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Self-tapping screw surface gas-phase cementation kinetics investigation, with EPDM washer

The kinetics of gas-phase cementation of goods – self-tapping metal screws for attachment of metal structures DIN 7982 / ISO 7050 with pointed ends from steels of SAE 1013-1018 to ASTM 510 grades was studied. The investigation was carried out by dynamic method with open volume, renewable gas phase, continuous delivery of natural gas to the flow system and its extraction from gaseous products of interaction. Chemical and phase composition of the surface coating was studied by means of low-vacuum raster electron microscope JSM 6490LV of JEOL company (Japan). A cementation of screws with washer from EPDM was examined. Experimental dependences of carbon concentration on the distance from the goods' surface for various exposures in the cementation process were received. It was found that hardness of all elements of the hardened goods is notably higher than of the hardened goods. Thread, head and drill, i.e. off set elements of the goods are subjected to the maximum hardening. It was also substantiated that there is substantial decarbonization of the surface coating at a depth of 0.2–0.4 mm depending on the exposure time in the process of traditional hardening heat of the self-tapping screws. This explains lower values of the hardness of the thread, head and drill, the protruding elements of the goods. In comparison with the hardening, the cementation allows raise the hardness of the surface to 64–73 HRC (hardness in Rockwell scale C). Ways to raise corrosion resistance using the cementation were aimed. Among goods with protective coating (galvanized, oxidized) at the initial stage, the case-hardened goods have the lowest weight loss and during the whole time of the experiment the weight loss is substantially lower than the standard values.

Keywords: cementation, protective coating, corrosion resistance, self-tapping screw.

Badania kinetyki cementacji z fazy gazowej powierzchni wkrętów samogwintujących z podkładką z EPDM

Zbadano kinetykę cementacji z fazy gazowej wkrętów samogwintujących do mocowania struktur metalowych DIN 7982/ISO 7050 z ostrymi końcami ze stali gatunków SAE 1013-1018 do ASTM 510. Niniejsze badanie zostało przeprowadzone za pomocą metody dynamicznej z otwartą objętościowo, odnawialną fazą gazową, ciągłym podawaniem naturalnego gazu do systemu przepływowego oraz jego ekstrakcją z gazowych produktów interakcji. Skład chemiczny i fazowy powłoki powierzchniowej był badany przy pomocy niskopróżniowego, rastrowego mikroskopu elektronowego JSM 6490LV firmy JEOL (Japonia). Zbadano także możliwość wykorzystania cementowanych śrub z nakrętkami, mającymi podkładki uszczelniające oparte na EPDM. Otrzymano doświadczalnie zależność stężenia węgla od odległości od powierzchni wyrobów dla różnych ekspozycji w procesie cementowania. Stwierdzono, że twardość wszystkich elementów wyrobów hartowanych jest istotnie wyższa niż wyrobów hartowanych. Gwint, główka i świderek gwintujący, tj. robocze elementy wyrobów poddawano maksymalnemu hartowaniu. Potwierdzono, że w procesie tradycyjnego hartowania cieplnego wkrętów samogwintujących zachodzi znaczna dekarbonizacja powłoki powierzchniowej na głębokość 0,2–0,4 mm, zależnie od czasu ekspozycji. To wyjaśnia niższe wartości twardości gwintu, główki i świdarka gwintującego, zewnętrznych elementów tych wyrobów. W porównaniu z hartowaniem, cementowanie pozwala na podwyższenie twardości powierzchni do 64–73 HRC. Zwrócono uwagę na sposoby podniesienia odporności na korozję przy użyciu cementowania. Spośród wyrobów mających powłokę ochronną (galwanizowanych, oksydowanych) na wstępnym etapie, wyroby hartowane mają najmniejszą utratę masy, a podczas całego eksperymentu utrata masy jest istotnie niższa od standardowych wartości.

Słowa kluczowe: cementacja, powłoka antykorozyjna, odporność na korozję, wkręty samogwintujące.

