ISSN 0208-7774 T R I B O L O G I A 6/2019 p. 73–80
DOI: 10.5604/01.3001.0013.7771

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THE INFLUENCE OF BORON IN THE SURFACE LAYER ON THE STRUCTURE AND THE TRIBOLOGICAL PROPERTIES OF IRON ALLOYS

WPŁYW BORU W WARSTWIE WIERZCHNIEJ NA STRUKTURĘ I WŁAŚCIWOŚCI TRIBOLOGICZNE STOPÓW ŻELAZA

Key words: diffusion and laser boriding, microstructure, microhardness, friction wear.

Abstract This paper presents two methods of introducing boron into the surface layer of iron alloys, namely diffusion boronizing by means of the powder method and laser alloying with a TRUMPF TLF 2600 Turbo CO_2 gas laser. Amorphous boron was used as the chemical element source. As regards diffusion drilling, the influence of temperature and time on the properties of the layer was tested. During the laser alloying, the influence of the thickness of the boriding paste layer as well as the power and laser beam scanning velocity was determined. How the carbon content in steel and alloying elements in the form of chromium and boron influence the structure of the surface layer was tested. To achieve this object, the following grades of steel were used: C45, C90, 41Cr4, 102Cr6, and HARDOX boron steel. The microhardness and wear resistance of the obtained boron-containing surface layers were tested. A Metaval Carl Zeiss Jena light microscope and a Tescan VEGA 5135 scanning electron microscope, a Zwick 3212B microhardness tester, and an Amsler tribotester were used for the tests. The structure of the diffusion- borided layer consists of the needle-like zone of FeB + Fe2B iron borides about 0.15 mm thick, with a good adhesion to the substrate of the steel subjected to hardening and tempering after the boriding process. After the laser alloying, the structure shows paths with dimensions within: width up to 0.60 mm, depth up to 0.35 mm, containing a melted zone with a eutectic mixture of iron borides and martensite, a heat affected zone with a martensitic-bainitic structure and a steel core. The microhardness of both diffusion-borided and laser-borided layers falls within the range of 1000 – 1900 HV0.1, depending on the parameters of the processes. It has been shown that, apart from the structure and thickness of the layer containing boron and microhardness, the frictional wear resistance depends on the state of the steel substrate, i.e. its chemical composition and heat treatment. The results of testing iron alloys in the borided state were compared with those obtained only after the heat treatment.

Słowa kluczowe: borowanie dyfuzyjne i laserowe, mikrostruktura, mikrotwardość, zużycie przez tarcie.

Streszczenie W pracy przedstawiono dwie metody wprowadzania boru do warstwy wierzchniej stopów żelaza, a mianowicie borowanie dyfuzyjne z zastosowaniem metody proszkowej oraz stopowanie laserowe za pomocą lasera gazowego CO_2 TRUMPF TLF2600 Turbo. Jako źródło pierwiastka użyto bor amorficzny. Zbadano w przypadku borowania dyfuzyjnego wpływ temperatury i czasu na właściwości warstwy. Przy stopowaniu laserowym określono oddziaływanie grubości warstwy pasty do borowania oraz mocy i szybkości posuwu wiązki laserowej. Przeanalizowano wpływ zawartości węgla w stali oraz dodatków stopowych w postaci chromu i boru na strukturę warstwy wierzchniej. W tym celu do badań zastosowano stale: C45, C90, 41Cr4, 102Cr6, stal borową HARDOX. Zbadano mikrotwardość oraz odporność na zużycie przez tarcie otrzymanych warstw wierzchnich zawierających bor. Do badań zastosowano mikroskop świetlny Metaval Carl Zeiss Jena i elektronowy mikroskop skaningowy Tescan VEGA 5135, mikrotwardościomierz Zwick 3212B oraz tribotester typu Amsler. Struktura dyfuzyjnej warstwy borowanej składa się z iglastej strefy borków żelaza $FeB+Fe_2B$ o grubości do ok. 0,15 mm o dobrej przyczepności z podłożem stali poddanej hartowaniu i odpuszczaniu po procesie borowania. Po stopowaniu laserowym w strukturze występują ścieżki o wymiarach: szerokość do 0,60 mm, głębokość do 0,35 mm, zawierające strefę przetopioną z mieszaniną eutektyczną borków żelaza oraz martenzytu, strefę wpływu ciepła o strukturze martenzytyczno-bainitycznej oraz rdzeń stali. Mikrotwardość warstw borowanych dyfuzyjnie i laserowo mieści się w zakresie 1000÷1900 HV0.1, w zależności od parametrów procesów. Wykazano, że poza strukturą i grubością warstwy zawierającej bor oraz mikrotwardością, odporność na zużycie przez tarcie zależy od stanu podłoża stali, czyli jej składu chemicznego i obróbki cieplnej. Wyniki badań stopów żelaza w stanie borowanym porównano z otrzymanymi tylko po obróbce cieplnej.

