, and practically the determination of matrix electrolytes after their thickening on the way of evaporating H_2O and HCl obtained in the process of electrolytic extraction of carbides showed that due to the carbides dissolution during austenitizing, the contents of chromium and tungsten in the NCWV/D3 quenched steel matrix have increased successively with the growth in austenitizing temperature, and time up to 30 minutes (Fig. 5).

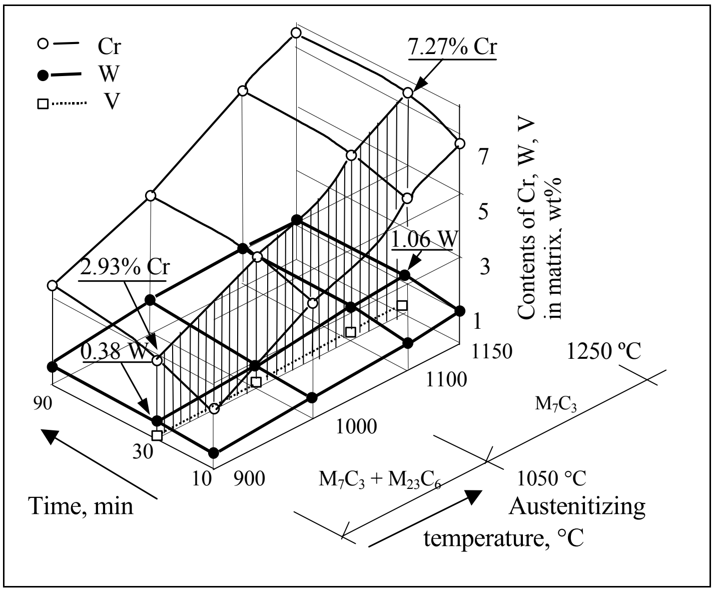


Fig. 5. Effect of austenitizing temperature and time on the contents of Cr, W, and V in matrix of the quenched NCWV/D3 steel (contents of Cr, W, V in matrix referred to the matrix)

It results from the determination of vanadium by colorimetric method that its contents in matrix increases though very slightly, that is from 0.045% after austenitizing at 900 °C up to 0.086% at 1150 °C. The results of chemical analyses show that the extention of austenitizing time at 900, 1000, 1100, and 1150 °C up to 90 minutes causes increase in the chromium and tungsten contents in matrix but on the level of hundredths per cent.

Based on the contents of carbides dissolved in austenite during austenitizing in the temperature range from 900 to 1150 °C and determination of Cr and W contents in matrix the analysis was carried out to find out a mathematical relationship between the contents of carbides dissolved in austenite and Cr and W contents in the steel matrix. This analysis was performed based on the least square method. Experimental data are presented in Table 3, and in Fig. 6. The obtained regression lines between the contents of dissolved carbides during austenitizing and Cr and W contents in matrix of the quenched steel are shown in Fig.6.

Table 3. *Calculation data to Fig. 6*

T_A, °C	$X_{c\text{-}da}$	$Y_{Cr\text{-}m}$	$Y_{W\text{-}m}$
900	3.32	2.93	0.38
1000	6.22	4.61	0.58
1100	9.24	6.64	0.94
1150	11.51	7.27	1.06

T_A – austenitizing temperature

$X_{c\text{-}da}$ – carbides contents in dissolved austenite, wt%

$Y_{Cr\text{-}m}$ – chromium contents in matrix of quenched steel, wt%, determined by potentiometric method of matrix electrolytes

$Y_{Cr\text{-}m}$ – tungsten contents in matrix of quenched steel, wt%, determined by colorimetric method of matrix electrolytes

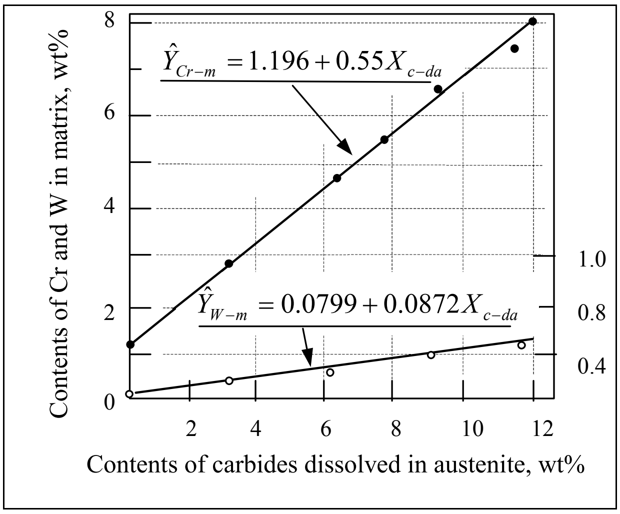


Fig. 6. Regression lines Y_{c-m} between the contents of dissolved carbides during austenitizing in the temperature range of 900 to 1150 °C throughout 30 minutes and the contents of chromium (-•-) and tungsten (-o-) in the NCWV/D3 quenched steel matrix

