

FORMATION OF NANOBAINITIC STRUCTURE IN CARBURIZED LAYER OF STRUCTURAL STEELS

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Abstract

Carburization, followed by quenching and tempering, is commonly used process to increase hardness and wear resistance of low carbon steels. Such process, besides improving mechanical properties and performance of steel surface, may cause however undesirable side effects such as: increased residual stress in surface layer, distortion of heat treated element or even initiation of cracking. The aim of the work was to reduce those possible risks by developing an alternative heat treatment of steel after carburization. Research data indicate that isothermal holding within the range of temperatures in which bainite is formed results in a reduction of residual stresses and distortion in comparison to quenching and tempering. Austempering of steels with increased content of silicon or aluminium at temperature slightly above the M_s may improve the mechanical properties as compared with quenching and tempering.. This is because the austempering performed at low temperatures range of bainitic transformation may lead to a nano-sized carbide-free bainite formation. Moreover the Si and Al additions hinders or even suppress the cementite precipitation which has a harmful effect on the resistance to cracking.

For the purpose of this study two structural steels were chosen: 35CrSiMn5-5-4 containing 1.3 wt.% of silicon and 38CrAlMo6-10 which contains 0.3 wt.% of silicon and 1 wt.% of aluminium. Both steels were subjected to vacuum carburization, then they were austenitized and austempered at temperature close to the temperature M_s of the surface layer. Transmission electron microscope (TEM) observations revealed that after austempering the carburized layer are composed of a nano-sized bainite plates separated by thin films of retained austenite. The results of hardness and wear resistance tests indicate that the austempering is advantageous as compared to the conventional quenching and tempering process.

Keywords: steel, vacuum carburization, austempering, nanobainite, retained austenite

1. INTRODUCTION

One of the most promising ways of new steels development is formation of nanocrystalline structure through bainitic transformation [1-5]. For certain steels with accurate chemical composition austempering at low temperature provides a carbide-free microstructure, containing nanometric laths of bainitic ferrite separated by films of retained austenite [2,5]. Such steels must contain 0.6÷1.1 wt.% of carbon and increased amount of silicon or aluminium to hinder cementite precipitation [1-3]. Steels that meet aforementioned conditions are reported to show, after austempering, tensile strength $R_m = 1926 \div 2098$ MPa, elongation of 3.1 ÷ 11.3 %, hardness HV30 = 590 ÷ 690 and fracture toughness $K_{IC} = 45 \div 135$ MPa m^{0.5} [5,6].

Recent works [7-9] shows that it is possible to achieve nanobainitic microstructure in carburized layers of low carbon steels. Replacing hitherto the conventional quenching and low tempering of carburized steels, by austempering may be beneficial in terms of reduction of residual stress level, reduction of distortion and increase of wear resistance [9].

The aim of this study was an attempt to produce a nanobainite in surface layer of two structural steels used commonly in industry: 38CrAlMo6-10 and 35CrSiMn5-5-4. Vacuum carburizing was performed beforehand to

acquire proper carbon content. Subsequently, part of samples was austempered while the other part was conventionally quenched and tempered. This permitted us to compare the wear properties of the two kinds of samples.

2. EXPERIMENTAL

2.1. Material

The chemical composition of both steels used in this study is given in table 1. Initially it was assumed that content of 1% Al in 38CrAlMo6-10 steel and 1.30% Si were sufficient to hinder carbides precipitation during austempering.

Table 1. Chemical composition of the investigated steels.

Steel	Weight %.										
	C	Mn	Si	P	S	Cu	Cr	Mo	V	Al.	Ni
38CrAlMo6-10	0.40	0.65	0.32	0.017	0.003	0.16	1.54	0.25	-	0.99	0.20
35CrSiMn5-5-4	0.35	0.95	1.30	0.012	0.007	0.15	1.31	0.018	0.006	0.04	0.14

2.2. Heat treatment

Samples in shape of flat bars were vacuum carburized using FineCarb® technology [10] by SECO/Warwick company in 15.0VPT-4022/24N vacuum furnace. As a carburizing agent the mixture of acetylene, ethylene and hydrogen was used. Process parameters were chosen on the basis of computer simulations performed using SimVac Plus® software. Carburization was carried out in 1000°C and divided into 10 alternating cycles of carburizing and diffusion, followed by 2h of diffusion. To reduce austenite grain size afterwards, pearlitization in 650°C was carried out, followed by austenitization in 840°C and cooling with the furnace. Carbon content distribution in carburized layers was measured by means of emission optical spectroscopy.

