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ANALYSIS OF THE AUSTENITE GRAIN GROWTH IN LOW-ALLOY BORON STEEL WITH HIGH RESISTANCE TO ABRASIVE WEAR

**ANALIZA ROZROSTU ZIARNA AUSTENITU W WYBRANEJ NISKOSTOPOWEJ STALI Z BOREM
O PODWYŻSZONEJ ODPORNOŚCI NA ZUŻYCIE ŚCIERNE.**

Today low-alloy steels with boron achieve high resistance to abrasive wear and high strength. These features are obtained by using advanced technology of manufacturing. This makes boron steels increasingly popular and their application more diverse. Application of these steels can extend the lifetime of very expensive machine construction in many industries such as mining, the automotive, and agriculture industries. An interesting subgroup of these materials is steel with boron intended for heat treatment. These steels are supplied by the manufacturer after cold or hot rolling so that it is possible for them to be heat treated in a suitable manner by the purchaser for its specific application. Very important factor that determines the mechanical properties of final product is austenite grain growth occurring during hot working process such us quenching or hot rolling. Investigation of the effect of heating temperature and holding time on the austenite grain size is necessary to understand the growth behavior under different conditions. This article presents the result of investigation of austenite grain growth in selected low-alloy boron steel with high resistance to abrasive wear and attempts to describe the influence of chemical composition on this process.

Keywords: Low alloy steels with boron, heat treatment, austenite grains, microstructural analysis

Obecnie stale z dodatkiem boru osiągają dużą odporność na zużycie ścierne w połączeniu z wysokimi parametrami wytrzymałościowymi. Te właściwości uzyskiwane są drogą zaawansowanej technologii wytwarzania. To sprawia, że stale z borem zyskują coraz większą popularność, a ich zastosowanie jest coraz szersze. Zastosowanie tego typu stali może wydłużyć trwałość często drogich maszyn przemysłu górnictwa, motoryzacyjnego, czy rolniczego. Interesującą podgrupę tych materiałów stanowią stale z dodatkiem boru przeznaczone do obróbki cieplnej. Producenci dostarczają je w stanie po walcaniu na gorąco do ewentualnie przeprowadzenia obróbki cieplnej przez kupującego odpowiedniej do konkretnego zastosowania. Ważnym czynnikiem, który istotnie wpływa na właściwości mechaniczne gotowego wyrobu, jest wielkość ziarna austenitu podczas przeprowadzania procesów obróbki cieplnej lub cieplno-plastycznej takich jak hartowanie, czy walcanie na gorąco. A zatem określenie wpływu temperatury i czasu austenityzowania na wielkość ziarna austenitu jest bardzo istotne. W artykule przedstawiono wyniki badań rozrostu ziarna austenitu przy różnych temperaturach i czasach austenityzowania w wybranej niskostopowej stali z borem o podwyższonej odporności na ścieranie oraz podjęto próbę opisania wpływu składu chemicznego na ten proces.

1. Introduction

Working conditions of machines elements exposed to abrasive wear require use of materials with high strength and high resistance to abrasive wear. Traditionally unalloyed steels which contain from 0.40% to 0.65% of carbon are used. They obtain sufficient strength and toughness due to quenching and tempering at high temperatures, but they are not sufficiently resistant to abrasion. In case of performing tempering at low temperature after quenching these steels are resistant to abrasion but often fracture after contact with hard materials during their work. The solution to this problem may be to replace them by modern structural

materials. Such as low-alloy boron steel with high resistance to abrasive wear.

In the 1930's it was already observed that the addition of boron significantly increases the hardenability of low and medium carbon steel. As a result of this research, the group of weldable bainitic steels with tensile strength to 1200 MPa were obtained. Production of boron steels with high resistance to abrasive wear began in the mid 1950's. At that time, Japanese ironworks JFE Steel Corporation began manufacturing of JFE EVERHARD steel plates, which were recommended for use in machine components exposed to abrasive wear [1]. In Europe, this type of steel was produced for the first time in 1970 by the Swedish steel company SAAB – Oxelösund [2].

