



Corrosion resistance of neodymium composite materials reinforced with metal powders

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Received 21.10.2012; published in revised form 01.12.2012

ABSTRACT

Purpose: The goal of the study is to investigate the corrosion resistance of hard magnetic composite materials Nd-Fe-B with 5%, 10% and 15% by weight of iron powder, casting copper alloy with tin CuSn10, steel corrosion-resistant X2CrNiMo17-12-2 and Epidian100 (2.5% by mass) as a binder in a humid environment at 40°C and 5% NaCl solution at 35°C and to determine their current-voltage characteristics.

Design/methodology/approach: The investigations of corrosion resistance of hard magnetic composite materials in climate chambers were carried out: test 1 (temperature 40°C, relative humidity 93%, duration 96 h), test 2 (temperature 35°C, 5% NaCl solution, duration 6 h). Pitting corrosion were made in an environment of 5% NaCl solution at 35°C.

Findings: The results of corrosion tests allows to determinate that the best corrosion show composite materials with addition of 15% of CuSn10 or X2CrNiMo17-12-2.

Practical implications: Composite materials Nd-Fe-B – polymer matrix can greatly expand the application possibilities of hard magnetic materials however further examination to obtain materials with improved properties are still needed.

Originality/value: Results show corrosion resistance of Nd-Fe-B - polymer matrix composite materials determined by different methods. Results are the base for further investigations of the impact of corrosion environment on the magnetic properties of such composite materials.

Keywords: Composites, Neodymium magnets, Epoxy resin, Corrosion resistance, Resistivity

Reference to this paper should be given in the following way:

M. Dziekońska, A. Ziębowicz, B. Ziębowicz, L.A. Dobrzański, Corrosion resistance of neodymium composite materials reinforced with metal powders, Archives of Materials Science and Engineering 58/2 (2012) 137-145.

PROPERTIES

1. Introduction

The era of modern hard magnetic materials containing metals of the rare earth group started in the 70s of the twentieth century [A, B, C, D, E]. The basis of these materials are compounds of neodymium and samarium with transition metals - iron and cobalt. The starting material for magnets manufacturing are intermetallic compounds $\text{Nd}_2\text{Fe}_{14}\text{B}$, SmCo_5 , $\text{Sm}_2\text{Co}_{17}$ [6,7]. Hard magnetic materials based on rare-earth metals have a high Curie temperature, large saturation anisotropy and saturation induction. High coercive force comes from the coupling of magnetic moments of rare earth metal and transition metal sub networks with crystal lattice of magnetically hard $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase. High enough spontaneous magnetization show phases containing cobalt or iron and light lanthanides Ce, Pr, Nd, Sm, La, and Y [6]. While a heavy lanthanides Gd, Tb, Dy cause antiparallel moments and their coupling phase with Co and Fe are characterized by low magnetization, which is an obstacle to their use for the production of permanent magnets [6,7].

Magnets Nd-Fe-B are a new generation of hard magnetic materials. They appeared on the world market in the 80s of the twentieth century, displacing the production of magnets Sm-Co, which previously accounted for more than 20% of the global market of hard magnetic materials [8]. Very good magnetic properties of three-phase compounds of rare earth elements with transition metals, allows improving the quality of applied magnetic systems and their miniaturization [9], which means that they are applied in new areas of technology. They are characterized by high saturation magnetization $J_s = 1.6 \text{ T}$ and a large axial magnetic anisotropy $H_A = 5.4 \text{ MA/m}$ [6]. The main technology of producing this type of magnets is a powder metallurgy and bonding them into composite materials with polymers [F, G, H, I, J]. These alloys among the basic components of the phase elements often contain additives, which can consciously design the magnetic properties and improve the technological properties [15].

The latest and most attractive hard magnetic materials in recent years become a magnet nanocrystalline Nd-Fe-B composed of grains which size at least one direction not exceed 100 nm [16]. Nanocrystalline magnets are divided into monophasic and biphasic. Magnets are made of single-phase intermetallic phase $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnetically hard, which grains can interact only magnetostatically, as in the case of magnets with microcrystalline structure. Phase grains are magnetically isolated by paramagnetic layer. The biphasic $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnets/ Fea , also known as nanocomposites include in its microstructure nanometric crystals of $\text{Nd}_2\text{Fe}_{14}\text{B}$ magnetically hard phase, arranged in a matrix of soft magnetic phase Fea and Fe_3B . The appropriate structure of nanocomposites with a grain size of 10 to 30 nm is obtained in the process of annealing of amorphous strip. Nanocrystalline hard magnetic materials are currently the most attractive group of materials in scientific and application field. While the anisotropic nanocrystalline materials could become the most popular magnets in twenty-first century [6,17].

