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Investigation of hardening and tempering deformations of carburized low alloy steel

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1. Introduction

Carbon saturation of the surface, i.e. carburizing, is usually applied technology for machinery of low carbon and low alloyed steel with Cr, Ni, Mo and other additives. The carbon content of such steel is commonly 0.1–0.25% [1-4]. Sometimes, carbon content can exceed 0.4%, when carburizing is applied for tools, high strength of core is required or the steel is alloyed.

Surface carburizing of products gives rather multiplex structure: at the very surface the hypereutectoid structure composes from pearlite and different amount of surplus carbides dependent on the degree of carbon saturation; next we have eutectoid structure of pearlite and lastly – hypoeutectoid ferrite/pearlite structure with rising amount of ferrite till the structure reaches the phase composition of the core. During quenching of the carburized parts, the martensite transformation at the low carbon core begins considerably earlier (M_s = 440 – 470°C) compared to the transformation at carburized surface (M_s = 160 – 245°C) [5]. When austenite turns to martensite, the relative volume increases ~ 1% at the low carbon core and signally more at the carburized surface [6]. These transformations precede great internal stress varying during quenching. Because of this stress, on the condition of transformation plasticity, big deformations of quenching occur, especially, when the surface is carburized asymmetrically.

Carburized parts with inhomogeneous structure are distinguished by the wide variety of micro-constituents and their properties through the all carburized layer. For example, quenched martensite in the carburized surface contains more carbon compared to the deeper volumes; therefore, surface martensite has bigger comparative volume and is formed from the smaller plates. Presence of different micro-constituents and their properties are related with temperature of heat treatment. During the hardening of carburized parts, they deform and precision is lost because of the volume mismatch, therefore, the vibration can occur in the elements coupling (e.g., coupling of gears) and resistance to the impact wear can decrease [7, 8]. Furthermore, all these processes are closely linked with alloying elements, especially with such strongly carbides constitutive ones as chromium, molybdenum, etc. [9].

Quenched and curved articles could be fixed by several ways:

-grinding of the overlaps, but then the thickness of carburized layer is declined;

-using hardening in the stamps or other form-fixing devices for the purpose to utilize stress relaxation and to avoid deformation of parts;

-quenched and curved articles could be fixed during the low tempering under the effect of transformation plasticity when huge deformations could be achieved under even small load because of temporal relaxation of atoms' binds in the lattice [10, 11].

This article presents the results of further research of carburized parts deformation kinetics [12], the investigation of transformation plasticity phenomenon during tempering of quenched and curved specimens. The results obtained could be applied for the development of heat treatment technologies of carburizing machinery.

2. Experimental procedures

The chemical composition of alloyed structural steel 12XH3A (GOST 4543-71) is listed in Table 1. An equivalent of steel grade is 15CrNi6 ISO 683/1-87.

Table 1
Chemical composition of steel 12XH3A, % (Fe balance). Data from factory certificate

C	Mn	Si	Cr	Ni	S	P	Cu
0.14	0.42	0.22	0.77	2.90	0.02	0.02	0.30

The critical temperatures of steel with such chemical composition are: A_{c1} = 695°C, A_{c3} = 800°C [5]. The phase composition of annealed steel is ferrite and pearlite.

The ingots for carburizing were manufactured from Ø12 mm diameter steel rods according to the scheme presented in Fig. 1.

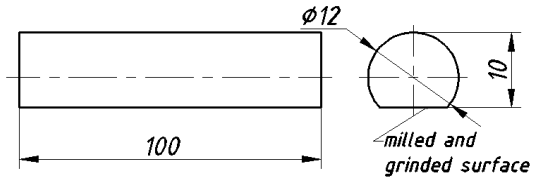

Fig. 1 Measurement of the ingot made-up for carburizing

The specimens were carburized at 930°C temperature 6 and 10 hours placed in hard carburizer. After carburizing they were kept cooling in furnace.

Carburized ingots were milled and grinded, and specimens with rectangular cross-section were manufactured. As dimensions of cross-section were used in further calculations of elasticity modulus, for the purpose to obtain all specimens with the same height and width of the cross-section, all specimens were grinded at the same setting. Then width and height of each specimen were verifying by

micrometer (accuracy 0.01 mm). Specimen parameters are shown in Fig. 2. The surface milled and grinded before carburization was unprocessed.

