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WEAR RESISTANCE OF STRUCTURAL STEELS AS A RESULT OF BORONISING OPERATIONS

ODPORNOŚĆ NA ZUŻYCIE STALI KONSTRUKCYJNYCH W EFEKCIE OPERACJI BOROWANIA

Key words: wear resistance, structural steels, structure, boronising, properties of boronised layers.

Abstract This paper presents the results of investigations on the effect of thermochemical treatment, boronising and chemical composition of selected structural steels on their wear in sliding friction process. The operation of boronising on C45, 37CrNiMo, 42CrMo6, 41Cr4, 50CrSi4-4 steels was performed by powder method at 950°C for 8 h. Following this operation, rod sections of the test steels were subjected to quench hardening from 850°C with isothermal holding at 300°C for 1h. The assessment of the construction, thickness and microhardness of boronised layers depending on the level of carbon and alloying elements in chemical composition of analysed steels was made. The testing for wear resistance of steels after boronising was carried out with the sliding friction method by applying a load of 150 N, counter-sample rotational speed of 1000 rpm and using aqueous solution of potassium chromate as a cooling medium. The metallographic observations of the structure and thickness measurement of the boronised layers were carried out using a light microscope, while the identification of phases was made by the X-ray qualitative analysis method. The hardness and microhardness measurements were taken by the Vickers method.

Słowa kluczowe: odporność na zużycie, stale konstrukcyjne, struktura borowanie, właściwości warstw naborowanych.

Streszczenie W publikacji przedstawiono wyniki badań wpływu obróbki cieplno-chemicznej, borowania i składu chemicznego wybranych stali konstrukcyjnych na zużycie w procesie tarcia ślizgowego. Operację borowania stali gatunku C45, 37CrNiMo, 42CrMo6, 41Cr4, 50CrSi4-4, przeprowadzono metodą proszkową w temperaturze 950ºC w czasie 8 godz. Po tej operacji odcinki prętów z badanych stali poddano hartowaniu z 850ºC z wytrzymaniem izotermicznym w temperaturze 300ºC w czasie 1 godz. Przeprowadzono ocenę budowy, grubości i mikrotwardości warstw naborowanych w zależności od stężenia węgla i pierwiastków stopowych w składzie chemicznym analizowanych stali. Badanie odporności na zużycie stali po borowaniu przeprowadzono metodą tarcia ślizgowego, stosując obciążenie 150 N, prędkość obrotową przeciwpróbki wynoszącą 1000 obr./min i roztwór wodny chromianu potasu jako środek chłodzący. Obserwacje metalograficzne struktury i pomiar grubości warstw naborowanych zrealizowano z wykorzystaniem mikroskopu świetlnego, a identyfikacji faz dokonano metodą rentgenowskie analizy jakościowej. Pomiary twardości i mikrotwardości zrealizowano metodą Vickersa.

INTRODUCTION

Boronizing is one of the thermochemical treatment operations whose main objective is to obtain a surface layer of high hardness (approx. 2000 HV), which is resistant to abrasion and gas corrosion at elevated temperatures [**L. 1–7**]. This treatment is used mainly for tools and parts of machines subject to wear by friction in their operating environment. In particular, boronized layers protect components against loose abrasive materials. Boronizing is suitable for the parts of vehicles (tractor axles, caterpillar components), parts of drilling tools and pumps, hot and cold work tools (stamping dies, moulds, drawing dies, pipe drawing plugs), parts of moulds for plastic materials, parts of casting machines, and parts of instrumentation such as sand spreader nozzles and retaining bushes for moulding boxes. Boronizing is primarily applicable to steels with carbon

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level of 0.35–0.45%. Higher carbon contents as well as alloying agents in the chemical composition of steel inhibit boron diffusion, thus affecting the thickness and hardness of boronized layers [**L. 8–14**]. The thickness of these layers depends on the boronizing parameters that affect boron diffusion, mainly the temperature (900–1000°C) and time of boronizing (from a few to several hours), and on the boronizing method that can be done in, but not limited to, a solid, liquid, or gas medium, also with use of the glow technology [**L. 2, 3**]. Boronized layers on common steels are characterised by thickness of up to 0.3 mm and on alloy steels of up to 0.008–0.15 mm.

