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THE EFFECT OF STEPPED AUSTEMPERING ON PHASE COMPOSITION AND MECHANICAL PROPERTIES OF NANOSTRUCTURED X37CrMoV5-1 STEEL

WPŁYW STOPNIOWEGO HARTOWANIA IZOTERMICZNEGO NA SKŁAD FAZOWY ORAZ WŁAŚCIWOŚCI MECHANICZNE STALI X37CrMoV5-1 O STRUKTURZE NANOKRYSTALICZNEJ

This paper presents the results of studies of X37CrMoV5-1 steel subjected to quenching processes with a one-step and a two-step isothermal annealing. The TEM observation revealed that steel after one-step treatment led is composed of carbide-free bainite with nanometric thickness of ferrite plates and of high volume fraction of retained austenite in form of thin layers or large blocks. In order to improve the strength parameters an attempt was made to reduce the austenite content by use of quenching with the two-step isothermal annealing. The temperature and time of each step were designed on the basis of dilatometric measurements. It was shown, that the two-step heat treatment led to increase of the bainitic ferrite content and resulted in improvement of steel's strength with no loss of steel ductility.

Keywords: heat treatment, austempering, steels, nanobainite, dilatometric test

W artykule przedstawiono wyniki badań stali X37CrMoV5-1 poddanej hartowaniu izotermicznemu jedno i dwu stopniowemu. Obserwacji TEM wykazały, że stal po jednostopniowym hartowaniu izotermicznym składa się z bainitu bezwęglikowego o nanometrycznej grubości płytek ferrytu poprzedzielanych cienkimi warstwami lub blokami austenitu. Wysoka plastyczność stali wynika z wysokiej zawartością fazy austenitycznej. W celu poprawy parametrów wytrzymałościowych postanowiono zmniejszyć ilość austenitu poprzez zastosowanie dwustopniowego hartowania izotermicznego. Parametry tego procesu zostały zaprojektowane na podstawie badań dylatometrycznych. Wykazano, że dwustopniowa obróbka cieplna prowadził do podwyższenia zawartości ferrytu bainitycznego i tym samym do poprawy wytrzymałości stali bez utraty plastyczności.

1. Introduction

Polycrystalline materials in which the grain size is less than 100 nm in at least one direction are called nanomaterials [1]. Bhadeshia and Cabalero have shown [2,3], that in steels with defined chemical composition after quenching with isothermal annealing at the lower range of temperature of bainitic transformation a nanostructured bainite can be formed. Steels which are susceptible to nanostructurisation contain increased amount of carbon (0.6-1.1%) and silicon (1,5-2%), which inhibits the cementite precipitation [4,5]. The process of nano structurisation consists of austenitization and quenching followed by an isothermal annealing. As a result, the structure of carbide-free bainite can be obtained with a nanometric thickness of grains and a high volume fraction of retained austenite [5,6]. Steels with such a structure are called nanobainitic steels and they have very beneficial mechanical properties [5,6]. Residual austenite in steel provides increased plasticity and fracture toughness [8,9,10,11]. However, if the austenite content is too high, the strength value decreases.

Originally, the structure of carbide-free bainite was produced in steels with specially designed chemical composition [3]. In recent years an attempt has been made to produce the carbide-free structure in some commercial steels [12]. One of the alloys considered for nanostructurisation was a X37CrMoV5-1 hot work tool steel, which is commonly used in industrial practice. This steel has been selected for nanostructuring process on the basis of its chemical composition, although it contains less carbon and far more chromium as compared to the nanobainitic steels which have been produced until now. However, it was demonstrated in our previous work [13], it is possible to obtain a nanobainitic structure by use of properly designed heat treatment process.

The aim of this paper was to prove that it is possible to control the phase composition and the mechanical properties of nanostructured X37CrMoV5-1 steel by means of appropriately designed thermal processing. The results of microstructural investigations and of mechanical tests of X37CrMoV5-1 steel quenched with one- and two-step isothermal annealing have been therefore presented and analysed.

