Corrosion resistance of the composite materials: nanocrystalline powder – polymer type in acid environment

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Materials

ABSTRACT

Purpose: The paper presents corrosion resistance of composite materials $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ – PEHD type in sulphuric acid and hydrochloric acid environments.

Design/methodology/approach: Composite materials $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ – PEHD type were manufactured by one-sided uniaxal pressing. The amount of polymer matrix was 2.5%, 5.0%, 7.5%, wt. Powder of the $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ was made by the high-energy grinding in the shaker type 8000SPEX CertiPrep Mixer/Mill for 1 h, 3 h, 5 h. Composite materials were placed in a corrosive environment and two tests were carried out as specified below: test at the temperature of 25°C, 0.1 M solution of hydrochloric acid HCl, time 348 h; test temperature 25°C, 0.1 M solution of sulphuric acid H_2SO_4, time 348 h, test temperature 25°C.

Findings: Obtained results of corrosion resistance allow to evaluate corrosion wear of composite materials FINEMET ($Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$) – PEHD in acidic solutions of 0.1M HCl and 0.1M H_2SO_4. It was found that the composite materials with 7.5% wt. of polyethylene portion show the best corrosion resistance.

Research limitations/implications: Composite materials $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$– PEHD type manufacturing greatly expand the application possibilities of soft magnetic materials nanocrystalline powders however further examination to obtain improved properties of magnetic composite materials and investigations of new machines and devices constructions with these materials elements are still needed.

Originality/value: Results allow to complete data concerning composite materials nanocrystalline powder – polymer type which are an attractive alternative for traditional materials with specific magnetic properties. Results are the base for further investigations of the impact of corrosion environment on the magnetic properties such composite materials.

Keywords: Composites; Corrosion resistance; Nanocrystalline powder; Polymer

1. Introduction

Designing new materials – composites – being a joint of two or more materials different from each other, the type or chemical composition in macro scale gives unlimited possibilities of designing their properties and simultaneously allows to broaden their application. [1-4]. The idea of new composite materials designing was the base for finding new materials with unique physical properties for the electrotechnical and electronic industry. It brings, among other things, to the development of the investigations on bonding powders of alloys with nanocrystalline structure which have unique soft magnetic properties with polymer materials [5-9].

Nanocrystalline magnetic materials are the competitors for conventional magnetic materials. Unfortunately, most often used manufacturing methods (mechanical alloying, melt quenching with controlled crystallization) significantly reduce the range of their application because of the geometrical form of manufactured nanomaterials (powder or thin tape). Only thin tapes can be used in electronics or telecommunication [2-4,10,11]. To broaden the application possibility of these materials it is obvious to manufacture composite materials. Taking into account metastable structure of nanocrystalline powder, methods of their consolidation into composite materials in which higher temperature is acting have limited application. In higher temperature the growth of crystallites is observed what has disadvantageous influence on the magnetic properties of these materials [10]. Regarding this the most adequate materials for nanocrystalline powder consolidation are chemo and thermo setting polymers [12-14]. Nanocrystalline components prepared to manufacture composite materials have the structure and properties adequate for further consolidation process with polymers materials.

Methods of nanocrystalline powders bonding into composite materials depends on the mixing of nanopowder with binding agent (polymer material) and forming into a planed shape in cold or hot forming methods (pressing, rolling, extrusion, injection, moulding).

New worked out materials should be investigated to collect a data base of their physical, chemical and mechanical properties. These data allow to broaden their practical application in many techniques fields. Unfortunately in the case of many materials, also composite materials nanocrystalline powder – polymer type,

the data base of their properties is incomplete and should be completed as soon as possible. Majority of papers concerning nanocrystalline alloys FINEMET ($Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$) and composite materials with them refers to their magnetic and mechanical properties [1,5-9,12-14] but there is a lack of literature concerning their corrosion resistance. This paper is an attempt to complete data base and it will bring, in the future, new application possibilities of these composite materials in the construction of new electric and electronic devices.