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INTRODUCTION

Nowadays, high requirements are being placed on the manufactured products in terms of the quality and functionality of the materials used. Surfaces of various constructions, machine elements, and tools are exposed to friction, corrosive-aggressive environments, or oxidation in high temperatures. The surface layers of products subjected to the above mentioned phenomena should be characterized by high hardness and strength, frictional wear resistance, as well as good adhesion to the substrate and a smooth transition to the core structure. Surface layers containing boron exhibit the presented properties **[L. 1–4]**.

Technologies of diffusional introduction of boron into material have been known for many years. They include powder, pastes, electrolytic, glow-discharge, and gas methods which were developed and used in practice in the 20th century. Many publications on the subject of boriding have appeared, including German and Russian, and in Poland monographs by K. Przybyłowicz **[L. 1]**, T. Wierzchoń **[L. 2]** and A. Pertek-Owsianna **[L. 3]**. Currently, there is still great scientific interest in boriding technologies, and many of the existing methods have been modified, as presented by M. Kulka in his monograph **[L. 4]**.

In the 21st century, along with the development of laser technology and the availability of lasers, including a CO_2 molecular laser, a diode one or a YAG laser, a method of laser-boriding appeared, consisting in alloying the substrate material with boron by means of a laser beam. This method is being increasingly used in surface engineering. Laser processing is an effective method, because, during the rapid heating and cooling, the alloyed material melts with the surface zone of the substrate and after solidification, the properties of the processed material increase due to the formation of fine-crystalline new structures in the surface layer, the exposure of these structures to defect creation process, and the emerging high compressive stresses **[L. 3–5]**. The advantage of laser technology is its eco-friendliness, the short duration of the process, and the possibility of performing a local surface treatment.

The classic diffusional boriding in gases **[L. 6, 7]**, powders **[L. 8–13]**, laser boriding **[L. 13–20, 24]**, as well as other varieties, e.g., with pastes and electrolytic plasma-supported boriding **[L. 21–23]** are still of great interest. The advantage of boriding is the possibility of using virtually all grades of structural, tool, unalloyed, and alloyed steels **[L. 6, 8, 9, 11, 13, 17, 18, 20, 22]**, the ones that are corrosion-resistant and heat-resistant **[L. 12, 24]**, as well as cast iron **[L. 16]**. Recently, it has also been used for other alloys, e.g., nickel and titanium **[L. 7, 21]**. Boriding is one of the well-known methods of increasing the durability of machine parts and tools in the machine and automotive industry, as well as in mining and agriculture **[L. 1, 3, 4, 5]**.

MATERIALS AND METHODOLOGY

The aim of the study was to test the influence of steel grade on the effects of boriding with the use of two methods: powder diffusion and laser boriding.

Steels to be tested were selected with the aim of determining the effect of carbon and alloying elements on the structure, hardness, and wear resistance of borided layers. The chemical composition of the tested steels is shown in **Table 1**. Among the tested steels there are known structural steels C45, 41Cr4, Hardox 450 and tool steels C90, and 102Cr6.

Table 1. The chemical composition of iron alloys [% wt]
Tabela 1. Skład chemiczny stopów żelaza [% mas.]

Steel	C	Mn	Si	Cr	Mo	Ti	B
C45	0.46	0.64	0.25	–	–	–	–
C90	0.96	0.31	0.21	–	–	–	–
41Cr4	0.37	0.75	0.23	1.04	–	–	–
102Cr6	1.00	0.31	0.21	1.44	–	–	–
Hardox 450	0.26	1.05	0.24	0.26	0.15	0.003	0.002

The samples subjected to the boriding process were ring-shaped with the following dimensions: 20 mm in external diameter, 12 mm in internal diameter, and 12 mm in height. The samples were borided by means of the gas-contact method using amorphous boron, KBF_4 as an activator as well as black carbon as filler in a temperature of 800–1000°C, for a time period of 1–4 hour. The temperature and time were selected in a way allowing one to obtain layers with a thickness of 100–150 µm. After being borided, the samples intended to be tested for friction wear were subjected to the hardening process in oil or water starting with a austenitization temperature of 850°C and then tempered in a temperature of 550°C (41Cr4, Hardox 450) or 150°C (C90 and 102Cr6 steels) for 1 hour.

