

K. WASILUK\*, E. SKOLEK\*, W. ŚWIĄTNICKI\*

## MICROSTRUCTURE AND PROPERTIES OF SURFACE LAYER OF CARBURIZED 38CrAlMo6-10 STEEL SUBJECTED TO NANOSTRUCTURIZATION BY A HEAT TREATMENT PROCESS

### STRUKTURA I WŁAŚCIWOŚCI WARSTWY NAWĘGLANEJ NA STALI 38CrAlMo6-10 PO PROCESIE NANOSTRUKTURYZACJI W WARUNKACH OBRÓBKI CIEPLNEJ

The aim of the study was to produce and characterize a nanobainitic microstructure in surface layers of carburized 38CrAlMo6-10 structural steel. Steel contained 1.% Al and 0.3% Si – elements hindering the cementite precipitation, which was considered to be adequate for obtaining a carbide free bainite. Steel samples were subjected to two different vacuum carburizing processes in order to obtain two different contents of carbon in surface layer. To produce a nanobainitic microstructure a heat treatment consisting of austempering at temperature slightly higher than the martensite start temperature ( $M_s$ ) of the layer was applied after each carburization process. It was found, that the obtained microstructure of carburized layer depends strongly on carbon content. In steel with surface layer containing lower carbon content a nanobainitic microstructure with carbon-enriched residual austenite was formed. In case of surface layer containing higher carbon content the ultra-fine grained lower bainite was obtained.

*Keywords:* carburization, austempering, low-temperature bainite, nanobainite

Celem pracy było wytworzenie w nawęglonej warstwie wierzchniej stali 38CrAlMo6-10 mikrostruktury nanobainitu. Stal ta zawiera dodatek 1,31% Al+Si – pierwiastków hamujących wydzielanie węglików, który został uznany za wystarczający by umożliwić powstanie nanobainitu. Próbki poddano dwóm procesom nawęglania do dwóch różnych zawartości węgla w warstwie wierzchniej. Zastosowana obróbka cieplna nanobainityzacji obejmowała hartowanie izotermiczne w temperaturach nieco wyższych niż  $M_s$  warstwy. Uzyskane wyniki pozwalają stwierdzić, że mikrostruktura warstwy wierzchniej po bainityzacji zależy silnie od zawartości węgla. W przypadku jednej warstwy uzyskano nanometrycznej wielkości listwy bainitu z filmem wzbogaconego w węgiel austenitu resztkowego, w drugiej mikrostrukturę ultra drobnoziarnistego bainitu dolnego.

## 1. Introduction

Technological progress in industry is strongly related with the development of new high-strength materials. Since economic factors of material manufacturing and exploitation are as important as its properties, high attention is paid to new generations of steel which combine high mechanical parameters with low costs of products. One of the most promising ways of new steels development is formation of a nanocrystalline microstructure through bainitic transformation [1-5]. For certain steels with accurate chemical composition the heat treatment consisting of low temperature austempering provides a carbide-free microstructure, containing nanometric plates of bainitic ferrite separated by thin layers of retained austenite [2,5]. Such steels should contain 0.6÷1.1 wt.% of carbon and increased amount of silicon and/or aluminium to hinder the 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  $HV_{30} = 590\div 690$  and fracture toughness  $K_{IC} = 45\div 135$  MPa  $m^{0.5}$  [5,6].

A low temperature austempering heat treatment applied for carburized low carbon steels has been reported by Zhang et al [7-9]. The authors have shown, that after austempering a low temperature bainitic structure has been formed in carburized surface layer and a lath martensitic microstructure in the centre [7,8]. Moreover it was found, that the sample with low temperature bainite produced on surface layer presented distinctly different wear behaviour than the martensitic sample under the same sliding wear process [9].

It can be assumed that replacing the conventional treatment of carburized steels consisting of quenching and low tempering, by austempering may also be beneficial in terms of reduction of residual stress level, reduction of distortion and prevention of cracking.

The aim of this study was to characterise the microstructure and wear properties of carbon-enriched surface layer of 38CrAlMo6-10 steel subjected to nanostructuring process by austempering heat treatment.