2. Material and methods

The experiments were made with the polymer matrix composite materials: FINEMET ($Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$) powder bonded with the high density pressureless polyethylene (PEHD). Powder of the $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ was made by the high-energy grinding in the shaker type 8000SPEX CertiPrep Mixer/Mill for 1h, 3h, 5h, structure of the powders was nanocrystalline [5]. The amount of polymer matrix was 2.5%, 5.0%, 7.5%, wt.. Advanced composite materials were compacted by the one-sided uniaxial pressing. The following compacting process parameters were used: pressure 350 MPa, pressing temperature 170°C, pressing time 0.25h [5,7].

Metallographic examinations were made on the LEICA MEF4A light microscope equipped with the computer image analysis system at the magnification of 500x. Figures 1-3 show the structure of composite materials observed on light microscope.

3. Results and discussion

Tables 1-9 show the mass loss and changes of samples' dimensions investigated in the solution of 0.1M H_2SO_4 and in the solution of 0.1M HCl after 24, 48, 96, 192, 384 hours of corrosion tests. Figures 4 and 5 show examples of composite materials surfaces after 384 hours of corrosion tests in the solutions of 0.1M H_2SO_4 or 0.1M HCl.

Besides, suitable magnetic and mechanical properties nanocrystalline materials including composite materials and polymer materials should show good corrosion resistance.
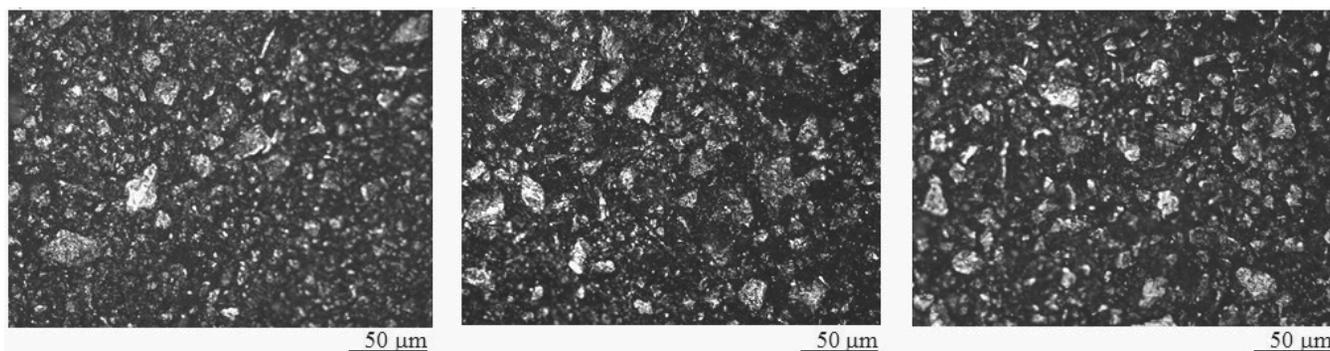
Fig. 1. Structure of composite materials FINEMET ($Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$) – PEHD: a) 2.5% wt. PEHD, b) 5.0% wt. PEHD, c) 7.5% wt. PEHD, milling time 1h of the $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ powder

These materials are more commonly used for transformers cores of generators and sensors. The majority of these application need also exploitation in humid air and in the atmosphere with sulphates and chlorides ions, often also in higher temperature, which favourite the corrosion of these materials. Choosing proper material for composites matrix is a very important factor influencing their corrosion resistance. Corrosion resistance is also determined by the structure and chemical composition of nanocrystalline powder [15-17].

Figures 6 and 7 show the mass loss of the composite materials FINEMET ($Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$) – PEHD. On the base of the

corrosion test results it was found that the mass loss of the composite materials is initially high and then tendency of decreasing is observed. It was found that the mass loss is stabilizing in time after 96 h corrosion test is made.