MATERIALS AND METHODS

Hot-rolled rods of unalloyed structural steel, C45, and of heat-treatable alloy steels, 37CrNiMo4-4, 42CrSi6-5, 41Cr4, and 50CrSi4-4, were used for testing. The chemical composition of the test steels is provided in **Table 1**.

Table 1. Chemical composition of test steels
Tabela 1. Skład chemiczny badanych stali

Steel designation	Concentration of elements [%wt]										
	C	Mn	Si	P	S	Cr	Ni	Mo	V	W	Ti
C45	0.48	0.65	0.21	0.019	0.014	0.08	0.08	0.007	0.001	0.020	0.020
37CrNiMo4-4	0.35	0.66	0.30	0.011	0.010	1.00	1.00	0.213	0.006	0.003	0.002
42CrSi6-5	0.39	0.48	1.10	0.018	0.007	1.40	0.08	0.010	0.010	0.020	–
41Cr4	0.41	0.55	0.25	0.007	0.002	0.85	0.18	0.049	0.003	0.007	0.026
50CrSi4-4	0.54	0.40	0.94	0.010	0.006	1.01	0.14	0.025	0.003	0.005	0.040

Two sections of 15 and 40 mm in length were cut off from each of the rods of the test steels. Having been grounded and polished, the sections were subjected to boronizing immediately followed by heat treatment, i.e. isothermal quenching, in the industrial process at the Mechanical Plant "Bumar" in Gliwice, Poland.

The thermochemical treatment of boronizing the test samples in the form of rod sections of the test steels was carried out in a solid medium by the powder method. The boronizing was made in powder containing 30% boron carbide as a source of boron, 68%, with calcined aluminium oxide as a filler and activators – 1% ammonium chloride and 1% sodium fluoride to reduce the duration of boronizing. The test samples for boronizing were put in steel containers filled with a powder mixture. The boronizing was carried out in an electric furnace using two-stage heating and holding. The initial holding took place at 600ºC for one hour, and in the other ones suitable for boronizing, the test steels were made at 950ºC for 8 hours. Following the thermochemical treatment, the containers holding the samples were taken out of the furnace and cooled in the air to ambient temperature. After boronizing, the samples were subjected to isothermal quenching. The heating to the austenitising temperature of 850ºC took place in an electric salt bath furnace, capable of keeping the temperature with an accuracy of ±10°. The cooling of the samples from the austenitising temperature and their isothermal holding at 300°C was performed in the salt bath furnace.

Test samples for metallographic examinations, X-ray examinations, hardness measurements, and abrasion tests were made out of rod sections after boronizing and isothermal quenching.

To determine the effect of boronizing on the abrasion resistance of selected structural steels as well as the impact of carbon and alloying element levels in these steels on the structure, thickness, and properties of boronized layers, the metallographic examinations, X-ray examinations, hardness measurements, and abrasion resistance tests were carried out.

The microscopic metallographic examinations were performed on longitudinal, transverse, and oblique microsections made out of samples subjected to the operation of boronizing and to the process of boronizing and isothermal quenching. The microsections for microscopic observations were prepared conventionally. The method for the preparation of test samples ensured that a proper sharpness of image in the near-surface areas of the samples was obtained, in particular, during the observations of the microstructure of boronized layers. The structures of the test steels and boronized layers were examined on microsections etched with the MiFe1 reagent. The etching was carried out at room temperature, with application times of 15 to 60 seconds. The structure observations were made with light microscopes, Leica MEF4A and Olympus GX71, and the measurement of thickness of boronized layers was taken by metallographic microscope, Neophot 2. The results of the layer thickness measurements were developed statistically by adopting the average of ten measurements for calculations.

The X-ray examinations were performed with an X-ray diffractometer DRON 2.0 to make phase identification of the surface layer formed when boronizing the samples.

These examinations were carried out by applying characteristic radiation λKα of a cobalt lamp and using a Fe filter at 40kV [**L. 15, 16**]. The X-ray photographs were taken for angles between 30° and 110° in the 2Θ scale, corresponding to interplanar distances between the test phases.

The hardness measurements were performed by the Rockwell method in HRB and HRC scales. The result of the hardness measurement is the average of seven measurements.