For the laser heat treatment (LHT), a technological laser TRUMPF TLF 2600 Turbo CO_2 with the nominal power 2.6 kW was used. The above mentioned laser is located at the Laser Technology Laboratory of Machining Treatment Institute of Poznań Technical University. Before the laser heat treatment (LHT), amorphous boron with water glass and distilled water in the form of paste with a thickness of approx. 40–120 µm was applied. Then the samples were subjected to exposure to a laser with a power of P = 1.04 – 1.82 kW, constant laser beam scanning velocity v = 3.84 m/min, at a constant beam diameter d = 2 mm. For the tests of frictional wear, the cylindrical friction surface was covered with multiple tracks with a distance of f = 0.50 mm between them, using a constant power P = 1.04 kW and alloying paste with a thickness of 40 µm.

The observation of the microstructure was performed by means of a Metaval light microscope manufactured by Carl Zeiss Jena and a scanning microscope Tescan Vega 5135. The boron content in the remelting zone of the laser-borided layer was measured by means of the PGT Avalon micro analyser by the EDS method. The thicknesses of the layers and their hardnesses on metallographic microsections were measured through Vickers method by means of a Zwick hardness tester using the load of 100G (HV0.1).

The tests of resistance to frictional wear were carried out by means of a tribometer MBT-01 type AMSLER with the following systems: the sample (a rotating ring) and the counter-sample (sintered carbide S20S with a hardness of 1430HV). Wear resistance was determined on the basis of the wear intensity coefficient Iw, calculated from the following correlation: $Iw = \Delta m/F \cdot t$ [$mg/cm^2 \cdot h$], where Δm – a mass loss [mg], F – a friction surface [cm^2], and t – friction time [h]. The tests were carried out under the following conditions: a sample load of 147 N, 250 rpm, and dry friction. Prior to the laser boriding process, the samples to be tested for friction wear were subjected to thermal improvement with the exception of the Hardox 450 steel with martensitic-bainitic structure in the initial state.

TEST RESULTS

The results of testing the microstructure of diffusion-borided layers formed on Hardox 450 and 41Cr4 structural steels are presented in **Fig. 1**. The layers contain two iron borides FeB and Fe_2B with a small proportion of FeB phase, revealed by the coloured etching **[L. 3]**, which causes etching of the layer while the substrate remains unetched (**Fig. 1a**). The use of Nital, a basic reagent (etchant) for steel, leaves a white layer – unetched, unlike the etched substrate (**Fig. 1b**). On other steels, similar structures are obtained with a good needle-like connection with the substrate.

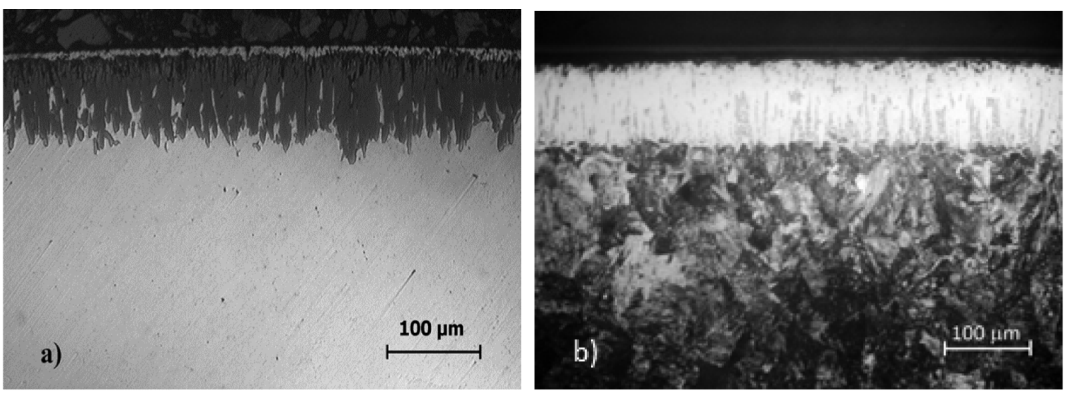

Fig. 1. The microstructure of Hardox 450 (a) and 41Cr4 (b) steel after diffusional boriding
Rys. 1. Mikrostruktura stali Hardox 450 (a) i 41Cr4 (b) po borowaniu dyfuzyjnym

The thickness of the borided layers is assumed to be the maximum extent of penetration of needles into the substrate (**Figs. 2, 3**). The tests show that it is possible to obtain layers with a thickness of up to approx. 200 µm for non-alloy steel C45, and up to approx. 150 µm for alloy steel 102Cr6. The increase in carbon concentration and alloying additives inhibit boron diffusion and the layer is narrower in thickness. The layers of optimal thickness of 100–150 µm were selected for testing their properties.