The reason for corrosion rate decrease (mass loss) versus time can be the fact that on the surface of composite materials a coating of corroded material in the form of iron oxides and hydroxides is deposited that makes the access to corrosive environment difficult into composite material. The best corrosion resistance is shown by samples with 7.5% wt. PEHD portion.
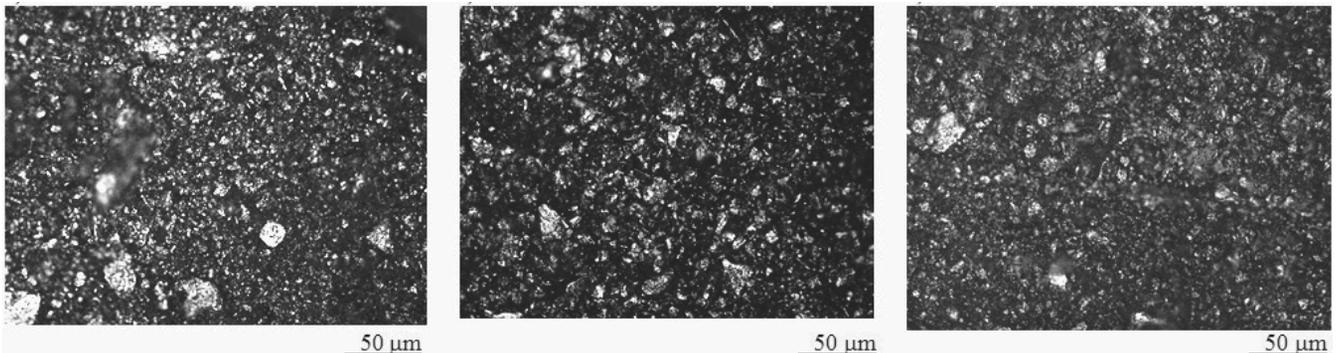
Fig. 2. Structure of composite materials FINEMET ($Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$) – PEHD: a) 2.5% wt. PEHD, b) 5.0% wt. PEHD, c) 7.5% wt. PEHD, milling time 3h of the $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ powder
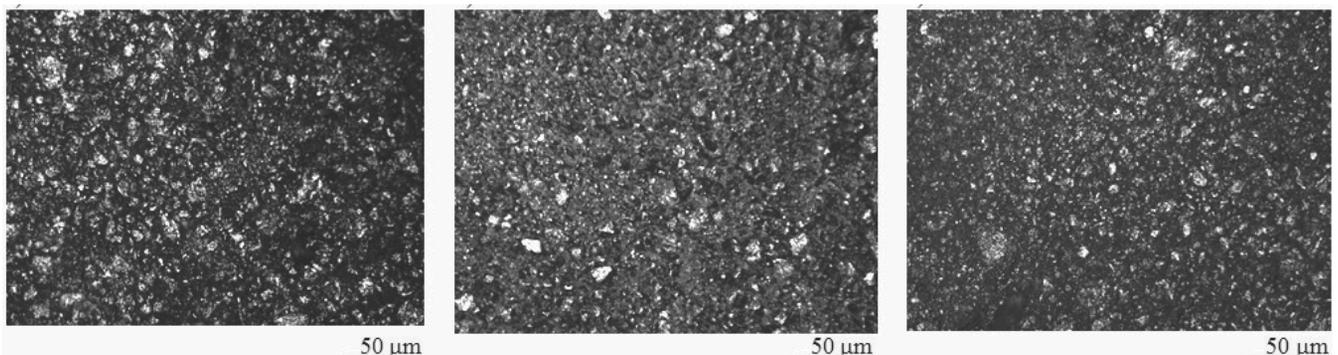
Fig. 3. Structure of composite materials FINEMET ($Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$) – PEHD: a) 2.5% wt. PEHD, b) 5.0% wt. PEHD, c) 7.5% wt. PEHD, milling time 5h of the $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ powder

