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INFLUENCE OF TEMPERING TEMPERATURE ON THE ABRASIVE WEAR OF HIGH-STRENGTH, MARTENSITIC BORON STEEL HARDOX EXTREME

WPŁYW TEMPERATURY ODPUSZCZANIA NA ZUŻYCIE ŚCIERNE WYSOKOWYTRZYMAŁEJ MARTENZYTYCZNEJ STALI BOROWEJ HARDOX EXTREME

Key words: wear-resistant steel, martensitic steel, abrasive wear testing, abrasive wear, tempering

Summary: The article discusses the microstructural and wear aspects of high-strength, martensitic boron steel Hardox Extreme. It is characterised by a hardness well in excess of 600 HBW and a static tensile strength R_m over 2000 MPa, which provides high resistance under abrasive wear conditions. However, such high mechanical properties reduce the steel's ductility parameters, including impact strength, elongation and area reduction. Examples of components exposed to abrasive wear, including ploughshares, cultivator teeth, excavator buckets or chutes, also require satisfactory resistance to impact wear. Subjecting Hardox Extreme steel to tempering treatments can enhance its performance characteristics by increasing its plastic properties while maintaining satisfactory mechanical indices. Therefore, it was decided to study the tribological properties of Hardox Extreme steel after stress-relieving and low-temperature tempering treatments. In the course of the work carried out, it was shown that with a reduction in hardness from 644 HBW to 508 HBW, it is possible to achieve satisfactory wear indices. The value of the coefficient of relative abrasion resistance k_b is equal to 1.36–1.12, respectively, for the as-delivered condition and after tempering treatments at 250°C.

Słowa kluczowe: stal odporna na zużycie ścierne, stal martenzytyczna, zużycie ścierne, odpuszczanie

Streszczenie: W artykule omówione zostały aspekty mikrostrukturalne i zużyciowe wysokowytrzymałej, martenzytycznej stali z borem Hardox Extreme. Charakteryzuje się ona twardością znacznie przekraczającą wartość 600 HBW i wytrzymałością na statyczne rozciąganie R_m powyżej 2000 MPa, co zapewnia wysoką odporność w warunkach zużycia ściernego. Jednakże tak wysokie własności mechaniczne powodują obniżenie parametrów plastycznych stali, uwzgledniających udarność, wydłużenie i przewężenie. Przykładowe elementy narażone na zużycie ścierne, w tym lemiesze pługa, zęby kultywatora, łyżki koparek czy zsuwnie kosza czerpakowego wymagają także zadowalającej odporności na ścieranie udarowe. Poddanie stali Hardox Extreme zabiegom odpuszczania może wpłynąć na zwiększenie jej cech użytkowych poprzez podwyższenie właściwości plastycznych przy jednoczesnym zachowaniu zadowalających wskaźników mechanicznych. W związku z powyższym zdecydowano się zbadać własności tribologiczne stali Hardox Extreme po przeprowadzeniu zabiegów odprężania i niskiego odpuszczania. W toku przeprowadzonych prac wykazano, że przy obniżeniu twardości z 644 HBW do 508 HBW możliwe jest uzyskanie zadowalającej odporności na zużycie ścierne. Wartość współczynnika względnej odporności na zużycie k_b wynosi 1,36–1,12, odpowiednio dla stanu dostarczenia i po przeprowadzeniu zabiegów odpuszczania w temperaturze 250°C.

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INTRODUCTION

Among the materials used for components subjected to abrasive wear are chromium cast iron, Hadfield steel, sintered carbides, hardfaced materials and high-strength martensitic steels. The last group includes, among others, the grades such as Hardox, Brinar, XAR, TBL, Creusabro, and HTK, which are characterised by high mechanical and ductile properties, what is determined by the low content of harmful elements (P and S), as well as the fine-grained structure obtained as a result of the manufacturing process by thermomechanical rolling. In addition, the above steels are alloyed with elements that retard the course of phase transformations (manganese, chromium, molybdenum), as well as boron microadditive, which, when added in an amount of 0.002%, causes an increase in hardenability that is equivalent to the content of 0.6% Mn, 0.7% Cr, 0.5% Mo or 1.5% Ni **[L. 1]**. In this way, due to the appropriately selected chemical composition, it is possible to obtain a homogeneous martensitic structure on the cross-section of sheets of considerable thickness – even 130 mm.

The static tensile strength R_m of higher-grade steels, i.e., Hardox 600 and Extreme, exceeds the value of 2000 MPa **[L. 2–3]**, being a comparable value relative to maraging and nanobainting steels. However, it should be noted that as the mechanical strength increases, the parameters defining plastic properties decrease, i.e., impact strength, elongation, and area reduction. The brittleness of the material, the limit of which is considered to be a value equal to 35 J/cm **[L. 4]**, can affect, first of all, the reduction of resistance to impact wear – a type of tribological wear resulting from the simultaneous occurrence of dynamic loads and abrasion, which occurs, among other things, during the drop of excavated material. Material selection is a complex issue that also requires analysis of the type of abrasive material, defined by hardness, shape, and particle size. For example, during in-service tests in the mining of lignite deposits, Hardox 500 steel did not show any significant wear compared to 18G2A steel hardfaced with Fe-Cr-C weld material **[L. 5–6]**. However, under laboratory conditions, the authors of the paper **[L. 7]** obtained contradictory results to the above position – when the applied abrasive material was quartz, Hardox 450 and 600 steel showed lower resistance to impact wear in relation to hardfaced layers. Similar conclusions were drawn in **[L. 8]**, where it was shown that the wear resistance of Hardox 500 steel is dependent upon the hardness of the abrasive material.