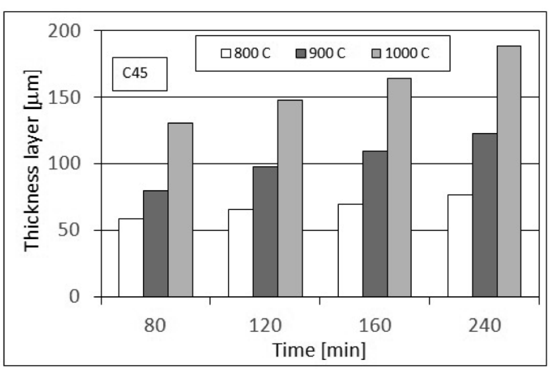

Fig. 2. The influence of temperature and time of diffusional boriding on the thickness of the layer on C45 steel
Rys. 2. Wpływ temperatury i czasu borowania dyfuzyjnego na grubość warstwy na stali C45

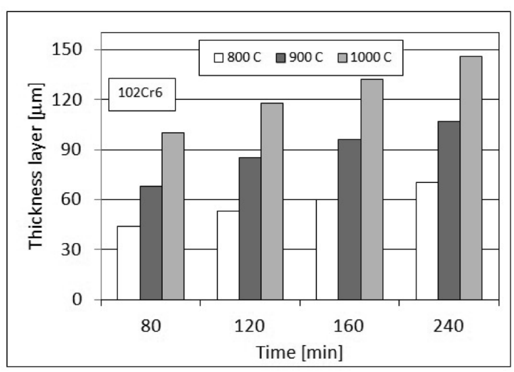

Fig. 3. The influence of temperature and time of diffusional boriding on the thickness of the layer on 102Cr6 steel
Rys. 3. Wpływ temperatury i czasu borowania dyfuzyjnego na grubość warstwy na stali 102Cr6

The results of the tests aimed at examining the microstructure of laser-borided layers formed on C45, C90 grades of steels are shown in **Fig. 4**. After the boriding process, the layer is obtained consisting of the following zones: the remelted zone (MZ), and the heat affected zone (HAZ), under which there is the core of the alloy. The microstructure of the remelted zone contains eutectics, which is a mixture of iron borides and martensite with a very fine-grained structure, and the heat affected zone demonstrates a martensitic-bainitic structure **[L. 17, 19]**.

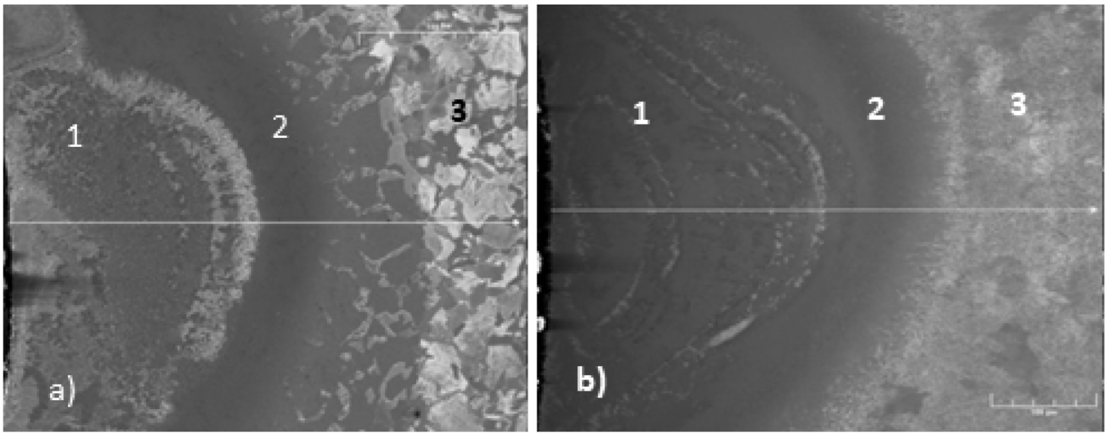

Fig. 4. The microstructure of laser-borided steels: C45 (a), C90 (b) 1-MZ (remelted zone), 2-HAZ (heat affected zone), 3-core