Table 1.
Mass loss and dimensions' changes of composite materials with powder $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ after 1h milling and with 2.5 wt of PEHD

	time [h]	height [cm]	diameter [cm]	volume [cm³]	mass [g]	mass loss [%]
0.1M H₂SO₄	0	0.69	1.70	1.56	7.485	-
	24	0.69	1.70	1.56	7.239	3.29
	48	0.69	1.70	1.56	7.218	3.58
	96	0.69	1.70	1.56	7.202	3.79
	192	0.62	1.69	1.40	7.189	3.97
	384	0.62	1.69	1.40	7.185	4.02
0.1M HCl	0	0.36	1.71	0.82	4.245	-
	24	0.36	1.71	0.82	4.160	1.99
	48	0.36	1.71	0.82	4.154	2.15
	96	0.36	1.71	0.82	4.150	2.24
	192	0.35	1.71	0.81	4.148	2.28
	384	0.33	1.69	0.75	4.147	2.30

Table 2.
Mass loss and dimensions' changes of composite materials with powder $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$ after 1h milling and with 5 wt of PEHD

	time [h]	height [cm]	diameter [cm]	volume [cm³]	mass [g]	mass loss [%]
0.1M H₂SO₄	0	0.82	1.70	1.86	9.368	-
	24	0.82	1.70	1.86	9.194	1.84
	48	0.81	1.70	1.83	9.146	2.36
	96	0.81	1.70	1.83	9.131	2.52
	192	0.80	1.69	1.79	9.124	2.60
	384	0.80	1.69	1.79	9.111	2.73
0.1M HCl	0	0.36	1.71	0.82	4.224	-
	24	0.36	1.71	0.82	4.156	1.61
	48	0.36	1.71	0.82	4.153	1.67
	96	0.36	1.71	0.82	4.149	1.77
	192	0.35	1.71	0.81	4.148	1.80
	384	0.35	1.69	0.80	4.140	1.98

Table 3.
Mass loss and dimensions' changes of composite materials with powder $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$ after 1h milling and with 7.5 wt of PEHD

	time [h]	height [cm]	diameter [cm]	volume [cm³]	mass [g]	mass loss [%]
0.1M H₂SO₄	0	0.57	1.70	1.29	6.156	-
	24	0.57	1.70	1.29	6.074	1.32
	48	0.56	1.69	1.25	6.059	1.57
	96	0.56	1.69	1.25	6.049	1.73
	192	0.53	1.69	1.18	6.046	1.78
	384	0.53	1.69	1.18	6.045	1.80
0.1M HCl	0	0.30	1.71	0.68	3.279	-
	24	0.30	1.71	0.68	3.223	1.72
	48	0.30	1.71	0.68	3.218	1.87
	96	0.30	1.71	0.68	3.214	1.97
	192	0.29	1.71	0.67	3.213	2.02
	384	0.29	1.69	0.66	3.210	2.15

Table 4.
Mass loss and dimensions' changes of composite materials with powder $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$ after 3h milling and with 2.5 wt of PEHD

	time [h]	height [cm]	diameter [cm]	volume [cm³]	mass [g]	mass loss [%]
0.1M H₂SO₄	0	0.78	1.71	1.80	8.813	-
	24	0.78	1.71	1.80	8.515	3.39
	48	0.78	1.71	1.79	8.492	3.65
	96	0.78	1.70	1.77	8.473	3.86
	192	0.74	1.69	1.67	8.457	4.05
	384	0.74	1.69	1.67	8.453	4.09
0.1M HCl	0	0.39	1.70	0.88	4.526	-
	24	0.39	1.70	0.88	4.433	2.04
	48	0.38	1.70	0.86	4.425	2.22
	96	0.38	1.70	0.86	4.421	2.31
	192	0.38	1.70	0.86	4.420	2.34
	384	0.35	1.69	0.79	4.419	2.36