The microhardness measurements were performed by the Vickers method using a PMT3 hardness tester with a load of 0.49 N. The hardness measurements of layers that occur after boronizing the test steels were taken on metallographic microsections using a diamond intender in the form of a quadrilateral regular pyramid, with an inclination angle of 136°, for 15 s. The value of impression diagonals was read using the hardness tester microscope's optical system. The microhardness (HV) was determined according to dependence $HV = 1.8543(F/d^2)$, taking the average length of diagonals (d) and average loading force (F).

The testing for wear resistance of steels during sliding friction was carried out using the tribological testing machine, Skoda-Savine, at the Department of Foundry in the Silesian University of Technology by applying a load of 150 N, counter-sample rotational speed of 1000 rpm and test duration of 5 min. The abrasion tests were carried out on boronized and isothermally quenched samples. The assessment of abrasibility of steels under the sliding friction conditions was being made based on the results of a test consisting in rotary movement of a counter-sample in the form of a sintered carbide disc against a fixed sample, while, at the same time, wetting and cooling the friction face with 0.5% solution of potassium chromate (K_2CrO_4) in distilled water – **Fig. 1**.

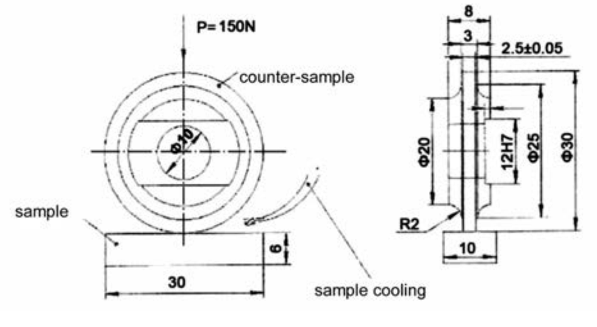

Fig. 1. Shape and dimensions of sample and counter-sample abrasion test

Rys.1. Kształt i wymiary próbki i przeciwpróbki do badań ścieralności

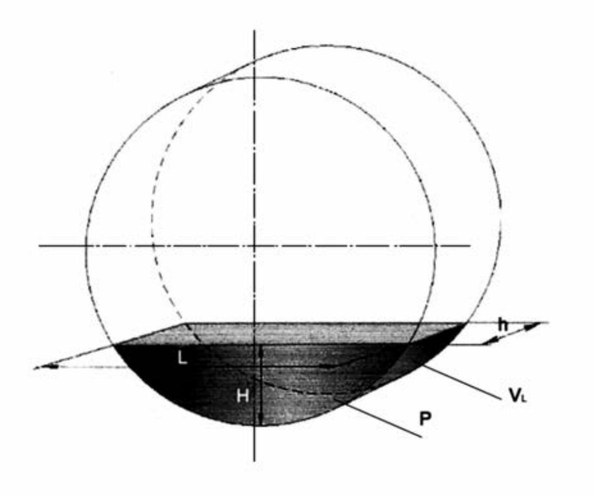

Fig. 2. Diagram of abrasion with symbols of for geometric quantities used for calculation of material abrasibility, VL – volume of abrasion, L – chord length, P, H – dimensions of a part of circular segment representing abrasion, h – disk width

Rys. 2. Schemat wytarcia i oznaczeń wielkości geometrycznych wykorzystanych do obliczeń ścieralności materiału; VL – objętość wytarcia, L – długość cięciwy, P, H – wymiary części wycinka koła odwzorowującego wytarcie, h – szerokość tarczy

The abrasion testing was carried out in three consecutive tests, calculating the specific volume of abrasion VL (worn layer of the sample) that mark the abrasibility of boronized surface layers on the test steels. The geometric quantity L was determined by microscopic method – **Fig. 2**. In calculations of the volume of the worn layer of the sample, the formulas for the determination of the volume of a disc section (**Fig. 2**) with a radius equal to that of the counter-sample (r), a chord equal to the abrasion length (L) (arithmetic mean of three measurements) and a thickness equal to the width of abrasive disc (h) were used.The presented formulas were used to determine the reaction of both the bearings of the shaft separately on the basis of their separate inner deformations.