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1. Introduction

Modern construction materials, used in various branches such as building, metal-processing, machine-building, are continuously developed in the direction of improvement of mechanical properties. Indicators of surface hardness, ultimate resistance are increased. All these require proper improvement of mechanical properties of attaching goods, which during assembly mechanically deform binding elements of a construction. Studied in this paper self-tapping metal screws for attachment of metal structures DIN 7982/ISO 7050 with pointed ends (Figure 1) refer to such goods [1]. According to the available technology for production of the self-tapping screws, the final stage is hardening with the following coating for protection against corrosion (galvanized, oxide, phosphate) [2]. The important quality index of the attaching goods is maintenance of the hardness of no less than 55 units by Rockwell HRC (Rockwell scale C). [1]. One of the problems of the hardening stage is that when heating the goods before the hardening, decarbonization of the metal's thread occurs [3]. By means of that, the hardness of the thread grows smaller and the joint efficiency reduces in whole. It is offered here to improve the technology by introduction of gaseous cementation of the attaching goods mentioned above. By means of formation of a carbide layer of the goods' surface it is possible to attain high hardness of the surface and protection against the corrosion. Thus, the protective finish stage is eliminated. This allows cut down expenses for the production. However, it remains to be seen to what extent the case-hardened goods will be protected against the corrosion in comparison with other coatings – phosphate, oxide, etc.

2. Materials and methods

The self-tapping metal screws for attachment of metal structures DIN 7982/ISO 7050 with pointed ends from steels of SAE 1013-1018 to ASTM 510 grades were used as the subject of the research (Figure 1) [1]. Chemical

composition of the steels is presented in Table 1. Natural gas was used as the bond. These goods were cemented in equilibrium conditions at a temperature of 940 °C on a unit (Figure 2). The investigation was carried out by dynamic method with open volume, renewable gas phase, continuous delivery of natural gas to the flow system and its extraction from gaseous products of interaction [2]. The unit involved automatic maintenance of constant pressure in the system by a pressure gauge 4 and pressure compensator 5. The gauge on the basis of a manometer has screen on a hand, closing photo resistance from the light flux 2. When changing the pressure one photo resistance is opened and a reversible motor 6 moves lever of the pressure compensator. Change in the volume, occurring before equilibration by the reversible motor 7, is registered by a potentiometer. Attainment of the constant dew point temperature of the gaseous mixture fixed by a moisture meter is also approximation indicator of the system to the equilibrium state. When attaining the dew point, at first dark moisture meter mirror begins to be radiant from the source 16. The photo resistance signal 12 is converted in a measuring circuit 13, 14. In order to avoid condensation of the moisture, all nodes of the apparatus, through which gas circulates, are arranged inside a thermostat, heated up to 130 °C. Circulation of the gaseous mixture is carried out by a circulating pump of membranous type. Analysis of the gas is carried out on a gas mass-spectrometer GCMS-QP 2010Ultra (Shimadzu, Japan). The goods, received in a result of the cementation, were hardened. The hardening was carried out from the cementation temperature into water without the following supply. In the part of chemical and phase composition of the surface coating the goods were studied by means of a low-vacuum raster electron microscope JSM 6490LV of JEOL company (Japan) with a system of energy-dispersive microanalysis INCA Energy-350 (OXFORD Instruments, Great Britain) and system of polycrystalline object structural analysis HKL Basis (OXFORD Instruments, Great Britain). A micro hardness of the case-hardened screws was also defined using micro indenters and metallographic microscope on

the surfaces of thread, rod, head, drill, recess. Besides, tests for resistance to the climatic external factors of corrosion effect by ISO 9227:2012, NEQ were carried out on the experimental unit (Figure 3) [5]. All the investigations were carried out using control samples gained by a simple hardening without any cementation, with applied galvanized coating and without it and untreated goods.

The corrosion resistance test of the case-hardened and control goods was carried out in a neutral salt spray [5]. The samples were cleaned directly before the test by acetone treating in ultrasonic bath. After the cleaning the goods were additionally rinsed by acetone and dried. Mass of the goods within the accuracy of 0.1 g and total surface area were defined. To define the surface area, volume of the goods was defined by the method

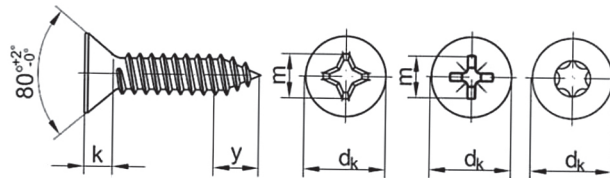


Fig. 1. Scheme of the self-tapping metal screw DIN 7982 / ISO 7050 with the pointed end [1]