Table 5.
Mass loss and dimensions' changes of composite materials with powder $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$ after 3h milling and with 5 wt of PEHD

	time [h]	height [cm]	diameter [cm]	volume [cm³]	mass [g]	mass loss [%]
0.1M H₂SO₄	0	0.82	1.70	1.86	9.592	-
	24	0.82	1.70	1.86	9.418	1.81
	48	0.81	1.70	1.83	9.385	2.15
	96	0.81	1.70	1.83	9.364	2.38
	192	0.79	1.69	1.78	9.352	2.49
	384	0.79	1.68	1.77	9.349	2.52
0.1M HCl	0	0.36	1.71	0.82	3.999	-
	24	0.36	1.71	0.82	3.923	1.89
	48	0.36	1.71	0.82	3.916	2.07
	96	0.35	1.71	0.81	3.912	2.17
	192	0.33	1.71	0.76	3.911	2.18
	384	0.32	1.69	0.72	3.910	2.20

Table 6.
Mass loss and dimensions' changes of composite materials with powder $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$ after 3h milling and with 7.5 wt of PEHD

	time [h]	height [cm]	diameter [cm]	volume [cm^3]	mass [g]	mass loss [%]
0.1M H$_2$SO$_4$	0	0.58	1.70	1.31	6.157	-
	24	0.56	1.70	1.28	6.079	1.26
	48	0.56	1.69	1.25	6.064	1.50
	96	0.56	1.69	1.25	6.053	1.67
	192	0.53	1.67	1.16	6.049	1.75
	384	0.53	1.67	1.16	6.047	1.78
0.1M HCl	0	0.19	1.71	0.43	2.154	-
	24	0.19	1.71	0.43	2.118	1.66
	48	0.19	1.71	0.43	2.116	1.82
	96	0.19	1.71	0.43	2.115	1.95
	192	0.18	1.70	0.41	2.113	1.92
	384	0.15	1.69	0.33	2.112	1.87

Table 7.
Mass loss and dimensions' changes of composite materials with powder $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$ after 5h milling and with 2.5 wt of PEHD

	time [h]	height [cm]	diameter [cm]	volume [cm^3]	mass [g]	mass loss [%]
0.1M H$_2$SO$_4$	0	0.83	1.70	1.88	9.355	-
	24	0.83	1.70	1.88	9.064	3.11
	48	0.83	1.70	1.88	9.029	3.48
	96	0.82	1.70	1.86	9.010	3.68
	192	0.81	1.69	1.81	9.002	3.76
	384	0.81	1.69	1.81	8.995	3.84
0.1M HCl	0	0.39	1.71	0.89	4.403	-
	24	0.39	1.71	0.89	4.325	1.77
	48	0.39	1.70	0.88	4.310	2.10
	96	0.38	1.70	0.86	4.308	2.15
	192	0.37	1.70	0.83	4.307	2.17
	384	0.33	1.69	0.75	4.307	2.17

Table 8.
Mass loss and dimensions' changes of composite materials with powder $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$ after 5h milling and with 5 wt of PEHD

	time [h]	height [cm]	diameter [cm]	volume [cm^3]	mass [g]	mass loss [%]
0.1M H$_2$SO$_4$	0	0.59	1.70	1.33	6.418	-
	24	0.58	1.70	1.31	6.327	1.41
	48	0.58	1.70	1.31	6.309	1.69
	96	0.58	1.69	1.30	6.297	1.87
	192	0.55	1.67	1.21	6.293	1.94
	384	0.55	1.67	1.21	6.289	1.98
0.1M HCl	0	0.29	1.71	0.66	3.292	-
	24	0.29	1.71	0.66	3.239	1.61
	48	0.29	1.70	0.65	3.228	1.96
	96	0.29	1.70	0.65	3.225	2.05
	192	0.28	1.69	0.62	3.223	2.10
	384	0.27	1.69	0.61	3.223	2.10