To select the optimal heat treatment parameters, the phase transformations that occur in carburized layer and in core of investigated steel samples were investigated using Bähr 805L quenching dilatometer. Based on above, the austempering temperature was chosen slightly above the Ms of carburized surface and the time of isothermal holding was set to finish the bainitic transformation. The parameters are given in table 2.

Table 2. Heat treatment parameters.

	Austenitization		Austempering	
	T [°C]	t [min]	T [°C]	t [h]
38CrAlMo6-10	930	15	250	48
35CrSiMn5-5-4	900	30	320	24

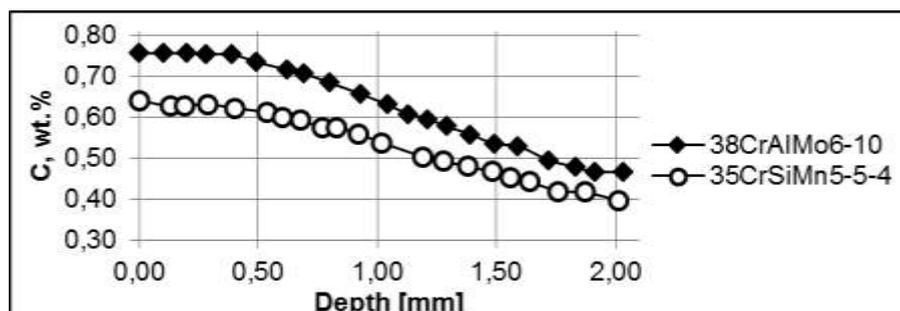


Fig. 1. Carbon content distribution in carburized layers of investigated steels.

Steel samples were austenitized in a controllable gas furnace with nitrogen atmosphere, then instantly immersed into Sn bath for austempering. A part of samples was quenched in oil and then tempered in 200°C. This permitted us to compare the wear properties of the two kinds of samples.

2.3. Characterization of microstructure

Microstructure observations were carried out using JEOL 1200 Transmission Electron Microscope operated at 120 kV. Thin foils of 250 µm thickness were cut from the carburized surface layer and from the core of the samples, then grinded to 100 µm thickness with a sandpaper and electropolished to perforation using Struers electropolisher with 100% glacial acetic acid.

2.4. Characterization of mechanical properties

Hardness distribution in carburized layer were investigated in Vickers scale with Zwick 3212002 hardness tester. The applied load was 200g.

Wear tests were performed according to ASTM G77 standard with T-05 tester, under loads of 200N and 400N. Wear samples were in shape of cuboids 6.35 mm thick. As a counterpart ring made of 100Cr6 steel with 62HRC surface hardness was used. Linear sliding velocity was calculated to be 0.25 m/s and rotation speed of the counterpart was 316 min⁻¹. Time of single wear test was 100 min. After wear slide, volume loss of tested samples was estimated according to wear width measurements.