\* WARSAW UNIVERSITY OF TECHNOLOGY, FACULTY OF MATERIALS SCIENCE AND ENGINEERING, POLAND

## 2. Experimental

### 2.1. Material

Table 1 shows chemical composition of the investigated steel. The total of alloying component of ~1% Al and 0.32% Si was assumed to be sufficient to suppress cementite precipitation during austempering. Steel samples prepared for treatment consisted of flat bars with 16×10 mm cross section. Two carburizing processes leading to different surface carbon content were performed. In one case the carbon content on the steel surface was equal to 0.76%C and in the second case it was 0.86%C. The obtained surface layers were labelled LC and HC respectively, as shown on Fig. 1. For dilatometric investigation a set of small rod-like samples was also prepared and submitted to carburizing processes in order to obtain a carbon content across the sample similar to that which exhibits surface layer of carburized massive samples. Both processes were performed using FineCarb® vacuum carburizing technology by Seco/Warwick company. As a carburizing agent the mixture of acetylene, ethylene and hydrogen was used.

TABLE 1  
Chemical composition of 38CrAlMo6-10 steel

Steel	Weight %									
	C	Mn	Si	P	S	Cu	Cr	Mo	Al	Ni
<b>38CrAlMo6-10</b>	0.40	0.65	0.32	0.017	0.003	0.16	<b>1.54</b>	0.25	<b>0.99</b>	0.20

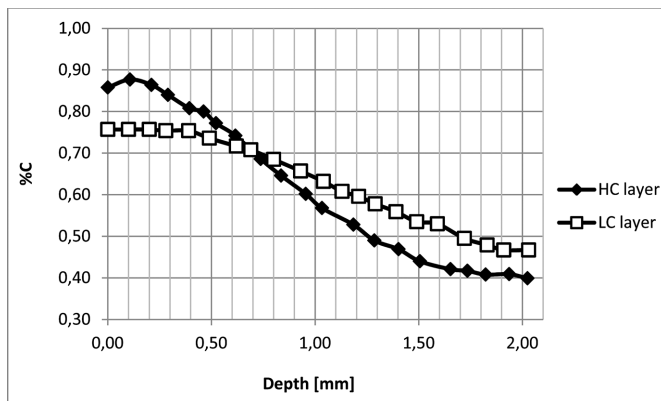


Fig. 1. Carbon content distribution in carburized surface layers of investigated steel

### 2.2. Heat treatment

In order to design the parameters of austempering heat treatment allowing to obtain a nanobainitic microstructure in carburised surface layers the dilatometric tests were performed. The aim of the tests was to determine the critical points and kinetics of phase transformations occurring in surface layers and in the core of carburized steel at various temperatures. The obtained results allowed to choose the optimal parameters for nanostructuring heat treatment. 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 of performed heat treatments are shown in Table 2. Steel samples

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

TABLE 2  
Heat treatment parameters

Designation of the process	Surface carbon content after carburizing [wt%]	Austenitization temperature [°C]	Austempering temperature [°C]
LC-A250 (austempering)	0.76	930	250
LC-A300 (austempering)	0.76	930	300
HC-A250 (austempering)	0.86	930	250
HC-A300 (austempering)	0.86	930	300
LC-QT (quenching and tempering)	0.76	930	
HC-QT (quenching and tempering)	0.86	930	

### 2.3. Characterization of microstructure

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

### 2.4. Characterization of mechanical properties

Hardness distribution in carburized layer were investigated in Vickers scale. The applied load was 200 g.

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

## 3. Results

### 3.1. Microstructure

The TEM observations revealed that the microstructure of LC surface layer after austempering at 250°C consisted of bainitic ferrite plates with thickness ranging between 9 nm and 2.3 μm. The plates were separated from each other by films of retained austenite of thickness varying from 9 nm to 1.6 μm (Fig 2). Mean width of bainitic ferrite plates was 414 nm ±111 nm and that of retained austenite was 233 nm ±95 nm. Moreover small austenite blocks of the maximum cross section

area of  $0.32 \mu\text{m}^2$  were occasionally observed. Overall volume fraction of retained austenite determined by the stereological analysis of TEM micrographs was equal to  $11\% \pm 2\%$ . Diffraction analysis revealed some reflexions from cementite though the cementite particles could not be observed.