Table 9.
Mass loss and dimensions' changes of composite materials with powder $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$ after 5h milling and with 7.5 wt of PEHD

	time [h]	height [cm]	diameter [cm]	volume [cm^3]	mass [g]	mass loss [%]
0.1M H$_2$SO$_4$	0	0.82	1.70	1.86	9.489	-
	24	0.81	1.70	1.83	9.383	1.11
	48	0.81	1.69	1.81	9.361	1.35
	96	0.81	1.69	1.81	9.349	1.47
	192	0.79	1.69	1.78	9.348	1.49
	384	0.79	1.69	1.78	9.346	1.50
0.1M HCl	0	0.39	1.70	0.88	4.443	-
	24	0.39	1.70	0.88	4.377	1.48
	48	0.38	1.70	0.86	4.371	1.62
	96	0.38	1.70	0.86	4.366	1.73
	192	0.36	1.70	0.82	4.364	1.76
	384	0.34	1.69	0.76	4.362	1.80

a)

b)

c)
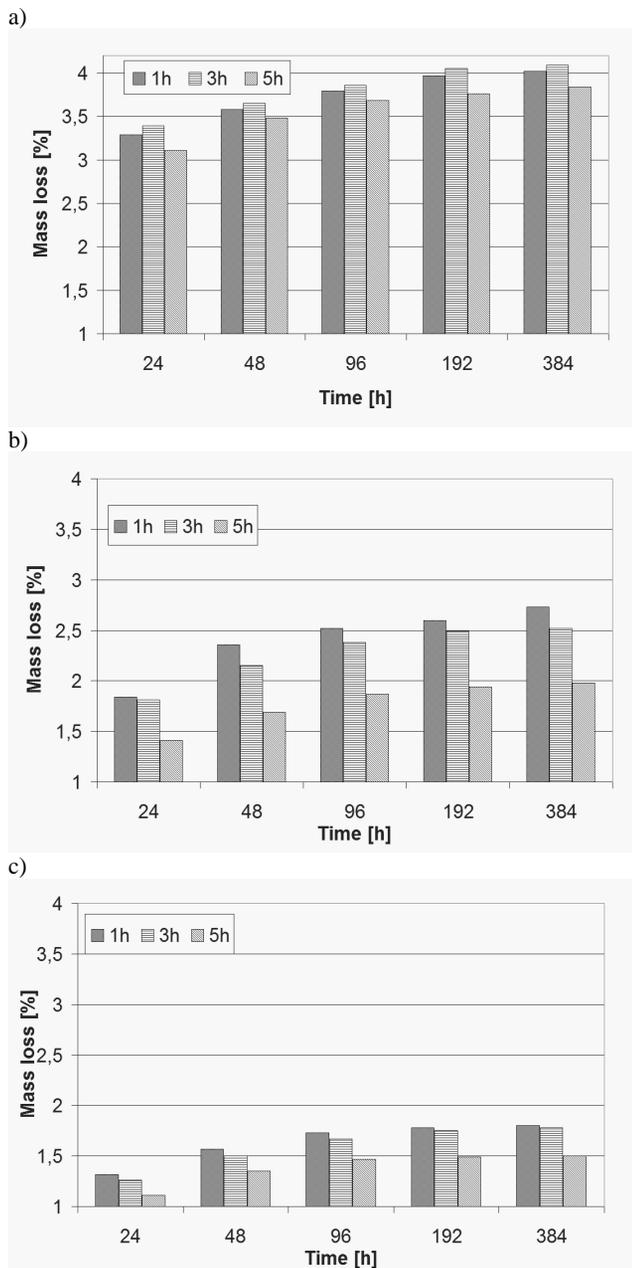
Fig. 6. Mass loss of composite materials FINEMET (Fe$_{73.5}$Cu$_1$Nb$_3$Si$_{13.5}$B$_9$) – PEHD during corrosion tests in solution 0.1M H$_2$SO$_4$, the portion of polyethylene in composite material: a) 2.5%, b) 5.0%, c) 7.5%