3. RESULTS

3.1. Microstructure

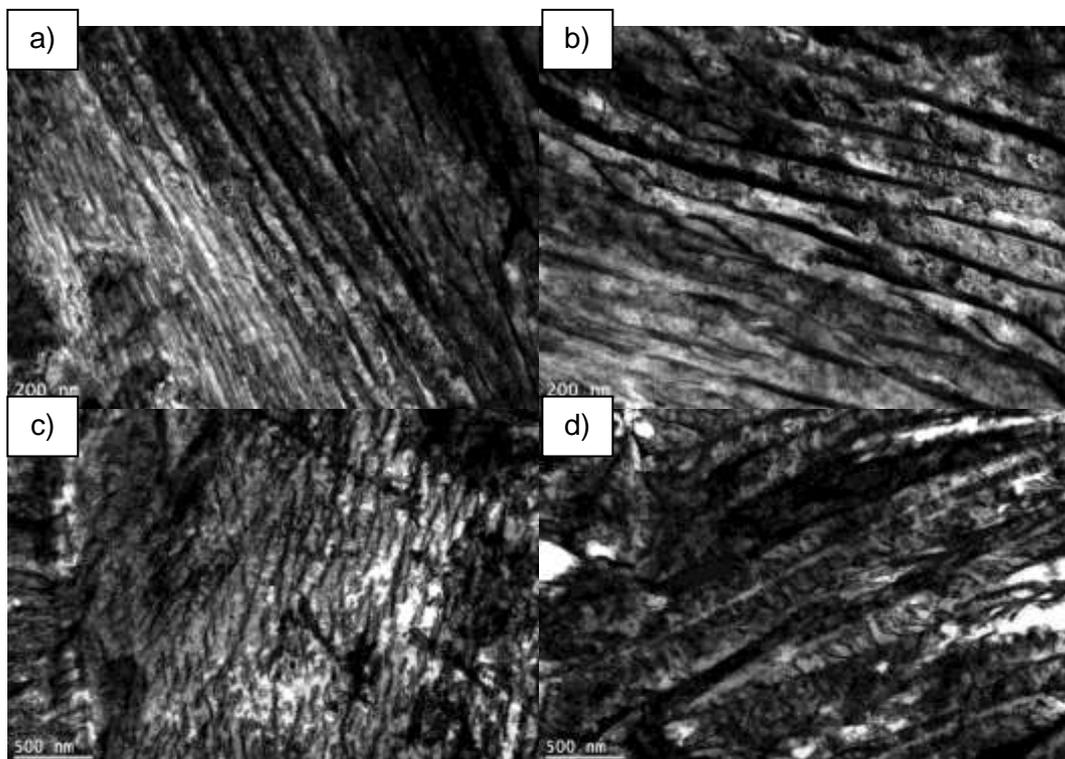


Fig. 2. TEM image of microstructure of 38CrAlMo6-10 steel after austempering at 300°C: a), b) - carburized layer, c), d) - core

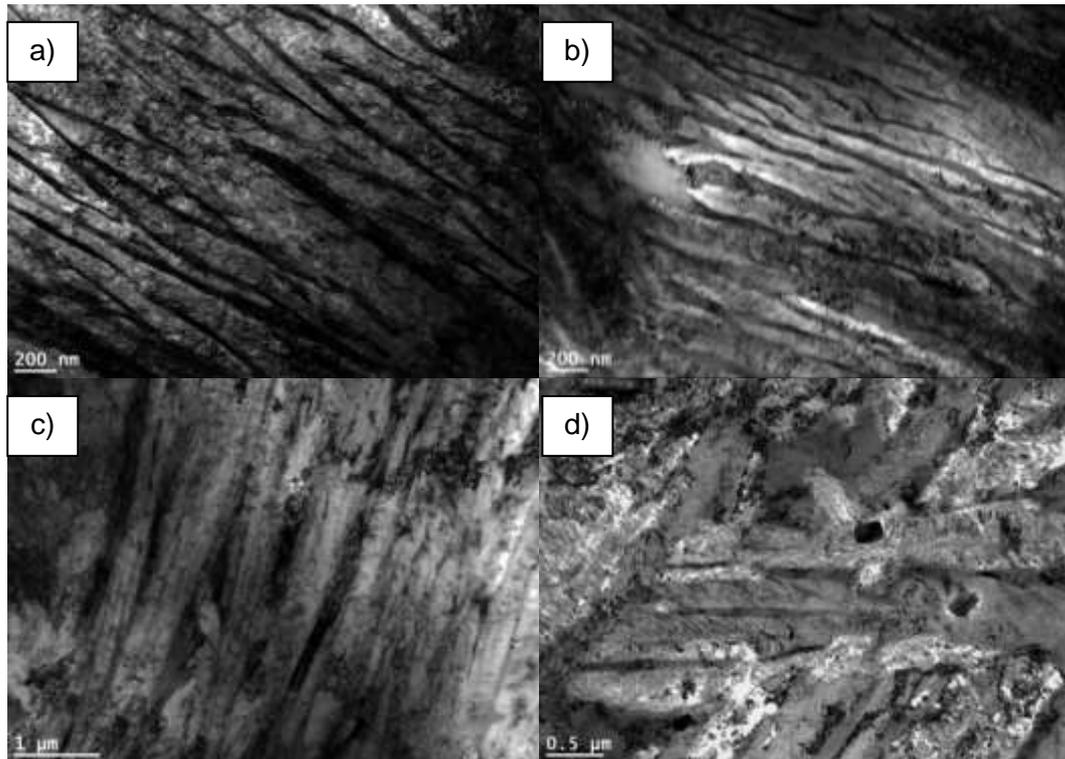


Fig. 4. Microstructure of the carburized surface layer on 35CrSiMn5-5-4 steel after austempering at 320 °C: a), b) - carburized layer, c), d) - core