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Today low-alloy steels with boron achieve high resistance to abrasive wear and high strength. These features are obtained by using advanced technology of manufacturing. This makes boron steels increasingly popular and their application more diverse. Application of these steels can extend the lifetime of very expensive machine construction in many industries such as mining, the automotive, and agriculture industries. An interesting subgroup of these materials is steel with boron intended for heat treatment. These steels are supplied by the manufacturer after cold or hot rolling so that it is possible for them to be heat treated in a suitable manner by the purchaser for its specific application. Manufacturers inform that to obtain high resistance to abrasive wear typical heat treatment of these materials is quenching or quenching and tempering at low temperature. In the material cards the manufacturer presents only the basic mechanical properties of the product, but more knowledge about these steels would be desirable by the customer for an accurate selection of heat treatment. By selecting the parameters of heat treatment of these steels, the desired microstructure and mechanical properties suitable for the particular machine elements can be reached. This would allow them to extend their range of applications. However, the different microstructures, which cause different properties, require careful decision-making to define the application of these steels in various states of heat treatment. In this case the parameters of the heat treatment, particularly the austenitizing temperature, should be properly defined and selected.

A very important factor to determine the microstructure is austenite grain growth rate. Austenite grain size strongly influences the kinetics of phase transformation during the cooling cycle [3,4]. Some investigations on the grain growth kinetics for low carbon steel and microalloyed steel have been already reported [4-6] but still some efforts have to be expended to understand the grain growth behavior and influence of alloy addition on the grain growth in low-alloy boron steels with high resistance to abrasive wear. With this state of knowledge it is necessary to study the effect of heating temperature and holding time on the growth of the austenite grains in low-alloy steels with boron intended to heat treatment. This article presents the results of investigation of austenite grain growth in selected low-alloy boron steel with high resistance to abrasive wear and attempts to describe the influence of chemical composition on this process.

2. Material and methods

For the tests, the material from a group of low-alloy boron steels with high resistance to abrasive wear was selected. It was produced by a leading metallurgical company using advanced technology methods. Selected mechanical properties of analyzed material according to the manufacturer's data and research data are presented in Table 1.

The performed tests included chemical analysis and metallographic studies using light microscope, scanning electron microscope and transmission electron microscope.

Microstructure in delivery condition of investigated steel was presented in Figure 1. Microstructure characterizes band distribution of ferrite and pearlite. Cementite in perlite is lamellar and partially coagulated (Fig. 1b).

Chemical composition of investigated steel obtained using the spectral method is summarized in Table 2. This composition is selected not only because of the need to obtain relatively high mechanical properties, but also to ensure its weldability and reduce price applied universality.

The heat treatment parameters are presented in Table 3. After each heat treatment, the specimens were tempered at temperature 250°C for 30 minutes to retain the detail of the austenite microstructure and to allow identification of the prior austenite grain boundaries. Samples were etched with reagent Mi7Fe in accordance to standard PN-H-04503:1961P. The measurements of austenite grain size were performed using the program NS Elements. Each average austenite grain size was evaluated from 100 measurements.

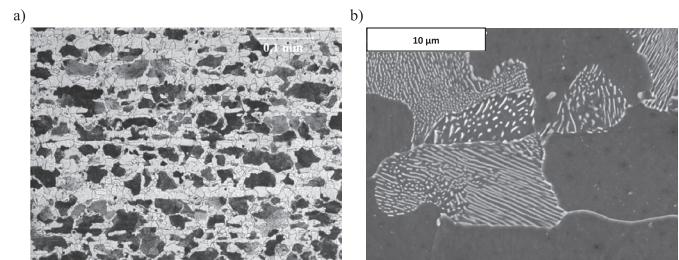


Fig. 1. Microstructure of investigated low-alloy boron steel with high resistance to abrasive wear in as delivered condition: a) light microscopy, etched state 5% HNO₃; b) scanning electron microscopy; etched state 5% HNO₃