Despite numerous studies showing a correlation between hardness and the resulting wear indices **[L. 9–11]**, this relationship must be used with caution, taking into account other factors, such as the degree of solid solution hardening. Enhancement of mechanical indices by substitutional alloying additives results in the occurrence of wear by microcutting **[L. 12–14]**, which can cause divergent wear characteristics even among materials of the same hardness in the as-delivered state **[L. 15–17]**.

It is also possible to properly associate a material's plastic and mechanical properties by applying additional thermal treatments. Tempering is a process used immediately after quenching to remove hardening stresses (stress relieving, up to 150°C) and increase impact strength (up to about 600°C). According to **[L. 18]**, carrying out tempering processes in low-alloy martensitic steels can favourably improve wear resistance due to the precipitation of fine, needle-like ε carbides, thereby also implying an increase in yield strength $R_{p0.2}$ and impact strength. However, increasing the heat treatment temperature above 180°C results in a decrease in plastic, mechanical and tribological properties due to the coagulation of the carbide into rod-like form. Similar conclusions were also presented in **[L. 19]**, where sliding wear tests conducted while heating NM600 steel showed the highest wear resistance at 150°C. Also, according to **[L. 20]**, the wear processes of steel with a hardness of 500 HBW are least intense after tempering treatments at 200°C. According to **[L. 21]**, medium and high-temperature tempering causes a decrease in abrasive and impact wear resistance. Isothermal quenching, in which autotempering processes occur while the material is held at a certain temperature, is also a common treatment. The above solution allows to maintain favourable plastic and tribological properties in the case of steels whose hardness exceeds 650 HV10 **[L. 22]**, as well as nanobainitic steels **[L. 23]**. With these conclusions in mind, the authors of this paper decided to investigate the possibility of subjecting Hardox Extreme steel to tempering treatments in the context of maintaining high indices of abrasion wear resistance, with Hardox 450 steel used as a reference material.

MATERIALS AND METHODS

Sheets of Hardox Extreme steel, supplied directly by an authorised distributor, were used for the tests. Samples were cut out to size using the high-energy abrasive water jet method, a technology that ensures the preservation of the microstructure formed at the stage of metallurgical processing.

Analyses of chemical composition were performed on the cross-sections of the analysed Hardox Extreme steels by means of a GDS500A glow discharge emission analyser from Leco, using the following parameters: U=1250 V, I=45 mA, 99.999% argon, where the obtained results were the arithmetic average of five measurements.

The heat treatment consisted of stress-relieving and low-temperature tempering treatments in the range of 100 – 250°C for 60 min. A Czylok FCF 12SHM/R gas-tight chamber quenching furnace was used for that purpose. After removal from the furnace, the material was cooled in the air. The heat treatment was performed on Hardox Extreme steel with a chemical composition designated as HE-II.

Microscopic studies were conducted with a Nikon Eclipse MA200 light microscope, while the material was etched with a 5% HNO_3 solution. Images of the microstructures were captured with a Nikon DS-Fi2 digital camera and then were analysed using NIS Elements software.

Hardness measurements were made by means of a Zwick/Roel ZHU universal hardness tester using the Brinell method, with a load of 187.5 kgf (1838.7469 N), using a carbide ball with a diameter of 2.5 mm, according to PN-EN ISO 6506-1:2014-12.

Wear tests were performed on a cross-section of the sheets by cutting samples in the core using the WEDM method. The experiments were fulfilled with the T-07 tribotester, designed for testing abrasive wear resistance in the presence of loose abrasive, in accordance with GOST23.208-79. The T-07 tester consists of a rubber-rimmed steel wheel with a diameter of Ø = 50 (+0.2) mm and a width of 15 (+0.1) mm, a hopper that allows the flow of abrasive, a controller that stops the device after a certain number of revolutions, and a counter lever with weights that holds the sample and generates a vertical force against the wheel in the friction zone (**Fig. 1**). The hardness of the rubber applied to the steel wheel lies in the range of 78-85 ShA. The tests were carried out under a constant load of F = 44N (ΔF = 0.25 N). Electocorundum with a particle size of #90 was used as an abrasive, according to the Polish standard PN-M-59115:1976. The duration of the test depended on the hardness of the tested material and was equal to 30 min (1800 rotary cycles). The dimensions of the samples were 30 mm × 30 mm × 3 mm. The coefficient of relative abrasion resistance k_b, calculated according to equation (1), was used to measure the abrasive wear resistance. According to GOST23.208-79, the reference sample was C45 steel in the as-normalised condition.