Permanent magnets Nd-Fe-B, produced from compounds of elements from a group of rare earth and transition metal, have a very good magnetic properties and low resistance to corrosion in environments, as well as in conditions of their production. High susceptibility of neodymium magnets to the aggressive corrosive

environment reduces the reliability of the equipment, in which magnets work and significantly reduces their applications area [7,9, 18-22]. Magnets Nd-Fe-B corrode very easily due to the presence in their chemical composition high metal content of the rare earth group. Moreover, the cause of low resistance to corrosion of permanent magnets is their multiphase structure with different chemical activity, which in turn leads to selective corrosion [20], consisting in the destruction of one or more phases of the alloy at a faster rate than the destruction of the matrix material [23]. Susceptibility to corrosion of various phases increases by sequence: ferromagnetic $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase - the phase of neodymium Nd_4Fe that fills the border areas between grains of hard magnetic phase - the phase of boron NdFe_4B_4 [9,19, 24-28].

Neodymium rich phase is the most electrochemically active phase, which favours its oxidation, thus reducing the corrosion resistance of the material. The mechanism of corrosion of magnets based on rare-earth metals comes from phases contact and consists of a privileged phase oxidation of neodymium located between the ferromagnetic phase grains, which results in dissolution of the border areas in the phase $\text{Nd}_2\text{Fe}_{14}\text{B}$ grains. Furthermore, the selective oxidation of the phase boundary Nd_4Fe is promoted by high density of crystal defects in this phase [29]. As a result of corrosion process the loss of whole grain agglomerates ferromagnetic phase and the surface of the magnet appeared (so-called corrosion of the agglomerates) [30]. Furthermore, the corrosion results in decreasing of magnetic properties [9,20,25, 29-32].

The mechanism of corrosion of neodymium magnets, and the rate of their destruction strongly depends on the type of environments in which they are located. In neutral aqueous environments on the surface of the magnet is formed passive layer consisting of oxides, which protects the magnetic material from further corrosion. Lanthanide oxides are relatively well soluble in weak acid solutions, and the dependence of corrosion rate in the acid is very strong with the pH environment. Furthermore, with increasing of neodymium content in the chemical composition of the magnet, magnetically hard material exhibits a greater tendency to passivation of the surface layers formed mainly of hydrated oxides of neodymium [18].

Materials with low neodymium content shows discontinuity of passive layers, caused by the porosity of the material, so they are less resistant to corrosion. The more magnet surface area is destroyed and the loss of coherence of the corrosion products is easier [30]. In addition, the presence of pure iron precipitates in the structure of neodymium magnets favours the formation of local corrosion microcells, causing decreasing of corrosion resistance [18,30]. In the acid environment the reaction results in the selective etching of neodymium-rich phase and a phase rich in boron, and then to separate the grain from the ferromagnetic phase from corrosive surface of the magnet. In addition, the phenomenon is accompanied by the extraction of hydrogen, which form the hydrides that damages the surfaced [24,29,30,33]. Thus, even a momentary acidification of the atmosphere leads to the degradation of the magnet surface [9,20], and with increasing acidity of the environment increases the corrosion rate [25]. In the range of $\text{pH} < 3$ comes to the catastrophic destruction of the magnets Nd-Fe-B surface and the process of corrosion occurs with the separation of grains and is actively controlled. In weakly acid solutions with a pH value within the limits of 3 to 5 the rate

of corrosion depends on the speed of transport of H⁺ ions, and neutral phase rich in neodymium is covered with compact oxide layer, which prevents its digestion, and thus protects the magnet against corrosion. Poor resistance to the aggressive agent magnet surface destroying results in pulverization in operating conditions (the disintegration of the material to a powder), the lack of passive, mechanical destruction due hydrogenation or loss of cohesion with the surface of the magnet [9,18,20,24,25,30,32,34].

Grain size, depending on the method of manufacturing neodymium magnets, also has a significant impact on the mechanism and rate of corrosion of these materials [18]. The smaller the grain of magnetic material, the greater its tendency to passivation. In addition, magnets of manometer grain corrode slower, and their corrosion resistance is higher in the sulphate environment, compared with magnets micrometric grains. Effect of grain size on the corrosion of magnets decreases with increasing pH corrosive medium.

The goal of this study is to investigate the corrosion resistance of hard magnetic composite materials Nd-Fe-B with 5%, 10% and 15% by weight of iron powder, casting copper alloy with tin CuSn10, alloy steel corrosion-resistant X2CrNiMo17-12-2 and Epidian100 (2.5% by mass) as a binder in a humid environment at 40°C and 5% NaCl solution at 35°C and to determine their current-voltage characteristics. Furthermore the structure of hard magnetic composite materials with the addition of these powders and damages of the magnet's surface after corrosion tests were evaluated.

