

STRUCTURE AND PROPERTIES OF THE CARBURISED SURFACE LAYER ON 35CrSiMn5-5-4 STEEL AFTER NANOSTRUCTURIZATION TREATMENT

STRUKTURA IN LASTNOSTI NAOGLJIČENE POVRŠINE JEKLA 35CrSiMn5-5-4 PO NANOSTRUKTURNI OBDELAVI

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Prejem rokopisa – received: 2014-10-08; sprejem za objavo – accepted for publication: 2014-12-09

doi:10.17222/mit.2014.255

The aim of the paper was to investigate the structure and properties of the carburized surface layer of the 35CrSiMn5-5-4 steel after the nanostructuring with the austempering heat treatment. During vacuum carburizing the surface layer of the steel was enriched with carbon above $w = 0.6\%$. Steel samples were subsequently austenitized, quenched at two different temperatures, 260 °C and 320 °C, and annealed at these temperatures for the time necessary for the completion of the bainitic transformation. For comparison, one set of carbonized samples was subjected to the conventional heat treatment: martensitic quenching and low tempering. The microstructural characterisation of the steel after different heat treatments was performed using scanning (SEM) and transmission (TEM) electron microscopes. It was shown that both austempering treatments led to a carbide-free nano-bainitic structure composed of nanometric ferrite plates separated by thin layers of retained austenite. The microhardness and wear resistance of three kinds of steel samples were investigated: the two subjected to the austempering treatment at different temperatures and the one subjected to the conventional treatment. It was shown that the nano-bainitic structure containing an increased amount of retained austenite displays a higher wear resistance than the tempered martensite. The results confirm that austempering can be a competitive method of thermal treatment, in comparison to the conventional heat treatment, for the steels after the carburizing process.

Keywords: vacuum carburizing treatment, carburized surface layer, carbide-free bainite, austempering, wear resistance

Namen tega dela je preiskati strukturo in lastnosti naogljčene površine jekla 35CrSiMn5-5-4 po nanostrukturiranju s toplotno obdelavo austempranja. Med naogljčanjem v vakuumu se je površina jekla obogatila z ogljikom nad masnim deležem $w = 0,6\%$. Vzorci jekla so bili segreti v avstenitno področje, gašeni na dve različni temperaturi: 260 °C in 320 °C, in zadržani na teh temperaturah, potrebnih za popolno bainitno pretvorbo. Za primerjavo je bila serija naogljčenih vzorcev toplotno obdelana po navadni metodi: gašenje v martenzit in popuščano pri nizki temperaturi. Karakterizacija mikrostrukture jekla po različnih toplotnih obdelavah je bila izvršena z vrstičnim (SEM) in presevnim (TEM) elektronskim mikroskopom. Izkazalo se je, da obe toplotni obdelavi austempranja povzročita nastanek nanobainitne strukture brez karbidov in sestavljene iz nanometrskih feritnih ploščic, ločenih s tanko plastjo zaostalega avstenita. Pri treh različnih vzorcih je bila izmerjena mikrotrdota in odpornost proti obrabi; pri dveh z austempranjem pri različnih temperaturah in enem z navadno toplotno obdelavo. Izkazalo se je, da ima nanobainitna struktura s povečanim deležem zaostalega avstenita večjo odpornost proti obrabi kot pa popuščeni martenzit. Rezultati so potrdili, da je toplotna obdelava z austempranjem boljša metoda toplotne obdelave kot navadna toplotna obdelava za jekla po postopku naogljčenja.

Ključne besede: naogljčenje v vakuumu, naogljčena plast, bainit brez karbidov, austempranje, odpornost proti obrabi

1 INTRODUCTION

Carbide-free bainite obtained in the bottom range of bainitic transformation is characterised by exceptionally high mechanical properties such as hardness and tensile strength.¹⁻⁵ Moreover, its ductility is kept due to a high volume fraction of the retained austenite.⁵⁻⁷ Simultaneously, a lack of cementite precipitations as well as the presence of a great amount of the retained austenite in the form of thin layers placed between the bainitic plates, result in a high fracture toughness.^{8,9} The presence of the retained austenite may also increase the frictional wear resistance.^{10,11} It was postulated¹⁰⁻¹² that during the wear tests austenite may transform into the strain-induced martensite due to the stresses occurring during the friction. This would increase the hardness in the contact

zone as well as the frictional wear resistance in comparison to the samples containing tempered martensite after the conventional treatment.¹⁰⁻¹²