TEM observations of surface layer in 38CrAlMo6-10 steel revealed a heterogeneous microstructure, containing a typical nanobainite mixed with occasional lower bainite laths. Small amount of cementite precipitated on ferrite-austenite interfaces and within bainitic ferrite was also found. The width of bainitic ferrite laths varied from 15 nm to 293 nm (mean value was 84 ± 6 nm). The laths of bainite were separated by retained austenite films with thickness varied from 7 nm to 101 nm (32 nm on average). There were also observed sparse, micrometric bainite laths containing coarse (up to 28 nm) cementite plates as well as blocks of untransformed austenite sized from $0.09 \mu\text{m}^2$ up to $0.33 \mu\text{m}^2$. Total austenite volume content was estimated to be $14.28 \% \pm 2.4 \%$.

Observation of the core of steel samples revealed heterogeneous microstructure consisted of martensite, carbide-free bainite and retained austenite. Some areas with typical nanobainite have been found. Bainite laths differed greatly in size., from 11 nm to 140 nm (mean value of $64 \text{ nm} \pm 13 \text{ nm}$). The thickness of retained austenite films varied from 9 nm to 19 nm (mean value of $15 \text{ nm} \pm 3 \text{ nm}$). These areas were bordered with relatively large, submicron bainitic ferrite laths (fig. 2c, d). A small areas of martensite were also found in microstructure. This can be explained by the austempering temperature which was slightly below M_s of the core of 38CrAlMo6-10 steel.

TEM investigation of carburized layer in 35CrSiMn5-5-4 steel revealed carbide-free nanobainite composed of ferrite laths separated by films of retained austenite (fig. 3). Measured ferrite laths width varied between 15 nm and 201 nm (mean $65 \text{ nm} \pm 4 \text{ nm}$) and austenite film width varied between 3 nm to 135 nm (mean $26 \text{ nm} \pm 2 \text{ nm}$). Blocks of residual austenite were also observed, varying in size from $0.09 \mu\text{m}^2$ to $0.93 \mu\text{m}^2$. Estimated from TEM micrographs austenite volume content was $20.2 \% \pm 3.5 \%$. Investigated microstructure was relatively homogenous, however the areas differing in austenite content were observed. Sparse equiaxial carbides remaining from initial microstructure were also found.

As a result of austempering a microstructure of carbide-free bainite with 11 - 370 nm wide ferrite laths was obtained in the core of the sample (fig. 4c). The mean bainitic ferrite lath width was calculated to be 140 nm \pm 10 nm. Retained austenite was present mostly as small blocks (from 0.086 μm^2 to 0.288 μm^2) or coarser layers 4 nm - 176 nm (mean 46 nm \pm 6 nm) thick. Austenite content was estimated at 11.38 % \pm 2.3 % and is less than in the carburized layer. Significant number of large bainitic ferrite laths (up to 0.5 μm) were observed (fig 4d). In papers [11, 12] it was found that if the time of austempering exceeded a critical value the coalescence of ferrite plates occurred and austenite blocks formed. Simultaneously, volume content of retained austenite decreased with the time of isothermal holding [12].

3.2. Hardness

Hardness distribution curves of carburized layers are shown on figure 5. Each point on the graph represents mean of three measurements. In 38CrAlMo6-10 steel hardness increased from 580 HV0.2 at the surface to about 660 HV0.2 at the distance of 300 μm from surface and remained at this level to the depth of 1300 μm . With further increase of the distance from the surface, a slight decrease of hardness to the value of 600 HV0.2 occurred. Lowered hardness at the surface of 38CrAlMo6-10 steel might result from microstructure heterogeneity or surface decarbonising. Hardness of 35CrSiMn5-5-4 was almost constant across the specimen varying between 520 HV0.2 at the surface to 525 in the core. The lack of hardening effect associated with the increased carbon content in surface layer is most probably due to the softening caused by the increased content of retained austenite in the surface layer. The retained austenite content decreases with increasing distance from surface, which results in constant hardness across the sample. Measured hardness of samples after quenching and tempering was respectively 690 HV0.2 for 38CrAlMo6-10 steel and 720 HV0.2 for 35CrSiMn5-5-4 steel.

