Iwona BAUER*

MICROSTRUCTURE AND RESISTANCE TO ABRASIVE AND CORROSIVE WEAR OF A CHROMOSILICONIZED LAYER ON C45 STEEL SUBSTRATE

MIKROSTRUKTURA I ODPORNOŚĆ NA ZUŻYCIE ŚCIERNO-KOROZYJNE WARSTWY CHROMOKRZEMOWANEJ NA STALI C45

Key words: chromosiliconized layer, wear, corrosion.

Abstract The paper presents the results of investigation of the microstructure and resistance to the abrasive and corrosive wear of a chromosiliconized layer on C45 steel substrate. The chromosiliconizing process was carried out at 1000°C for 6 hours by the powder pack method. Microstructure, chemical and phase composition of the layer as well as surface roughness, thickness, and hardness measurements were carried out. Tribocorrosion tests were conducted with the use of a wear tester composed of three rollers and a cone. The frictional resistance of a chromosiliconized layer was investigated under exposure to a corrosive medium of an aqueous suspension of quartz sand. Chromosiliconizing of C45 steel influenced the resistance to wear of the layers during friction processes in the abrasive and corrosive environment.

Słowa kluczowe: warstwa chromokrzemowana, zużycie, korozja.

Streszczenie Omówiono wyniki badań dotyczące mikrostruktury warstwy chromokrzemowanej wytworzonej na podłożu stali C45 w kształtowaniu odporności na zużycie podczas tarcia ścierno-korozyjnego. Proces chromokrzemowania realizowano przy temperaturze 1000°C przez 6 godzin metodą proszkową. Przeprowadzono badania mikrostruktury, składu chemicznego i fazowego warstwy, a także pomiary chropowatości jej powierzchni oraz grubości i twardości. Badania zużycia podczas tarcia warstwy chromokrzemowanej narażonej na oddziaływanie zawiesiny wodnej piasku kwarcowego przeprowadzono za pomocą tribometru typu trzy wałeczki/stożek. Chromokrzemowanie stali C45 zwiększyło jej odporność na zużycie w procesie tarcia w warunkach oddziaływania korozyjnego.

INTRODUCTION

The degradation of devices as a result of mechanical and corrosive factors and their combined impact constitute a significant problem concerning many branches of industry. Designing machine parts, which would guarantee their reliability, emerges as a challenge for constructors in many aspects, for example, the chemical composition of the material or applied production technology. Materials used for friction nodes should exhibit good functional properties, which are discussed, among others, in the works **[L. 1–6].** The contact of machines with natural resources and the aggressive environment in which they work in various industrial sectors are the causes of their excessive wear. In many research facilities, research is being conducted to improve the technology of the creation of surface layers that affect the life of the equipment as a result mechanical loads and wear during abrasive-corrosive friction. The beneficial effect of surface treatment technologies such as chrome plating, hybridization, and laser alloying on the increase in their performance has been presented in the research papers **[L. 7–11].** The pack cementation method is a subject of studies in research facilities **[L. 12–13]**. Due to the obtained results, it will be applied on parts of machines and devices in many branches of industry, e.g., American Company Alon Pressing INC, for the diffusional aluminization of pipes used in chemical systems. Taking into account the economic aspect, i.e. the low manufacturing costs and the increase

* University of Warmia and Mazury in Olsztyn, Faculty of Technical Sciences, ul. Oczapowskiego 11, Olsztyn 10-719, Poland, e-mail: iwona.bauer @uwm.edu.pl.

in the functional properties of machine parts, the powder chromosiliconization process meets a number of expectations **[L. 14–17].** Very few studies have been devoted to the wear of chromosiliconized layers in the friction process under corrosive conditions **[L. 18–24]**; therefore, conducting research in this field is of utilitarian significance. Machines and devices working in open-pit mining, construction, the glass industry, and agriculture are exposed to excessive wear as a result of the impact of quartz sands and gravels.