Initially, the carbide-free structure was produced in the types of steel with specifically designed chemical compositions. These contained the appropriate amounts of carbon, silicon and manganese.¹³ However, recently an attempt was made to obtain such a structure also in the commercial types of steel.^{5,14,15} The main objective of the present study was to produce a structure of carbide-free bainite in the surface layer of the 35CrSiMn5-5-4 steel after the carburising. The second objective was to determine the effect of the isothermal-quenching temperature on the structure and the surface properties of the examined material.

2 EXPERIMENTAL PROCEDURE

2.1 Vacuum carburizing

The carburizing process was carried out in a 15.0VPT-4022/24N vacuum furnace at the Seco/Warwick Company. The carburizing treatment was conducted with the FineCarb® technology in accordance with the patent.¹⁶ It was divided into ten subsequent processes of saturation and diffusion. The times of particular saturation and diffusion processes were selected in accordance with the simulations conducted with the SimVaC Plus® programme. The carburising atmosphere was a mixture of acetylene, ethylene and hydrogen. Due to the possibility of a significant growth of austenite grains, the pearlitisation process was carried out directly after the carburisation process. Afterwards, the furnace feed was heated up to 850 °C, held for 20 min and, finally, slowly cooled to the room temperature at the furnace-cooling rate.

2.2 Carbon-content measurements

The amount of carbon after the carburisation process was determined on the cross-section of the layer, using the Magellan Q8 Bruker optical emission spectrometer with spark excitation. The study was conducted from the front of the sample. 21 measurements were carried out at 0.1 mm – a surface layer of about 0.1 mm was removed after each measurement to obtain the cross-section of the carbon content. The measurements were performed in an accredited laboratory, in accordance with the 3/CHEM procedure, 3 ed., Sep 2010, with a precision of 0.02 % (0.001 % for sulphur and phosphorus).

2.3 Heat treatment

In order to form a nano-bainitic microstructure, the carburised samples of steel were austenitized for 30 min

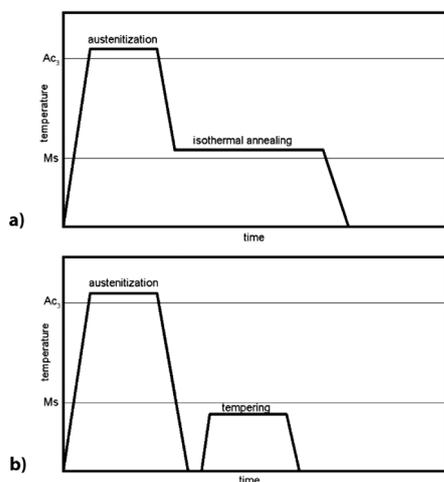


Figure 1: a) Scheme of the austempering and b) quenching and low-tempering treatment

Slika 1: Shematski prikaz toplotne obdelave: a) austempranje, b) gašenje in popuščanje pri nižji temperaturi

at a temperature of 900 °C, then cooled to the temperature range of the bainitic transformation and, finally, annealed at this temperature (**Figure 1**). The cooling medium used for isothermal quenching was composed of a liquid alloy of Sn-Ag heated up to the temperature of the isothermal step (260 °C and 320 °C). The time of isothermal quenching was selected in the way that ensured the end of the bainitic transformation at the temperature of isothermal annealing. The conventional heat treatment consisting of the martensitic quenching from the temperature of 900 °C and the subsequent tempering process at a temperature of 200 °C for 1h was performed in order to compare the properties of the layers obtained with two treatments. In this case, the oil quenching (WODOL) intended for structural steels was used as a cooling medium.

2.4 Microstructural observations

The microstructures of the austempered samples were examined with the use of light microscopy, SEM Hitachi S-3500N scanning electron microscopy and the TEM JEOL 1200 transmission electron microscope working at 120 kV. The LM and SEM observations were conducted just under the surface of the layer. At first, 50 µm of the examined material of the sample surface were grinded. Subsequently, the surfaces were etched with the Nital agent. The TEM observations were conducted at the surface zone of the layers at a depth of about 50–100 µm from the surface and in the core of the samples. Thin plates with a thickness of 200 µm were cut out of the samples, grinded to a thickness of 100 µm and, finally, electrolytically polished until perforation occurred. Phase constituents were identified according to the images of the electron diffraction analysis. The observations were carried out both in the bright field (BF) and in the dark field (DF), using the reflexions obtained from different phases.