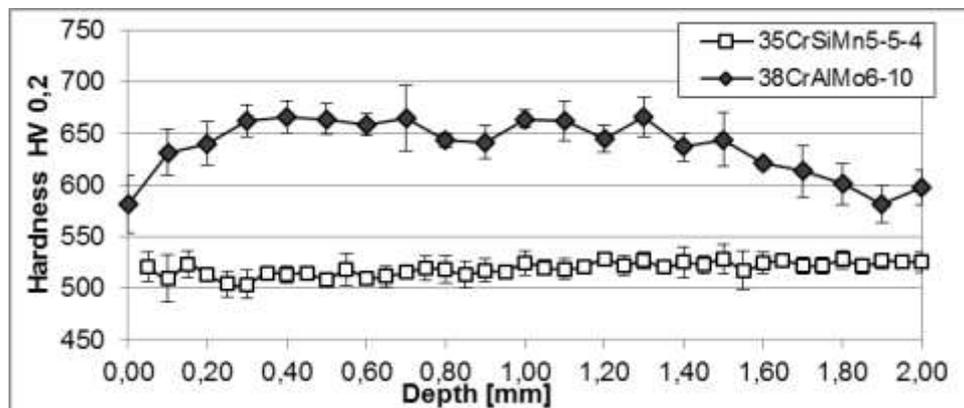


Fig. 5. Hardness distribution in carburized layers after austempering.

3.3. Wear analysis

Figure 6 shows volume loss of steel samples after wear tests as a function of applied load. In general, 38CrAlMo6-10 steel demonstrated less material loss than 35CrSiMn5-5-4 in all conditions. Both carburized steels manifested lower wear rate after austempering than after traditional heat treatment. Austempered 38CrAlMo6-10 steel samples exhibited lower volume loss of about 30 % under 200 N and 40 % under 400 N load than samples after quenching and tempering. In the case of 35CrSiMn5-5-4 steel the volume loss were respectively 70 % and 45 % lower for austempered samples than for the samples quenched and tempered. Better wear resistance of nanobainitic microstructure compared to tempered martensite may be interpreted as a result of relatively high volume fraction of retained austenite in carburized surface layer after austempering. The stresses induced during wear test at the carburized surface layer caused the transformation of austenite into martensite due to the TRIP effect [9, 13]. This resulted in a drastic increase of hardness in wear area, which led to the wear resistance improvement.

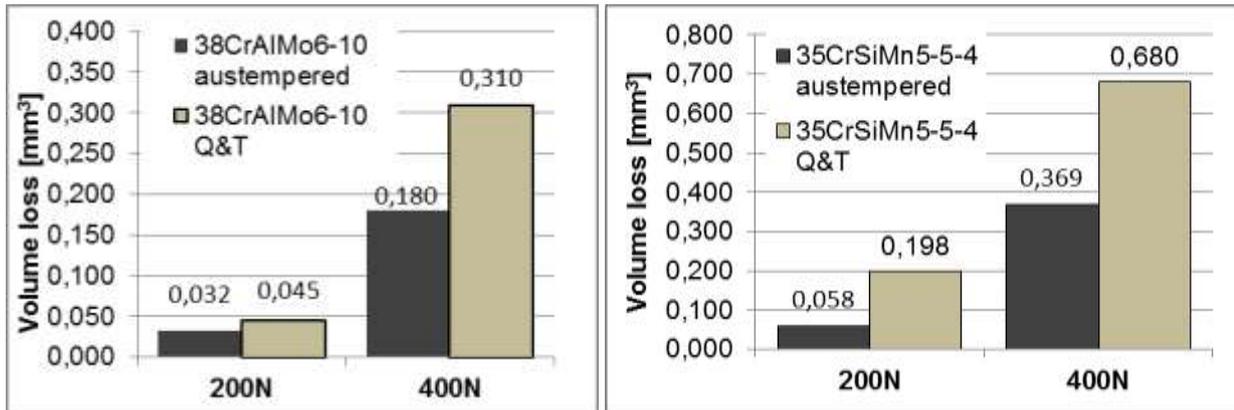


Fig. 6. Volume loss after wear sliding test.