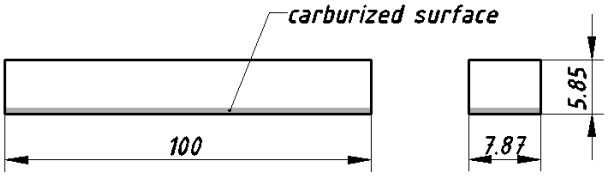

Fig. 2 Simplified design of specimen manufactured for bending test

For the determination of carburized layer depth and observation of steel microstructure, cross-cuts were made from only carburized and carburized and quenched specimens. Cross-cuts were mounted by the resin Technovit 4071, then grinded, polished with dough of chromium oxide and soup, and etched with 4% HNO_3 solution in ethanol. The microstructure was examined by the laser micro-analyzer LMA10 Carl Zeiss.

The recommended temperature of quenching of carburized specimens is 800–860°C [5, 13]. We have chosen two temperatures of quenching – 800°C (this temperature is optimal for carburized volume) and 860°C (optimal for low carbon containing volume), heating 30 min in the protective ambience and cooling in the water.

Values of hardness HRC were determined after quenching by measuring at three places of each specimen. Measuring device was Rockwell meter TK-2 with 150 kg load. Hardness of no carburized surfaces was obtained 52-54 units. The carburized surfaces gave 62-64 HRC. Higher values of hardness were appropriated to 800°C hardening temperature.

Water quenching causes especially great deformations of carburized specimens. The initial deflection of specimen y_0 was gauged with the accuracy of 0.01 mm at the fixed position according to the presented scheme (Fig. 3). Water quenching was chosen for the purpose to obtain the most possible quenching deformations by such an extreme cooling that later would be repaired with the help of transformation plasticity effect [10, 12, 14].

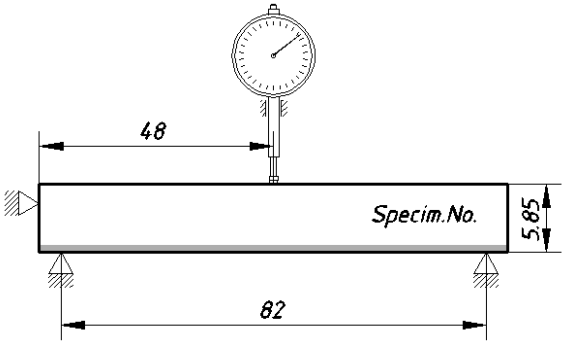

Fig. 3 Schematic view of the measuring of specimen curvature

The transformation plasticity experiment of specimen bending during low tempering was carried out after quenching and hardness measurement. The tempering temperature was 200°C. The required load and stresses were calculated every time evaluating the initial curvature of specimen (carburized, quenched and tempered rod with Ø10 mm diameter has $R_{0.2}$ = 1080 MPa, R_m = 1220 MPa [13]). The equipment used for the experiment of bending during tempering has been described in the earlier works [14]. During experiment of bending and tempering the graph of specimen deformation has been written measuring the deflection of transformation plasticity y_{tp} with the accuracy of 0.01 mm. After tempering, when the specimen was cooled down, the plastic deflection of specimen y_p was measured and remained plastic deflection y_p was calculated estimating the initial hardening deflection y_0:

$$y_p = y_m - y_0. \quad (1)$$

Micro-hardness of micro-constituents was measured by Vickers meter PMT-3M1.

3. Results and discussion

3.1. Microstructure analysis

The microstructure of carburized surface is presented in Fig. 4. For the control, the approximate average content of carbon was determined in the specimen core using standard photographs of microstructures. It was obtained ~ 0.14–0.15% of carbon, this allows an assumption that only the surface was saturated with carbon.

Examining the microstructures, the approximate depth of carburizing was determined. There was obtained that the depth of carburizing of specimens was approximately 0.45 mm and 1.30 mm, when carburizing duration was 6 and 10 respectively. The microstructure analysis had showed carbides undistinguished in the hypereutectoid zone because of high dispersion, also the boundary was not visible between hyper- and eutectoid zones.