The photographs showing the surfaces subjected to abrasive wear testing were taken with a Phenom XL electron microscope using BSE imaging and an accelerating voltage of 15 kV. The photographs were taken with magnifications within a range of 5000 – 10 000×.

$$k_b = \frac{Z_{ww}\rho_b N_b}{Z_{wb}\rho_w N_w} \tag{1}$$

where:

kb – coefficient of relative abrasion resistance (dimensionless);

Zww – mass consumption of the standard sample (g);

Zwb – mass consumption of the tested sample (g);

Nw – number of rotations of the rubber-rimmed steel wheel during the test of the standard sample;

Nb – number of rotations of the rubber-rimmed steel wheel during the test of the tested sample;

ρw, ρb – material density of the standard sample and tested sample (g/cm).

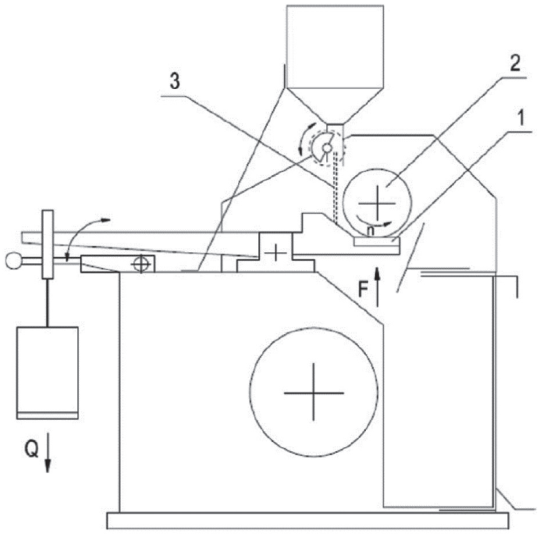

Fig. 1. Layout of tribotester T-07: 1 – examined specimen, 2 – counter-specimen, 3 – abrasive material. Reprinted from [L. 16]

Rys. 1. Schemat urządzenia T-07: 1) badany materiał, 2) ogumione koło; 3) ścierniwo. Na podstawie **[L. 16]**

RESULTS AND DISCUSSION

Chemical and microstructural analysis

Table 1 shows the chemical composition of Hardox Extreme steel for two production batches. Studies on the effect of tempering temperature on the abrasive wear were conducted on the material designated as HE-II. Analysis of the chemical composition showed a carbon content of 0.45%, classifying the steel as medium carbon. The main element that increases hardenability is manganese, whose content is equal to 0.49 – 1.0%, representing a lower value compared to steels with increased hardenability, such as 22MnB5, which can be justified by the negative effect of manganese on the grain growth of former austenite and the lowering of the martensite start temperature Ms. The content of silicon is low (about 0.14 – 0.16%), thus ensuring the preservation of satisfactory plastic properties – however, for tempering treatments it is advisable to use higher amounts since this element causes a delay in the course of transformations due to the blocking of carbon atoms diffusion. The analysed material is enriched with the addition of nickel at the level of 0.7%, which improves hardenability, provides significant solid solution strengthening and affects the lowering of both austenitising and plastic-brittle transition temperatures. However, this content is 1.4% lower than that of the production batch of steel designated HE-I, with lower values also observed for chromium and molybdenum. Boron is an additional element that enhances hardenability, which is present in a concentration typical of low-alloy martensitic steels (0.002%). The hardening effect of boron is intensified by chromium, nickel, manganese, and molybdenum. In addition, trace amounts of titanium and aluminium can be observed, which ensures the preservation of a fine-grained structure due to the tendency to form intermetallic phases that block the migration of grain boundaries at high temperatures. Vanadium has a similar effect while also helping to improve fatigue resistance, which the formation of micrometric titanium compounds can reduce. The content of harmful admixtures (P and S), which reduce mechanical properties, is negligible.

Table 1. Chemical composition of two analysed production batches of Hardox Extreme steel. HE-I – based on [L. 3]

Tabela 1. Skład chemiczny dwóch partii produkcyjnych stali Hardox Extreme. HE-I – na podstawie **[L. 3]**

	C	Mn	Si	P	S	Cr	Ni	Mo
HE-I	0.44	0.49	0.16	0.006	0.002	0.83	2.01	0.14
HE-II	0.45	1.00	0.14	0.006	–	0.07	0.70	0.07
	V	Cu	Al	Ti	Nb	Co	B	Zr
HE-I	0.008	0.018	0.04	0.008	0.001	0.02	0.0021	–
HE-II	0.008	0.005	0.04	0.003	–	0.01	0.0011	–