The purpose of the work was to assess the effect of the chromosiliconized layer on the increase in the wear resistance of C45 steel in the friction processes under conditions of the interaction with a corrosive medium, which in this experiment, was an aqueous suspension of quartz sand.

MATERIALS

Experimental specimens were made of C45 steel. The chromosiliconized layer was produced in the Labotherm LH 15/14 laboratory furnace. The technological process was carried out at 1000°C for 6 hours. A powder mixture (wt.%) containing ferro-chromium with SiC (70%), kaolin (29.5%) and ammonium-chloride (0.5%) as an activator was applied in order to form a layer. Samples of C45 steel were placed in previously prepared powder mixture in boxes made of stainless steel X6CrNiTi18-10. The boxes were closed and sealed hermetically with enamel to prevent charge oxidation.

To assess the construction and properties of the chromosiliconized layer (three samples were subjected to each test):

– Microstructure analyses and thickness measurements were made using an Olympus IX70 inverted system microscope, and the samples made of C45 steel with dimensions of 4.0×8.0×12.00 mm were subjected to metallographic investigation of their chemical compositions.
– The quality of the surface, i.e. their shine tint and surface damage were assessed by means of SEM and visual inspection.
– Analysis of the chemical composition of the chromosiliconized samples were conducted using SEM with X-ray EDS microanalysis.
– Analysis of the chemical composition of the diffusion layer was performed using glow discharge optical emission spectroscopy (GDOES).
– Porosity measurements along the cut-off length l_c = 0.8 mm and the length of travel l_t = 4.8 mm were taken with the use of the Hommel Tester T1000E. The following values were registered: *Ra* – arithmetic average deviation from the mean line, *Rz* – height at ten roughness profile peaks, *Sm* – mean spacing of roughness profile peaks.
– Analysis of the phase composition of the chromosiliconized layer was conducted using a Phillips X-Pert X-ray diffraction system using monochromatic $CuK_α$ radiation, with a tube power at 40 kV and 30 mA.
– Hardness tests HV0.05, were made according to standard PN-EN ISO 6507-1: 2007 **[L. 25].**
– Tribocorrosion tests were performed to determine the wear of samples with the use of a I-47-K-54 wear tester composed of three rollers and a cone. The samples were made of a chromosiliconized layer and C45 steel with the dimensions of Ø 8.0×21.0 mm with a cone-shaped toughened to 48HRC.

The samples for tribocorrosion tests (three samples for every surface load) were hardened in oil at T = 840°C and tempered at T = 500°C for a period of 2 hours. The following parameters were applied in the tribocorrosion test: friction velocity – 0.58 m/s, cone rotational speed – 576 rpm, force per unit area – 50 MPa, 100 MPa, 200 MPa. Test time was *t* = 100 min, which corresponded to a friction path of *s* = 3470 m. Wear was evaluated microscopically to the nearest 5 μm at 10 minute intervals. The corrosive medium was an aqueous suspension consisting of quartz sand administered in the amount of 30 drops/minute with pH 6.8 and the following phase composition (wt. %): SiO_2-88.5%; Fe_2O_3-0.02%; Al_2O_3- 0.74%; TiO_2-0.74%; H_2O-10%.

RESULTS AND DISCUSSION

The surfaces of the samples made of C45 steel were subjected to both visual and microscopic analyses after chromosiliconizing. The samples displayed a grey coloured surface with a slight shine effect. An exemplary image of the surface of C45 steel following chromosiliconizing is shown in (**Fig. 1).**