Rys. 1. Schemat metalowego wkrętu samogwintującego DIN 7982 / ISO 7050 z ostrym końcem [1]

Table 1. Chemical composition of the steels by ASTM 510 [5]

Tabela 1. Skład chemiczny stali wg normy ASTM 510 [5]

Steel grade	Ladle analysis		In the finished steel	
	Carbon content, %	Manganese content, %	Carbon content, %	Manganese content, %
SAE 1013	0.11–0.16	0.50–0.80	0.09–0.18	0.47–0.83
SAE 1015	0.13–0.18	0.30–0.60	0.11–0.20	0.27–0.63
SAE 1016	0.13–0.18	0.60–0.90	0.11–0.20	0.57–0.93
SAE 1017	0.15–0.20	0.30–0.60	0.13–0.22	0.27–0.63
SAE 1018	0.15–0.20	0.60–0.90	0.13–0.22	0.57–0.93

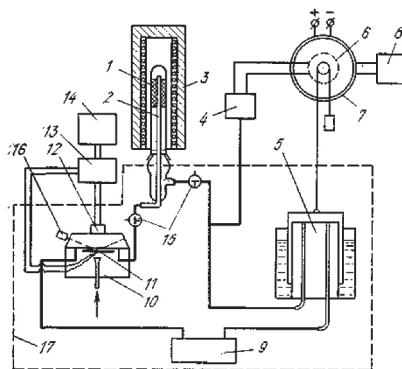


Fig. 2. Scheme of the experimental unit for investigation of the gas-phase cementation: 1 – case-hardened goods, 2 – reaction tube, 3 – furnace, 4 – pressure gauge, 5 – pressure compensator, 6 – reversible motor, 7 – rheochord of the reversible motor, 8 – volume change recorder, 9 – circulating pump, 10 – moisture meter, 11 – thermal couple, 12 – photo resistance, 13 – electronic control of the moisture meter, 14 – water recorder, 15 – valves, 16 – light source, 17 – thermostat

Rys. 2. Schemat eksperymentalnego stanowiska do badania cementacji z fazy gazowej: 1 – wyroby hartowane, 2 – rurka reakcyjna, 3 – piec, 4 – manometr, 5 – kompensator ciśnienia, 6 – silnik nawrotny, 7 – reostat silnika nawrotnego, 8 – rejestrator zmiany objętości, 9 – pompa cyrkulacyjna, 10 – miernik wilgotności, 11 – termoelement, 12 – fotorezystor, 13 – elektroniczny kontroler miernika wilgotności, 14 – rejestrator wody, 15 – zawory, 16 – źródło światła, 17 – termostat

of displacement at submersion. Further, the goods were presented as a cylinder with the height equal to the length of a rod and the mean diameter was calculated. Area of the surface was calculated by the definite geometrical sizes. The goods were placed in the center of each of the experimental unit quadrants (Figure 3). The upper part of the chamber was prepared in such a way that agglomerating on it drops of the solution couldn't flow down the goods. Air to the chamber was intaken under a pressure of 110 kPa through a lavsan filter. Base for the goods was made from polyvinylchloride. The lower edge of the goods was on a level with the salt spray collector. Duration of the test consisted 48 hours and more.

The test was carried out by continuous spraying of 5% salt spray in water. Dispersion degree of the spray was determined on the basis of picking rate in the solution collector 0.2 ml/hour. Weight loss per unit area was defined on the morrow of the test. To this end, the goods were extracted from the chamber and

corrosion products were removed by 50% solution of hydrochloric acid etching with addition of a retarding agent – hexamethylenetriamine. After the etching, the goods were washed with water, acetone and dried. The goods were weighted within the accuracy of 0.1 g and weight loss was calculated in grams per square meter.

The test was carried out on single product and on threaded screw with EPDM washer after thermal treatment as well.

Thermal treatment of washers is conducted in same conditions as screws. EPDM is modern elastomer which is used in industry more than 30 years. This material is used in construction industry instead of neoprene and as a sealing material. The primary reason of changing the use of neoprene into EPDM is its' higher harsh weather conditions resistance.

Technical parameters of the material used for testing are shown in Table 2.