The thickness of the ferrite plates and the austenite layers, observed on the TEM images, was determined in accordance with the following stereological Equation:¹⁷

$$d = \frac{2}{\pi} L \quad (1)$$

where d stands for the real size of the element of the microstructure (in the analysed case, the real thickness of a plate) and L means the size of the microstructure measured on the TEM image (in this case, the width measured on the image). The plate widths (L) were measured perpendicularly to the interphase boundaries.

The relative volume fraction of the phases was calculated on the assumption that the volume fraction of a given phase is equivalent to its area fraction observed on the image. Therefore, the n numbers of the secants of the l length were marked on the image of the microstructure. The volume fractions of phases V_V were calculated with the following formula:

Table 1: Chemical composition of the 35CrSiMn5-5-4 steel in mass fractions, w/%**Tabela 1:** Kemijska sestava jekla 35CrSiMn5-5-4 v masnih deležih, w/%

	C	Cr	Mn	Si	Ni	Cu	Al	Mo	W	Fe
w/%	0.35	1.31	0.95	1.3	0.14	0.15	0.04	0.018	<0.03	balance

$$V_v = \frac{\sum c_{ik}}{nl} \quad (2)$$

where $\sum c_{ik}$ is the sum of the widths of all the intersections of the secant line l with a given phase, l – the length of the secant line.

2.5 Microhardness measurements

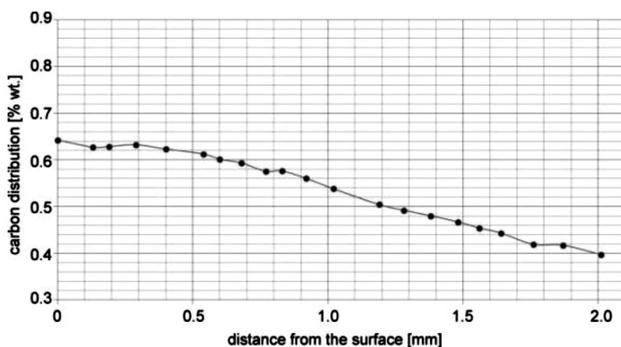
The measurements of the microhardness on the intersection of a layer were conducted with a LECO LM 248AT semi-automatic microhardness tester with the AMH43 v1.81 software in accordance with the PN-EN ISO 6507-1 norm.¹⁸ 1.96 N and 9.81 N loads, indenting the material for 10 s, were used in the investigation.

2.6 Wear-resistance measurements

The wear resistance measurements were carried out with the block-on-ring method using a T-05 tester, in conformity with the ASTM G77 norm.¹⁹ The examined sample was of a block shape and a width of 6.35 mm. The ring with an outer diameter of 34.99 mm was used as a counter specimen. The ring used in the research was made of the 100Cr6 bearing steel with a hardness of 62 HRC. The investigation lasted for 100 min and the applied unit load was 200 N mm⁻² and 400 N mm⁻². The rotation speed was 316 r/min (5.26 Hz) and the rubbing speed was 0.25 m/s. The track of the wear radius was 17.5 mm. Lux 10 oil was used as the grease.

3 RESULTS AND DISCUSSION

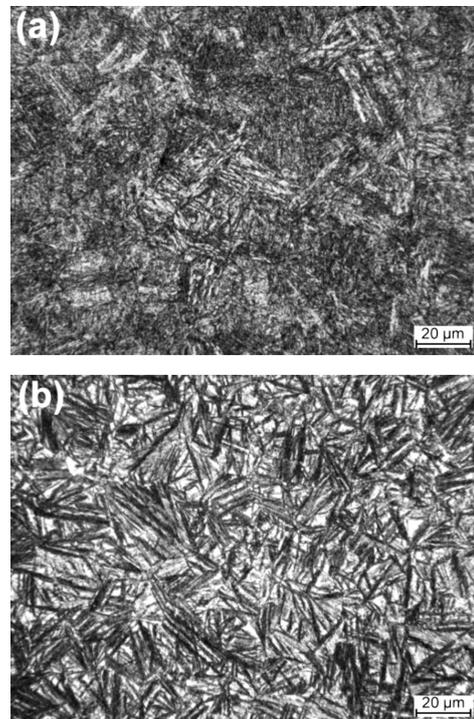
The previous research conducted on the 35CrSiMn5-5-4 steel indicated that the formation of carbide-free bainite structure with nanometric or submicron grain sizes can be achieved during isothermal quench-