TABLE 1

Selected mechanical properties of investigated steel in as delivery condition

	R _m [MPa]	R _{p0.2} [Mpa]	A _s [%]	Hardness	KCV ₄₀ [J/cm ²]	KCV ₂₀ [J/cm ²]	KCV ₀ [J/cm ²]	KCV ₂₀ [J/cm ²]
Manufacturer's data [7]	620	420	22	170 HB	no data	no data	no data	no data
Research data [8]	541	372	27	155 HV10	7	9	17	39

TABLE 2

Chemical composition of investigated steel obtained using the spectral method

Element	C	Mn	Si	P	S	Ni	Cr	V	Al
wt.%	0.230	1.330	0.270	0.009	0.009	0.060	0.370	0.016	0.030
Element	Ti	Nb	B	W	Cu	Co	Mo	N	
wt.%	0.040	0.013	0.001	0.007	0.040	0.040	0.008	0.001	

TABLE 3

Heat treatment parameters of the investigated steel

Temperature [°C]	Holding time [min]			Quenching medium
900	20	40	60	water
930	20	40	60	water
950	20	40	60	water
1000	20	40	60	water
1050	20	40	60	water
1100	20	40	60	water
1150	20	40	60	water
1200	20	40	60	water

3. Results and discussion

Comparison of the austenite grain size attained after holding at temperature range from 900°C to 1200°C for 20, 40 and 60 minutes is presented in Figure 2. After holding at temperature 950°C some abnormal austenite grains were observed. In this reason it was decided to evaluate the size of abnormal and normal grains separately (Figs. 3 and 4). The effect of occurrence of abnormal grains can be connected with the presence of minimum two types precipitates that inhibit growth of austenite grains. As it was improved by the authors of the research [9] the non-dissolved AlN precipitates block only parts of the austenite grain boundaries to inhibit the austenite grain growth, which is the cause of the inhomogeneity of the austenite grains distribution in the steel.

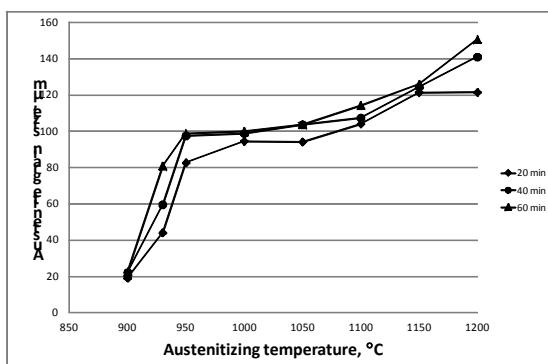


Fig. 2. Comparison of the austenite grain size attained after holding at temperature range from 900°C to 1200°C for 20, 40 and 60 minutes

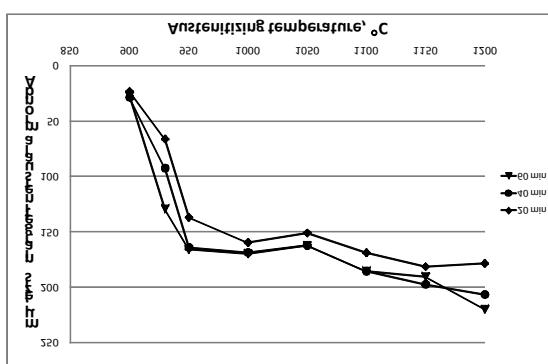


Fig. 3. Comparison of the abnormal austenite grain size attained after holding at temperature range from 900°C to 1200°C for 20, 40 and 60 minutes