2. Material for research

The experiments were made with the polymer matrix hard magnetic composite materials reinforced with nanostructured particles of the powdered rapid quenched Nd-Fe-B strip MQP-B type made by Magnequench (Nd_{14.8}Fe₇₆Co_{4.95}B_{4.25}). Powders of metals and their alloys: iron, CuSn10 casting alloy of copper with tin, and of the X2CrNiMo17-12-2 high-alloy steel (5, 10, 15% wt.) were added to the composite material. The heat-hardening epoxy resin was used as the matrix (2.5% wt.). Zinc stearate (0.2% wt.) was used to ensure slip during compaction and pulling the test pieces out of the die. To obtain the composite materials the Nd-Fe-B powder was mixed with

the epoxy resin powder. The composite materials with addition of metal and metal alloys powders were fabricated by mixing the Nd-Fe-B powder in a mechanical mixer with the addition powder, and next by mixing them with the epoxy resin powder (Table 1).

Portions of the matrix and slip agent powders, compacting pressure, as well as the temperature and curing time of the polymer material decide the technological conditions of magnets manufacturing. The following compacting process parameters were used: unilateral uniaxial compaction, room temperature, pressure 800-900 MPa. The polymer matrix of the composite materials was cured at the temperature of 180°C for 2 hours after compacting.

3. Research methods

Observations of morphology of powders used iron, CuSn10 casting alloy of copper with tin, and of the X2CrNiMo17-12-2 high-alloy steel and the observation of surface topography of composite materials were made DSM 940 OPTON in scanning electron microscope at the maximum magnification of 100 and 200x using the secondary electron detection at the 20 kV accelerating voltage.

The investigations of corrosion resistance of hard magnetic composite materials in climate chambers were carried out in accordance with standards: IEC 50 (221) + A1: 1999 for test 1 (temperature 40°C, relative humidity 93%, duration 96h) and PN-EN ISO 8040:1999 for test 2 (temperature 35°C, 5% NaCl solution, duration 6 h).

Samples surface before being placed in a climatic chamber were cleaned by grinding and degreasing in ethanol. In order to characterise damages of the magnet surface, microscopic observations were carried out using LEICA MEF4A optical microscope. The Computer Assisted Image Analysis was then employed to evaluate the impact of corrosion on the surface during the test and after the test has finished. A series of observation of the magnet surfaces were made after the periods of time:

- Test No. 1 - 24h, 48h, 96h, respectively,
- Test No. 2 - 1h, 2h, 6h, respectively.

Table 1.
Chemical composition of composite materials

No.	Composite material	Reinforcement of composite materials		Matrix of composite	Density [g/cm ³]
		Mass portion of	Mass portion of	Mass portion of matrix	
1	Nd-Fe-B	100	0		5.58
2	Nd-Fe-B/5%Fe	95	5		5.82
3	Nd-Fe-B/10%Fe	90	10		5.85
4	Nd-Fe-B/15%Fe	85	15		5.88
5	Nd-Fe-B/5%CuSn10	95	5	2.5	5.76
6	Nd-Fe-B/10% CuSn10	90	10		5.94
7	Nd-Fe-B/15% CuSn10	85	15		6.03
8	Nd-Fe-B/5%X2CrNiMo17-12-2	95	5		5.83
9	Nd-Fe-B/10% X2CrNiMo17-12-2	90	10		5.86
10	Nd-Fe-B/15% X2CrNiMo17-12-2	85	15		5.98

Corrosion tests by gravimetric method involving the exposure of samples in corrosive environments, and then after a certain time of corrosion test, measuring the masses of these samples and determining their weight changes were also carried out. To measure the mass changes was applied an analytical weight with accuracy of 0.0001. Pitting corrosion evaluation in an environment of 5% NaCl solution at 35°C, consists in determining the electrochemical anodic polarization curves, showing the potential of the electrode and correlation of current flow through the interface material - solution. The surfaces of samples subjected to the corrosion test were initially smoothed by grinding and cleaned in an ultrasonic washer with acetone for 3 minutes. Electrochemical study of pitting corrosion resistance determined the potentials of the stationary states in no current conditions. The values of corrosion potentials were set in 10 minutes, and then were plotted anodic polarization curves.

4. Results and discussion

In Figs. 1, 2 the morphology of the powders used in the manufacture of composite materials and composites manufactured surface topographies are shown. Nd-Fe-B powder is characterized by irregular morphology, grains are in the form of flakes with sharp edges. Iron powder has a uniform morphology, the grains show a flaky structure with comparable diameters. Morphology of CuSn10 powder is characterized by a grain close to spherical shape with average diameter about 100 microns. Steel X2CrNiMo17-12-2 powder exhibits a regular morphology with a very small, spherical grains of similar size.