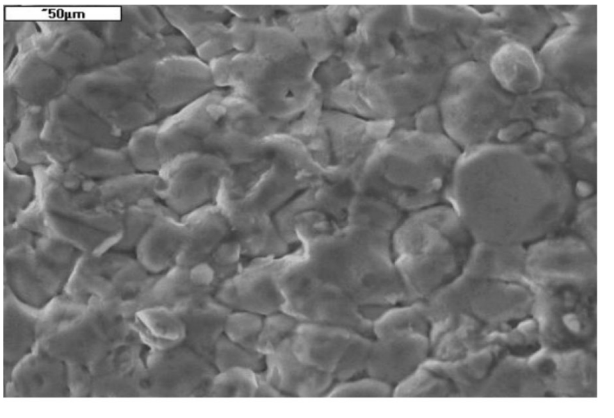

Fig. 1. Surface of C45 steel, after chromosiliconization, SEM, magnification 500×, etched with 2 vol. % of HNO_3 in H_2O

Rys. 1. Powierzchnia stali C45 po chromokrzemowaniu; obraz SEM, powiększenie 500×, trawiono roztworem 2% obj. HNO_3 w H_2O

The identification conducted in the micro-regions on the surface of the chromosiliconized sample revealed the presence of the following elements: Cr, Si, Al, and C **(Fig. 2)**.

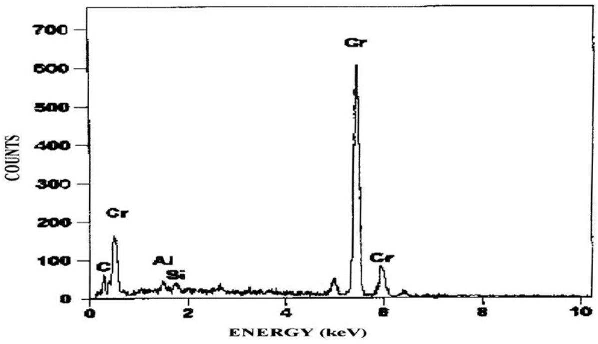

Fig. 2. Energy spectrum of electrons ejected from the chromosiliconized surface layer on C45 steel substrate obtained with use of the EDS attachment

Rys. 2. Widmo energii elektronów wyrzuconych z powierzchni warstwy chromokrzemowanej na stali C45 otrzymane przy pomocy przystawki EDS w mikroskopie skaningowym

The roughnesses of the samples were measured. The data obtained with a profilograph for C45 steel without a layer and following chromosiliconizing are presented in **Table 1** (the results are the averages of three measurements). Examples of profilograms of C45 steel with the layer and after chromosiliconizing are presented on **(Fig. 3)**.

Table 1. Stereometric parameters characterizing the topography of C45 steel without a surface layer and after chromosiliconizing process

Table 1. Parametry stereometryczne charakteryzujące topografię powierzchni stali C45 bez warstwy i po procesie chromokrzemowania

Steel C45	Ra,µm	Rz,µm	Sm,µm
Without layer	0.13 ±0.05	1.2 ±0.2	56 ±10
Chromosiliconized layer	0.80 ±0.08	6.7 ±0.6	108 ±10

a)

b)

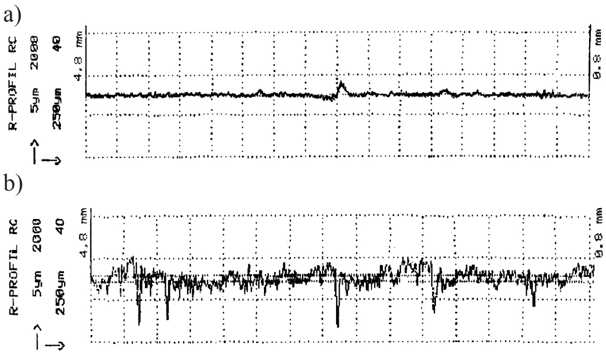

Fig. 3. An example of a profilogram of the surface roughness of C45 steel samples: a) without layer, b) with a chromosiliconized layer

Rys. 3. Wybrany profilogram chropowatości powierzchni próbek stali C45: a) bez warstwy b) z warstwą chromokrzemowaną

Observations of the microstructure were performed on transverse metallographic specimens under a light microscope revealed the presence of a bright non-etching layer with an estimated thickness of 20 µm, which was separated from the base **(Fig. 4)**, while the sections beneath were partially decarburized. The phenomenon was a result of the migration of carbon from the steel core to the layer being produced, which was caused by the lower activity of carbon due to uphill diffusion.