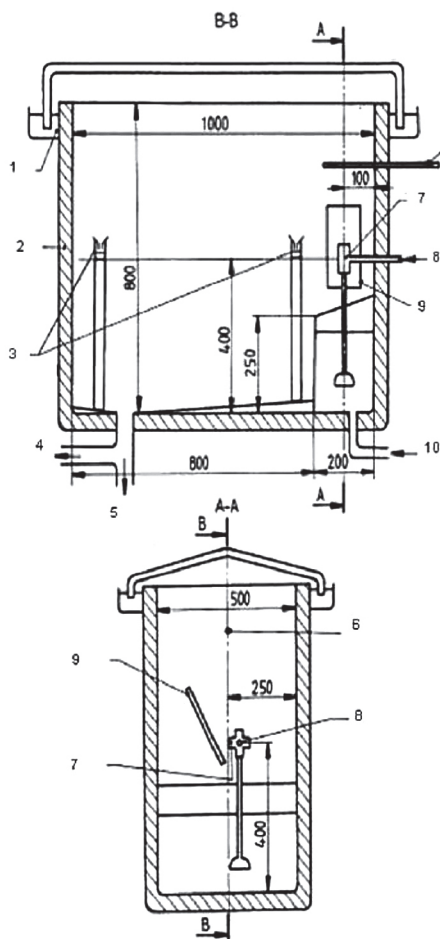


Fig. 3. Scheme of the spray chamber for studying of the corrosion resistance in the neutral salt spray of the self-tapping screws: 1 – hydraulic lock, 2 – heating heat-insulation wall, 3 – collectors, 4 – air tap, 5 – water export, 6 – thermometer, 7 – spray port, 9 – deflector plate, 10 – salt solution supply

Rys. 3. Schemat komory natryskowej do badania odporności korozyjnej wkrętów samogwintujących w obojętnym roztworze solanki: 1 – uszczelka hydrauliczna, 2 – podgrzewana ścianka termoizolacyjna, 3 – kolektory, 4 – upust powietrza, 5 – odpływ wody, 6 – termometr, 7 – otwór natryskowy, 9 – płytka odchylająca, 10 – podawanie roztworu solanki

EPDM comparing to neoprene is much better considering following aspects:

- resistance to ozone;
- resistance to UV rays;
- resistance to weather conditions;
- resistance to water;
- resistance to low temperature;
- resistance to ageing;
- flexibility;
- low specific weight.

In initial stage of production, EPDM has similar physical properties comparing to neoprene.

EPDM physical properties:

Temperature resistance	from -40 °C to + 120 °C
Tension resistance	high
Resistance to hitting	high
Resistance to grinding	high
Resistance to deformation	very high
Resistance to water	very high
Resistance to acids	very high
Resistance to chemical compounds	very high
Resistance to weather conditions	very high
Resistance to action of fats	very high
Resistance to high temperatures	very high
Resistance to UV	very high

protected by galvanized coating and untreated control samples [6, 7, 8]. In Figure 4 dependence graphs of the carbon concentration on different distance from the goods surface at different duration of the cementation are presented. In Figure 5 microphotograph of the case-hardened goods' surface at the exposure time of 12 minutes is shown.

As is seen from the electronic microphotograph (Figure 5), surface of the goods is saturated by the cementite in consequence of that it is possible to expect increase in the surface hardness. On the microphotograph, white grains of the cementite on the surface have small dark inclusions of ferrite. With deepening inside the goods, amount of the dark inclusion increases, alternating with the grains of perlite. It is seen from the Fig. 4a that the carbon concentration near the surface substantially increases with increase in the exposure time. At the initial section of the surface layer, the metal is presented as hypereutectoid steel. In this connection, before hardening of the goods it is necessary to cool down the goods till a temperature of $A_1 + 30$ °C, i.e. 770 °C to attain fine grain microstructure [2]. Distribution of the carbon in the horizontal projection on different sections of the goods remains constant both for the cementation case and hardening heat in the oxidizing conditions. Depth of the layer, to which

Table 2. Technical characteristics EPDM washers according to norms

Tabela 2. Techniczne charakterystyki podkładek EPDM, zgodne z normami

USA norms	EPDM
Resistance to pulling ASTM 412	11,9 N/mm ²
Shore hardness acc. to ASTM D 2280	70–78
Flexibility ASTM D 412 300%	700 N/cm ²
Dislayering strenght ASTM D 624	294 N/cm
Deformation strenght	25 %
Temperatures limits	from -40 °C to +120 °C
Max short time temperature	+ 150 °C
Resistance to atmosphere effects	very high