Topographical image of the Nd-Fe-B composite material structure shows a compact surface of the material. Nd-Fe-B powder grains are visible in the form of irregular flakes and places between the grains of powder Nd-Fe-B, which is a binding material. Surface topography of Nd-Fe-B/15% Fe material is characterized by large flaky grains of magnetic powder and smaller grains of iron powder. On the magnet surface the grain boundaries are visible, and a binder located on the borders of powder particles.

The composite material Nd-Fe-B/15% CuSn10 is characterized by a surface with irregular edged flakes of Nd-Fe-B powder which is homogenous distributed on the surface of the magnets. The topographic image of this material shows grain boundaries of Nd-Fe-B and CuSn10 powders. Surface topography of Nd-Fe-B/15%X2CrNiMo17-12-2 composite material shows

regular distribution in the polymer matrix of Nd-Fe-B grains with visible boundaries of grains and steel powder with very small spherical grains.

Table 2 shows the increase in mass of materials during and after the test corrosion in water conditions, temperature 40°C (test 1), while Table 3 shows the increase in mass of materials during and after the test corrosion in NaCl 5% solution, temperature 35°C (test 2). Topography of the hard magnetic composite materials Nd-Fe-B with the 15% mass addition of iron, CuSn10 and X2CrNiMo17-12-2 powders during the test corrosion in water at 24 and 96 hours are shown in Fig. 3 and for NaCl 5% solution at 1 and 6 hours are shown in Fig. 4, respectively.

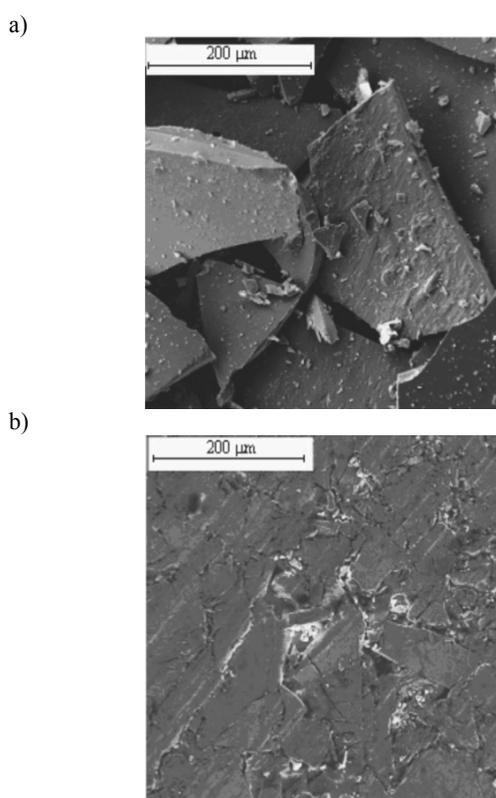


Fig. 1. Nd-Fe-B powder: a) morphology, b) topography of composite materials surface without addition powders

Table 2.

Samples mass changes for test 1 (temperature 40°C, relative humidity 93%)

No.	Composite material	Mass changes after 24h in H ₂ O [%]	Mass changes after 48h in H ₂ O [%]	Mass changes after 96h in H ₂ O [%]
1	Nd-Fe-B	0.10	0.11	0.13
2	Nd-Fe-B/5%Fe	0.05	0.07	0.09
3	Nd-Fe-B/10%Fe	0.08	0.14	0.18
4	Nd-Fe-B/15%Fe	0.07	0.11	0.13
5	Nd-Fe-B/5%CuSn10	0.02	0.04	0.06
6	Nd-Fe-B/10% CuSn10	0.13	0.31	0.45
7	Nd-Fe-B/15% CuSn10	0.93	0.99	1.01
8	Nd-Fe-B/5%X2CrNiMo17-12-2	0.09	0.15	0.17
9	Nd-Fe-B/10% X2CrNiMo17-12-2	0.74	0.77	0.83
10	Nd-Fe-B/15% X2CrNiMo17-12-2	0.79	1.15	1.16