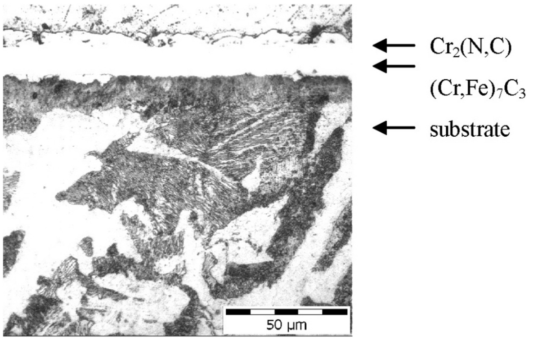

Fig. 4. Microstructure of a polished section normal to the surface of the C45 steel after chromosiliconization. Light microscope, magnification 500×, sample surface etched with 2 vol.% of HNO_3 in H_2O

Rys. 4. Mikrostruktura zgładu poprzecznego stali C45 z warstwą chromokrzemowaną. Mikroskop świetlny, powiększenie 500×, trawiono 2% obj. HNO_3 w H_2O

Using X-ray phase analysis in chromosiliconized layer, the presence of $(Cr, Fe)_7C_3$ carbide and Cr_2 (N,C) carbide nitride was detected **(Fig. 5).**

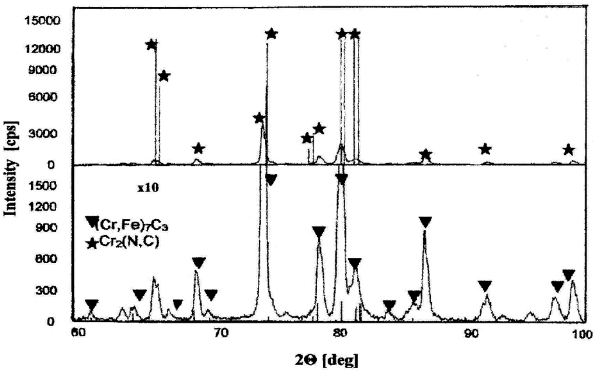

Fig. 5. X-ray diffraction pattern from the surface of a chromosiliconized layer produced on C45 steel substrate in 2Θ range 60–100 deg

Rys. 5. Dyfraktogram rentgenowski z powierzchni warstwy chromokrzemowanej wytworzonej na stali C45 w zakresie kąta 2Θ od 60 do 100 deg

The measurements of the microhardness of the chromosiliconized layer, taken in accordance with the Vickers hardness test (HV0.05) in metallographic sections perpendicular to the surface, revealed a 5.5-fold increase in relation to the core material and were set at 1338 HV0.05. The results are presented in **Table 2** (the results are the averages of three measurements).

Table 2. Hardness of C45 steel
Tabela 2. Twardość próbek ze stali C45

Steel C45	Hardness HV0.05
Without layer	240 ±20
Chromosiliconized layer	1338 ±50

The chemical composition of the chromosiliconized layer was determined in a quantitative profile analysis and indicated the following concentrations of chemical elements: Cr, Fe and Si in the surface zone (wt%): ~78% Cr, ~12% Fe and ~1% Si. The estimated chemical composition of the layer near the substrate determined by on the analysis was as follows (wt.%) ~ 45% Cr, ~45% Fe and ~ 0% Si **(Fig 6).**