RESULTS OF INVESTIGATIONS

The results of the investigations allowed specifying the effect of boronizing and isothermal quenching on the abrasibility determined by the structure and properties of the surface layer, which depend substantially on chemical composition of the C45, 37CrNiMo4-4, 40CrSi5-6, 41Cr4, and 50CrSi4-4 structural steels. Moreover, the results of investigations made it possible to assess the structure and hardness of the internal layer of the test steels after thermochemical and heat treatment. The documentation of the metallographic examinations of the steels after boronizing and isothermal quenching is presented in the microphotographs – **Figs. 3** and **4**. The results of X-ray identification of phases occurring in boronized layer are shown in the X-ray pattern – **Fig. 6** and in **Table 2**. The results of thickness measurement of boronized layers are provided in the form of the histogram in **Fig. 7**, and the values of microhardness are summarised in **Table 3** and of abrasibility – in the histogram in **Fig. 6**.

Following the operation of boronizing at 950ºC for 8h, a boronized layer with a columnar structure (**Fig. 3**) occurs on the surface of the test steels. In the near-surface part of this layer, there is an intensely etched porous area – **Figs. 3a–d**. On the base-material side of the steel, the acicular boron compounds penetrate into the steel material – **Figs. 3b, c**. The highest thickness of the porous area with dark colour in the boronized layer is observed in the C45 steel – **Fig. 3a**. It is approx. 50–80 μm. The minimum thickness of the porous area of approx. 30 μm occurs in the 37CrNiMo4-4 and 50CrSi4-4 steels.

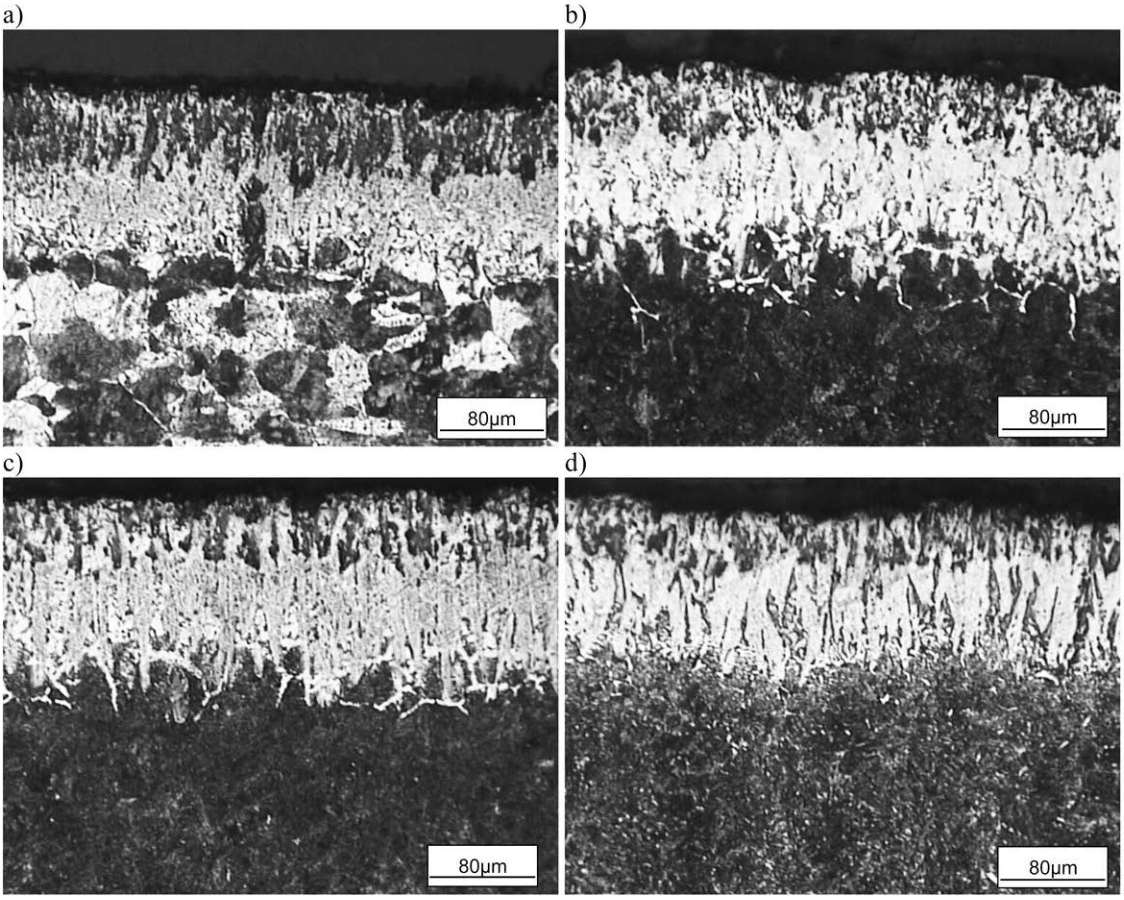

Fig. 3. The structure of boronized layer on the test steels: a – C45, b – 37CrNiMo4-4, c – 41Cr4, d – 50CrSi4-4