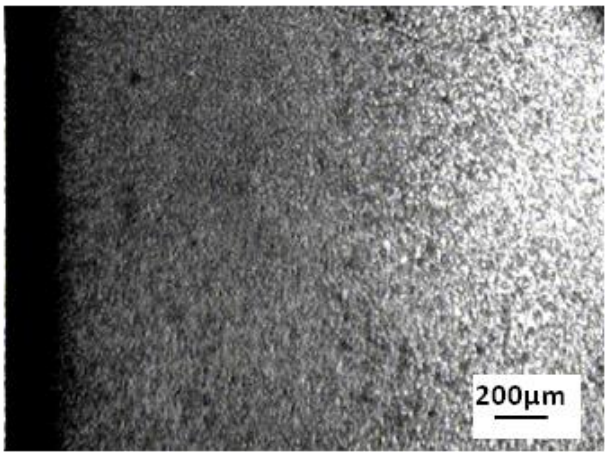

Fig. 4 Carburized surface of specimen after 10 h carburization

Carburization of 10 h at 930-950°C temperature allows surface assimilation with carbon up to ~1.6% when steel contains ~0.7% chromium [15]. When $T_{carb.}$ = 980-1000°C and content of chromium is 0.7%, then surface contains approximately 1.55% carbon after 10 h carburization, i.e., it doesn't amount 1.6%.

The content of carbon in the surface layer may be determined by the intersection of line SE from Fe-C diagram and horizontal line drawn from temperature point

T = 930°C [6]. Estimation according this method had resulted 1.4% C content in the carburized layer. Anyways, hypereutectoid structure with pearlite and disperse carbides was assured at the first zone of carburized layer of steel 12XH3A specimens after carburization at 930°C temperature.

Martensite structure formed in the carburized layer after quenching from 800°C or 860°C temperature was obtained fine therefore the retained austenite wasn't visible. At the very surface (approximately till 100 μm depth) bright grains of 10-20 μm size with clear boundaries were detected. Furthermore, much more bright grains were formed at 860°C quenching temperature (Fig. 5, b) compared to 800°C, when a few grains were found at ~20 μm depth of the carburized surface (Fig. 5, a). The pictures of microstructures discovered in some technical manuals and albums had allowed an assumption of the presence of surplus carbides [7, 16]. Their micro-hardness measuring test gave 965 HV. As micro-hardness of cementite is about 1000 HV units [2], thus an acceptance of carbide particles being was made. Alloyed cementite $(Fe, Cr)_3C$ containing up to 5% chromium (in some ways and $(Cr, Fe)_7C_3$) may form when concentration of chromium is marginal (0.77%) and steel contains near 1% carbon [9, 15]. Anyways the temperature of carbides complete dissolution in austenite is rather high: for cementite Fe_3C – 950-1000°C, for alloyed cementite $(Fe, Cr)_3C$ – 1050-1200°C [17].

Eventually there were accepted that quenching from 800°C or 860°C temperatures enabled the formation of martensite structure and enlarged coagulated particles of alloyed cementite (up to 15% vol. [17]) in the carburized surface layer. The conditions of carbides coagulation were more favourable at higher 860°C temperature, thus the final number of coagulated carbides was obtained greater compared to quenching at 800°C. At the extreme surface of specimen the retained austenite couldn't be formed because of lower content of carbon and alloying elements (caused by carbides growth and coagulation) but in the deeper volumes (~0.2 mm) with small carbides the amount of retained austenite might be 15-20% [3, 4, 8].

The observation of microstructure of not carburized material volume has showed lath martensite after quenching from 860°C (Fig. 6, b). It was formed with finer plates compared to 800°C quenching temperature, when brighter areas of ferrite were detected (Fig. 6, a).