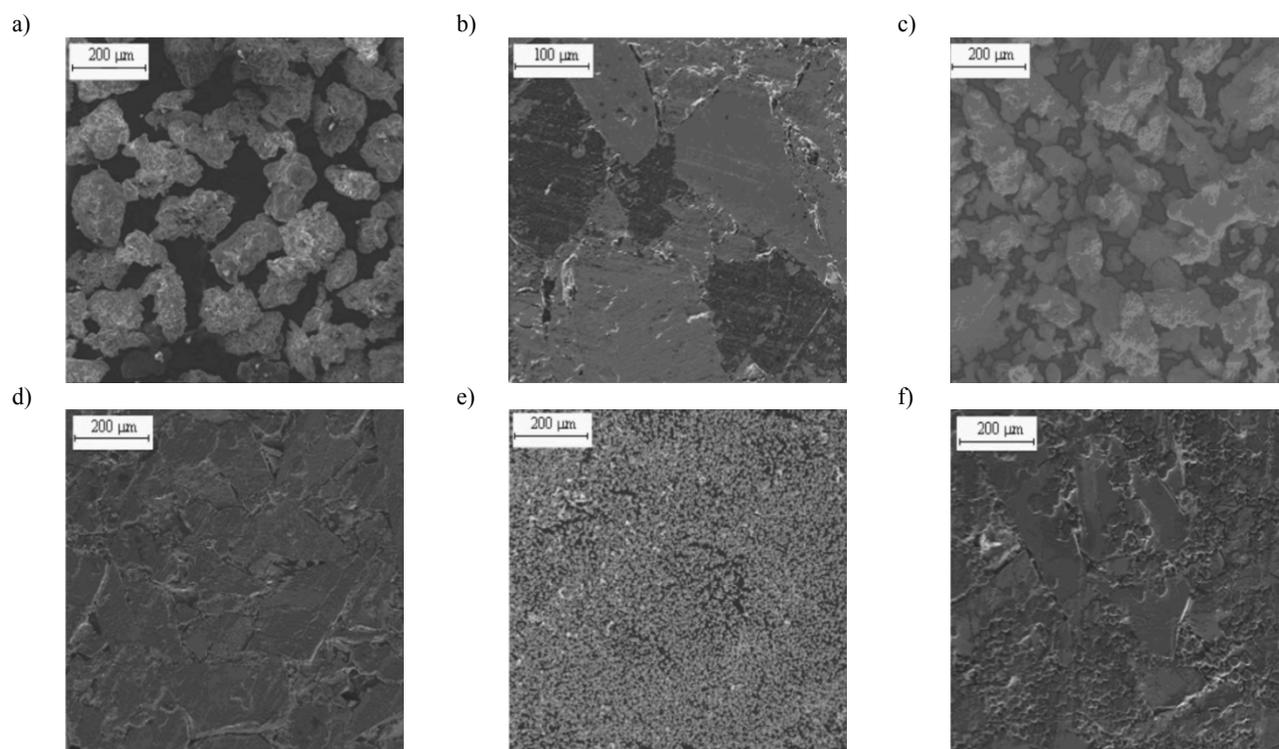


Fig. 2. Iron powder: a) morphology, b) topography of composite materials surface Nd-Fe-B/15%Fe; CuSn10 casting alloy of copper with tin powder: c) morphology, d) topography of composite materials surface Nd-Fe-B/15%CuSn10; X2CrNiMo17-12-2 high-alloy steel powder: e) morphology, f) topography of composite materials surface Nd-Fe-B/15% X2CrNiMo17-12-2

Table 3.

Samples mass changes test 2 (temperature 35°C, NaCl 5% solution)

No.	Composite material	Mass changes after 24h in H ₂ O [%]	Mass changes after 48h in H ₂ O [%]	Mass changes after 96h in H ₂ O [%]
1	Nd-Fe-B	0.11	0.49	0.80
2	Nd-Fe-B/5%Fe	0.07	0.52	0.56
3	Nd-Fe-B/10%Fe	0.82	0.96	1.00
4	Nd-Fe-B/15%Fe	0.16	0.19	0.60
5	Nd-Fe-B/5%CuSn10	0.25	0.34	0.49
6	Nd-Fe-B/10% CuSn10	0.21	0.39	0.49
7	Nd-Fe-B/15% CuSn10	0.93	1.14	1.42
8	Nd-Fe-B/5%X2CrNiMo17-12-2	0.18	0.32	0.38
9	Nd-Fe-B/10% X2CrNiMo17-12-2	0.42	0.69	0.84
10	Nd-Fe-B/15% X2CrNiMo17-12-2	1.05	1.79	2.44

Table 4.