**Figure 2:** Carbon-content profile for the carburized surface layer on 35CrSiMn5-5-4 steel**Slika 2:** Profil vsebnosti ogljika v naogljčeni plasti na jeklu 35CrSiMn5-5-4

ing.¹⁵ However, the amount of the retained austenite is relatively low due to a relatively low carbon content in this steel.¹⁵ In order to increase the carbon content in the surface layer, the 35CrSiMn5-5-4 steel with the chemical composition presented in **Table 1** was submitted to the vacuum carburizing treatment using the injection method. The increase in the carbon content facilitated the formation of a nano-bainitic structure in the steel.

The analysis of the chemical composition of the cross-section of the carburised layer of a steel sample indicated that the carbon content at the surface is $w = 0.64\%$. This value remains constant up to a depth of 0.5 mm from the surface. At a depth of 2 mm the carbon content is $w = 0.39\%$ (**Figure 2**).

A fine-grained acicular structure was formed during the isothermal quenching at the temperature of 260 °C, in the carburised layer of steel. The bainitic ferrite plates within this structure were either parallel to each other or arranged at different angles to each other. They formed groups or packages. An increase in the temperature of the treatment resulted in the microstructure in which small, unetched areas of the retained austenite occurred,

**Figure 3:** Microstructure of the carburized surface layer on 35CrSiMn5-5-4 steel after austempering at: a) 260 °C and b) 320 °C – LM images**Slika 3:** Mikrostruktura naogljčene površine jekla 35CrSiMn5-5-4 po austempranju na temperaturi: a) 260 °C in b) 320 °C (svetlobna mikroskopija)

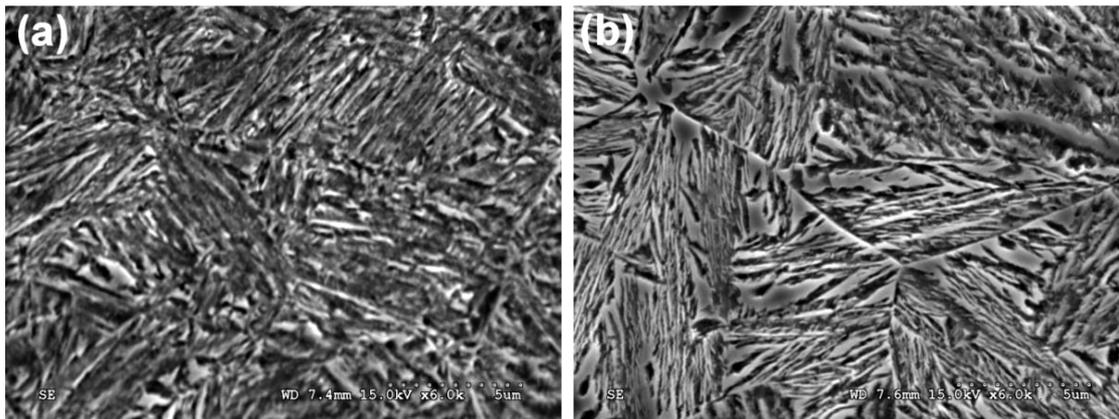


Figure 4: Microstructure of the carburized surface layer on 35CrSiMn5-5-4 steel after austempering at: a) 260 °C and b) 320 °C – SEM images
Slika 4: Mikrostruktura naogljicene plasti jekla 35CrSiMn5-5-4 po austempranju na temperaturi: a) 260 °C in b) 320 °C (SEM-posnetka)