As the result of calculations for the variable system X_{c-da}-Y_{Cr-m} the following regression equation was obtained:

$$\hat{Y}_{Cr-m} = 1.196 + 0.55X_{c-da} \quad (3)$$

where:

X_{c-da} – contents of carbides dissolved in austenite

$\hat{Y}_{Cr-m}$ – contents of chromium in matrix determined based on the regression equation

For the variable system X_{c-da} - Y_{W-m} the following equation of regression line was obtained

$$\hat{Y}_{W-m} = 0.0799 + 0.0872X_{c-da} \quad (4)$$

where:

X_{c-da} – contents of carbides dissolved in austenite

$\hat{Y}_{W-m}$ – contents of tungsten in matrix determined based on the regression equation.

Correlation factors between the contents of carbides dissolved in austenite during austenitizing in the temperature range of 900 to 1150 °C throughout 30 min and the contents of chromium and tungsten in the matrix of quenched NCWV/D3 steel are r = 0.9906 and r = 1.002, respectively, what means the complete/full correlation.

The presented literature review indicates that steels containing about 2% C and 12% Cr, in these also with the additives of tungsten, molybdenum and vanadium in the annealed state contain from about 17 to 27% of carbides resulting generally from different contents of carbon. In some of them, apart from M_7C_3 carbides, the alloying cementite [11, 13] and/or carbides of $M_{23}C_6$ type [6-9, 11-13] may occur. Comparison of the authors' own results of studied NCWV/D3 steel, concerning the effect of austenitizing temperature on carbides contents, with the results obtained by Sato et al. [1], Głowacki [2], Berns [4], and Kałuża [6,7] for steels of 2% C and 12% Cr type, and authors' own results, are presented in Fig. 7.

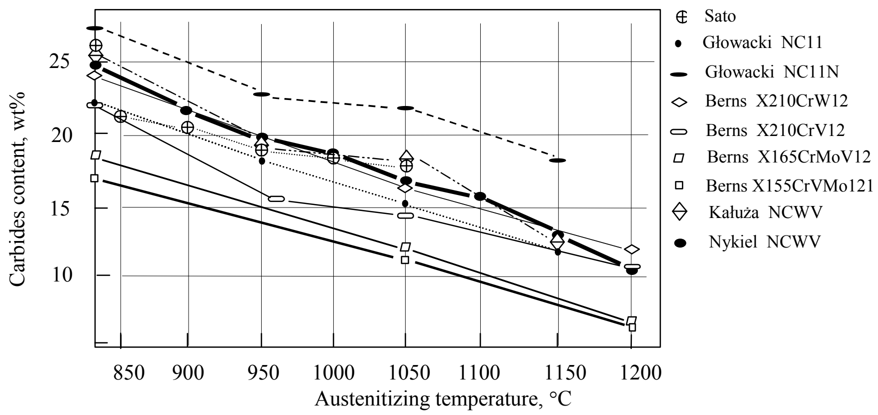


Fig. 7. Effect of austenitizing temperature on the carbides contents in the following steels: 2.25% C and 12.03% Cr, τ_A = 60 min, after Sato [1]; NC11 and NC11N (with doubled amount of nitrogen of 0.06% N; the steel for the experiment only), τ_A = 30 min, after Głowacki [2]; X210Cr12, X210CrW12, X165CrMoV12, X155CrVMo12 1, τ_A = 15 min, after Berns [4]; NCWV/D3 [6,7], NCWV/D3, τ_A = 30 min, the authors' own investigations [8, 9]