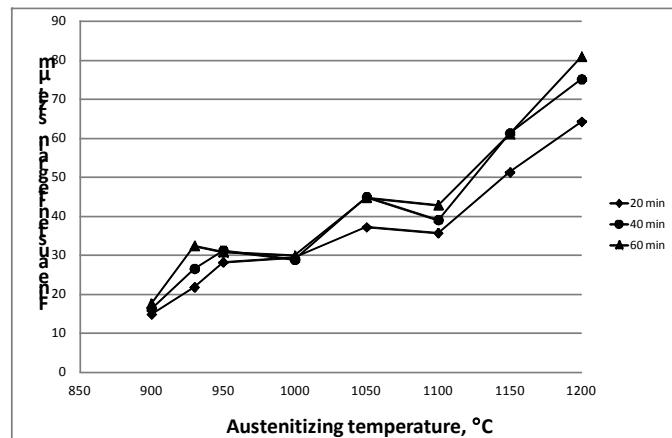


Fig. 4. Comparison of the fine austenite grain size attained after holding at temperature range from 900°C to 1200°C for 20, 40 and 60 minutes

In addition in Fig. 5 the main effects plots were presented to compare the effects of holding temperature and time on average austenite grain size.

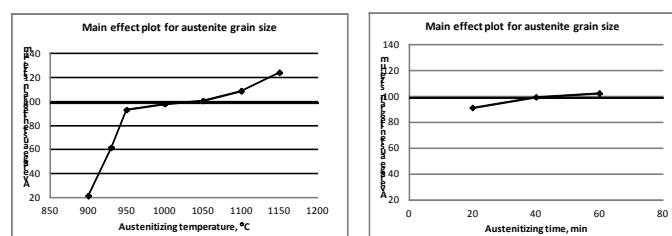


Fig. 5. Comparison of the effects of holding temperature and time on average austenite grain size

Austenite grain growth rate during the hot working process such us quenching or hot rolling is an important factor that determines the mechanical properties of final product. Investigation of the effect of heating temperature and holding time on the austenite grains size is necessary to understand the growth behavior under different conditions. Based on curves shape presented in Fig. 2, the austenite grain growth at temperature range from 900°C to 1200°C can be divided in to three phases: phase of rapid growth (900–950°C), phase of stable growth (950–1050°C) and again phase of rapid growth (1050–1200°C). Separate analysis of curves shape for abnormal and fine austenite grains shows that growth of