In the as-delivered state, the microstructure of Hardox Extreme steel consists of fine-grained martensite with a structure typical of medium-carbon steels (**Fig. 2**). A hierarchical structure is characteristic, i.e., based on the fragmentation of the grain of former austenite successively into packets, blocks and laths – during cooling, the austenite grain is divided into packets, each of which shows a different habitus plane, forming wide-angle boundaries. The packets are made up of blocks with approximate crystallographic orientation (narrow-angle boundaries), while these are composed of laths, constituting the basic crystallographic structure of the martensite. In addition, there are no areas composed of untempered martensite and non-martensitic phases observed. In comparison, the microstructure of Hardox 450 steel is also characterised by lath martensite, but due to its lower carbon content, autotempering processes do not result in the precipitation of a significant amount of carbides between the martensite blocks. For the same reason, the hierarchical structure is also more pronounced (i.e., a contrast is observed between individual packets) – steels with a lower carbon content tend to form a thick-lath martensitic structure, with individual packets being etched with varying intensity due to a change in crystallographic orientation. The higher degree of fragmentation in medium- and high-carbon steels makes it much more difficult to distinguish boundaries between packets and blocks. Low-

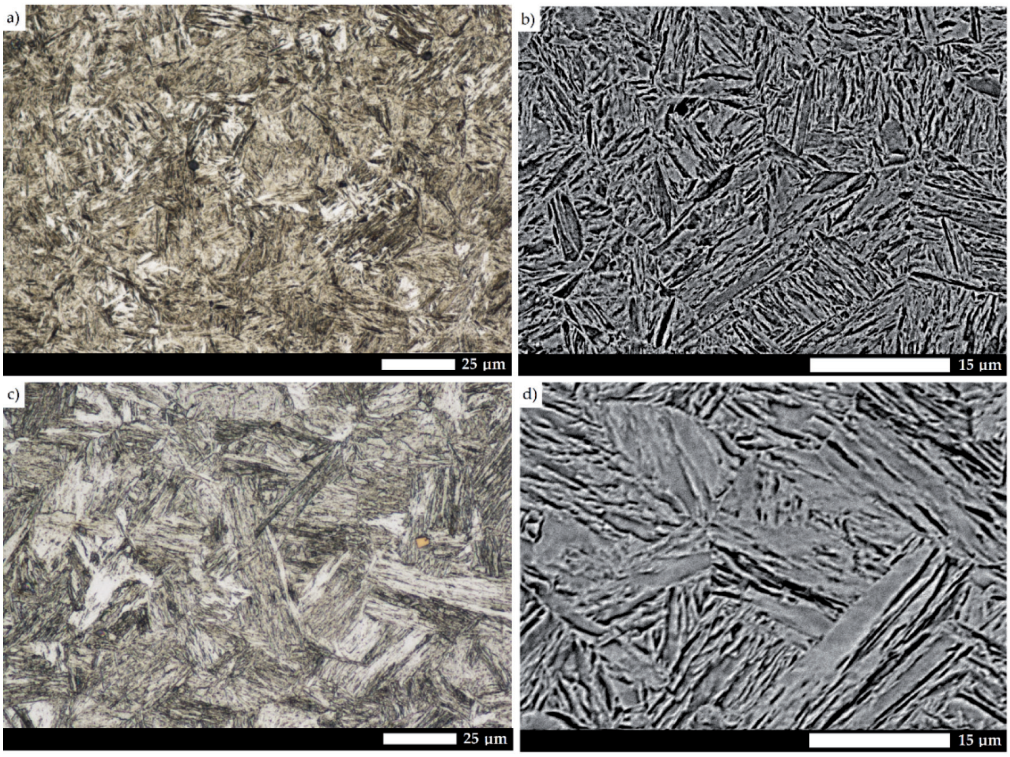

Fig. 2. Microstructures of Hardox Extreme (a–b) and Hardox 450 (c–d) steels in the as-delivered state. Light and electron microscopy etched with 5% HNO_3

Rys. 2. Mikrostruktury stali Hardox Extreme (a–b) i Hardox 450 (c–d) w stanie dostarczenia. Mikroskopia świetlna i elektronowa, trawiono 5% HNO_3