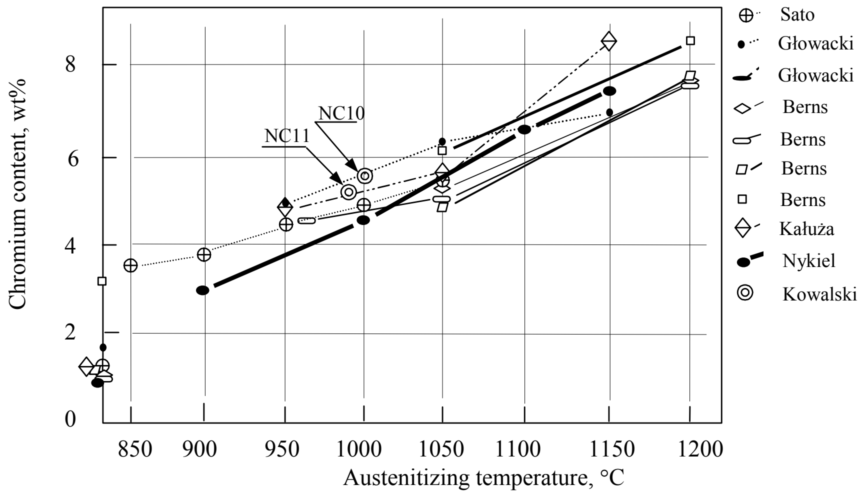
It results from the Fig. 7 that in steel NCWV/D3 the course of changes in carbides contents as the function of temperature is qualitatively very similar to the changes occurring in other steels of this type. Besides a good convergence of the amount of carbides dissolved in NCWV/D3 and X210CrW12 steels with the amount of carbides which dissolves in NC11 steel after austenitizing under the same conditions proves that additives of tungsten and vanadium with the amounts usually used in these steels do not affect essentially the kinetics of the dissolution of $(Cr,Fe,W,V)_7C_3$ carbides present in these steels. The exception is NC11N (with nitrogen) steel, in which carbides dissolve in considerably less degree because nitrogen dissolved in carbides causes an increase in stability/durability. In case of Sato's studies [1] the carbides dissolve more intensively than in other steels during austenitizing at 900 °C.

Comparison of the results of chemical analyses of matrix in the quenched NCWV/D3 steel concerning the effect of austenitizing temperature on its chemical composition with the results obtained by other authors, and of Kowalski [3], are presented in a graphical form in Fig. 8 (a and b).

It results from Fig. 8 that in ferritic matrix of annealed steels of about 2% C and 12% Cr type the chromium contents is found to be in the interval from 1 to 2%, with the exception of X155CrVMo12 1 steel in which the chromium contents is 3.1%. In these steels the most intensive increase of chromium contents in matrix occurs during austenitizing at 900 °C and it is the effect of dissolution of M_7C_3 carbides of the smallest sizes. Also it results from Fig. 8 that increase of Cr contents in matrix of these steels after austenitizing at 900 to 1200 °C, with the exception of NC11 steel, is approaching the rectilinear course. On the other hand, the Cr contents in matrix of these steels quenched to a maximum hardness, that is after austenitizing at 980 to 1000 °C during 15 to 30 minutes, dependent on the sort of steel, is placed in the interval from about 3.8% to 5.3%.

Small contents of vanadium in the analysed steels, with simultaneous its high concentration in the big primary carbides, which begin to dissolve at the temperature above 1200 °C [8] indicate/cause the percentage of this component in matrix to be very small, that is at the level of 0.01 to 0.034%. From the presented in Fig. 8 data results that the saturation with vanadium increases with temperature growth but with insignificant degree. Of the analysed steels, the highest amount of vanadium equalled 0.5% occurs in matrix of X165CrVMo12 1 steel, quenched after austenitizing at 1200 °C.

a)



b)