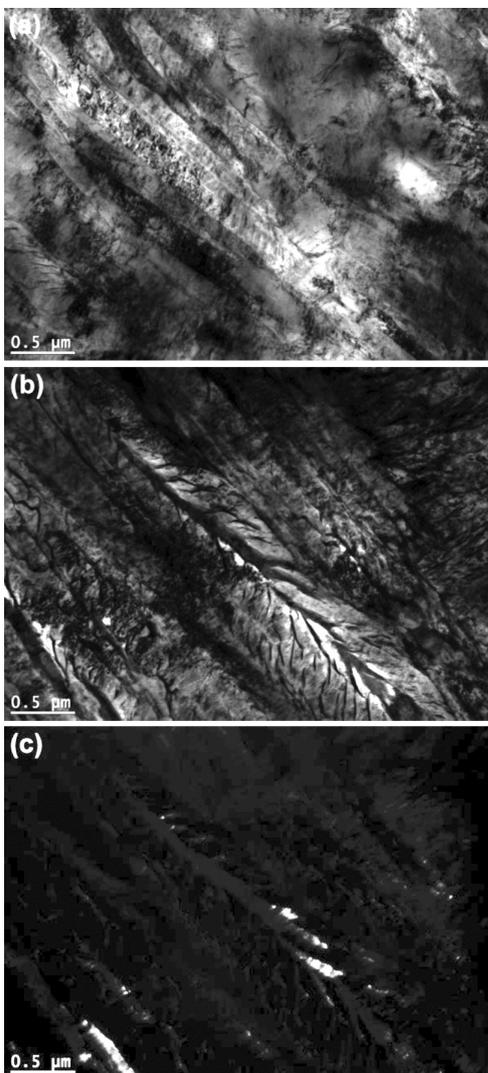


Figure 5: a) Microstructure of the carburized surface layer on 35CrSiMn5-5-4 steel after austempering at 260 °C, b) TEM BF image, c) TEM DF image of austenite reflection
Slika 5: a) Mikrostruktura naogljicene plasti na jeklu 35CrSiMn5-5-4 po austempranju na temperaturi 260 °C, b) TEM BF-posnetek, c) TEM DF-posnetek odboja avstenita

apart from the fine bainite plates arranged parallelly to each other (**Figures 3 and 4**).

A detailed investigation, conducted with TEM, of the microstructure of a steel sample treated at the temperature of 260 °C revealed the presence of martensite and carbide-free nano-bainite. The martensite and bainite were separated from each other with the layers of retained austenite (**Figure 5**). The average measured width of the ferritic and martensitic plates was (102 ± 7) nm, whereas the width of the layers of the retained austenite was (33 ± 3) nm. The increase in the temperature of the

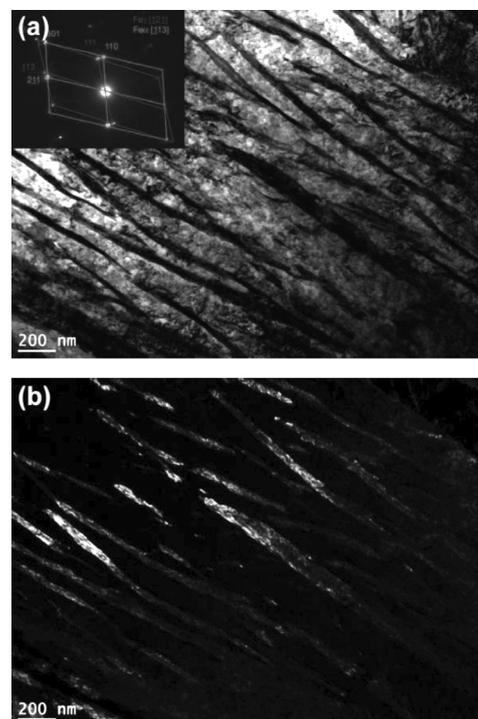


Figure 6: Microstructure of the carburized surface layer on 35CrSiMn5-5-4 steel after austempering at 320 °C: a) TEM BF image, b) TEM DF image of austenite reflection
Slika 6: Mikrostruktura naogljicene površine jekla 35CrSiMn5-5-4 po austempranju na 320 °C: a) TEM BF-posnetek, b) TEM DF-posnetek odboja avstenita



Figure 7: Block of retained austenite in the carburized surface layer on 35CrSiMn5-5-4 steel after austempering at 320 °C

Slika 7: Kos zaostalega avstenita v naogljiveni površini jekla 35CrSiMn5-5-4 po austempranju na 320 °C

treatment to 320 °C allowed the formation of a structure consisting of carbide-free bainite with the retained austenite. The bainitic plates have the average width of $65 \text{ nm} \pm 4 \text{ nm}$, whereas the layers of the retained austenite are $26 \text{ nm} \pm 2 \text{ nm}$ thick (**Figure 6**). Additionally, blocks of austenite with an area not exceeding $1 \mu\text{m}^2$ were observed (**Figure 7**).