3. Results and discussion

The cementation time was varied in the course of the investigations: 1, 3, 5, 12 minutes. Change in the carbon (cementite) concentration on different distance from the surface was studied in the gained case-hardened goods. Values of the micro hardness on different sections of the goods for different variants of thermochemical treatment were also studied. Change in the corrosion rate in the neutral salt spray in time was also studied. The results were compared with similar indicators of the goods after thermal treatment in an oxidizing atmosphere by traditional technology for production of the screws,

the hypereutectoid steel microstructure is spread, also depends on the cementation time and consists 0.03; 0.6; 0.15; 0.22 mm for the cementation time: 1, 3, 5, 12 minutes respectively. Growth rate of the layer with the hypereutectoid structure in the cementation process substantially decreases with increase in the exposure time from 20 minutes on 1 mm in the 1st minute, to 40 minutes on 1 mm to the 12th minute. However, the rate is multiply higher in comparison with the traditional cementation, as in our case the process was carried out at the supercritical pressure of the gaseous reagent and using of the gaseous activator of the process, forming active complexes on the surface, from which the cementite grain growth occurred.

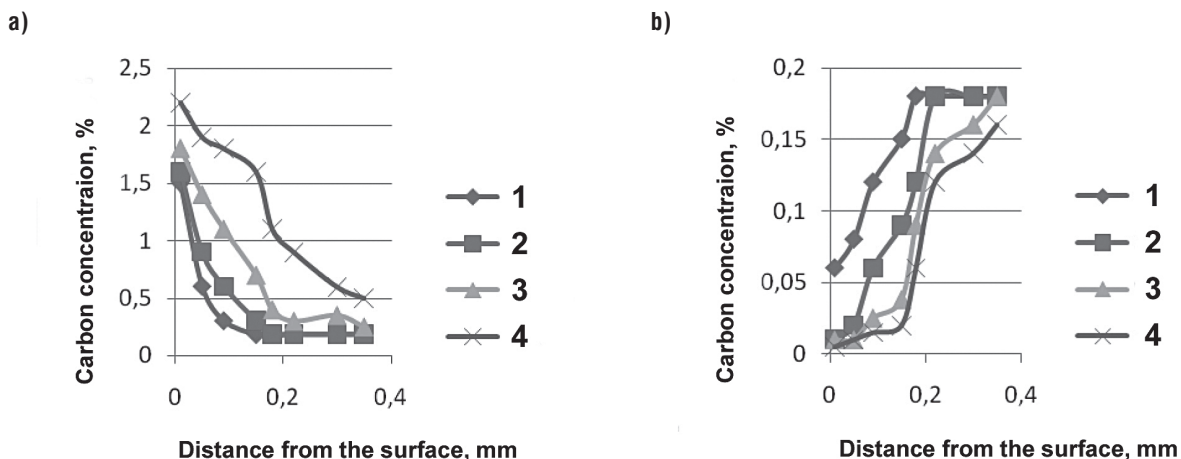


Fig. 4. Dependence of the carbon concentration on the distance from the goods' surface for different exposure times: a) in the cementation process, b) in the heating under the hardening in the oxidizing atmosphere; exposure time: 1 – 1 minute, 2 – 3 minutes, 3 – 5 minutes, 4 – 12 minutes

Rys. 4. Zależność zawartości węgla od odległości od powierzchni wyrobów przy różnych czasach ekspozycji: a) w procesie cementacji, b) podczas podgrzewania, pod hartowaniem w atmosferze oksydacyjnej; czas ekspozycji: 1 – 1 minuta, 2 – 3 minuty, 3 – 5 minut, 4 – 12 minut