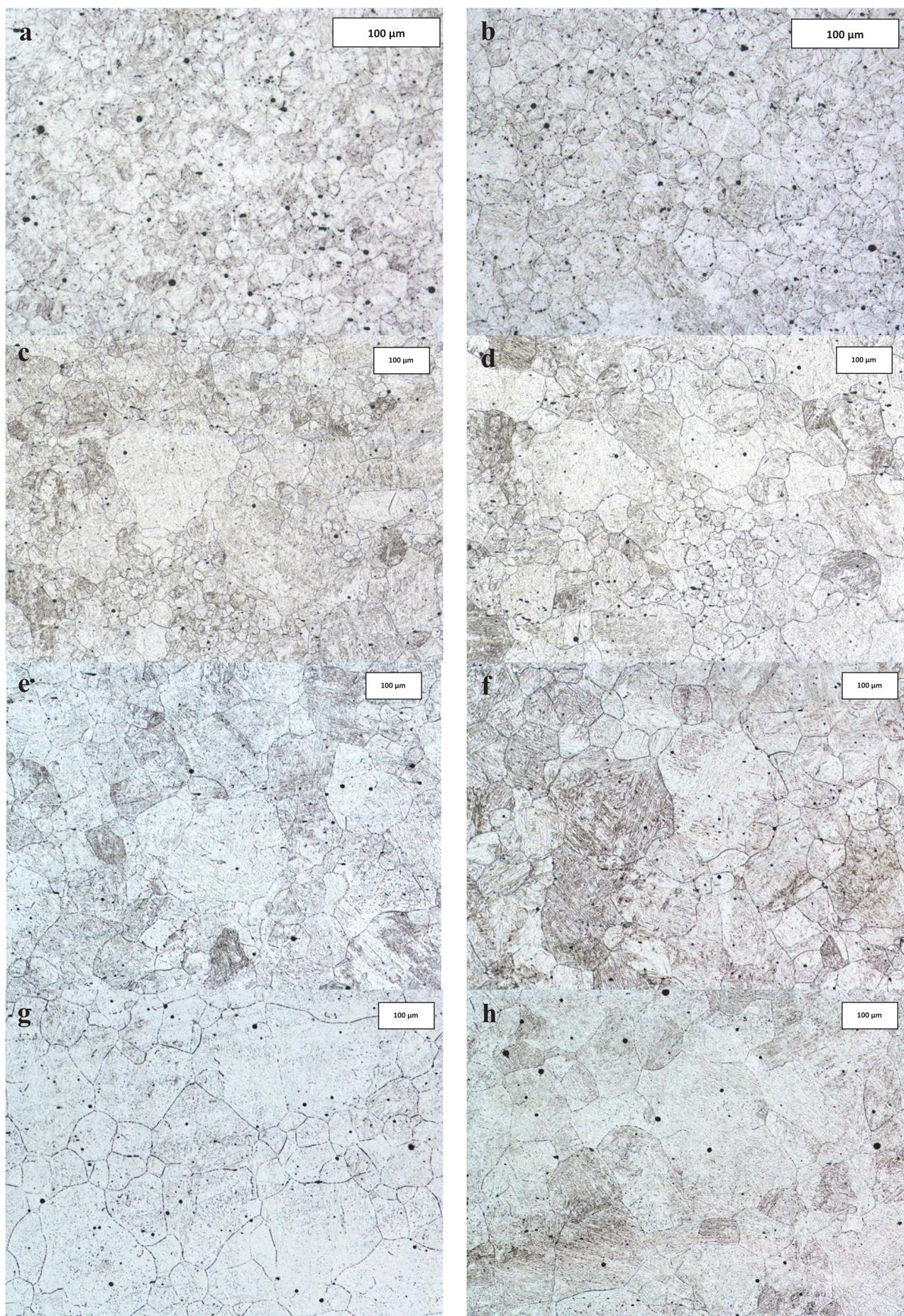


Fig. 6. Micrographs of austenite grain boundaries under different annealing conditions: a) 900°C, 20 min; b) 930°C, 20 min; c) 950°C, 20 min; d) 1000°C, 20 min; e) 1050°C, 20 min; f) 1100°C, 20 min; g) 1150°C, 20 min; h) 1200°C, 20 min. Etched state, light microscope

abnormal grains is rapid only at temperature range $900 \div 950^\circ\text{C}$ and at higher temperature level is rather gradual. However, the growth of fine austenite grains is rapid at three small temperature ranges: $900 \div 950^\circ\text{C}$, $1000 \div 1050^\circ\text{C}$ and $1100 \div 1200^\circ\text{C}$.

Based on curves' shape (Fig. 2) it was stated that the growth behavior of austenite grain at different temperature is similar for holding time of 20, 40 and 60 minutes. The same situation can be observed in case of growth of abnormal and fine austenite grains (Figs. 3 and 4). This observation was confirmed with analysis of the main effect plots for austenite grain size. The plots were prepared to compare the means to look for differences within factors - holding temperature and holding time. Based on this the sources of output variability could be identified. As it is shown in Fig. 5 the main effect on grains size has holding temperature. For different holding time the variability of austenite grain size was evidently smaller. Holding time should be shorter than 20 minutes to be able to observe the variability. As it was stated in the research [10-11] the increase in the austenite grains growth is initially rapid and then becomes slow over long times. After holding at temperature of 900°C for holding time of 20, 40 and 60 minutes the average of austenite grains size is similar (between 19 and 22 μm). After holding at 950°C growth of the grains becomes rapid and average size of all grains is between 82 and 98 μm for different holding time. This was caused by abnormal growth of some austenite grains. The size of abnormal grains at this holding temperature was from 137 to 166 μm . Due to the initial phase of grains growth is rapid it was necessary to perform the analysis of austenite grain size at additional holding temperature of 930°C . Abnormal growth of austenite grains is observed at temperature range from 950°C to 1200°C , for all holding times (20, 40 and 60 minutes). The temperature of 950°C can be indicated as a start temperature of the abnormal austenite grain growth. In phase of stable growth, the range of average size of all austenite grains was between 83 and 100 μm at three holding times. In this holding temperature range abnormal grains growth is rather gradually (abnormal grains size changes from 137 to 170 μm) but size of fine austenite grains changes by about 60% (from 28 to 45 μm). In third phase, at temperature of 1050°C , austenite grain growth again increases rapidly. While the growth of abnormal austenite grains is still gradual (its size changes from 151 to 220 μm for different holding times), the growth of smaller grains is rapid (its size changes from 37 to 81 μm at different holding times).