Rys. 4. Mikrostruktura stali borowanych laserowo: C45 (a), C90 (b) 1-SP (strefa przetopiona), 2-SWC (strefa wpływu ciepła), 3-rdzeń

The microanalysis tests, shown in **Fig. 5**, indicate that the average surface content of boron in the remelted zone of C45 steel is 7.52% wt. ±2.16% wt., and in steel C90 – 7.77% wt. ±2.29%wt. According to a diagram of Fe-B equilibrium **[L. 25]**, these levels of boron content indicate the presence of the followings borides in the melted zone: Fe_2B with a boron content of 8.83% wt. as well as non-equilibrium Fe_3B, containing 6.06% wt. of boron.

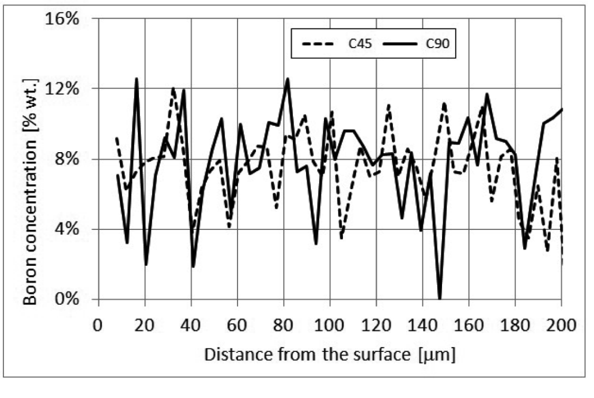

Fig. 5. Boron concentration profiles in the laser-borided layer of C45 and C90 grades of steel

Rys. 5. Profile stężenia boru w warstwie borowanej laserowo stali C45 i C90

The dimensions of the laser tracks depend on the parameters of the treatment, namely, laser beam power and the amount of the boron related to the thickness of the alloying paste. For C45 steel, width *a* and depth *b* of the remelted zone measured in the axis of the track increase along with the increasing laser power (**Fig. 6. 7**), at the same time with the increase in a depth from approx. 150 µm to approx. 350 µm, and the increase in a width from approx. 500 µm up to approx. 600 µm.

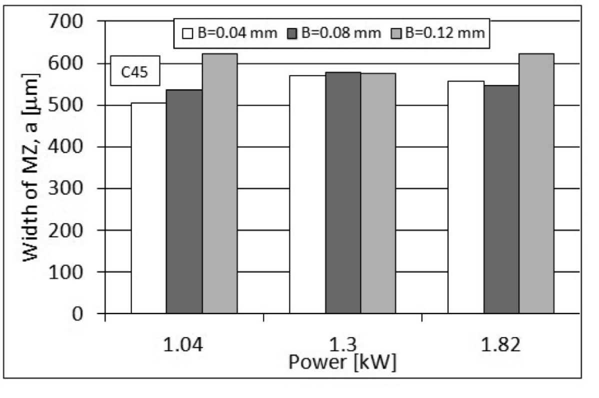

Fig. 6. The influence of laser power P and the thickness of boron paste B on the width of remelted track a

Rys. 6. Wpływ mocy lasera P i grubości pasty boru B na szerokość ścieżki przetopionej a

As the thickness of the boron paste increases, regardless of the iron alloy (C45 and C90), at a constant power level, the width of the tracks does not increase significantly, and the depth decreases (**Figs. 8, 9**); therefore, further tests were carried out for layers obtained for the smallest paste thickness of about 40 µm. The heat affected zones for tested grades of steel have similar dimensions – about 100 µm.