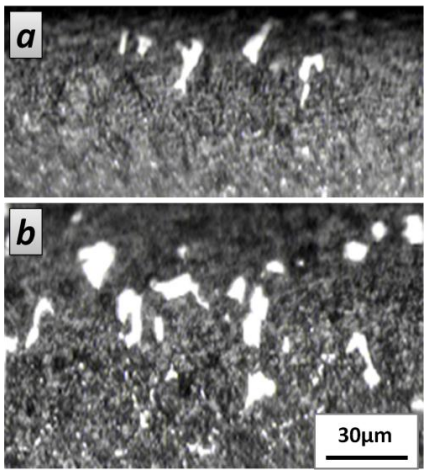

Fig. 5 Surface microstructure of carburized specimens quenched from: a – 800°C; b – 860°C

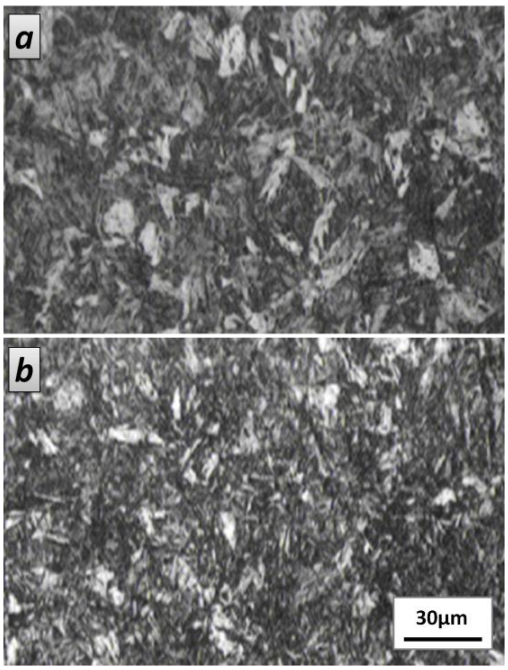

Fig. 6 Microstructure of not carburized volumes after quenching from: a – 800°C, b – 860°C

3.2. Quenching deformations

Independent of quenching temperature the hardened specimens were curved, mostly "hump" up, in percent: 73% specimens had deflection in the direction of „∩"and 27% specimens – „∪".

While heating at 800°C quenching temperature such material structure related with carbon content was formed: as the critical temperature of the steel is A_{c3} = 800°C [5], probably, some content of ferrite may remained not dissolved (Fig. 7).

Heating at 860°C has maintained structure of not carburized volume of 100% austenite (Fig. 7).

Sudden water cooling produces polymorphous transformations, new phases formation, thus the curved specimen having a variety of micro-constituents has been looked like Fig. 8 presents.

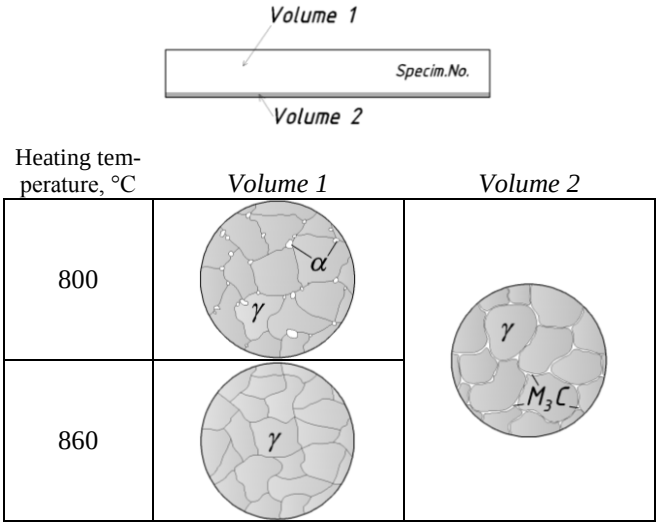

Fig. 7 Schematic view of micro-constituents of carburized specimen during heating at 800°C and 860°C: α – ferrite, γ – austenite, M_3C – alloyed cementite

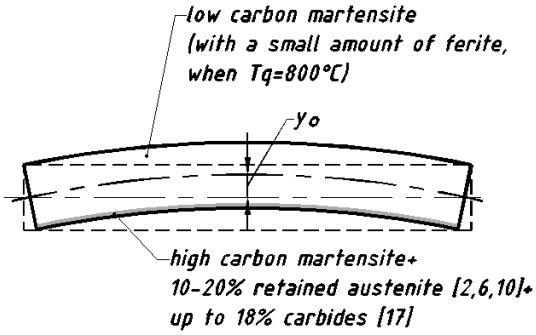

Fig. 8 Design of quenched and curved specimen with formed new phase's distribution at room temperature