The relative volume fraction of the retained austenite in the surface layer, estimated on the basis of the TEM images was $(20.5 \pm 3.5) \%$ and $(20.2 \pm 3.5) \%$ for the samples isothermally quenched at 260 °C and 320 °C, respectively. The microstructure obtained through isothermal quenching at 320 °C was relatively homogenous

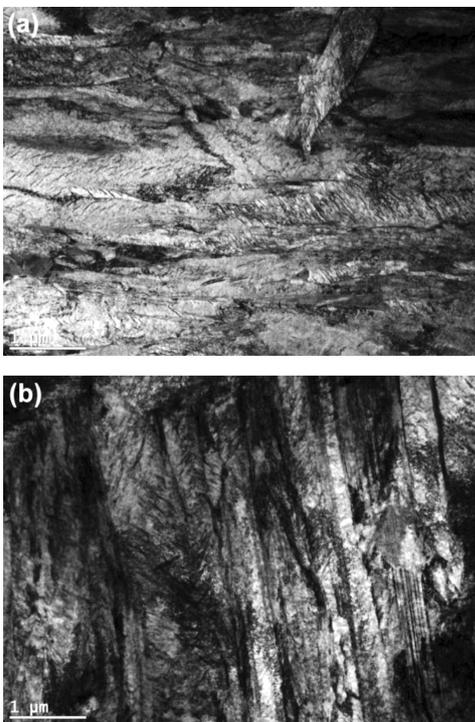


Figure 8: Microstructure of the core of the carburized steel after austempering at 260 °C

Slika 8: Mikrostruktura jedra pri naogljivenem jeklu po austempranju na 260 °C

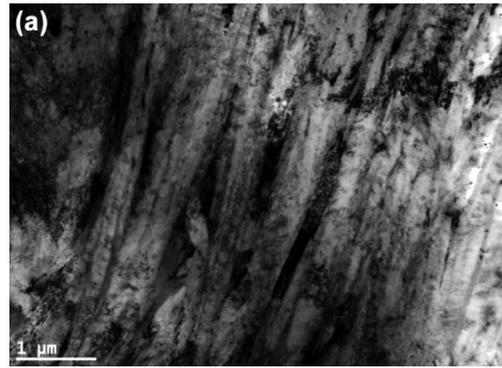


Figure 9: Microstructure of the core of the carburized steel after austempering at 320 °C

Slika 9: Mikrostruktura jedra pri naogljivenem jeklu po austempranju na 320 °C

in terms of the grain size. However, areas with different amounts of austenite were observed in the structure, as well as some individual secondary carbides that did not form clusters.

In the cores of the samples treated at 260 °C a martensitic-bainitic structure was formed. The presence of martensite resulted from the M_s temperature of 307 °C which is higher than the austempering temperature. The presence of highly dense carbides in martensite laths indicates that the tempering process occurred in the core of a sample during the austempering (**Figure 8**). The

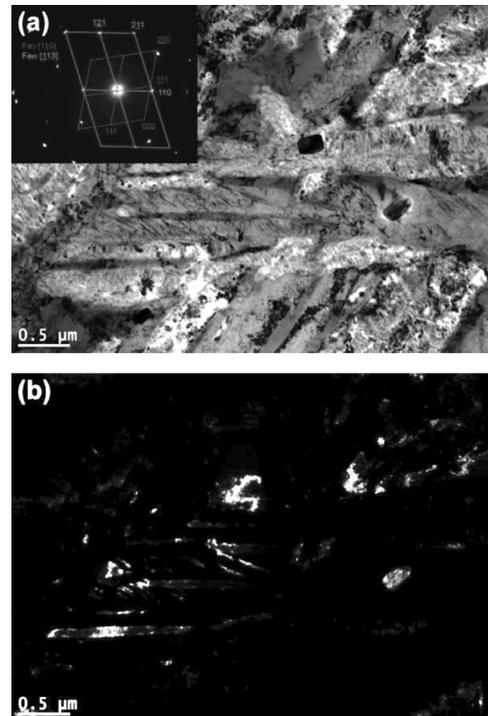


Figure 10: Austenite areas in the core of the carburized steel after austempering at 320 °C: a) TEM BF image, b) TEM DF image of austenite reflection