Values from Sterna and Tafel graph

No.	Composite material	Corrosion potential E_{kor} [mV]	Polarization resistivity R_p [$\Omega \cdot \text{cm}^2$]	Density of corrosion current i_{kor} [mA/cm ²]
1	Nd-Fe-B	-749.7	86.81	0.0185
2	Nd-Fe-B/5%Fe	-768.0	345.01	0.0790
3	Nd-Fe-B/10%Fe	-748.2	166.29	0.1402
4	Nd-Fe-B/15%Fe	-688.4	82.04	0.3148
5	Nd-Fe-B/5%CuSn10	-733.6	160.20	0.1556
6	Nd-Fe-B/10% CuSn10	-729.8	180.59	0.0903
7	Nd-Fe-B/15% CuSn10	-693.8	353.86	0.0626
8	Nd-Fe-B/5%X2CrNiMo17-12-2	-752.6	127.23	0.2061
9	Nd-Fe-B/10% X2CrNiMo17-12-2	-700.4	220.50	0.1054
10	Nd-Fe-B/15% X2CrNiMo17-12-2	-676.9	460.50	0.0391

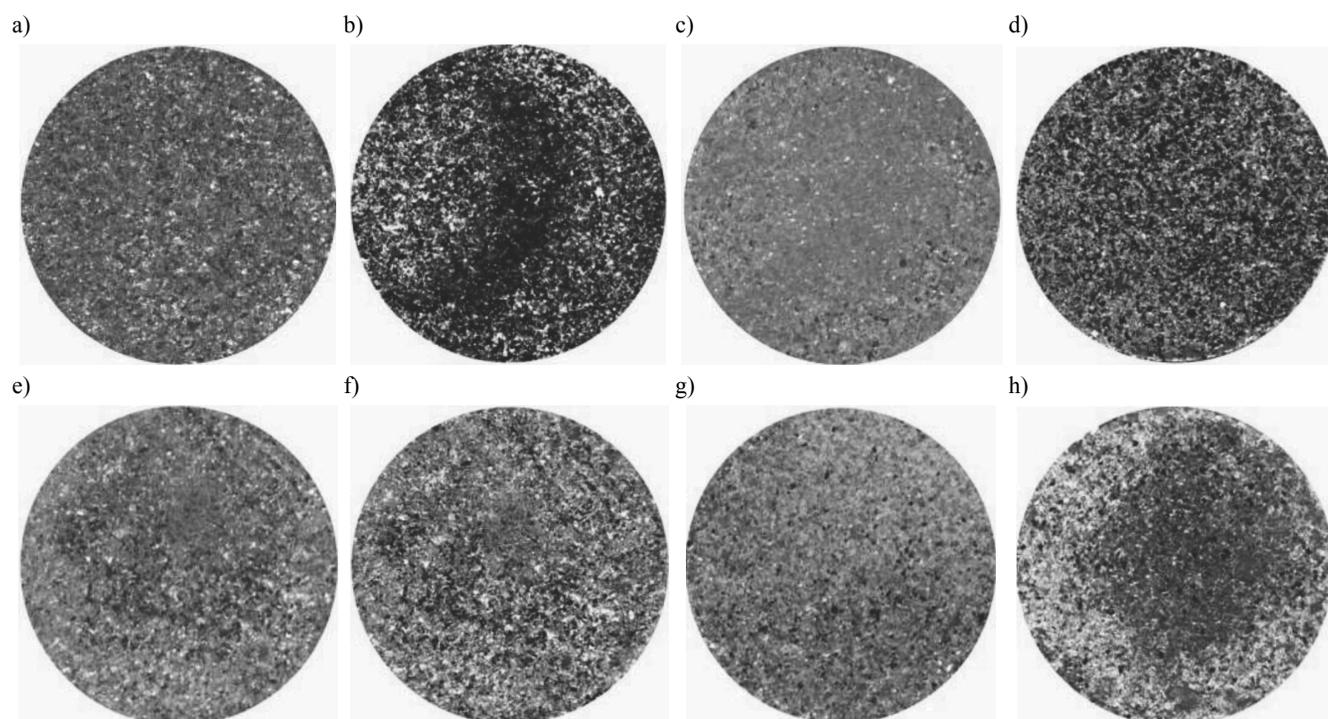


Fig. 3. Topography of composite materials surface during corrosion test 1 (temperature 40°C, relative humidity 93%) Nd-Fe-B - a) 24h, b) 96h, Nd-Fe-B/15%Fe B - c) 24h, d) 96h, Nd-Fe-B/15%CuSn10 - e) 24h, f) 96h, Nd-Fe-B/15% X2CrNiMo17-12-2 - g) 24h, h) 96h