The question is why dominant curvature of quenched specimens was obtained in the direction of „∩"? Probably this fact is related with different temperatures of martensite transformation start M_S in carburized and low carbon volumes; i. e. when T_q = 860°C, martensite formation starts at M_S = 100°C in carburized part of specimen. For not carburized part this temperature is M_S = 420°C [5]. Cooling from quenching temperature produces transformation of martensite first of all in the not carburized part of specimen. Its volume increases (specimen lengthens), yield strength of steel decreases significantly because of transformation plasticity effect. Carburized part of specimen remains austenitic and has greater thermal expansion coefficient α_T and higher yield strength. Material remains in this state in the range of temperatures from 420°C till 100°C producing stress field in the structure that causes plastic curvature of specimen in the direction of „∩" [18]. The yield strength of carburized part remains high enough to resist deformation. Later, when martensitic transformation begins in carburized part, its volume increases forcing specimen to curve in reverse direction „∪". Martensite structure already formed in not carburized volumes resists this curvature. The following interactions cause the curvature of specimen as Fig. 8 has showed. Thermal stresses generated because of sudden and uneven cooling have also significantly influence on the size of curvature of specimens.

3.3. Specimens' deformation during tempering

The deflection of asymmetrically carburized and quenched specimens has changed during tempering without bending load. Kinetics of the deflection change was observed during heating of specimen according to the schema shown in Fig. 9. The specimen with initial curvature in the direction „∩" was placed in the furnace for heating at 200°C temperature. At the first 2-3 min, the specimen has tried to straighten up, i.e. its centre point was going down (Fig. 10). Maximum deflection at that moment was only 0.02 mm and 0.01 mm when carburized depth was s = 0.45 mm and s = 1.3 mm, respectively.

The determination of causes of initial straightening requires the following experiments. According scientific literature data, one of them could be different coefficient of thermal expansion of different micro-constituents – austenite, martensite with various carbon content, carbides and ferrite in carburized volumes and not carburized ones [15, 17].

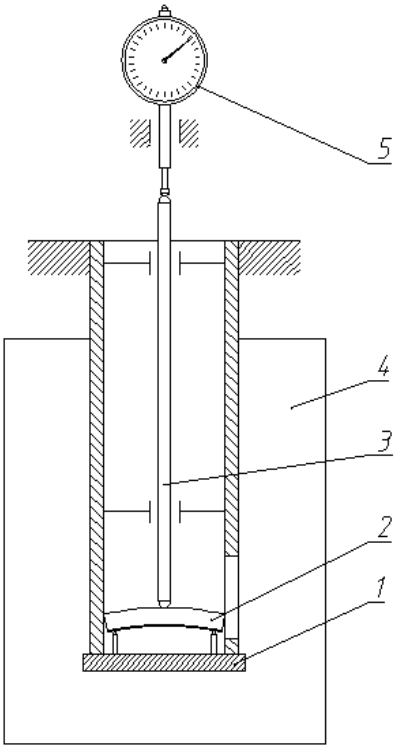

Fig. 9 Schema of determination of specimen deflection change during tempering: *1* – prism support; *2* – specimen placed in direction of carburized part downwardly, *3* – measuring rod; *4* – furnace; *5* – indicator

When temperature lays in 20-250°C range, the coefficient of thermal expansion of ferrite is (14.3 – 14.5) × 10^6 [15, 17]. Also, it is known that chromium decreases the coefficient, e.g. when ferrite contains 3.5% chromium, α = 12.8 × 10^6 [17]. The coefficient of thermal expansion of steel depends also on carbon content (Fig. 11).

Coefficient of thermal expansion of austenite is significantly greater compared to ferrite's one – even up to 20 × 10^6 [15, 17].