Slika 10: Avstenitna področja v jedru pri naogljivenem jeklu po austempranju na 320 °C: a) TEM BF-posnetek, b) TEM DF-posnetek odboja avstenita

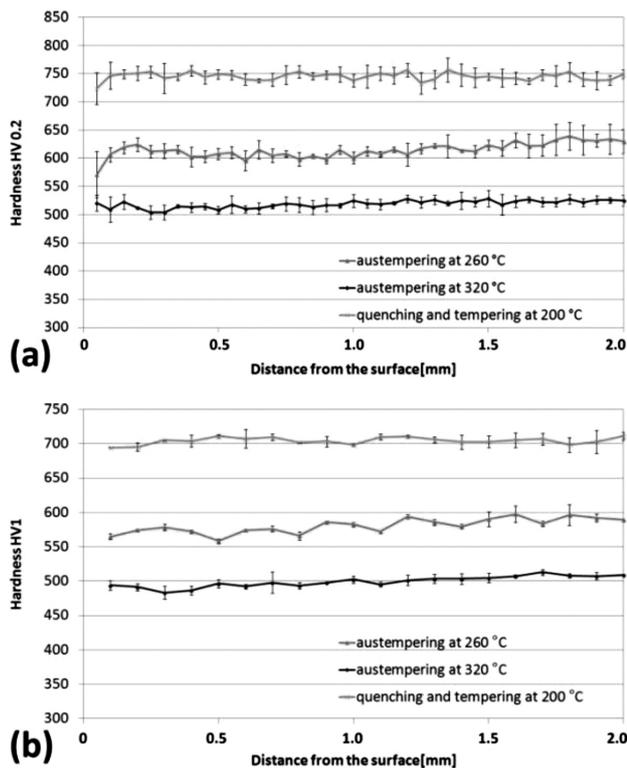


Figure 11: Hardness: a) HV_{0.2} and b) HV₁ profiles for the carburized surface layer on 35CrSiMn5-5-4 steel after various heat treatments
Slika 11: Profili trdote: a) HV_{0.2} in b) HV₁ v naogljivičeni plasti jekla 35CrSiMn5-5-4 po različnih toplotnih obdelavah

average grain size was (152 ± 23) nm. During the austempering at 320 °C carbide-free bainite of a submicron grain size $140 \text{ nm} \pm 10 \text{ nm}$ was formed in the core (**Figure 9**). However, a relatively large number of larger ferrite grains of about $0.5 \mu\text{m}$ were also observed (**Figure 10**). In both cases, the relative austenite volume fraction in the core, equal to $(13 \pm 3) \%$ and $(11.4 \pm 2.3) \%$ for the austempering at 260 °C and 320 °C, respectively, was significantly lower than in the carburised layer. Austenite mainly occurred in three different forms: as very thin layers $(29 \pm 4) \text{ nm}$ in the case of the treatment carried out at 260 °C; as thicker layers $(46 \pm 6) \text{ nm}$; and small blocks $(0.086 \mu\text{m}^2\text{--}0.288 \mu\text{m}^2)$ in the case of the treatment conducted at 320 °C.

In the case of the austempering at 320 °C, which is higher than the M_s temperature, various factors may affect the structure of the core as well as the volume fractions of certain phases. The previous studies conducted on the 35CrSiMn5-5-4 steel revealed that the time necessary for completing the process of the bainitic transformation at 320 °C was slightly below 2 h.¹⁵ According to the literature,^{20,21} extending the time of isothermal treatment over the critical value results in a coalescence of ferritic grains and leads to the formation of austenite in the form of blocks. The increase in the grain sizes of particular phases may be also affected by the temperature of the treatment.²¹ Simultaneously, the