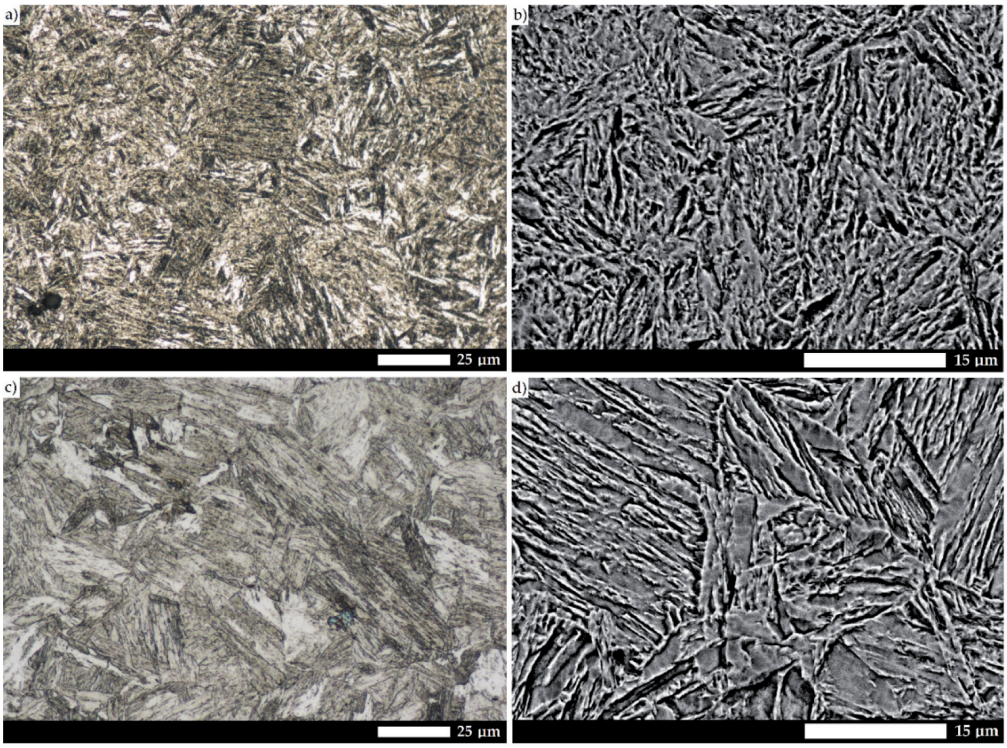

Fig. 3. Microstructures of Hardox Extreme (a–b) and Hardox 450 (c–d) steels after tempering treatments. at 250 °C. Light and electron microscopy etched with 5% HNO_3

Rys. 3. Mikrostruktury stali Hardox Extreme (a–b) i Hardox 450 (c–d) po przeprowadzeniu zabiegów odpuszczania w temperaturze 250°C. Mikroskopia świetlna i elektronowa, trawiono 5% HNO_3

carbon steels are also less susceptible to tempering processes, so no significant structural changes are visible in Hardox 450 after applying heat treatment at 250°C. In comparison, the structure of Hardox Extreme steel consists of tempering martensite – despite maintaining a structure with a martensitic orientation, the finely dispersed carbides are also observed (**Fig. 3**).

The observations described above also hold true for the course of hardness changes (**Fig. 4**). In the case of Hardox Extreme steel (production batch HE-II), the hardness of the material in the as-delivered state is 644 HBW, while increasing the tempering temperature causes a sharp and non-linear decrease, to a value equal to 504 HBW after heat treatment at 250°C. Taking the mechanical parameters as the primary criterion influencing the results obtained during wear tests, it can be expected that the resistance of steel subjected to low-temperature tempering treatments will be similar to the indices obtained for steels with a hardness of 500–550 HBW. The decreases in hardness for Hardox 450 steel are negligible, ranging from a value of 419 HBW for the as-delivered condition to 386 HBW after applying a tempering temperature of 250°C.

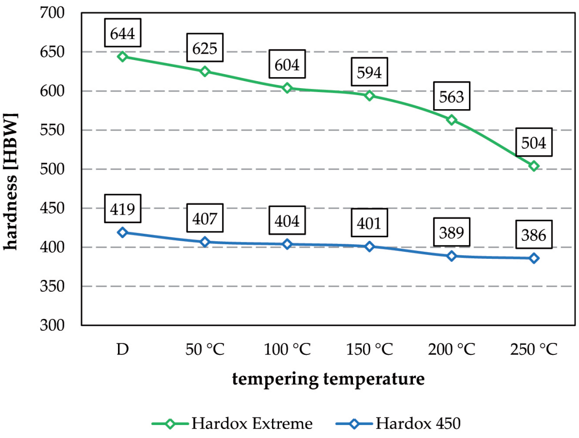

Fig. 4. Tempering curves of Hardox Extreme and Hardox 450 steels

Rys. 4. Krzywe odpuszczania stali Hardox Extreme i Hardox 450

Analysis of abrasive wear resistance

Tribological analysis carried out using the T-07 tester with electrocorundum applied as an abrasive showed that as the tempering temperature rose, the amount of weight loss increased (**Fig. 5**, **Tab. 2**). The value of the coefficient of relative abrasion resistance k_b of Hardox Extreme steel for the two analysed production batches is 1.36. In contrast, this value for the material subjected to tempering at 250°C is equal to 1.12. In the case of Hardox 450 steel, the obtained indices are similar to those of C45 steel in the as-normalised condition, confirming the conclusions drawn in **[L. 5]**, where it was shown that the coefficient k_b for steels with a hardness not exceeding 450 HBW is close to the value of 1.0. The slight increase in wear resistance after annealing at 100°C may be the effect of the removal of hardening stresses, which are the direct cause of the occurrence of wear mechanism by brittle fracture. It should be noted that due to the high mechanical properties of the analysed material, which are the result of the highest carbon content in Hardox Extreme steel among all Hardox steels, differences in the content of individual substitutional elements (nickel, chromium, molybdenum) do not show any significant effect on the amount of weight loss.