2. Experimental

The study was performed on the X37CrMoV5-1 tool steel in the softened state (ferritic structure with carbide precipi-

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TABLE 1

Chemical composition of the X37CrMoV5-1 steels

	C [%]	**Si [%]**	**Mn [%]**	**Cr [%]**	**Mo [%]**	**V [%]**	**Ni [%]**
X37CrMoV5-1	0,37	1,01	0,38	4,91	1,2	0,34	0,19

tates). Chemical compositions of this steels are given in TABLE 1.

This steel is often applied in the production of tools and parts that work in elevated temperatures. The heat treatment of this steel consist usually of quenching and high tempering.

In the present study, steel was subjected to nanostructurisation treatment in order to obtain a nanobainitic structure. For this purpose the samples were subjected to austenitization followed by two kinds of processes: single and two-step austempering treatments. The parameters of both heat treatments were determined on the basis of dylatometric studies with the use of quenching dilatometer DIL 805 L. One and two-step austemparing treatments were performed with the use of tin bath. Design of the heat treatment parameters is described in the next section.

After both heat treatments were performed, the analysis of a microstructure was carried out with the use of the transmission electron microscope TEM JEOL 1200 EX. Moreover, the hardness of the samples were measured.

In order to characterise the proprieties of steel after single-step austempering the static tensile tests were performed, with the initial strain rate of 10^{-3} s^{-1} on samples with the diameter of 6 mm on the Zwick/Roell Z250 testing machine with the use of an extensometer.

Two-step heat treatment was performed on samples for the static tensile test only. The results of mechanical tests were compared with the properties of samples submitted to the conventional quenching and tempering heat treatment. The parameters of tempering was selected in such a manner so as to obtain the hardness similar to the hardness after austempering.

3. Results

3.1. Design of one-step isothermal annealing

The proper parameters of thermal treatment were determined on the basis dilatometric studies of phases transformations occurring in steel. The key issue was to fix a proper temperature of the isothermal stop, which should be slightly higher than the martensitic start (Ms) temperature in order to gain relatively low size of bainitic ferrite plates. The dilatometric test indicated that the Ms temperature is equal to 295°C, Fig. 1. During quenching below Ms temperature, two additional effects can be observed. The proper interpretation of these effects needs however additional investigations, which will be the subject of another research work. Therefore, for isothermal annealing the temperature of 300°C was applied.

One-step austempering consisted of austenisation of steel samples at 1030°C, cooling down in tin bath at 300°C and 19-hour long annealing at that temperature. During conventional heat treatment the austenitization parameters were the same as during one-step isothermal quenching (1030°C). After austenitization steel samples were quenched in oil and then tempered at 585°C for 2 hours, Fig. 2.