After austempering of LC sample at  $300^\circ\text{C}$ , a typical nanobainitic microstructure was formed in surface layer (Fig. 3). However, the obtained microstructure was not totally carbide-free, in some areas very fine carbides were found within ferrite plates, on ferrite grain boundaries or on ferrite/austenite interfaces. Ferrite plates width varied from 15 nm to 293 nm with the mean value of  $84 \text{ nm} \pm 6 \text{ nm}$ . The width of retained austenite films was between 7 nm to 101 nm with the mean value of  $32 \text{ nm} \pm 4 \text{ nm}$ . Austenite blocks with the size of cross section area up to  $0.33 \mu\text{m}^2$  were also observed in the microstructure. Total amount of retained austenite was  $14\% \pm 2\%$ .

The microstructure formed at  $250^\circ\text{C}$  in surface layer of HC samples was highly heterogeneous. It consisted mostly of lower bainite regions (Fig. 4), though in some areas a nanobainitic microstructure was found. A huge density of cementite precipitates were observed within both bainitic ferrite plates and on ferrite/austenite interfaces. Moreover the nodular

carbide particles were revealed within some of ferrite plates by TEM dark field observations (Fig. 5). Mean thickness of bainitic ferrite plates was  $95 \text{ nm} \pm 6 \text{ nm}$  (thickness ranged from 16 nm to 492 nm). The films of retained austenite present in nanobainitic regions varied in thickness from 4 nm to 195 nm with mean value of  $33 \text{ nm} \pm 3 \text{ nm}$ . Small blocks of austenite were also visible. The overall austenite volume fraction determined by the stereological analysis of TEM micrographs was  $19\% \pm 4\%$ .

HC sample after isothermal annealing at  $300^\circ\text{C}$  consisted of bainitic ferrite plates containing carbide precipitates i.e.: the lower bainite regions adjacent to nanobainitic areas with retained austenite films (Fig. 6). The volume fraction of nanobainitic areas seem to be greater than in HC sample austempered at a temperature of  $250^\circ\text{C}$ . Ferrite plates thickness varied from 9 nm to 323 nm (mean  $94 \text{ nm} \pm 4 \text{ nm}$ ). The width of austenite films present in nanobainitic regions varied from 4 nm to 166 nm with mean  $32 \text{ nm} \pm 2 \text{ nm}$ . Few austenite block were observed and total amount of austenite was  $24\% \pm 4\%$ .

The surface layers of LC and HC samples subjected to quenching and tempering (QT) treatment were composed of tempered martensite.

TABLE 3

Quantitative characterization of microstructure of carburized layer of 38CrAlMo6-10 steel after austempering processes

Samples	Austempering temperature [°C]	Ferrite laths thickness [nm]	Austenite films thickness [nm]	Block austenite size [ $\mu\text{m}^2$ ]	Volume fraction of retained austenite [%]
LC layer	250	$414 \pm 111$	$233 \pm 95$	0.32	$11 \pm 2$
	300	$84 \pm 6$	$32 \pm 4$	0.33	$14 \pm 2$
HC layer	250	$14 \pm 2$	$33 \pm 3$	–	$19 \pm 4$
	300	$94 \pm 4$	$32 \pm 2$	–	$24 \pm 4$

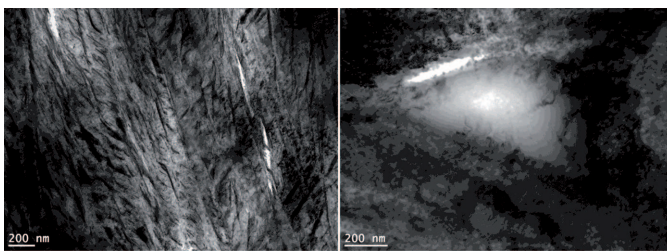


Fig. 2. TEM micrograph of carburized layer in LC sample after austempering at  $250^\circ\text{C}$  a) nanobainite, b) block of retained austenite

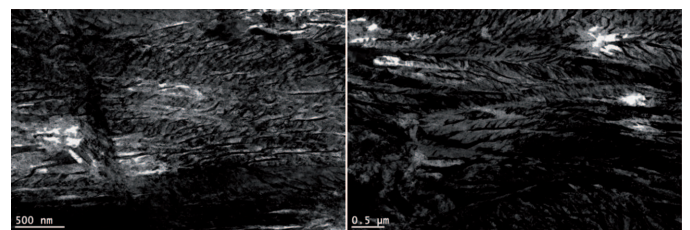


Fig. 4. TEM micrograph showing a lower bainite in carburized layer of HC sample after austempering at  $250^\circ\text{C}$