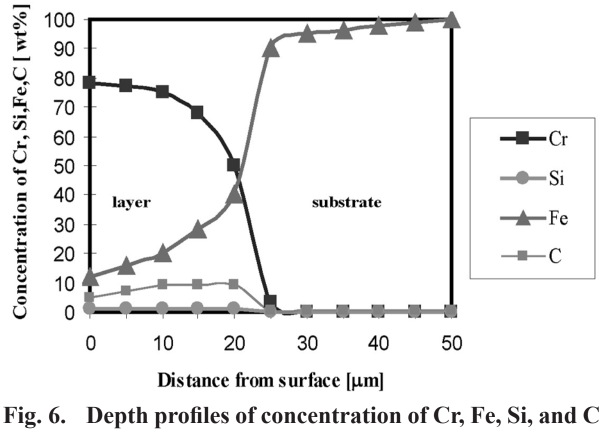

Fig. 6. Depth profiles of concentration of Cr, Fe, Si, and C atoms, down from surface of the chromosiliconized layer produced on C45 steel
Rys. 6. Profile stężeń atomów Cr, Fe, Si oraz C pod powierzchnią warstwy chromokrzemowanej wytworzonej na stali C45

In order to evaluate the structure and properties of the chromosiliconized layer and its influence on the wear resistance of C45 steel in friction processes, the following tests were conducted with three samples subjected to each. In friction resistance tests in an aqueous suspension of quartz sand, the friction elements came into contact with the aggregate surface of three rollers and counter samples. The abrasive-corrosion medium during the tests was administered at 30 drops/min. Linear wear over time was adopted as the main criterion of the chromosiliconized layers resistance to corrosion wear. At the first stage of the procedure (5 min), there was a loss of lustre but no signs of corrosion were characterized. No signs of accelerated wear were observed under the pressure of 50 MPa. The examined layers underwent uniform wear at successive stages of a pressure increase of 100 MPa. A pressure increase to 200 MPa led to accelerated and individual symptoms of uniform corrosion after 60 minutes of the test. The steel core was exposed due to surface cracking after 80 minutes under a pressure of 200 MPa. Surface pits were reported. The depth of wear marks exceeded the thickness of chromosiliconized layer. Linear wear was determined at 20.36 µm. Subject to the applied load and test time, the linear wear of C45 steel with a chromosiliconized layer was determined at the following times, (the results are the averages of three measurements):

- 3.9–12.5 µm for 50 MPa load and a test time of 10–100 min,
- 6.7–16.9 µm for 100 MPa load and a test time of 10–100 min,
- 7.9–20.3 µm for 200 MPa load and a test time of 10–80 min.

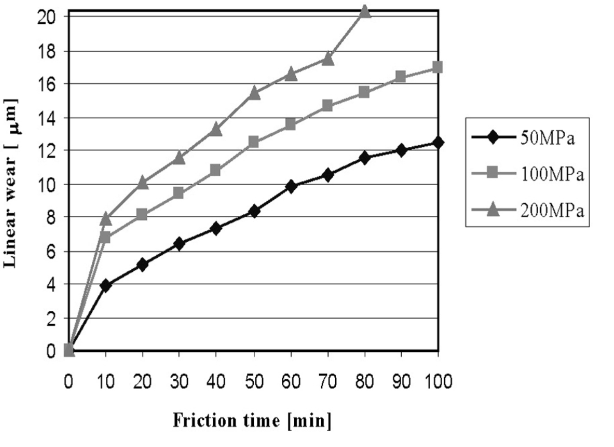

Fig. 7. Linear wear of C45 steel samples with a chromosiliconized layer as a function of durability of the abrasive -corrosive test at different load
Rys. 7. Zużycie liniowe próbek ze stali C45 z warstwą chromokrzemowaną w zależności od czasu tarcia dla różnych nacisków jednostkowych

An analysis of the linear wear of C45 steel with and without a chromosiliconized layer resulting from exposure an aqueous suspension of quartz sand for 100 minutes under 50 MPa revealed that the chromosiliconized layer showed a 1.5 increase in wear resistance under abrasive-corrosive friction conditions **(Fig. 8)**.