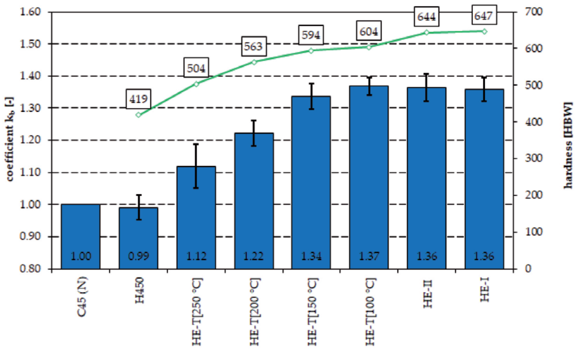

Fig. 5. The coefficient of relative abrasion resistance k_b and the results of hardness measurements of Hardox 450 and Hardox Extreme steels in different variants of heat treatment. C45 (N) – C45 steel in a normalised state with a hardness of 220 HBW, T – tempering; H450 – Hardox 450; HE – Hardox Extreme

Rys. 5. Wartość współczynnika k_b oraz wyniki pomiarów twardości stali Hardox 450 i Hardox Extreme w różnych wariantach obróbki cieplnej. C45 (N) – stal C45 w stanie normalizowanym o twardości 220 HBW, T – odpuszczanie; H450 – Hardox 450; HE – Hardox Extreme

In addition, analysis of the surfaces subjected to abrasive wear tests indicates the occurrence of increasingly severe tribological changes with a decrease in material hardness (**Fig. 6**). Abrasive wear of Hardox 450 steel occurs mainly by microploughing, the formation of wear debris and the separation of larger fragments due to the interaction (impacts) of loose abrasive particles (**Fig. 7**a). In the case of Hardox Extreme steel, the main mechanism of wear is microcutting; however,

Table 2. Average weight loss [g] of Hardox 450 and Hardox Extreme steels in different heat treatment variants along with the value of standard deviation. T – tempering; H450 – Hardox 450; HE – Hardox Extreme

Tabela 2. Średni ubytek masy stali Hardox 450 i Hardox Extreme w różnych wariantach obróbki cieplnej wraz z wartością odchylenia standardowego. T – odpuszczanie; H450 – Hardox 450; HE – Hardox Extreme

	C45	H450	HE – T [250°C]	HE – T [200°C]	HE – T [150°C]	HE – T [100°C]	HE – II	HE - I
average [g]	0.260	0.257 ±0.04	0.232 ±0.07	0.212 ±0.04	0.194 ±0.04	0.190 ±0.03	0.190 ±0.04	0.191 ±0.04