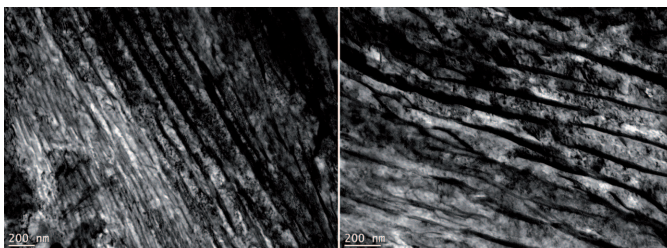


Fig. 3. TEM micrograph showing a typical nanobainitic structure in carburized layer of LC sample after austempering at  $300^\circ\text{C}$

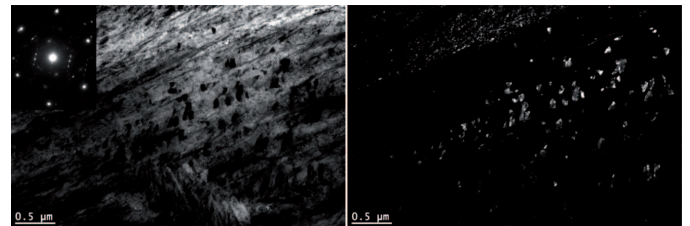


Fig. 5. Microstructure of carburized layer of HC sample after austempering at  $250^\circ\text{C}$  – a) bright field image, b) dark field image from diffraction spot of cementite – nodular carbides visible



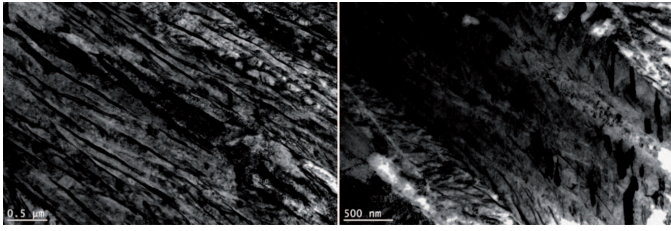


Fig. 6. TEM micrograph showing a mixture of nanobainite and lower bainite in carburized surface layer of HC sample after austempering at 300°C

### 3.2. Hardness distribution

Hardness distribution curves of LC and HC carburized layers are shown on Figures 7 and 8 respectively. Each point on the graph represents mean of three measurements. Austempering at 250°C of LC sample resulted in hardness about 600 HV0.2 on the very surface. In the cross section of carburized layer hardness values showed little difference, varying in between 540÷560 HV0.2.

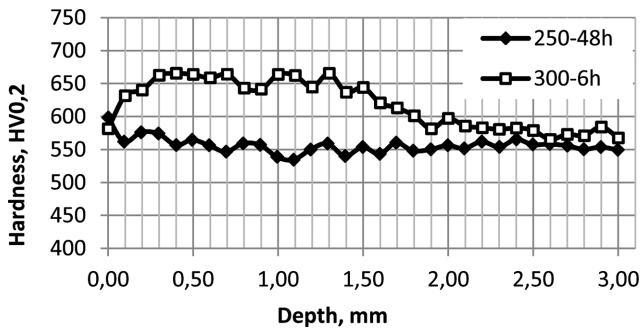


Fig. 7. Hardness distribution in cross section of LC sample

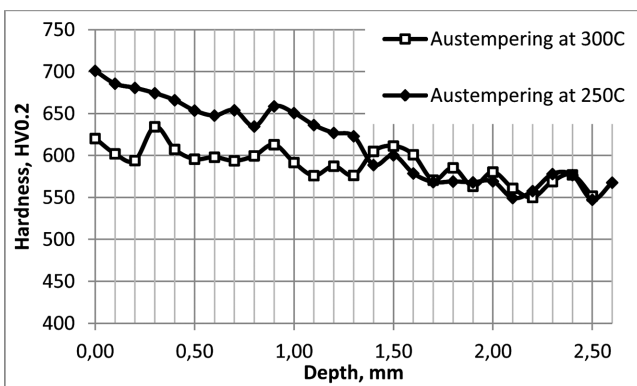


Fig. 8. Hardness distribution in cross section of H C sample

In LC sample austempered at 300°C hardness increased from 580 HV0.2 at the surface to about 660 HV0.2 at the distance of 300  $\mu\text{m}$  from surface and remained at this level to the depth of 1300  $\mu\text{m}$ . 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 this sample might result from microstructure heterogeneity or surface decarburizing, which could occur during austenitization.