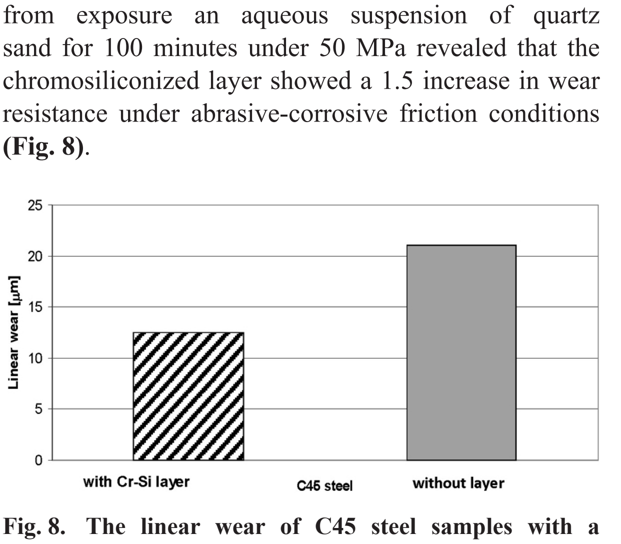

Fig. 8. The linear wear of C45 steel samples with a chromosiliconized layer and without a layer (after heat treatment) for 100 min friction time and 50 MPa unit pressure
Rys. 8. Zużycie liniowe próbek stali C45 z warstwą chromokrzemowaną i bez warstwy (po obróbce cieplnej) dla czasu tarcia 100 min przy nacisku 50 MPa

CONCLUSIONS

The wear resistance during abrasive-corrosion friction of a chromosiliconized layer prepared by the powder-pack method on C45 steel was tested in this study. The corrosive medium was an aqueous suspension of quartz sand. Our research revealed the increase in the wear resistance of the chromosiliconized layer. The results of the tests performed support the formulation of the following conclusions:

1. The powder -pack method supports the production of a chromosiliconized layer with the thickness of around 20 µm.
2. Chromosiliconizing plating caused an increase in the surface roughness of C45 steel compared to the surface roughness of steel before processing.
3. Chromosiliconizing of C45 steel supported the production of a layer composed mainly of $(Cr,Fe)_7C_3$ carbides and Cr_2 (N,C) carbide nitride.
4. The produced layer had a hardness of (1300) HV0.05 and was about 5.5 times thicker than the hardness of the substrate of C45 steel before chromosiliconization.
5. The chromosiliconized layer showed a 1.5 increase in wear resistance in aqueous suspension of quartz sand compared to the wear resistance of C45 steel without chromosiliconization.
6. The studies showed an increase in corrosion resistance in the friction process of the chromosiliconized layer in the water environment of a quartz sand suspension in relation to C45 steel without the layer.
7. The observed increase in wear resistance after chromosiliconization of C45 steel enables its application on parts of machines and devices intended for work in an abrasive-corrosive environment.

The next stage of studies will be carried out on the optimization of technological parameters of the chromosiliconization process in terms of the exploitation of the chromosiliconized layer – the substrate of C45 steel in the process of friction under corrosive conditions.