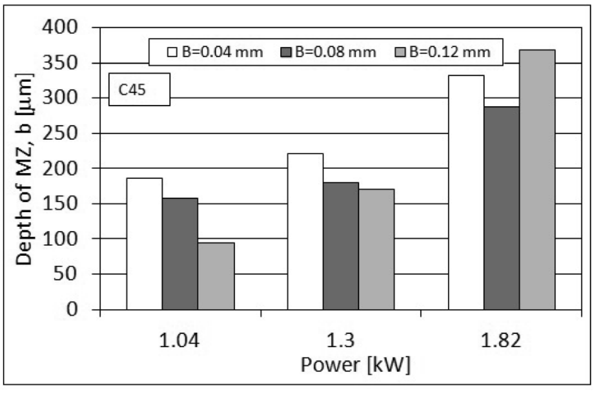

Fig. 7. The influence of laser power P and the thickness of boron paste B on the depth of remelted track b

Rys. 7. Wpływ mocy lasera P i grubości pasty boru B na głębokość ścieżki przetopionej b

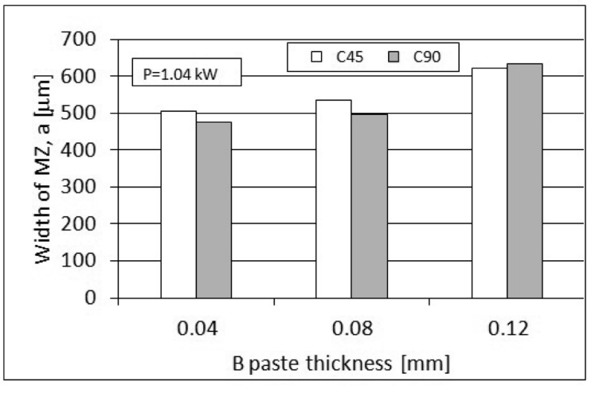

Fig. 8. The influence of the thickness of boron paste B and the grade of steel on the width of melted track a

Rys. 8. Wpływ grubości pasty boru B i rodzaju stali na szerokość ścieżki przetopionej a

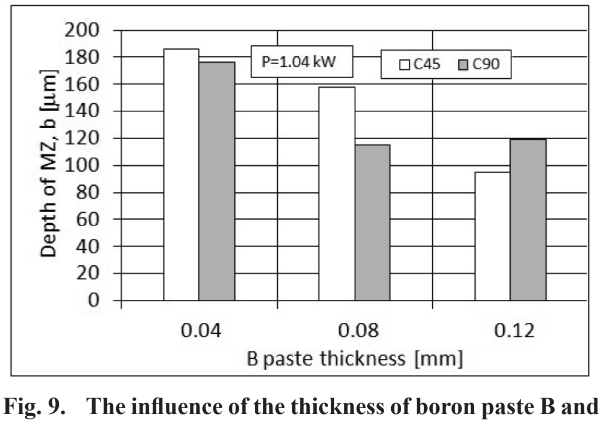

Fig. 9. The influence of the thickness of boron paste B and the grade of steel on the depth of melted track b

Rys. 9. Wpływ grubości pasty boru B i rodzaju stali na głębokość ścieżki przetopionej b

Figure 10 shows the results of microhardness measurements of 41Cr4, C90, and 102Cr6 grades of steel after diffusion boriding, and **Fig. 11** presents the comparison of the microhardness of 41Cr4 steel after diffusion and laser boriding. The tests show that iron boride layers demonstrate a similar microhardness of approx. 1200–1900 HV0.1, regardless of the grade of steel, which drops sharply towards the core in 41Cr4 thermally improved steel (up to approx. 350 HV0.1), and in C90 and 102Cr6 steels, this decrease is much smaller, because these steels, after being thermally hardened, have a high core hardness of up to approx. 950 HV0.1 (**Fig. 10**). 41Cr4 laser-borided steel (**Fig. 11**) has a lower microhardness in the melted zone of 1000–1400 HV0.1, compared to iron boride layer after diffusional boriding, which decreases gradually through the heat affected zone with a microhardness of 600–800 HV0.1 towards the core.

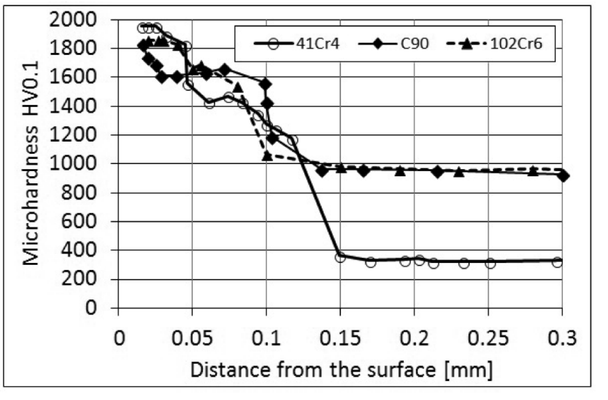

Fig. 10. Microhardness profiles of diffusion-borided grades of steel: 102Cr6 and C90