Rys. 3. Struktura warstwy borowanej na badanych stalach: a – C45, b – 37CrNiMo4-4, c – 41Cr4, d – 50CrSi4-4

After boronizing with cooling in the air, the internal structure with pearlite and ferrite grains of varying sizes and forms, corresponding to conditions after normalising (**Fig. 4**) is observed in the test steels. In the C45, 37CrNiMo4-4 and 42CrSi4-5 steels, the existence of ferritic-pearlitic structure arranged in bands with an average grain diameter of approx. 85 μm and hardness of 85 HRB, 77 HRB and 81 HRB, respectively, was determined, while in the structure of the 41Cr4 steel, large pearlite colonies of approx. 80 μm with a small amount of ferrite (**Fig. 4a**) and hardness of 81 HRB and in the 50CrSi4-4 steel, pearlite colonies with average grain size of approx. 120 μm and hardness of 91 HRB were observed – **Fig. 4b**.

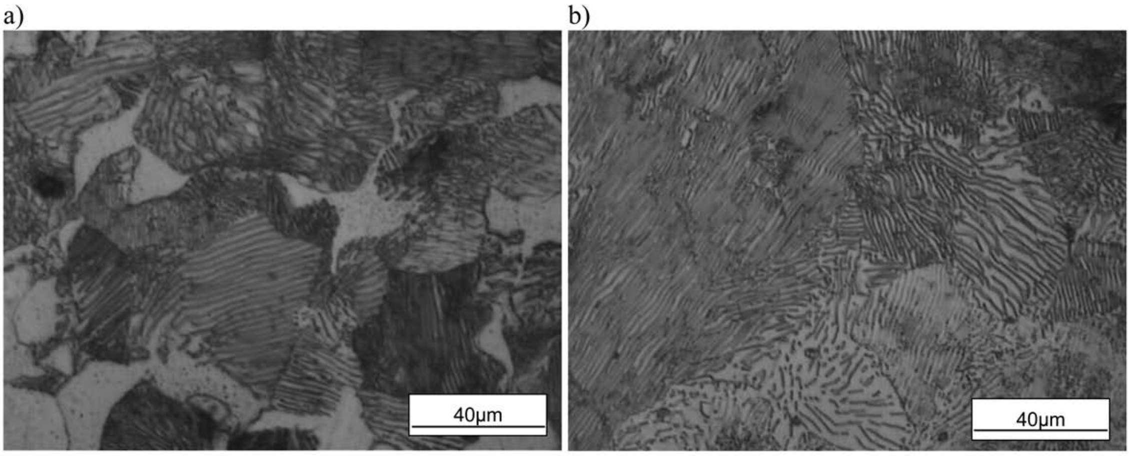

Fig. 4. The structure of internal layers of the 41Cr4 (a) and 50CrSi4-4 (b) steels after boronizing
Rys. 4. Struktura wewnętrznych warstw stali 41Cr4(a) i 50CrSi4-4(b) po borowaniu

After boronizing and isothermal quenching, the test steels are characterised by a tempered martensite structure, with the exception of the steel with higher carbon, chromium, and silicon contents, where the structure of bainite with tempered martensite occurs – **Fig. 5**. In these steels, the time of isothermal holding during quench hardening was too short for the complete transformation to take place [**L. 1, 2**].