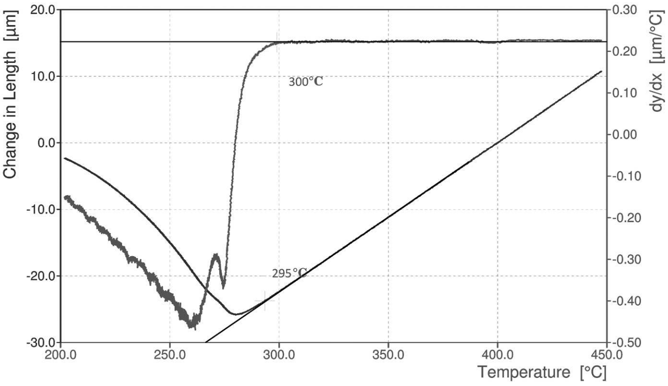

Fig. 1. Differential and dilatometric curve obtained during rapid cooling

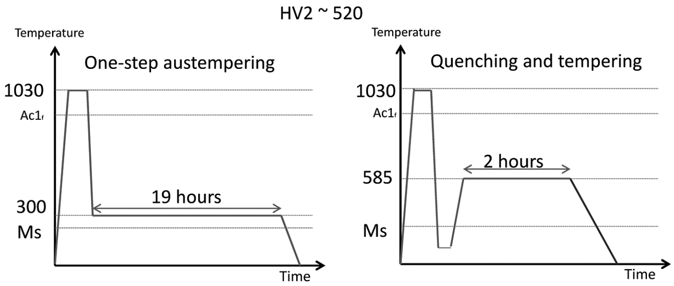

Fig. 2. Diagram applied heat treatments

3.2. Microstructure and mechanical properties of steel after one-step austempering

The microstructure of steel formed during the one-step isothermal processing is composed of overlapping layers of bainitic ferrite and retained austenite. The typical microstructures, as observed by TEM, are presented on Fig. 3. No carbides were found in a microstructure of steel.

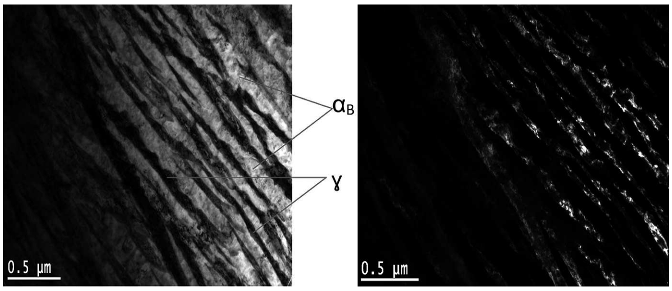

Fig. 3. Microstructure of X37CrMoV5-1 steel after one-step austmpering at 300°C (left), dark field image for austenite reflection (right)

Austenite occurred not only in form of thin layers, but also in form of large blocks partially transformed to martensite,

Fig. 4. The surface area of austenite blocks as observed by TEM varied from 0.13 μm^2 to 4.33 μm^2. The average thickness of the bainitic ferrite plates was 89 nm±6 nm, whereas the average thickness of the retained austenite layers was 31 nm±2 nm, It means that the one-step austempering leads to a carbide-free nanobainitic structure in steel.

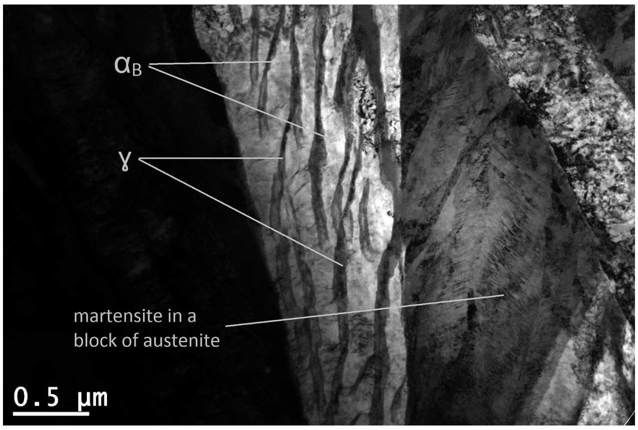

Fig. 4. The block of austenite with partial martensitic transition in X37CrMoV5-1 after one-step austmpering

The stereological analysis of the microstructure images obtained by TEM revealed that the volume fraction of austenite amounts to ca. 45%.

The X37CrMoV5-1 steel after the isothermal quenching and after the continuous quenching and high tempering exhibited similar hardness of about 520 HV2. On the contrary, steel with nanobainitic structure showed higher total elongation to fracture with slightly increased strength as compared to steel with tempered martensite, Fig 5. This can be explained by a great volume fraction of austenite in a nanobainitic microstructure after one-step austempering. Austenite is known as a more ductile phase as compared to bainitic ferrite or tempered martensite. Moreover, the austenite in form of layers can inhibit crack propagation and the austenite in form of blocks can increase the plasticity of steel due to a Transformation Induced Plasticity (TRIP) effect [12,13]. To further improve strength of steel, it was necessary to transform a fraction of retained austenite, which is a softer phase, to a harder bainitic ferrite in a nanobainitic structure.