Rys. 10. Profile mikrotwardości w borowanych dyfuzyjnie stalach: 41Cr4, 102Cr6 i C90

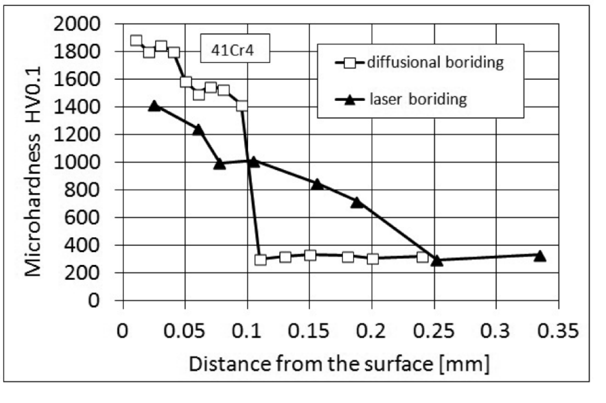

Fig. 11. Microhardness profiles of diffusion and laser-borided steel 41Cr4

Rys. 11. Profile mikrotwardości w borowanej dyfuzyjnie i laserowo stali 41Cr4

The microhardness of the surface layer after laser–boriding depends on the laser heat treatment (LHT) parameters (**Figs. 12, 13**). As the power increases, the average HV value measured for C45 and C90 grades of steel decreases from approx. 1500 HV0.1 (for P = 1.04 kW) to approx. 1000 HV0.1 (for P = 1.82 kW). The content of alloyed boron expressed in the thickness of boriding paste does not affect the hardness of the layer.

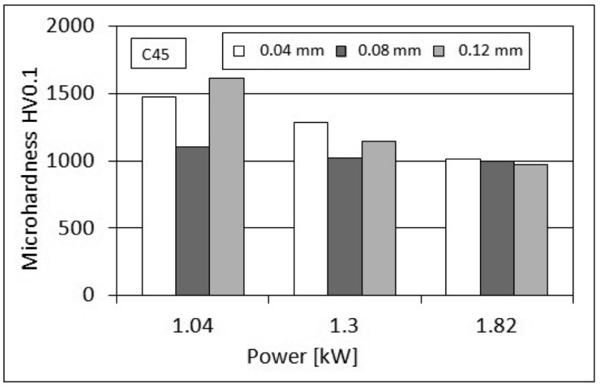

Fig. 12. The influence of laser power P and the thickness of boron paste B on the melted track microhardness of C45 steel

Rys.12. Wpływ mocy lasera P i grubości pasty boru B na mikrotwardość ścieżki przetopionej stali C45

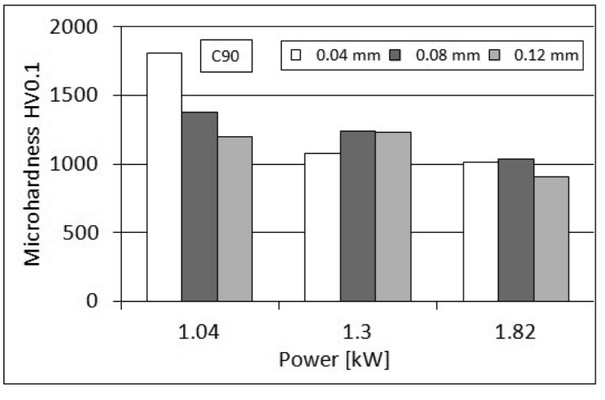

Fig. 13. The influence of laser power P and the thickness of boron paste B on the melted track microhardness of C90 steel

Rys. 13. Wpływ mocy lasera P i grubości pasty boru B na mikrotwardość ścieżki przetopionej stali C90

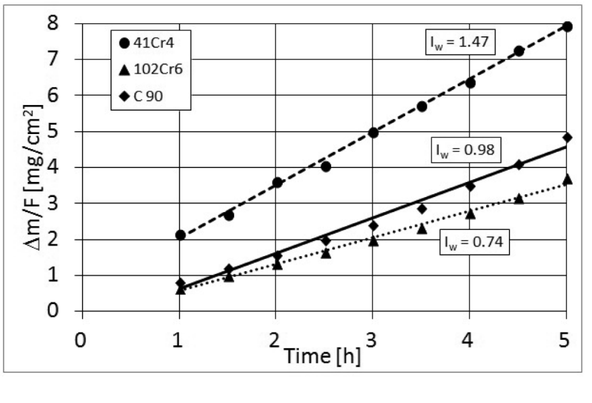

Fig. 14. Friction wear resistance after diffusion boriding of steels: 41Cr4 and C90, 102Cr6