REFERENCES

1. Szczerek M., Wiśniewski M.: Tribologia i tribotechnika. Wydawnictwo Instytutu Technologii Eksploatacji, Radom 2000.
2. Solski P., Zięba S.: Zagadnienia zużycia elementów maszyn spowodowanego tarciem. PWN, Warszawa 1969.
3. Spałek J.: Problemy inżynierii smarowania maszyn w górnictwie. Monografia. Wydawnictwo Politechniki Śląskiej, Gliwice 2003.
4. Stachowiak A.: Problemy modelowania zużywania tribokorozyjnego w układach ślizgowych. Wydawnictwo Naukowe Instytutu Technologii Eksploatacji – PIB, Radom, 2012.
5. Klich A., Gospodarczyk P., Kalukiewicz A., Kotwica K., Pawlik K.: Maszyny i urządzenia dla inżynierii budownictwa podziemnego. Wyrobiska korytarzowe i szybowe w górnictwie.Wydawnictwo Śląsk, Katowice 1999.
6. Wyciszczok S.: Maszyny i urządzenia górnicze. Część 1.Wydawnictwo REA, Warszawa 2013.
7. Kasprzycka E., Bogdański B., Senatorski J., Gębski P., Więczkowski A.: Tribological properties of the hybrid layers produced in chromizing process by pack powder method and PVD treatment. Tribology 2, 2014, 79–88.
8. Smolik J., Mazurkiewicz A.: Rozwój hybrydowych technologii powierzchniowych w oparciu o praktyczne zastosowania przemysłowe. Problemy Eksploatacji 3, 2010, 105–114.
9. Kasprzycka E.: Antiwear properties of medium carbon steel with Cr/CrN type hybrid coating produced by the PVD method. Tribologia 6, 2018, 63–69.
10. Paczkowska M., Selech J.: An investigation of the influence of laser alloying of the surface layer on abrasive wear resistance of cast iron elements. Tribologia 6, 2018, 107–117.
11. Paczkowska M.: The analysis of the influence of laser heat treatment of the crankshaft journal on wear resistance of the bearing. Tribologia 2, 2016, 87–100.
12. Y. Wang, D. Liu, S. Feng, Y. Zhang, T. Ouyang, J. Suo: Preparation of tritium permeation barrier consisting of titanium by the pack cementation method. Surface & Coating Technology, 307 (2016) 271–277.
13. Bogdański B., Kasprzycka E.: Tribological characterizations of chromized carbide layers produced by the pack powder method at low pressure, Tribology 3, 2015, 9–19.
14. Mikhailin V. N., Kogan Ya. D., Nowikov A. Yu.: Diffusion chromizing and chromosiliconizing of fastenings parts. IVUZ, Mašinostroenie 1–3, 1991, 51–55.
15. Honchi M., Yamazaki T.: Chromium/silicon composite pack cementation agent and treatment using the same. Patent JP 2002129304, 2002.
16. Agarwal S., Jain A., Lal C., Ganesan V., Jain I.P.: Surface morphology and the phase formation at Cr/Si system. Applied Surface Science, 253 (10), 2007, 4721–4726.
17. Fujimura H., Nishimoto A., Akamatsu K.: Simultaneous diffusion coating of Cr and Si on stainless steel using fluoride-free activator. Journal of the Japan Institute of Metals, 77 (8), 2013, 334–338.
18. Gurvich L., Zhirnov A.: Corrosion resistance of nickel alloyed with chromium and silicon to the fuming nitric acid. Zascita Metallov 31, (3), 1995, 256–261.

19. Hoshiyama Y., Li X., Dong H., Nishimoto A.: Characterization of hot-steam oxidation tested chromosiliconized heat-resistant austenitic stainless steel. Materials Transactions, 53 (6), 2012, 1090–1093.
20. Granat K.: Wieloskładnikowe stopy Fe-C-Cr-Si odporne na zużycie przeznaczone na odlewy i warstwy napawane. Pr. Nauk. Inst. Tech. Masz. i Aut. Pol. Wroc. (86-26), 2001, 175.
21. Zhongrong G., Haixin W., Chengbing W., Lipimg W., Guangan Z.: Effect of Si content on the tribological properties of CrSi films in air and water environment. Tribology International, 79, 2014, 140–150.
22. Bauer I.: The effect of microstructure on the tribocorrosive properties of chromosiliconized layers. Physico-Chemical Mechanics of Materials 7, 2008, 293–295.
23. Bauer I.: Właściwości warstw chromokrzemowanych w procesie tribokorozji. Technical News 2010, 31, 32 (1, 2), 143–145.
24. Bauer I.: The influence of the microstructure of a chromosiliconized layer on the tribocorrosive properties exposed to mine water. Tribologia 1, 2017, 17–21.
25. PN-EN ISO 6507-1: 2007: Metallic materials-Vickers hardness test. Part 1:Test method.
26. PN-H-04302: 1983: The strength tests of metals. The friction test in 3 rollers-cone system.