The HC sample exhibited 700 HV0.2 at the surface. In cross section, hardness decreased steadily from 700 HV0.2

at the surface to 560 HV0.2 in the core, throughout whole carburized layer,

After austempering of HC sample at 300°C, the surface hardness was lower than after austempering at 250°C and equal to 620 HV0.2. Hardness decreased slightly in carburized layer from surface to the core of the sample in carburized layer to about 560 HV0.2 which is a value similar to that of a core of sample austempered at 250°C.

Surface hardness was also measured on carburized samples given to quenching and low tempering and it was 690 HV0.2 and 730 HV0.2 in FC and HC samples respectively.

### 3.3. Wear analysis

In order to compare the wear resistance of steel samples which were subjected to different heat treatments, volume loss of each sample were measured after abrasion test.

For LC samples after austempering at both applied temperatures the volume loss during wear tests were significantly smaller than for quenched and tempered QT samples (Fig. 9). After treatment at 250°C samples had fourfold greater wear resistance under 200 N load and threefold under 400 N load than after conventional QT treatment. For samples austempered at 300°C, the volume loss during wear test, despite its finer microstructure, was greater than in a previous case but it was still distinctly lesser than for QT samples. Such result, in addition of decreased surface hardness on 300°C sample, may indicate surface decarburization. Lowered carbon content in the surface resulted in decreased wear resistance. It is possible that properly performed heat treatment (avoiding the decarburization) with austempering at 300°C would result in better wear resistance. Lesser volume loss of both austempered LC samples compared to the QT samples may, besides microstructure refinement, result from very low presence of cementite precipitates on one hand and from high volume fraction of retained austenite in carburized surface layer after austempering on the other. The stress level induced during wear test at the carburized surface layer was sufficient for initiation the transformation of austenite into martensite due to the TRIP effect [10, 11].

The wear behaviour of HC samples is different than that of LC samples. For the test run under 200 N load the volume loss was highest in sample treatment at 250°C. For samples austempered at 300°C and for QT samples the measured volume loss was the same in this test. After tests run under 400 N load, the wear of sample austempered at 250°C was similar as in QT sample. The sample austempered at 300°C demonstrated about 44% lower volume loss than both other samples tested. Better wear resistance of sample austempered at higher temperature was most probably related with higher retained austenite content in sample annealed at 300°C ( $V(\gamma_r) = 24\%$ ) than in sample treated at 250°C ( $V(\gamma_r) = 19\%$ ) which could lead to the stress-induced martensitic transformation. Under higher load (400 N) more austenite would transform into the martensite by TRIP effect during the test, which could explain much lower volume loss of sample austempered at 300°C as compared to the QT sample with tempered martensite. Such effect was not however observed for HC sample treated at 250°C which exhibited lower wear resistance than other sam-

ples. This could be due to a high density of cementite precipitates present in this sample after austempering.