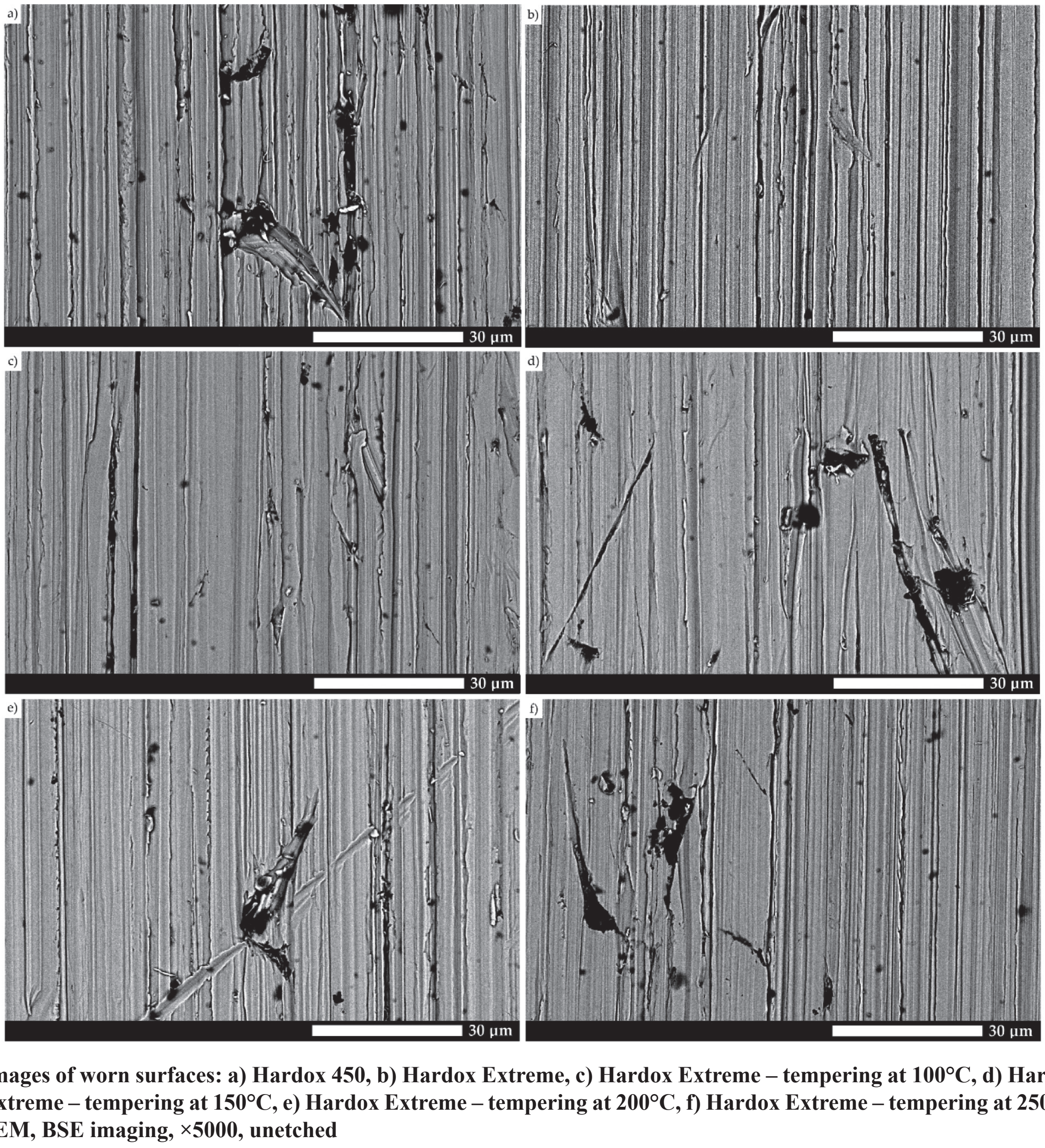

Fig. 6. Images of worn surfaces: a) Hardox 450, b) Hardox Extreme, c) Hardox Extreme – tempering at 100°C, d) Hardox Extreme – tempering at 150°C, e) Hardox Extreme – tempering at 200°C, f) Hardox Extreme – tempering at 250°C. SEM, BSE imaging, ×5000, unetched

Rys. 6. Powierzchnie poddane testom zużycia: a) Hardox 450, b) Hardox Extreme, c) Hardox Extreme – odpuszczanie w 100°C, d) Hardox Extreme – odpuszczanie w at 150°C, e) Hardox Extreme – odpuszczanie w 200°C, f) Hardox Extreme – odpuszczanie w 250°C. SEM, obrazowanie BSE, ×5000, stan nietrawiony

some features of plastic deformation of the material are also observed (**Fig. 7b**). As the tempering temperature is increased, these changes become more intense, taking on the character of wear by debris formation and microploughing for the material subjected to stress-relieving heat treatment (**Fig. 7c**). After low-temperature tempering, the observed marks are much wider and manifested by