Thermal expansion of steel could be evaluated with calculation of volume increase of different micro-constituents during heating from 20°C to 200°C by formula [20]:

Austenite.....$0.12282 + 8.56 \times 10^{-6} \times T + 2.15 \times 10^{-3} \times C_p$;
Ferrite.....$0.12708 + 5.528 \times 10^{-6} \times T$;
Martensite.....$0.12708 + 4.45 \times 10^{-6} \times T + 2.79 \times 10^{-3} \times C_p$;
Cementite..... $0.13023 + 4.88 \times 10^{-6} \times T$,

where T is temperature, °C; C_p are carbon content, wt. %.

Relative volumes of micro-constituents were calculated at 20°C (before heating) and at 200°C (when reached T of the test). Since carbon content has not been examined in solid solution though the approximate content was picked in conformity with other results of scientific data. Conclusively, the precise amount of carbon isn't required for this calculation, because only the tendency of the relation between carbon content and volume changes of micro-constituents while heating is needed in this instance. Amount of carbon various in the surface from maximal (~1.3-1.5%) to similar of not carburized part (0.14%).

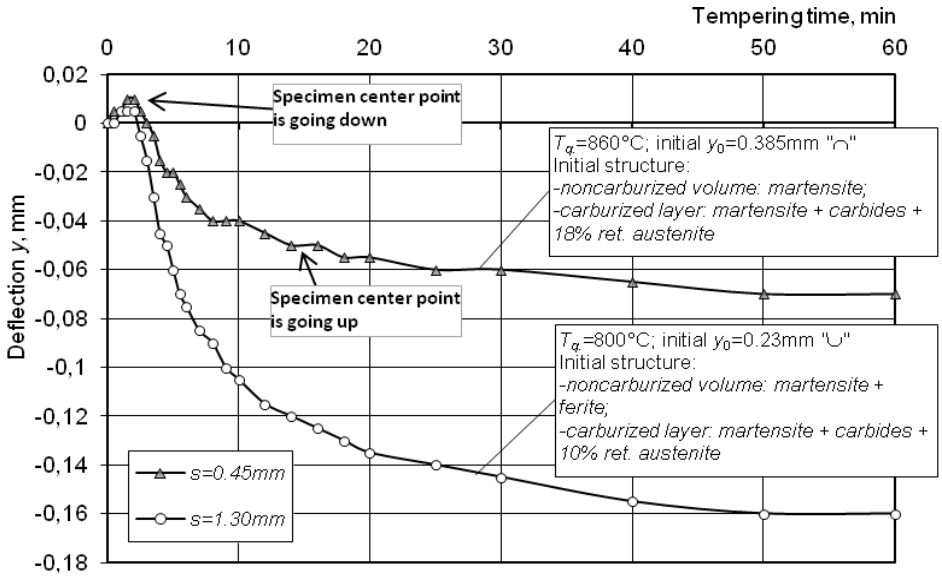

Fig. 10 Kinetics of deflection change of carburized and quenched specimens obtained during tempering at 200°C. Mentioned value *s* is carburized depth. Content of retained austenite is taken from literature [19]

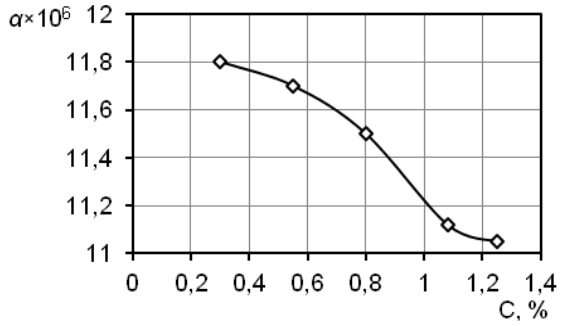

Fig. 11 Dependence between coefficient of thermal expansion of steel and carbon content [17]

There were two marginal values of carbon content used for calculation: 1.2% – of carburized surface [16] and 0.14% – of not carburized part. The results of calculation are presented in Table 2.