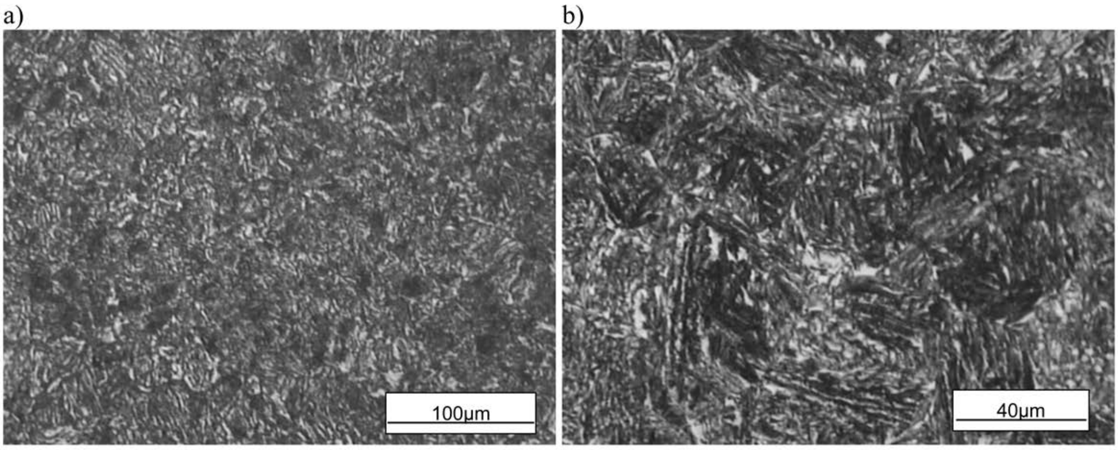

Fig. 5. The internal structure after boronizing and isothermal quenching of steel; a – 37CrNiMo4-4, b – 50CrSi4-4
Rys. 5. Struktura wewnętrzna po borowaniu i hartowaniu izotermicznym stali; a – 37CrNiMo4-4, b – 50CrSi4-4

In the C45, 37CrNiMo4-4 and 40CrSi6-5 steels, there is a tempered martensite structure (**Fig. 5a**) with a hardness of 31, 30, and 34 HRC, respectively, while, in the 41Cr4 and 50CrSi4-4 steels, there is a structure of tempered martensite with bainite marked by hardness of 37 and 41 HRC, respectively – **Fig. 5b**. Tempered martensite with high structure refinement was found in the structure of the C45 and 37CrNiMo4-4 steels – **Fig. 5a**. High-tempered martensite is a structure that provides steel with high impact strength and tensile strength [**L. 1–3**]. Such properties are required for axles, stamping dies, moulds, vehicle axles where surfaces are hardened by boronizing.

The structural condition of steels after boronizing and heat treatment have a significant impact on the strength properties and wear resistance of machine components and parts as well as tools. Therefore, it is important not only to use the appropriate boronizing parameters, but also to carry out a heat treatment after boronizing with optimum parameters to ensure the expected properties of the internal layers of the material.

The decisive factor for the properties of material is its structure. As a result of boronizing, the existence of two-phase area comprised of FeB and Fe_2B borides is observed in the surface layers of the test steels – **Fig. 5**. FeB borides occur directly at the surface, forming a porous layer and are etched a little more intensely than the Fe_2B boride layer observed at a slightly lower level – **Fig. 3**.

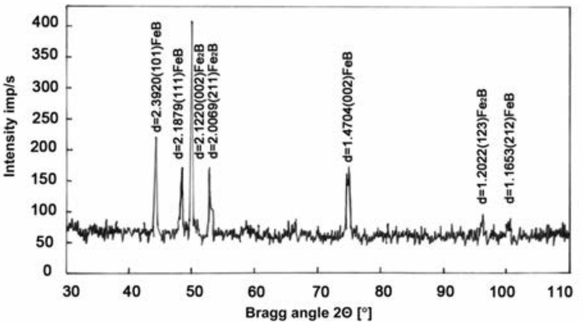

Fig. 6. The X-ray pattern of the surface layer of 40CrSi6-5 steel after boronizing

Rys. 6. Dyfraktogram rentgenowski warstwy wierzchniej stali 40CrSi6-5 po borowaniu

The identification of phase composition of the boronized layers was carried out based on the results of X-ray examinations. In the boronized layers of the test steels, the existence of FeB and Fe_2B phases was found. The results of the identification of phase composition for the boronized layer on the 40CrSi6-5 steel are shown in **Table 2**. The X-ray pattern for the surface layer of the 40CrSi6-5 steel after boronizing shows diffraction lines from the (101), (111), (002), and (212) planes of the FeB phase and from the (002), (211), and (123) planes of the Fe_2B phase – **Fig. 6** and **Table 2**. The existence of three diffraction lines with the maximum intensity of identified boron compounds in the X-ray pattern confirms the presence of FeB and Fe2B phases in the surface layer.