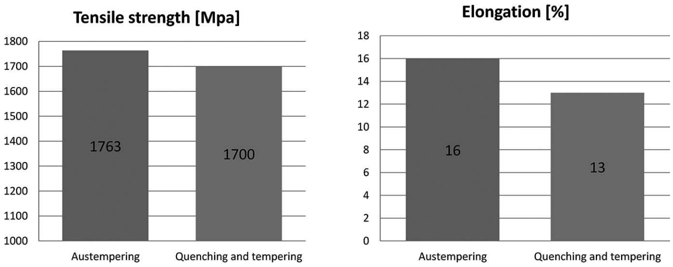

Fig. 5. Mechanical properties studied during static tensile tests

3.3. Designing of two-step isothermal annealing

To obtain a steel with increased amount of bainitic ferrite it was decided to modify the process of austempering. According to the T0 line theory [16] this can be done by the decrease in the temperature of isothermal annealing, at which the nanobainitic structure forms.

Bainitic transformation, which occurs during annealing at the temperature of 300°C, causes an enrichment of austenite in carbon leading to increase its stability and decrease the Ms temperature of the remaining austenite. This effect is visible in the dilatometric graph, Fig. 6, which shows a change in dilatation which is related with bainitic transformation. As a result, the Ms temperature decreases down to a new value of 235°C, Fig. 6.

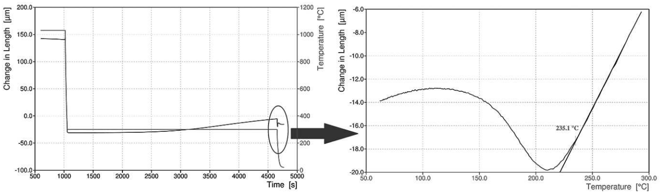

Fig. 6. Dilatometric curve showing change of Ms with the progress of bainitic transformation

In order to increase the volume fraction of bainite, a two-step isothermal quenching was applied. After the first step of isothermal annealing at 300°C for 1h the Ms temperature was decreased to 260°C and the second step of annealing were conducted at this temperature for 12h. During the second step the bainitic transformation continued as shown on Fig. 7.

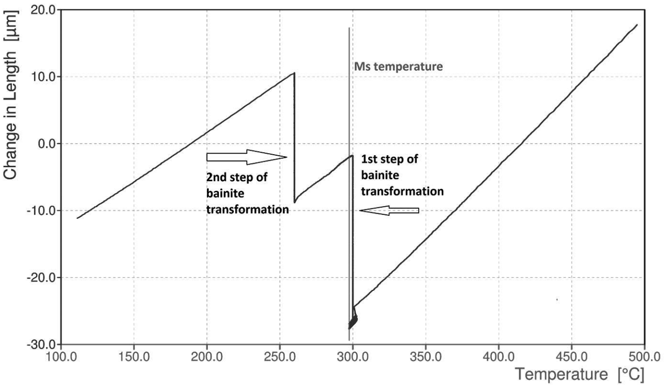

Fig. 7. Dilatometric curve showing the first bainite transformation above the initial Ms temperature and the second bainite transformation below the initial Ms temperature

The use of the two-step isothermal quenching permitted forming of a greater volume fraction of bainitic ferrite in the final microstructure, which was confirmed by a greater total elongation during the dilatometric test. To sum up, the total elongation of a dilatometric sample in the whole range of isothermal annealing was greater than during the single process. In the one-step transformation the elongation was 34,8 μm while in the two-step process it reached 44,2 μm, Fig. 8.