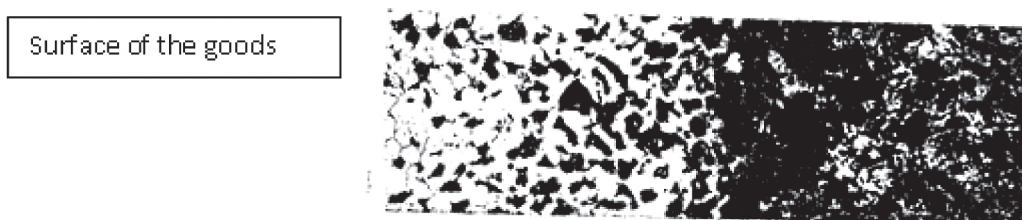


Fig. 5. Microphotograph of the surface case-hardened layer of the self-tapping screw
Rys. 5. Mikrofotografia warstwy hartowanej powierzchni wkręta samogwintującego

Figure 4b shows that substantial decarbonization of the surface layer on a depth of 0.2–0.4 mm depending on the exposure time occurs in the traditional hardening heat of the self-tapping screws. Indeed, such phenomenon may result in the loss of the surface hardness and inductile deformation of the screw thread when twisting-in the screw. In this case,

the joint efficiency will be substantially reduced. For the partial compensation of the surface layer hardness loss, correction of the hardening temperature to a higher extent providing requirement $A_3 + (30-50)^\circ\text{C}$ for the hypereutectoid steels was made [2].

Table 3 presents data for studying of the goods' micro hardness on different sections of the surface.

Table 3. Micro hardness by Rockwell HRC of different sections of the self-tapping screws' surfaces
Tabela 3. Mikrotwardość wg Rockwella HRC różnych sekcji powierzchni wkrętów samogwintujących

Thermal treatment way	Exposure time, minutes	Thread	Rod	Head	Drill	Recess
Cementation $A_1 + (30-50)^\circ\text{C}$, hardening	1	52	56	61	51	51
	3	64	61	63	56	59
	5	69	64	70	69	61
	12	71	68	73	72	64
Heating $A_3 + (30-50)^\circ\text{C}$ in the oxidizing conditions, hardening	1	45	49	49	41	52
	3	46	53	51	44	53
	5	46	54	52	45	53
	12	47	51	48	43	52

As is seen from these data, the hardness of all elements of the case-hardened goods is substantially higher than that of the hardened goods. The thread, head and drill, i.e. off set elements of the goods are subjected to the maximum hardening. This can be explained by less thickness of the laminar film at the flowing of the goods by the gas flow at the protruding elements [8]. The gas flow rate, and therefore, the diffusion rate is substantially decreased in valleys of the rod and recess surfaces. Although analysis of the carbon concentration has not showed differences by the whole goods surface, but differences probably presented. They exerted their influence on the distribution of the hardness values on the goods' surface. Values of the goods hardness increase with increase in the exposure time. However, at the time of 12 minutes, the growth rate substantially decreases. It tells that the further increase in the cementation time is inexpedient.

For the heating in the oxidizing conditions and the following hardening it should be noted that the protruding elements such as thread, head and drill vice-versa become hard. This fact can be explained by increased oxidation of the carbon in these elements by means of low diffusion resistance. Influence of the exposure time on the hardness is controversial. When increasing the time of curing, even abating is possible. This is explained by substantial development of the oxidizing processes in the metal structure.

Figure 6 shows the results of the studies of the goods corrosion resistance with different types of the surface preparation.

As is seen from the results of the studies of corrosion kinetics of the goods from the steel of SAE 1013-1018 to ASTM 510 grades without any thermal treatment and anticorrosive coating the weight loss at 24 and 48 hours' exposure shows values matching the standard 40 and 80 g/m² respectively. However, further, by the reason of heavy increase in the active corrosion centers the rate substantially increases, and dependence takes the exponential form. Among the goods with protective coating at the initial stage, the case-hardened goods have the least loss in the weight and during the whole time of the experiment the loss in the weight is substantially lower the standard values. It should be noted that beginning from the 48th hour of the investigation, in the brake field of the corrosion rate among all surfaces, the case-hardened goods' weight loss rate becomes the highest. The case-hardened goods' weight loss rate is two times higher than for the galvanized and oxidized goods. At that, the absolute values are kept on the low levels that eliminates risk of excess of the standard values in the time limits inaccessible for the studies.