The migration of grain boundary can be compared to a diffusion process. So, the grain growth rate increases with raising of holding temperature. In some cases, the migration of austenite grains can be inhibited by presence of second phases particles. More information about austenite grain growth character can be received after identification of the precipitations and inclusions. After comparison of the effect of temperature and time on average austenite grain size (Fig. 5) it was stated that the main effect on austenite grain size had holding temperature. In this case the typical micrographs of austenite grain boundaries under different heating temperatures were shown only for holding time of 20 minutes (Fig. 6), as example. It can be seen in Figs. 6a and 6b that the austenite grains growth is gradual when the

temperature increases from 900 to 930°C . With increasing of holding temperature some abnormal grains appear. The abnormal grains are already presented in the microstructure after heating at temperature 950°C (Fig. 6c). These abnormal austenite grains can be also noticed in microstructure after holding at temperature 1000 , 1050 , 1100 , 1150 and 1200°C (Figs. 6d \div 6h). The difference between these microstructures is in number of fine grains. The decrease number of fine grains indicates that bigger austenite grains can merge smaller ones and grow gradually with the increase of holding temperature. The abnormal grains are developed in areas of fine grains embedded in regions. These regions of fine grains surround abnormal grains. When the fine grain areas disappear, normal growth of the coarse grains can take place.

The decreases of abnormal grain size at temperature of 1050°C relative to the abnormal grain size at 1000°C (Fig. 3) and decrease of fine austenite grain size at temperatures of 1000°C and 1100°C (Fig. 4) relative to the temperatures of 950°C and 1050°C respectively, require additional explanation especially when the average size of all austenite grains increases (Fig. 2). This is due to the fact that at 1050°C takes place the significant increase in number of abnormal grains and their size is smaller than the size of abnormal grains that are formed as the first at lower temperature, and whose size does not change dramatically at temperature of 1050°C . Their participation in the calculated arithmetic average grain size is significant, which could cause the confusing impression that at 1050°C abnormal grain size decreases. The situation is similar with regard to the calculating the arithmetic average of fine austenite grain size at temperatures of 1000°C and 1100°C . Some fine austenite grains which were presented at 950°C or 1050°C are abnormally growth at temperatures of 1000°C and 1100°C but in the structure the smallest grains, whose size has not changed, still remain. They surround the abnormal grains (ie. compare Figs 5c and 5d) and their size has a significant participation in the calculation of the arithmetic mean of fine austenite grain size that remained at temperatures 1000°C and 1100°C .

The most common microalloying elements binging of nitrogen, preventing a formation of boron nitrides, reducing the austenite grain size and increasing the strength of steel without reducing the toughness and plasticity of steel are aluminum and titanium. There are the barriers that inhibit the grain growth. Titanium and aluminum added to steel as a deoxidizer reduce the content of free nitrogen by forming aluminum or titanium nitrides (AlN, TiN). Next to Al additions, the most effective in avoiding to coarse of the austenite grains at high temperature are the elements that form stable, highly dispersed carbides. These elements are vanadium, niobium, chromium and also titanium. There are common microalloying elements in low-alloy steels that control the austenite grain growth and increase the mechanical properties. The austenite grain growth is possible only after the dissolution of these precipitates.