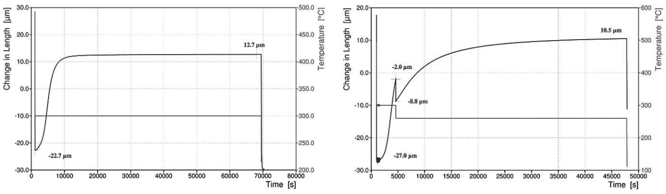

Fig. 8. Dilatometric curves comparison (left) one-step quenching and (right) two-step quenching, which shows sample elongation during the bainitic transformation

3.4. Microstructure and mechanical properties of steel after two- step austempering

Microscopic observations confirmed that the two-step isothermal annealing resulted in the carbide-free nanobainite with retained austenite, Fig. 9. This structure was very similar to that obtained during one-step austempering, however the stereological analysis indicated that the content of austenite was lowered by 10% and reaches 35%. Moreover, the thickness of ferrite plates and austenite layers was higher than in one-step austempering, TABLE 2. These results were obtained by means of stereological analysis of TEM images. Because the observation area is very small, these results, especially regarding austenite content, can display a significant error. Qualitative comparison of the microstructure of both samples reveals however, that the austenite content is significantly lower in sample after two-step isothermal quenching with respect to the sample after single isothermal quenching. These observations are consistent with the results of dilatometric tests.

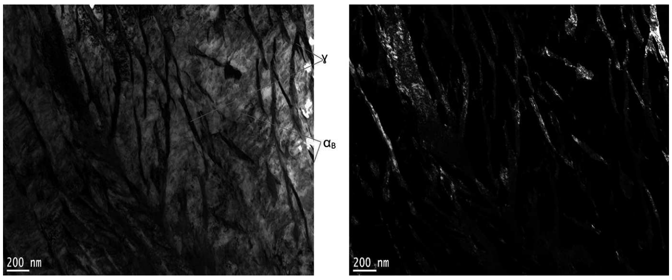

Fig. 9.

After two-step austempering, the martensite was still observed in the blocks of austenite despite of its greater carbon enrichment. However, the martensitic transformation was not detected in the dilatometric tests, which suggests that it could occur during the preparation of the sample for TEM observations. This transformation might have occurred due to the stress relaxation in a microstructure during thinning down of TEM samples.

We can infer from the measurements of the thickness of bainite plates that the transformation in the second step of austempering led to thickening of bainie plates up to 130 ±9 nm, TABLE 2.

TABLE 2

Results of stereological measurements basing on TEM studies

	Thickness of bainitic ferrite plates [nm]	Thickness of retained austenite layers [nm]	Cross section area of austenite blocks [μm]	Content of austenite [%]
One-step austempering	89±6	31±2	0.13 to 4.33	45.3±3.1
Two-step austempering	130±9	51±4	0.15 to 3.5	35.0±5.5

Fig. 10 shows the tensile test results for the steel samples after the conventional treatment as well as after one- and two-step austempering. It should be underlined, that in spite of the increase in plate thickness, the hardness and strength parameters increased after the two-step isothermal quenching. The increase of yield point and tensile strength was of 150 MPa and 100 MPa respectivelyas compared to samples after one-step annealing, Fig. 11. This effect can be due to the decrease of the volume fraction of the soft austenitic phase after two-step austempering.

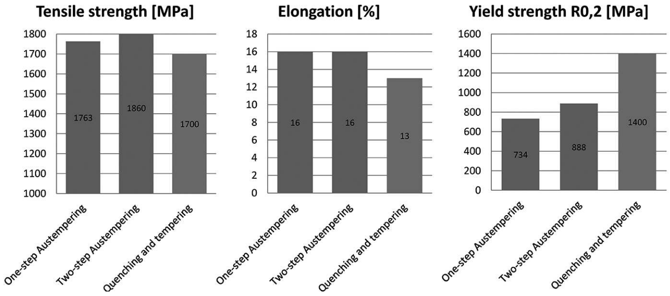

Fig. 10. Comparison of mechanical properties tested during static tensile tests

It should be also noticed that the increase in strength did not cause the drop in ductility of steel.

However, the yield strength is lower after both isothermal processes than after quenching and tempering treatment. The low values of yield point and high ductility of steel after austempering can be explained by high volume fraction of austenite in nanobainitic samples.

The high hardening observed on tensile curves and resulting high values of tensile strength, Fig. 11, may be due to the austenite transformation into martensite during tensile deformation (TRIP effect) [15].