The boronized layer in the C45, 37CrNiMo4-4, 40CrSi6-5, 41Cr4, and 50CrSi4-4 steels have different thickness, which depends mainly on the chemical composition of steel, in particular, on the content of carbon and carbide-forming elements – chromium and molybdenum.

Table 2. The results of X-ray examinations of the boronised layer in the 40CrSi6-5 steel

Tabela 2. Wyniki badań rentgenograficznych warstwy naborowanej w stali 40CrSi6-5

Experimental data					Phase identification			
No.	2θ [°]	Θ [°]	Sinθ	d$_{cal}$ [Å]	d$_{tbl}$ [Å]	I/I$_o$ [%]	(hkl)	Phase
1	43.9	21.95	0.3739	2.3901	2.3920	100	101	FeB
2	48.3	24.15	0.4091	2.1879	2.1879	74	111	FeB
3	49.9	24.95	0.4218	2.1221	2.1220	23	002	Fe_2B
4	52.8	26.40	0.4446	2.0132	2.0069	100	211	Fe_2B
5	74.8	37.40	0.6074.	1.4739	1.4740	100	002	FeB
6	96.2	48.10	0.7443	1.2026	1.2022	12	123	Fe_2B
7	100.3	50.15	0.7677	1.1208	1.1653	71	212	FeB

The boronized layer thickness in the test steels ranges between 121 µm and 139 µm. The boronized layer with the highest thickness occurs in the C45 steel and amounts to 139 µm, while the layer with the lowest thickness of 121 µm is observed in the 50CrSi4-4 steel. In the 37CrNiMo4-4, 40CrSi6-5, and 41Cr4 steels, the boronized layer thickness is 128 µm, 134 µm, and 138 µm, respectively – **Fig. 7**.

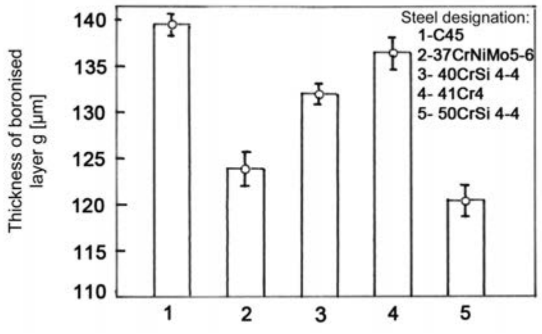

Fig. 7. Thickness of boronized layer vs. chemical composition of steel

Rys.7. Zależność grubości warstwy naborowanej od składu chemicznego stali

The microhardness measurement in steels after boronizing and isothermal quenching was taken in the boronized layer, transition layer, and base material. The results of the measurement are provided in **Table 3**.

The maximum microhardness is observed in the boronized layer of the C45, 37CrNiMo4-4, and 50CrSi4-4 steels, amounting to 1933 HV, 1927 HV, and 1975 HV, respectively. The microhardness of boronized layer in the 40CrSi6-5 steel is 1827 HV, and in the 41Cr4 steel, it is 1872 HV. Microhardness in the transition layers reaches the level of 506 to 612 HV, and, in the internal layers of steels, it ranges between approx. 276 HV and 359 HV – **Table 3**.

Table 3. The results of microhardness measurement in the boronized, transition and internal layers of steel

Tabela 3. Wyniki badania mikrotwardości w warstwie naborowanej, przejściowej i w warstwie wewnętrznej stali

Steel designation	Microhardness HV0.05		
	Boronised layer	Transition layer	Inner layer of steel
C45	1933	506	292
37CrNiMo4-4	1927	549	276
40CrSi6-5	1827	571	298
41Cr4	1872	518	319
50CrSi4-4	1975	612	359

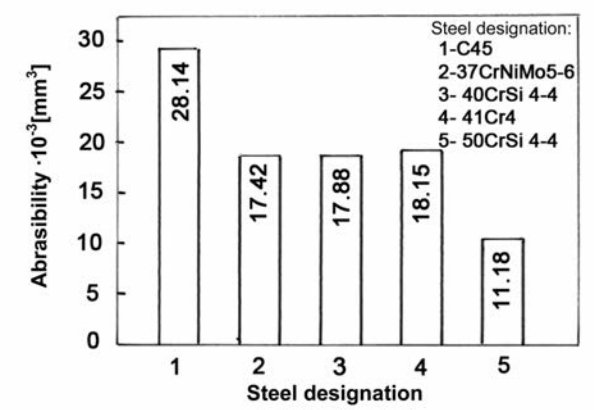

Fig. 8. The results of abrasion tests of the test steels after boronizing and isothermal quenching