When carburized and quenched from 860°C specimen is placed in the furnace for heating at 200°C its initial structure is such as it was shown in Fig. 8. When the depth of carburized layer is s = 1.3 mm, the carburized volume forms approximately 22% part and not carburized one – ~78% of all volume of specimen. Not carburized volume (low carbon martensite) increases 0.67% when is heating. At carburized layer such volume increase of different micro-constituents is obtained:

- high carbon martensite – 0.61%;
- retained austenite – 1.22%;
- carbides – 0.67%.

In total carburized part of specimen increases approximately 0.75%, i.e. 20% more than not carburized part. The conclusion is that different increase of volumes during heating from 20°C to 200°C is a reason of specimen autobending downwardly (i.e. "crooked" specimen is straightening) at the first minutes of experiment.

When quenching temperature is 800°C the not carburized part of specimen may contain some ferrite because it's A_{c3} = 800°C [5]. Tempering of such quenched specimen at 200°C temperature provides the change of volume increase of different micro-constituents i.e. not carburized volume increases more than carburized one because of 0.8% expansion of ferrite. Carburized part of specimen contains less retained austenite – up to 10% [19], less carbides (Fig. 5, a), therefore the expansion of carburized layer is also less. The curve (-○-) in Fig. 10 proves this assumption.

Table 2
Relative volume increase during heating from 20°C to 200°C temperatures, g/cm^3

T_q, °C	Total C, %	T, °C	Austenite	Ferite	Martensite	Cementite
860	0.14	20			0.127560	
		200			0.128361	
	1.2	20	0.1259912		0.130517	0.1303276
		200	0.1275320		0.131318	0.1312060
800	0.14	20		0.127191		
		200		0.128186		

The specimen has started humping after ~2 min from the beginning of heating at 200°C. Net deflection of humping composed the most part of the total deflection – about 88% when T_q = 860°C and 97% when T_q = 800°C. Such increase of curvature of specimens' may be related with different changes of volumes of specimen parts with various amount of carbon during tempering process. When carburized surface is affected by tempering processes the martensite extricates a big amount of carbon so the tetragonality of martensite lattice and following volume increase shrinks more compared with the shrinkage in not carburized part. The difference of such unequal volume decrease influents humping of the specimen.

When specimen is loaded (100 MPa and 200 MPa) and tempered at 200°C, it humps nevertheless but the more diminutively the higher stress are (Fig. 12). Once the stress has reached 300 MPa the specimen stopped humping and after 4-5 min. the deflection of humped specimen hasn't changed. In consequence, the stresses formed

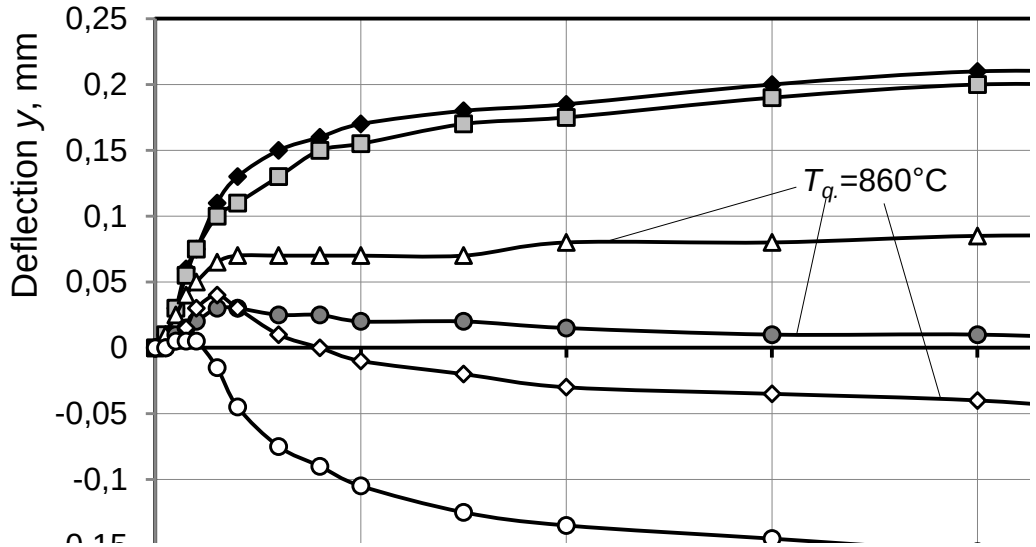

Fig. 12 Bending curves of carburized and quenched specimens during bending at 200°C by various bending stress. Depth of carburization is 1.3 mm

inside the specimen because of unequal volumes decrease of tempered structure are higher than 200 MPa. The following increase of load produces bigger deflection in ↓ direction.