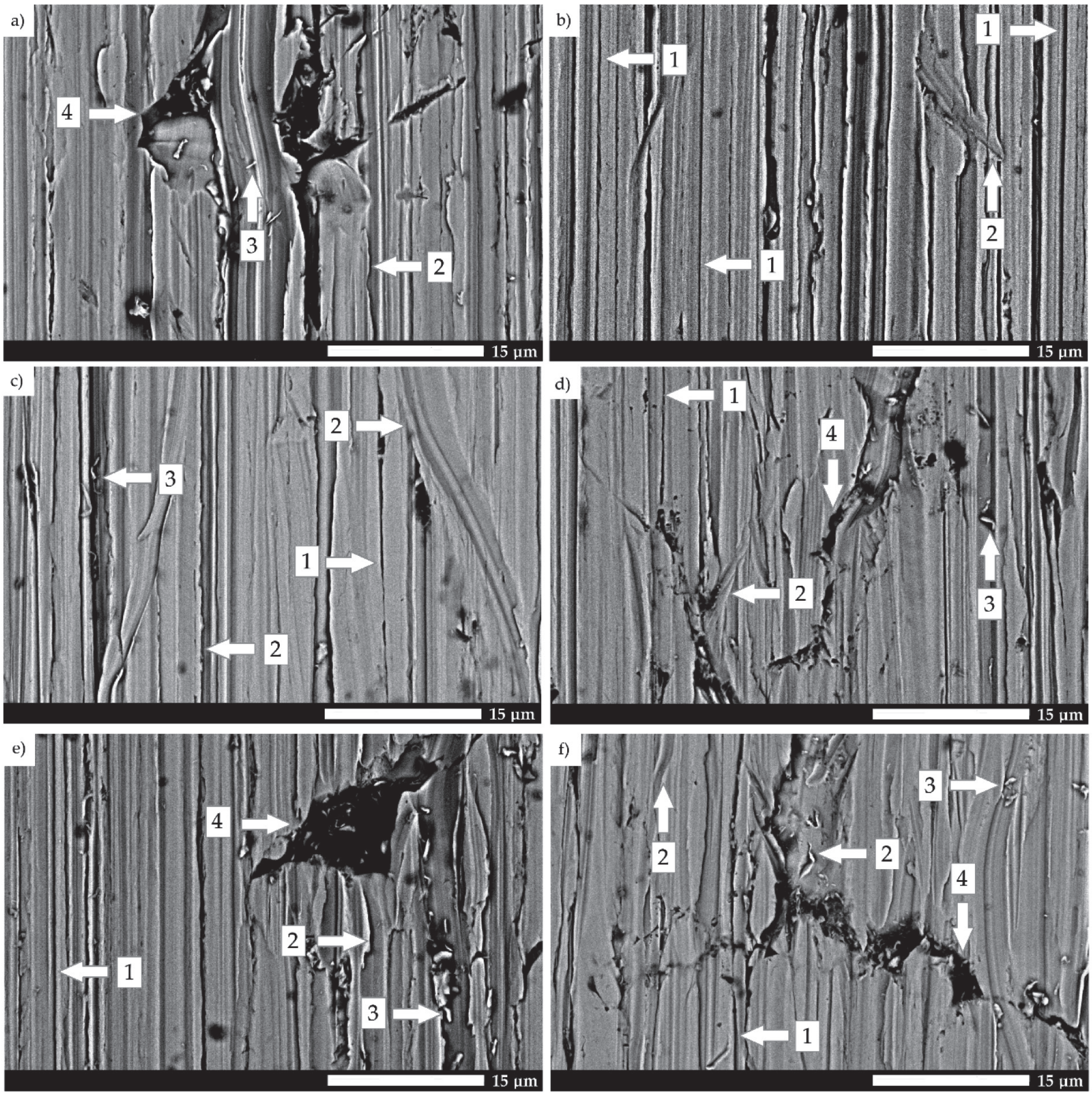

Fig. 7. Images of worn surfaces: a) Hardox 450, b) Hardox Extreme, c) Hardox Extreme – tempering at 100°C, d) Hardox Extreme – tempering at 150°C, e) Hardox Extreme – tempering at 200°C, f) Hardox Extreme – tempering at 250°C. SEM, BSE imaging, ×10.000, unetched. 1 – microcutting; 2 – microploughing; 3 – wear debris; 4 – detached material.

Fig. 7. Powierzchnie poddane testom zużycia: a) Hardox 450, b) Hardox Extreme, c) Hardox Extreme – odpuszczanie w 100°C, d) Hardox Extreme – odpuszczanie w at 150°C, e) Hardox Extreme – odpuszczanie w 200°C, f) Hardox Extreme – odpuszczanie w 250°C. SEM, obrazowanie BSE, ×5000, stan nietrawiony. 1 – mikroskrawanie; 2 – bruzdowanie; 3 – rysowanie; 4 – odłamanie większych fragmentów materiału.

the presence of furrows, what indicates an increase in the material's plastic characteristics (**Fig. 7d–f**). However, it should be noted that the detachment of larger fragments of material (gouges) accounts for a significant share of the abrasion processes taking place, and their frequency of occurrence is as intense as in the case of Hardox 450 steel.

CONCLUSIONS

The above article analysed the possibility of subjecting Hardox Extreme steel to tempering heat treatments in the context of obtaining high abrasive wear resistance indices. Based on the presented study, the following conclusions can be drawn:

- Hardox Extreme steel belongs to the group of medium-carbon steels, while the important alloying additives that increase hardenability are manganese, nickel, chromium, and boron. It should be noted that differences are observed between the content of the above elements when conducting chemical analysis on material from different production batches. However, this does not show a significant effect on the obtained results of abrasive wear tests.

- In the as-delivered state, the microstructure of Hardox Extreme and Hardox 450 steels consists of fine lath martensite. Hardox 450 steel is less susceptible to tempering processes, so no significant structural changes are observed after heat treatment at 250°C. In the case of Hardox Extreme steel, the structure is composed of tempered martensite – even though the structure with a martensitic orientation is maintained, it comes to precipitation of finely dispersed carbides.
- The hardness of Hardox Extreme steel in the as-delivered state is 644 HBW while increasing the tempering temperature causes a sharp and non-linear drop to a value equal to 504 HBW after heat treatment at 250°C. The decreases in hardness for Hardox 450 steel are negligible, ranging from a value of 419 HBW for the initial state to 386 HBW for the low-temperature tempered state.
- Analysis of wear tests showed that the amount of weight loss increases as the tempering temperature rises. The value of the coefficient of relative abrasion resistance k_b for Hardox Extreme steel for the two analysed production batches is 1.36, while for the material tempered at 250°C, it is 1.12. The obtained indices are still more favourable than those of Hardox 450 steel. It may be assumed that increased wear resistance is determined by higher hardness.
- The abrasive wear of Hardox 450 steel comes mainly through microploughing and separation of larger fragments of the material due to the action of loose abrasive particles. In the case of Hardox Extreme steel, the main wear mechanism is microcutting; however, some plastic deformation features are also observed. After low-temperature tempering treatments, the observed traces are much wider and are manifested by the presence of furrows, which indicates an increase in the material's plastic characteristics. However, it should be noted that the detachment of larger fragments of the material accounts for a significant share of the abrasion processes taking place.

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