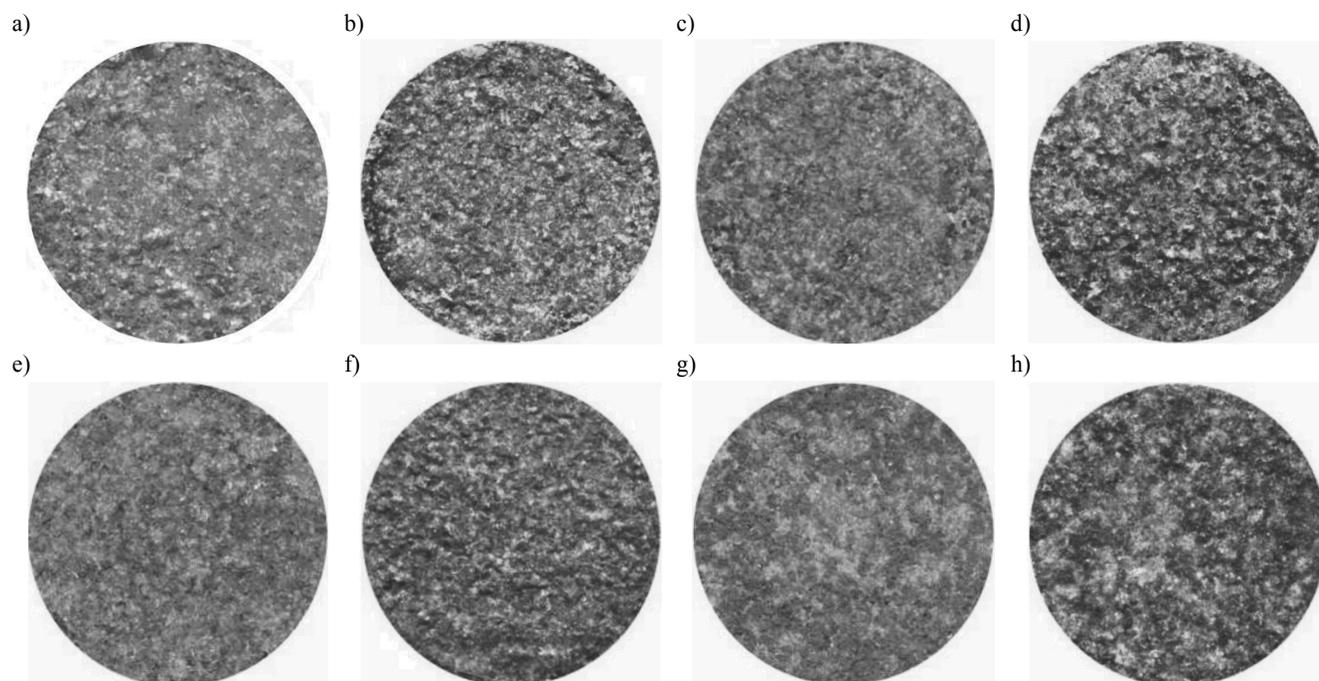


Fig. 4. Topography of composite materials surface during corrosion test 2 (temperature 35 ° C, 5% NaCl solution) Nd-Fe-B - a) 1h, b) 6h, Nd-Fe-B/15%Fe B - c) 1h, d) 6h, Nd-Fe-B/15%CuSn10 - e) 1h, f) 6h, Nd-Fe-B/15% X2CrNiMo17-12-2 - g) 1h, h) 6h

In test 1, in an water (93% relative humidity) at 40°C, the results shown an increase in mass of the composite material Nd-Fe-B: after 24 hours exposure in water, its weight growth is about 0.10%, and after the test at 0.13% in comparison with the sample before testing corrosion. In test 1, in an water (93% relative humidity) at 40°C, the results shown an increase in mass of the composite material Nd-Fe-B: after 24 hours exposure in water, its weight growth is about 0.10%, and after the test at 0.13% in comparison with the sample before testing corrosion. The mass growth for composite material containing iron (Nd-Fe-B / 5% Fe) during the test, is lower which may indicate that in the on the surface creates less product corrosion of the substrate (corrosion products are not so adhesive to the composite surface) (Fig. 5).

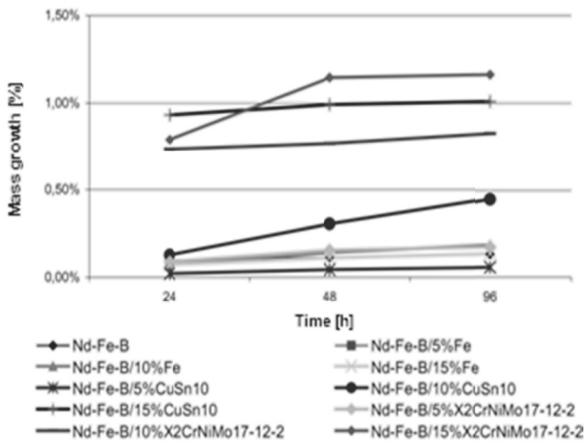


Fig. 5. Dependence of weight changes during the exposure time in aqueous environments (test 1)

The results of analysis show that the higher percentage of CuSn10 alloy powder, the higher increase of their mass, what could be associated with higher participation of corrosion products compacted with the surface.