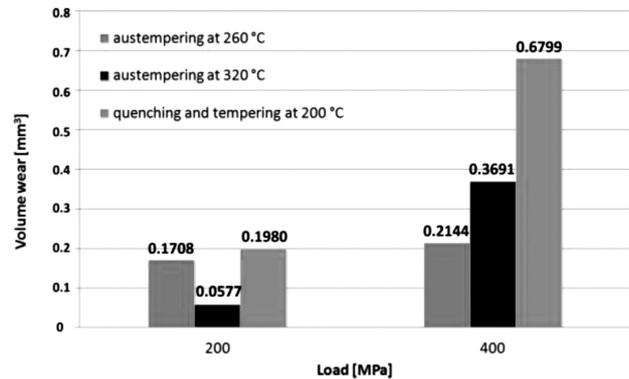


Figure 12: Volumetric wear of the carburized surface layer on 35CrSiMn5-5-4 steel after various heat treatments

Slika 12: Volumenska obraba naogljivičene plasti jekla 35CrSiMn5-5-4 po različnih toplotnih obdelavah

relative volume fraction of the retained austenite has a tendency to decrease with the increase in the time of the treatment.²⁰

The heat treatment described in the present study lasted for 24 h. Therefore, the grain sizes of ferrite and austenite in the core increased, whereas the relative austenite volume fraction decreased during the austempering in comparison with the previous treatment of this type of steel conducted for 2 h.¹⁵

The hardness of the layers was measured at different loads. In the case of a carburised, quenched and low-tempered layer, the hardness was higher than the hardness of the carburised steel submitted to isothermal quenching at 260 °C of approximately 130 HV_{0.2} and 125 HV₁ units. It was also higher than the hardness of the steel after the treatment at 320 °C of about 225 HV_{0.2} and 205 HV₁ units (**Figure 11**). This may be associated with a high amount of the relatively soft retained austenite in the nano-bainitic structure. The hardness does not change on the cross-section of a layer in the case of the steel subjected to the conventional treatment. After the nanostructuring process, the hardness of the steel slightly increased when the distance from the surface increased. With the increasing distance from the surface, the grain size also increases which should result in a decrease in the hardness. However, both the carbon content and the relative austenite volume fraction in the nano-bainitic structure decreased; therefore, the hardness in the core of the austempered samples is slightly higher.

The results of the investigations on the frictional wear resistance with the applied loads of 200 MPa and 400 MPa are presented in **Figure 12**. The level of the volume wear increases with an increase in the applied load. The wear tests indicate that the layer that was carburised and then austempered is characterised by a significantly higher frictional wear resistance in comparison to a much harder carburised layer, quenched and tempered in the conventional way. This difference is particularly pronounced in the case of the layer austempered at 260 °C. Most likely, this is associated with the amount of

the retained austenite in the layer with a nano-bainitic structure. Although the austenite is a relatively soft phase, it may transform into martensite under the stresses occurring during the friction due to the TRIP effect.⁹⁻¹² As a result of this effect, the hardness in the contact zone would increase significantly. This, in turn, may improve the frictional-wear resistance in comparison with the samples treated in the conventional way.¹⁰⁻¹² However, in order to prove this theory, a further study of the phase composition performed with XRD is required.

4 CONCLUSIONS

The austempering process conducted on the carburised 35CrSiMn5-5-4 steel allowed the formation of a nano-bainitic structure in the carburised layer as well as the formation of a martensite or carbide-free bainite structure with submicron grain sizes in the core of the examined material.

The increase in the carbon content in the layer during the carburizing favours the formation of the retained austenite, which may undergo a martensitic transformation due to the stresses during the wear test. The formed martensite may significantly increase the frictional-wear resistance of the examined steel.

Low-temperature isothermal quenching may be a new heat-treatment method applied on steel after carburisation. It is an alternative to the conventional treatment of quenching and low tempering. It seems beneficial in terms of the properties of a layer such as the increase in the frictional-wear resistance and lower quenching distortions.

Acknowledgements

The results presented in this paper were obtained within the project "Production of nanocrystalline steels using phase transformations" – NANOSTAL (contract no. POIG 01.01.02-14-100/09 made with the Polish Ministry of Science and Higher Education). The project is co-financed by the European Union from the European Regional Development Fund, within Operational Programme Innovative Economy 2007-2013.

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