4. Conclusion

As is seen from the results of the carried out investigations, the self-tapping screw cementation is promising direction of the thermochemical treatment has not used earlier for these purposes. In comparison with the hardening, the cementation allows increase hardness of the surface to 64–73 HRC depending on

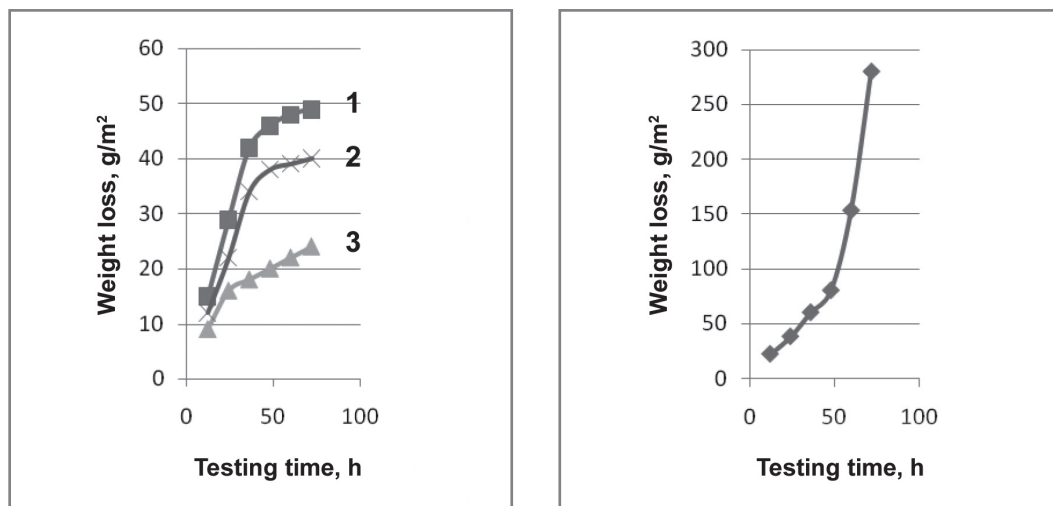


Fig. 6. Corrosion resistance of the goods with different way of treatment: a) with protective coating: 1 – galvanized, 2 – oxidized, 3 – cementation; b) without any thermal and anticorrosive coating

Rys. 6. Odporność na korozję wyrobów przy różnej metodzie obróbki: a) z powłoką antykorozyjną: 1 – wyroby ocynkowane, 2 – wyroby oksydowane, 3 – wyroby cementowane; b) wyroby bez powłoki antykorozyjnej i obróbki cieplnej

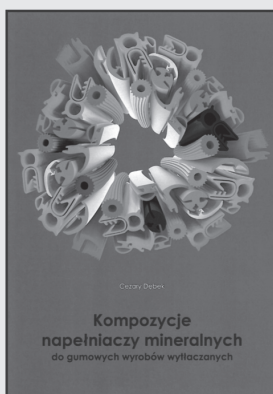
the goods' elements. Indicator of the goods' corrosion resistance in comparison with the goods with galvanized and oxidized coating at the initial stage of exploitation is also improved. However, there are risks connected with insignificantly high corrosion rate with increase in the test time. This requires the following investigation of this problem.

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„Kompozycje napelniaczy mineralnych do gumowych wyrobów wytłaczanych”



Monografia kierowana jest do szerokiego grona odbiorców: specjalistów zajmujących się technologią gumy, zwłaszcza projektowaniem i modyfikowaniem mieszanek kauczukowych, a także do studentów i nauczycieli akademickich kierunków technicznych związanych ze technologią elastomerów.

W pracy przedstawiono napelniacze mineralne powszechnie używane w przemyśle gumowym: krzemionkę, kaolin, talk, kredę, węgiel magnezu, siarczan baru i inne. Szczególną uwagę poświęcono minerałom będącym potencjalnymi, nowymi surowcami w technologii gumy: sepiolit, pirofilit i montmorylonit.

Na podstawie wymienionych minerałów opracowano nowe napelniacze (kompozycje napelniaczy). Zastosowanie tych napelniaczy pozwala na poprawę aspektów technologicznych otrzymywania mieszanek kauczukowych, ich reologii w połączeniu z zachowaniem bądź poprawą właściwości fizykochemicznych wulkanizatów, szczególnie otrzymywanych na drodze wytłaczania, w porównaniu z mieszanekami zawierającymi tradycyjne napelniacze, zwłaszcza krzemionkę.

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