The lower corrosion show materials with steel powder addition, as indicated by the high percentage increase in weight of the samples, probably due to good compaction of corrosion products with the substrate. Tight adhesion to the substrate and the tight structure of corrosion products, may be responsible for preventing the flow of corrosive agent to the surface of the sample, and thus decrease the rate of corrosion.

The increase in mass of the magnetic material of Nd-Fe-B after 1 hour corrosion test in NaCl solution was 0.11%, and with increasing time of the study, after 6 hours, the increase in weight of the sample is 0.80% of the initial sample weight. Weight gain of composite materials with metal powder additives in the case of this test are significantly higher than in the case of the test in an aqueous medium (Fig. 6).

Table 4 shows the values read from the graph Stern and Tafel during the test pitting corrosion electrochemical method. For composite material without additives powder, corrosion potential is equal to -794.7 mV polarization resistance 86.81 Ω cm, and the corrosion current density is 0.0185 mA/cm². This material is characterized by the lowest resistance to electrochemical

corrosion. Additive powders of iron, CuSn10 alloy and stainless steel X2CrNiMo17-12-2 influence the changes of electrochemical properties of materials. Iron powder causes only minor changes in the corrosion potential. However CuSn10 alloy casting powders and stainless steel X2CrNiMo17-12-2 increase the value of this potential, which is responsible for increasing the corrosion resistance of composite materials. The increase in the amount of the additive powder results in an improvement of their resistance to corrosion. The best electrochemical properties of the material is obtained for material containing 15% of the steel X2CrNiMo17-12-2. Corrosion potential for this material is -676.9 mV, the corrosion current density is 0.0391 mA/cm². In addition, composite materials with 15% of the powder additive show the highest corrosion resistance among all materials tested for electrochemical corrosion (Fig. 7).

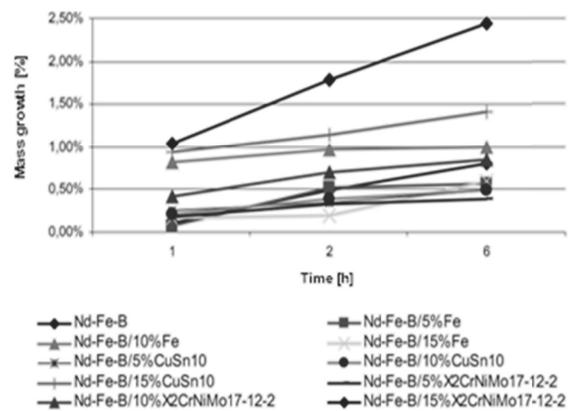


Fig. 6. Dependence of weight changes during the exposure time in NaCl solution environments (test 2)

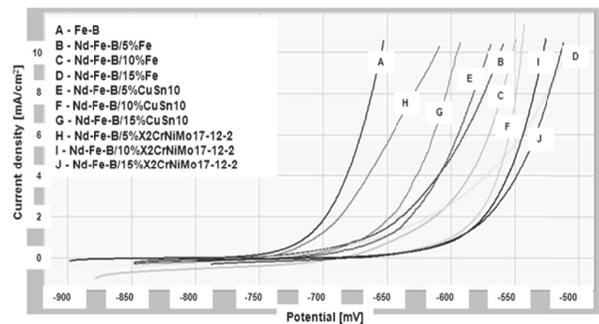


Fig. 7. Dependence of current potential and current density for the samples

5. Conclusions

On the base of the investigations the followings can be concluded: Investigations of the powder morphology and the structure of hard magnetic composite materials revealed that the powder Nd-Fe-B and powders of additions are evenly distributed in the polymer matrix.

Results of corrosion tests made in climatic chambers allows to evaluate that the best corrosion resistance shows composite materials with addition 15% of CuSn10, and 15% of X2CrNiMo17-12-2 steel in water test and composite material with 15% of X2CrNiMo17-12-2 steel in 5% NaCl solution. The products of corrosion deposited onto the surface of composite materials show dielectric character. Potentiodynamic investigations in the 5% NaCl solution allows to evaluate corrosion potential of composite materials. The best corrosion resistance show material with addition of 15% X2CrNiMo17-12-2 steel what correspond to the results of gravimetric corrosion.

Investigated hard magnetic composite materials are an interesting alternatives for other hard magnetic materials. Their properties, especially taking into consideration their corrosion resistance, show that they can be applied with success in different devices in which neodymium magnets are needed Application of these materials cause the increase of devices effectiveness and their miniaturization. The knowledge of their corrosion resistance will optimize the technological process of these devices manufacturing and their conditions of service, which will greatly improve the quality.

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