It can be caused by the fact that polyethylene is resistant to the acids in which corrosion test were made and its higher portion in composite material increases isolation of Fe$_{73.5}$Cu$_1$Nb$_3$Si$_{13.5}$B$_9$ particles (Figs. 1-3). The important factor influencing unfavourably on the corrosion resistance is the flaky shape of Fe$_{73.5}$Cu$_1$Nb$_3$Si$_{13.5}$B$_9$ alloy powder particles (Figs. 1-3). Near–spherical shape seems to be more optimal taking corrosion

resistance of composite materials into account. Unfortunately near–spherical shape influences unfavourably on other properties (mechanical and magnetic) of these materials [2-4]. Flaky shape of Fe$_{73.5}$Cu$_1$Nb$_3$Si$_{13.5}$B$_9$ alloy powder particles in composite materials allows for better optimization of physical and mechanical properties with corrosion resistance of worked out composite materials.

a)

b)

c)
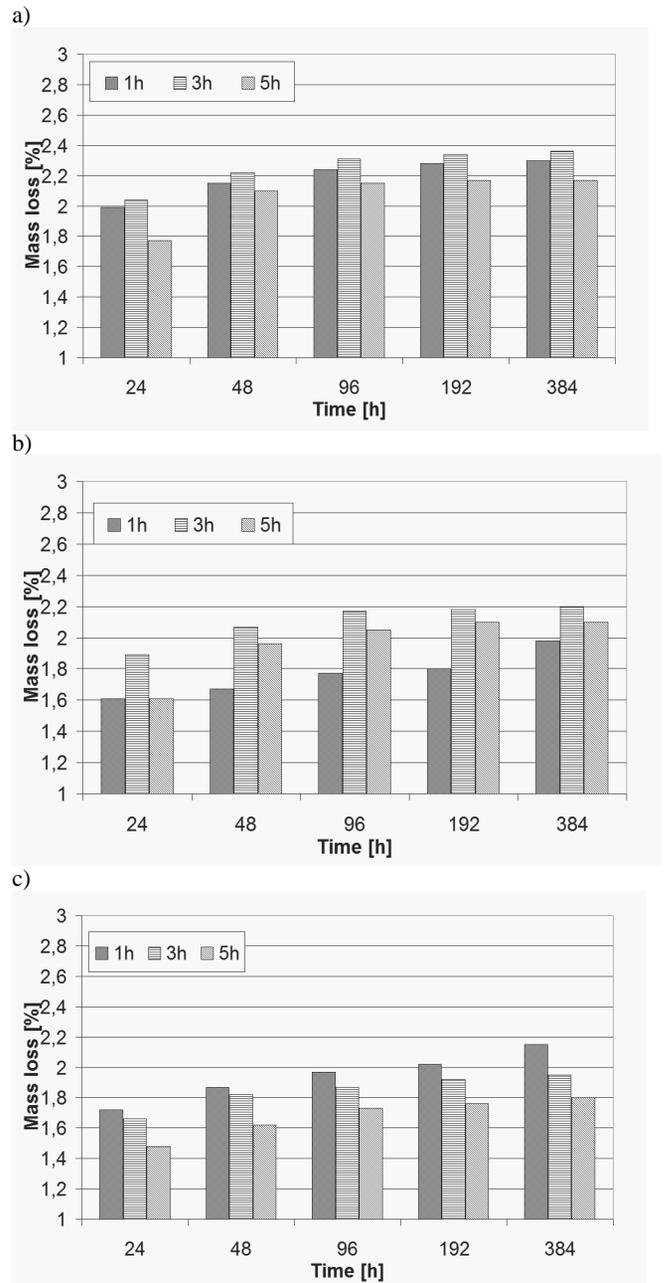
Fig. 7. Mass loss of composite materials FINEMET (Fe$_{73.5}$Cu$_1$Nb$_3$Si$_{13.5}$B$_9$) – PEHD during corrosion tests in solution 0.1M HCl, the portion of polyethylene in composite material: a) 2.5%, b) 5.0%, c) 7.5%

The analysis of composite materials surface topography allows to determine damages occurring as a result of corrosion environment activity. Figures 4 and 5 show corrosion wear of chosen composite materials samples with 2.5%, 5.0%, 7.5% wt. polyethylene portion. After corrosion tests in 0.1M HCl and 0.1M H_2SO_4 solutions on the composite materials surface it was found that small corrosion centre are occurring, which at the beginning do not show tendency to expansion. After 96h of corrosion tests, their growth and connecting is observed. The lower portion of polyethylene in composite material the higher is progress of corrosion.