Understanding the impact of secondary interstitial phases on the austenite grain growth during hot working process requires the knowledge of the mechanism of their formation and their stability in austenite [12]. The dependence of MX phase solubility (concerns simple nitrides or carbides) as a function of temperature, taking into consideration the Arrhenius equation, can be presented as follows [12, 13]:

$$\log [M] \cdot [X] = B - A/T, \quad (1)$$

where: $[M]$ and $[X]$ - mass fraction of metallic microaddition and metalloid dissolved in austenite at T temperature, respectively, A and B - constants connected to free enthalpy of MX phase formation.

The values of A and B for selected nitrides and carbides (possible to be formed in analyzed steel) and calculated solution-precipitation temperature are summarized in Table 4. Formation of nitrides of these elements with high affinity to the carbon and kinetics of the precipitates were not taken into consideration. Based on this that the consumption of nitride and carbon by different elements takes place simultaneously calculated dissolution temperatures can be slightly lower.

TABLE 4
Values of the constants A and B in the equation (1) for selected carbides and nitrides [14, 15, 16]

MX phase	A	B	Temperature [°C]
AlN*	6770	1,03	946
TiN	14400	5,00	1259
TiC	10745	5,33	1186
VC	9500	6,72	765
NbC	7290	3,04	1037

*Based on assessment of Wilson and Gladman [17] these values of A and B are "most likely" one for AlN.

As it was stated during analysis of the austenite grain growth diagram two phases of rapid grain growth could be identified. In first temperature range 900 \div 950°C the growth of austenite grain can be caused by decreasing volume fraction of aluminum nitrides. Complete its dissolution at temperature of 950°C (Table 4) starts the abnormal growth of some grains. As it was mentioned the abnormal grains are developed in areas of fine grains embedded in regions. Growth of fine grains is most rapid at temperature ranges 1000 \div 1050°C (when niobium carbides are completely dissolved) and 1100 \div 1200°C (when titanium carbides are completely dissolved). This indicates that growth of austenite grains is inhibited by precipitation of AlN, TiC and NbC but dissolving of aluminum nitrides causes abnormal growth. Even if dissolving temperature of TiN is relatively high, this type of nitride is not effective inhibitors of grain growth in case of analyzed steel. The presence of some inclusions containing titanium and nitrogen was confirmed by analysis of the examined steel in delivery state. Example is shown in Fig. 7.

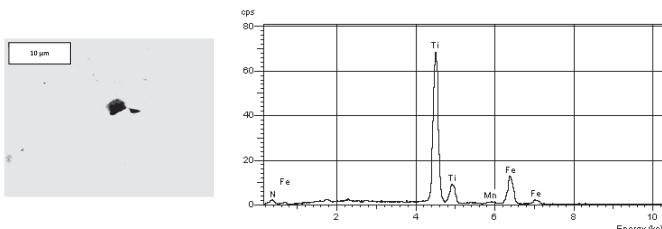


Fig. 7. EDX Spectrum of selected inclusion

Using the equation (1) dissolving temperature can be calculated only for precipitates type MX. Due this, additional TEM observations of the microstructure were performed to identification of the complex precipitates. Their dissolving temperature cannot be calculated based on equation (1) but they can also inhibit the grow of austenite grains. As can be seen in chemical composition of analyzed steel (Tab. 1) content of chromium is more than 0,37% but chromium formats complex carbides type M₃C₂, M₇C₃, M₂₃C₆. In analyzed steel some chromium carbides like Cr₃C₂ and Cr₂₃C₆ type were identified using the electron diffraction method (Fig. 8).