Rys. 8. Wyniki badań ścieralności badanych stali po borowaniu i hartowaniu izotermicznym

The results of abrasion tests allowed the effect of the chemical composition of the test steels and the process of boronizing with isothermal quenching on their wear to be determined. The results of abrasion test are shown in **Fig. 8**.

The wear resistance of the test steels determined in the abrasion tests under the limit sliding friction conditions is varied – **Fig. 8**. The minimum abrasibility of $11.18 \cdot 10^{-3}$ mm^3 is observed in the 50CrSi4-4 steel; whereas, the values of abrasibility for the 37CrNiMo6-5, 40CrSi4-4, and 41Cr4 steels are comparable and amount to approx. $17.42 \cdot 10^{-3}$ mm^3, $17.88 \cdot 10^{-3}$ mm^3, and $18.15 \cdot 10^{-3}$ mm^3, respectively. The abrasibility of the alloy steels is significantly lower than that of the unalloyed one. The C45 unalloyed structural steel was found to reveal the maximum abrasibility of $28.14 \cdot 10^{-3}$ mm^3.

Table 4 shows the summary measurement results for abrasibility, the thickness of the boronized layer, and the hardness of this layer and the internal layers of the test steels. It was found that the results obtained in individual tests are significantly affected by the content of elements in the test steels and the process of boronizing with isothermal quenching as a result of which the formed structure of surface and internal layers determines the tested properties of structural steels.

Table 4. The summary results of investigations on structural steels after boronizing and isothermal quenching

Tabela 4. Zbiorcze wyniki badań stali konstrukcyjnych po borowaniu i hartowaniu izotermicznym

Steel designation	Concentration of elements [% wt]						Hardness HV0.05		g [µm]	Abrasiveness $\cdot 10^{-3}$ [mm^3]
	C	Mn	Si	Cr	Ni	Mo	Steel	Boronised layer		
C45	0.48	0.65	0.21	0.08	0.08	0.007	292	1933	139	28.14
37CrNiMo4-4	0.35	0.66	0.30	1.00	1.00	0.213	276	1927	128	17.42
40CrSi4-4	0.39	0.48	1.10	1.40	0.08	0.010	298	1827	134	17.80
41Cr4	0.41	0.55	0.25	0.85	0.18	0.049	319	1872	138	18.15
50CrSi4-4	0.54	0.40	0.94	1.01	0.14	0.025	359	1975	121	11.18

CONCLUSIONS

The examinations carried out as well as the obtained results and their analysis allowed for the following conclusions to be drawn:

- The process of boronizing with isothermal quenching of structural steels makes it possible to obtain wear resistance on the surface of 50CrSi4-4, 37CrNiMo4-4, 40CrSi4-4, 41Cr4, and C45 steels determined by abrasibility amounting to $11.18 \cdot 10^{-3}$ mm^3, $17.42 \cdot 10^{-3}$ mm^3, $17.88 \cdot 10^{-3}$ mm^3, $18.11 \cdot 10^{-3}$ mm^3, and $28.14 \cdot 10^{-3}$ mm^3, respectively.
- The obtained abrasion test results show the summary effect of the boronizing process and chemical composition on the resulting surface layer properties.

- As a result of the thermochemical treatment with regard to boronizing in powders with use of the boron-bearing substance – B_4C boron carbide, a layer with a thickness of 121–139 µm and a hardness of approx. 1827–1975 HV was produced in the test steels.
- The boronized layer is characterised by good quality and correct structure with no cracking and splinters, which proves good layer adhesion and properly completed thermochemical treatment.
- The maximum hardness is observed in the near-surface layer of the 50CrSi4-4 steel and amounts to 1975 HV.

In the transitional layer, the hardness is 612 HV, and in the core, it is 359 HV.

The X-ray qualitative phase analysis of boronized surface layer in the test steels revealed the existence of two-phase layer of FeB and Fe_2B borides.

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