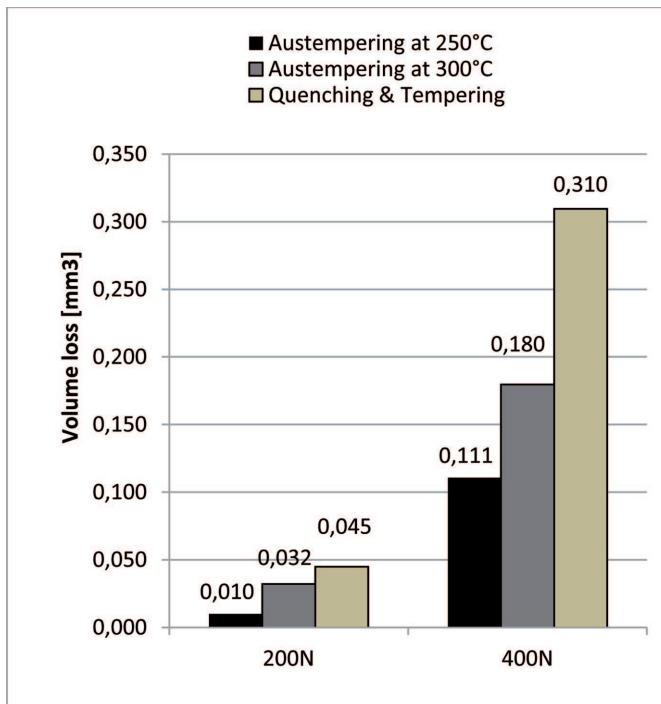


Fig. 9. Volume loss after wear test of LC samples

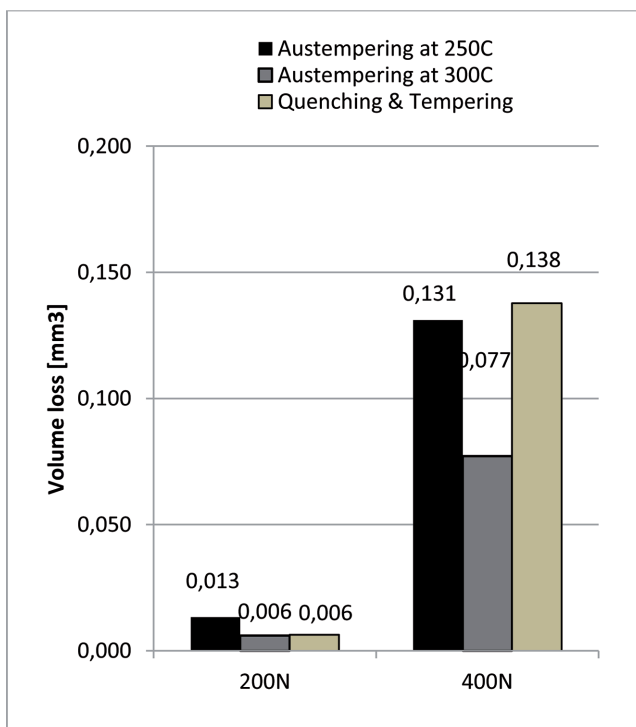


Fig. 10. Volume loss after wear test of HC samples

QT and HC samples austempered at 300°C showed generally better wear resistance than the LC samples after the same heat treatments. This is due to higher carbon content resulting in higher solution strengthening as well as the higher amount of retained austenite in HC samples. On the contrary the HC sample austempered at 250°C which contains high density of cementite precipitates displayed smaller wear resistance than

LC sample after austempering at 250°C with very small cementite precipitates. The intensive precipitation of cementite occurring in HC sample during austempering at 250°C could lead to a decrease of carbon content in bainitic ferrite and in retained austenite. This effect can reduce the wear resistance of this sample as compared to HC samples subjected to austempering at 300°C which has a higher volume content of carbide-free nanobainitic microstructure.

#### 4. Conclusions

Austempering of 38CrAlMo6-10 steel samples carburized to 0.76% C resulted in formation of nano- or ultra-fine bainitic microstructure in carburized surface layer. The nanobainitic microstructure was composed of ultra-thin ferrite plates in film-like retained austenite matrix.

Samples (HC) carburized to a higher carbon content presented after austempering at 250°C a high density of carbides in bainitic ferrite and highly heterogeneous bainite morphology. This observation, in addition with evidence of low cementite presence in austempered 0.76%C (LC) samples, indicates that addition of 1% of Al was not sufficient to inhibit the carbide precipitation of steel with high carbon content.

Steel samples with the nanobainitic microstructure obtained after austempering in, though having lower hardness than tempered martensite, exhibited significantly lower volume loss after sliding wear test, compared to the latter. Such a high wear resistance could result from both microstructure refinement as well as from presence of retained austenite, which transforms into a martensite by TRIP effect under shear stress occurring during wear tests.

The wear tests indicate that steel samples with lower bainite containing carbides exhibit lower wear resistance when compared to samples with carbide free nanobainite and to samples with tempered martensite.

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