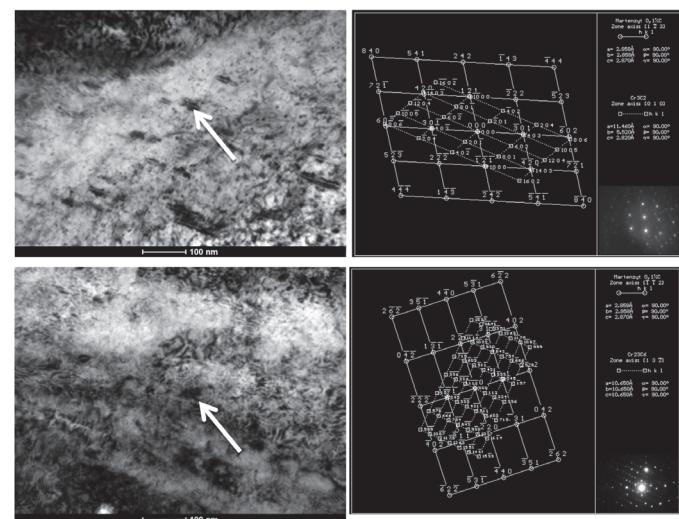
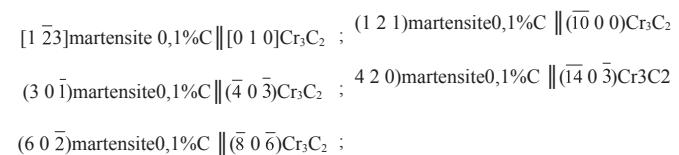


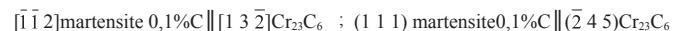
Fig. 8. Thin foils microstructures and corresponding electron diffraction patterns. Arrows indicate analyzed precipitates. Chromium carbides partially coherently coupled with martensite 0.1% C. Transmission electron microscopy

Corresponding electron diffraction patterns show partial coherence of these carbides with martensitic matrix responsible for the effect dispersion hardening.

Determined relationship between the martensitic matrix containing 0,1% C and Cr₃C₂ are:



Crystallographic relationship between the 0,1%C martensitic matrix and Cr₂₃C₆ carbides are determined:



In addition, chromium carbide precipitations, coupled coherently or partially coherently with martensitic matrix, block the dislocations and cause strengthening of steel. This kind of distribution of the precipitates also excludes the possibility of its chipping from the metal matrix under conditions of dynamic loads, thus preserving the high hardness and resistance to abrasive wear. This is very important in case of analyzed steel whose high resistant to abrasive wear is guaranteed by manufacturer.

4. Conclusion

This article examines austenite grain growth at different holding temperature and time in selected low-alloy boron steel with high resistance to abrasive wear and attempts to describe the influence of chemical composition on this process.

1. The austenite grain growth at temperature range from 900°C to 1200°C can be divided into three phases: phase of rapid growth (900 ÷ 950 °C), phase of stable growth (950 ÷ 1050°C) and again phase of rapid growth (1050 ÷ 1200°C).
2. Separate analysis of curves shape of abnormal and fine austenite grains shows that growth of abnormal grains is rapid only at temperature range 900 – 950°C and at higher temperature level is rather gradual. However, the growth of fine austenite grains is rapid at three small temperature ranges: 900 ÷ 950°C, 1000 ÷ 1050°C and 1100 ÷ 1200°C.
3. Based on the main effect plots for austenite grain size it was stated that the main effect on grains size has temperature. For different holding time the variability of austenite grain size was evidently smaller.
4. Abnormal growth of austenite grains is observed in the temperature range 950°C ÷ 1200°C, for a holding time from 20 minutes to 60 minutes. The temperature of 950°C can be indicated as a start temperature of the abnormal austenite grain growth.
5. The abnormal grains are surrounded by fine grains embedded in regions. The decrease number of fine grains indicates that bigger austenite grains can merge smaller ones and grow gradually with the increase of heating temperature.
6. In first temperature range from 900 to 950°C the growth of austenite grains can be caused by the decreasing volume fraction of aluminum nitride. Complete its dissolution at temperature 950°C starts the abnormal growth of some grains.
7. Growth of fine grains is most rapid in temperature ranges 1000 ÷ 1050°C (when niobium carbides are completely dissolved) and 1100 ÷ 1200°C (when carbides of titanium are completely dissolved).
8. Some chromium carbides like Cr₃C₂ and Cr₂₃C₆ type were identified using the electron diffraction method. Corresponding electron diffraction patterns show partial coherence of these carbides with martensitic matrix responsible for the effect dispersion hardening.

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