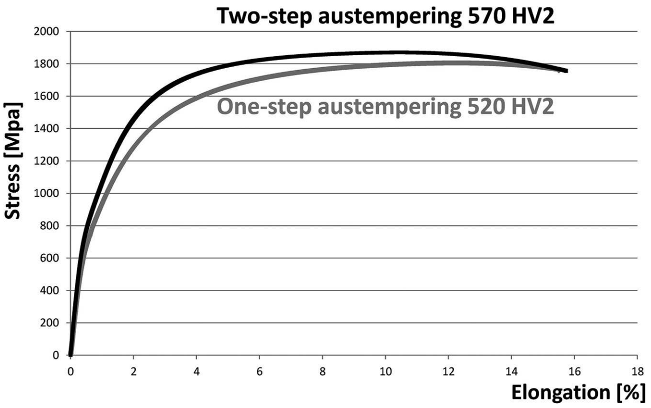

Fig. 11. Exemplary curves of extension. The green line – the one-step austempering and the black the two-step austempering

4. Conclusions

The isothermal quenching of the X37CrMoV5-1 steel at the temperature which is slightly above the Ms temperature allows obtaining of a structure composed of nanometric plates of carbide-free bainite separated by thin layers of residual austenite – called nanobainite.

Nonetheless, steel shows improved strength and ductility after one-step austempering as compared to the steel after quenching and tempering treatment.

The application of the two-step austempering process leads to the increase in the volume fraction of bainitic ferrite at the expense of austenite. The increase of bainitic ferrite occurs by widening of ferrite plates thickness.

The second-stage of two-step austempering process, during which the bainite transformation occurs below the initial Ms temperature, allows for the increase in the strength of the steel without deterioration of its ductility.

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REFERENCES

[1] K.J. Kurzydłowski, M. Lewandowska (Eds), Nanomateriały inżynierskie konstrukcyjne i funkcjonalne, Warsaw 2010.
[2] H.K.D.H. Bhadeshia, Proceedings A, Royal Society 466, UK (2010).
[3] F.G. Cabalero, H.K.D.H. Bhadeshia, K.J.A. Mawela, D.G. Jones, P. Brown, Mater. Sci. Tech. **18**, 279 (2002).
[4] H.K.D.H. Bhadeshia, C. Garcia-Mateo, P. Brown, Bainite steel and methods of manufacture thereof, United States Application US20110126946 (2011).
[5] W. Burian, J. Marcisz, B. Garbarz, L. Starczewski, Arch. Metall. Mater. **59**, 1211 (2014).
[6] C. Garcia-Mateo, F.G. Cabalero, T. Sourmail, M. Kuntzc, J. Cornide, V. Smanio, R. Elvira, Mater. Sci. Eng. A **549**, 185 (2012).
[7] M.N. Yoozbashi, S. Yazdani, T.S. Wang, Mater. Design **32**, 3248 (2011).
[8] A. Barbacki, E. Mikołajski, J. Mater. Process. Tech. **78**, 18 (1998).
[9] J. Pacyna, Steel Res. **58**, 87 (1987).
[10] A. Kokosza, J. Pacyna, Archives of Materials Science and Engineering **31**, 87 (2008).
[11] L. Kucerova, H. Jirkova, B. Masek, Arch. Metall. Mater. **59**, 1189 (2014).
[12] http://www.nanostal.eu/
[13] W.A. Świątnicki, K. Pobiedzińska, E. Skołek, A. Gołaszewski, Sz. Marciniak, Ł. Nadolny, J. Szawłowski, Mater. Eng. **6**, 524 (2012).
[14] H.K.D.H. Bhadeshia, Mater. Sci. Eng. A **481**, 36 (2008).
[15] C. Garcia-Mateo, F.G. Caballero, Mater. Trans. **46**, 1839 (2005).
[16] H.K.D.H. Bhadeshia, Bainite in Steels: Transformations, Microstructure and Properties, 2001.

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