CONCLUSION

Austempering of carburized 38CrAlMo6-10 and 35CrSiMn5-5-4 structural steels resulted in formation of nanobainite in carburized surface layer. The nanobainite was composed of ultra-thin ferrite laths in film-like retained austenite matrix. Nanobainite in carburized layer of 35CrSiMn5-5-4 steel was slightly finer and more homogeneous compared to this, which was formed in 38CrAlMo6-10 steel surface. Samples of both steels contained in the core a bainitic ferrite plates of various sizes mixed with retained austenite and, in the case of 38CrAlMo6-10 steel, a small quantity of martensite. Presence of the carbides in 38CrAlMo6-10 steel indicates that addition of 1 % of Al was not sufficient to inhibit the carbide precipitation. The nanobainite, obtained during austempering, despite its lower hardness than tempered martensite, exhibited significantly better wear resistance, compared to the latter. Improvement of wear resistance could result from microstructure refinement as well as from presence of retained austenite, which transformed into martensite under applied load during wear tests.

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LITERATURE

- [1] CABALLERO F. G., BADESHIA H. K. D. H., MAWELLA K. J. A.: Very strong low temperature bainite, *Mat. Sci and Technol.* 18 (2002), 279÷284
- [2] GARCIA-Mateo C., CABALLERO F. G., BADESHIA H. K. D. H.: Acceleration of low-temperature bainite, *ISIJ Int.* 43 (2003) 1821÷1825
- [3] GARCIA-MATEO C., CABALLERO F. G., BADESHIA H. K. D. H.: Development of hard bainite, *ISIJ Int.* 43 (2003) 1238÷1243
- [4] DHARAMSHI H. K., BADESHIA H. K. D. H., GARCIA-MATEO C., BROWN P.: Bainite Steel and Methods of Manufacture Thereof, Patent Application Publication, No US 2011/0126946 A1, 2 June 2011
- [5] ŚWIĄTNIKI W.A., POBIEZIŃSKA K., SKOLEK E., GOŁASZEWSKI A., MARCINIĄK Sz., NADOLNY Ł., SZAWŁOWSKI J.: Otrzymywanie struktury nanokrystalicznej w stalach przy wykorzystaniu przemiany bainitycznej, *Inżynieria Materiałowa*, rok XXXIII, nr 6 (2012) 524-529

- [6] CABALLERO F. G., BADESHIA H. K. D. H.: Very strong Bainite, *Curr. Opin. Solid State Mat. Sci.* 8 (2004) 251÷257
- [7] ZHANG F. C., WANG T. S., ZHANG P., ZHENG C. L.: A novel method for the development of a low-temperature bainitic microstructure in the surface layer of low-carbon steel, *Scripta Mat.* 59 (2008) 294÷296
- [8] ZHANG P., ZHANG F. C., WANG T. S.: Preparation and microstructure characteristics of low-temperature bainite in surface layer of low carbon gear steel, *Applied Surface Sci.* 257 (2011) 7609÷7614
- [9] ZHANG P., ZHANG F. C., YAN Z. G., WANG T. S., QIAN L. H.: Wear property of low-temperature bainite in the surface layer of a carburized low carbon steel, *Wear* 271 (2011) 697÷704
- [10] Kula P., Olejnik J., Heilman P.: European Patent No.: EP1558780 (2007), United States Patent No.: US 7513958 (2009)
- [11] BHADESHIA H.K.D.H., Nanostructured bainite, *Proc. R. Soc. A* 466 (2010) 3-18.
- [12] LIU C., ZHAO Z., BHOLE S.D., Lathlike upper bainite in a silicon steel, *Materials Science and Engineering A* 434 (2006) 289–293
- [13] DE COOMAN B.C., Structure–properties relationship in TRIP steels containing carbide-free bainite, *Current Opinion in Solid State and Materials Science* 8 (2004) 285–303