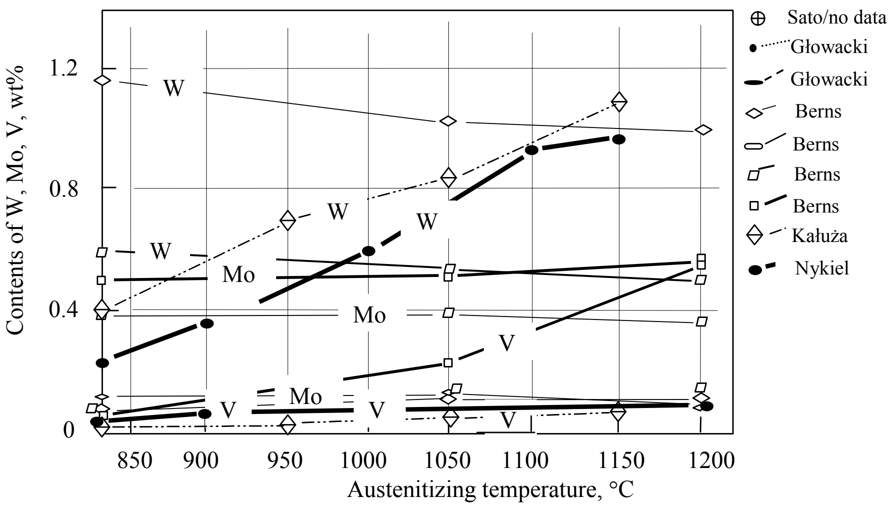


Fig. 8. Effect of austenitizing temperature on the contents of Cr (Fig. 8a), and W, Mo, V (Fig. 8b) in matrix of the quenched steel of 2% C and 12% Cr type

The percentage of tungsten in matrix of these steels under annealed state fluctuates from 0.25% in NCWV/D3 steel up to 1.14% in X210CrW12 steel. In NCWV/D3 steel the percentage of tungsten in matrix increases with the temperature growth up to 1.06% after austenitizing at 1150 °C, whereas acc. to Berns [4] in X210CrW12 and X165CrMoV12 steels it decreases of 0.17% and 0.08%, respectively (after austenitizing at 1200 °C) in relation to the content occurring in matrix of these steels in the annealed state.

Summarizing it should be noticed that a full/complete analysis concerning dissolution of carbides in the steels of 2% C and 12% Cr type as well as of the change of chemical composition of matrix is impossible mainly due to inadequate amount of investigations in this area. Apart from this such an analysis should take into consideration some other factors such as e.g. the degree of refinement/fineness of carbides.

CONCLUSIONS

The following conclusions may be drawn based on both theoretical and experimental research carried out:

(1) During austenitizing of the investigated NCWV/D3 steel in the temperature range of 900 to 1150 °C the amount of carbides with intercepts up to 1.4 μm long have distinctly decreased with the highest degree of intercepts up to 1.0 μm. Dissolution of these carbides affects the contents of C, Cr, W, and V in matrix of the quenched steel.

(2) Percentage of carbides in the studied NCWV/D3 steel has decreased with austenitizing time and temperature increase from 24.84% in the annealed steel down to 13.33% after austenitizing at 1150 °C during 30 minutes; the content of carbides in the steel after austenitizing in the temperature range from 900 to 1150 °C and constant time equalling 30 minutes may be determined from the following empirical dependence:

$$cb[wt\%] = 28.60 - 0.46e^{0.003T}$$

(3) The highest hardnesses of steel after quenching, with very slight fluctuations, are obtained after austenitizing at 1000 °C and times of 10 to 30 minutes; the effect of austenitizing time up to 30 minutes at 1000 °C on carbides contents may be determined from the following empirical relationship:

$$cb[wt\%] = 18.33 + 4.25e^{-0.706\tau} + 2.26e^{-0.072\tau}$$

(4) In the studied NCWV/D3 steel after austenitizing in the temperature range from 900 to1150 °C and constant time of 30 minutes a rectilinear correlation occurs between the carbides contents dissolved in austenite and the contents of Cr and W in matrix after quenching, with the correlation factors equalling r = 0.9906 and r = 1.002, respectively. The approximate contents of Cr and W in matrix may be determined from the following empirical dependences:

$$\hat{Y}_{Cr-m} = 1.196 + 0.55X_{c-da} \quad \text{and}$$

$$\hat{Y}_{W-m} = 0.0799 + 0.0872X_{c-da}$$

(5) In the soft annealed steels of 2% C and 12% Cr type, dependent on the carbon contents, the amount of carbides equal from about 17% (in X155CrVMo12 1 steel) to about 27% (NC11N experimental steel). In the steels containing 2.25% C and 12.03% Cr, and NC11, X210CrW12, NCWV, quenched after austenitizing in the temperature range of 900 to 1150 °C (for steel containing 2.25% C and 12.03% Cr, up to 1050 °C) the carbides contents are very similar (on the same level).

(6) The contents of Cr, W, and V in ferritic matrix of analyzed steels under soft annealed state are in the intervals from about 1-2% Cr, 0.25-1.14% W, and 0.01-0.05% V. Percentages of chromium in the matrix of these steels under quenched state increase with the temperature growth up to about 6.9 to 8.5% Cr after austenitizing at 1150 °C during 15-30 minutes. The matrix of the steels quenched to the maximum hardness contains, dependent on the steel type, from 3.8 to 5.3% Cr, about 0.5-1% W, and about 0.06% V.

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