Rys. 14. Odporność na zużycie przez tarcie po borowaniu dyfuzyjnym stali: 41Cr4, C90, 102Cr6

Based on the analysis of the results so far, samples covered with boronizing paste with the smallest thickness of 40 µm were selected to be tested for frictional wear resistance and subsequently treated by means of a laser beam with a power of 1.04 kW.

Figure 14 shows the results of the tests determining frictional wear resistance performed on diffusion- borided grades of steel. After 1 hour of running testing, all the samples exhibited signs of the end of the friction period. The further (to 5 h) mass wear related to the surface of friction was linear. C90 and 102Cr4 steels with the highest hardness of the core (**Fig. 10**) have the lowest wear, while alloy steel has the highest frictional resistance. **Figure 15** compares the wear intensity indicators Iw of two structural steels – conventional steel 41Cr4 and the newer generation steel with increased strength – Hardox 450. The tests show that Hardox has better properties than 41Cr4 after heat treatment and only as well as after diffusion and laser boriding.

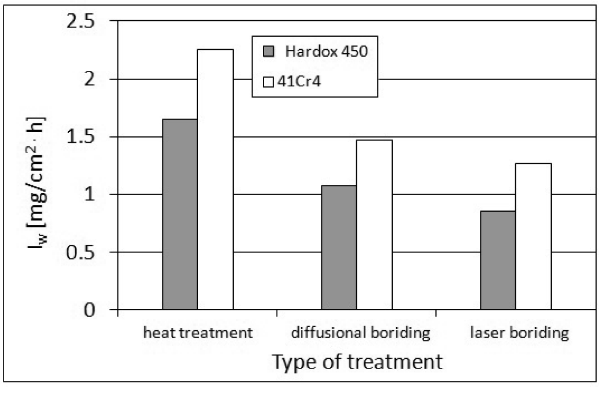

Fig. 15. Friction wear resistance after diffusion and laser boriding of steels: 41Cr4, Hardox 450

Rys. 15. Odporność na zużycie przez tarcie po borowaniu dyfuzyjnym i laserowym stali: 41Cr4, Hardox 450

Table 2 compiles the results of the tests determining the wear intensity of the tested steels after diffusion and laser boriding. The most favourable tribological properties among all tested steels are demonstrated by the following: structural steel Hardox 450 and tool steel 102Cr6.

It has been shown that lower coefficients of wear intensity are obtained after laser boriding. Compared to grades of steel subjected to only heat treatment, and the results of tests on 41Cr4 and Hardox 450 grades of steel in a borided state showed an increase in wear resistance by 1.5–2 times.

Table 2. The results of tests determining frictional wear resistance of boriding steels
Tabela 2. Wyniki badań odporności na zużycie przez tarcie borowanych stali

Steel	Type of boriding	Iw [mg/cm²/h]
41Cr4	diffusional	1.47
	laser	1.27
Hardox 450 **[L. 13]**	diffusional	1.08
	laser	0.86
C90 **[L.19]**	diffusional	0.98
	laser	1.11
102Cr6	diffusional	0.74

CONCLUSIONS

The conducted tests showed that the tribological properties of boron-containing surface layers obtained by diffusion and laser boriding are affected by the following: the chemical composition of the iron alloy, the state of the steel core (which depends on the type of heat treatment as well as the structure), and the thickness and hardness of the surface layer, in accordance with the principle of synergism **[L. 5]**.

Laser-borided steels have lower coefficients of wear intensity. It is influenced by the structure and the phase composition of the surface layer. After LHT, laser tracks with a melted zone are formed with a microhardness of 1000–1500 HV0.1 (without FeB boride with a microhardness of up to 1900 HV0.1) and with high brittleness contributing to a reduction in wear resistance in diffusion- borided layers. Therefore, diffusion- borided layers should not exceed a thickness of 150 μm, because they then tend to flake of when subjected to pressures **[L. 3]**. Whereas, laser boriding allows creating considerably thicker surface layers, with a lower microhardness, with a milder hardness gradient between the surface and the steel core, which, in practice, allows extending the service life of the work pieces.

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