There was a try to straighten a part of curved quenched specimens by using a transformation plasticity effect [10, 12, 14]. The past results of specimen bending test were used for this experiment. For this purpose modulus of transformation plasticity E_{tp} was calculated that has evaluated elastic-plastic properties of material during transformation:

$$E_{tp} = \frac{Pl^3}{48 I_x y}, \quad (2)$$

where: P is load, N; l is distance between supports (l = 82 mm), mm; I_x is moment of inertia (h = 5.85 mm; b = 7.87 mm), mm^4; y is total deflection after bending test during heating, mm. Reliability of results obtained was up to 10%. Furthermore, huge auto-deformations (sometimes more than 100%) were observed when already tempered and bent specimens were tempered second time, i.e. the specimens had restored the initial curved shape. The attempt of fixing of curved specimens has no point. The following experiments and investigations will be carried out for better understanding of peculiarities of difficult carburized structure and its behaviour during tempering and bending at the same time.

4. Conclusions

1. Asymmetrically carburized specimens have curved much more than specimens with homogeneous structure. The reason is the difference between M_S temperatures of high and mild carbon saturated volumes and related volume mismatch during martensite transformation and tempering.

2. There was not found enough data in world scientific literature to explain exactly mechanism of auto-deformation of carburized parts. Many fields still remain undiscovered – fundamentals of carbides formation in carburized layer, effect of internal and external stresses on deformation of parts and especially auto-deformations of quenched and bent during tempering specimens. For the purpose of the research these items, the area of investigation methods will be extended by using X-ray, dilatometric, electron microscopy and other testing.

3. The results of the experiment could be used for the development of heat treatment technologies of the carburized production.

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ĮANGLINTO LEGIRUOTO PLIENO GRŪDINIMO IR ATLEIDIMO DEFORMACIJŲ TYRIMAS

R e z i u m ė

Straipsnyje pateiktas mažaanglio nedidelio legiruotumo plieno bandinių su įanglintu paviršiumi plastinių savybių tyrimas žemojo atleidimo metu. Įanglinti grūdinti bandiniai dažnai deformuojasi dėl vidinių įtempių, kuriuos sukelia struktūros heterogeniškumas, tūriniai pokyčiai, skirtinga M_S temperatūra įanglintame ir neįanglintame tūryje, išorinės apkrovos, skirtingu laipsniu vykstantys virsminiai procesai, virsminio plastiškumo efektas ir kiti faktoriai. Ištirtas nesimetriško įanglinimo poveikis plieno virsminio plastiškumo efektui, įvertinant struktūrinius gniuždymo ir tempimo įtempius, atsiradusius dėl tūrinių pokyčių. Nustatyta, kad vidiniai įtempiai plieno bandinio struktūroje viršija 300 MPa. Gautus tyrimų rezultatus galima panaudoti įanglinamų detalių (velenų, krumpliaračių, kumštelių) terminio apdirbimo technologijoms tobulinti.

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INVESTIGATION OF HARDENING AND TEMPERING DEFORMATIONS OF CARBURIZED LOW ALLOY STEEL

S u m m a r y

The article presents the investigation of plastic properties of carburized steel specimens during low tempering. Carburized articles often deform during quenching. The reasons of curvature are internal stresses produced by heterogeneity of structure, volume mismatch, different M_S temperature of carburized and not carburized parts, uneven transformations, effect of transformation plasticity and other factors. The effect of asymmetrical carburization on steel transformation plasticity was examined evaluating the influence of structural compressive and tensile stress, which occurred because of volume mismatch. There was determined that internal stresses of carburized specimen exceed 300 MPa. The results of the experiment could be used for the development of heat treatment technologies of the carburized production – shafts, gears, cams, etc.

Keywords: steel, carburization, plastic deformation.

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