Higher corrosion resistance was observed in the corrosion tests in the solution of 0.1M HCl. It can be caused by the fact that corrosion products formed on the samples surface – chlorides – make more coherent layer with the sample's solution of 0.1M H_2SO_4. In the case of 0.1M HCl solution the portion of polyethylene do not significantly influence on a slight increase of corrosion resistance (about 20%). In the case of the 0.1M H_2SO_4 solution the influence of the polymer material portion is more visible. The growth of corrosion resistance for composite materials with 7.5% polyethylene is about 60% in comparison with composite materials with 2.5% of polyethylene.

The size of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ powder particles influences corrosion resistance in smaller level than the portion of polyethylene. Particles size is about 20.4 µm for the $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy milled 1h, 15.3 µm for alloy milled 3h and 8.1 µm for alloy milled 5h. The smaller powder particles the higher corrosion resistance of composite material. It is caused by the fact smaller particles are easier isolated by polymer matrix.

4. Application possibilities

The range of soft magnetic materials application, to which composite materials nanocrystalline is also included $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ powder and polyethylene growths with the improvement of their magnetic, mechanicals, electrical, thermal properties and corrosion resistance. They are used more and more commonly in many fields of human life and not always we realize they exist. Table 10 shows possibilities of soft magnetic composite materials applications. The application of magnetic composite materials allows to miniaturizing magnetic elements, construction simplification and lowering both manufacturing and material costs (Fig. 8) [2, 10, 11].

Table 10.
Application of soft magnetic composite materials

Soft magnetic composite materials
magnetic cores in high frequency transformers, transformers impedance coils, magnetic and current sensors, instruments for power measurements, frequency converters, signal transformers, read-write head of digital devices, magnetic screen, magnetostriction converters, computers power packs, telephone exchange power packs, magnetostriction transducers, telecommunication engineering

Miniaturization requires better and better materials which not always were taken into consideration. In these properties corrosion resistance is also included. Smaller devices are more mobile and work in more aggressive environments. Figure 9 shows the division of soft magnetic materials market.

Fig. 8. The main direction of miniaturization in modern world

Taking their application field into account, it is so important to investigate corrosion resistance to increase possibilities of their application and to extend their exploitation time.

Although slight but constantly increasing the participation world-wide, the marked interest of these materials is very important because of very good magnetic properties [11]. Application of nanocrystlalline alloys Finemet, Hitperm, Nanoperm will increase in the next years because of the composite materials bonded by polymer materials manufacturing.
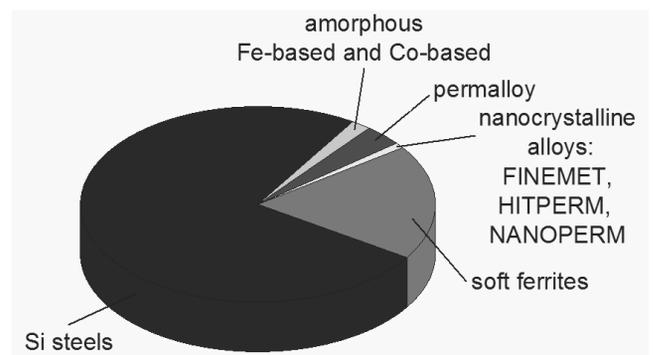
Fig. 9. Division of global market of soft magnetic materials

5. Conclusions

The dynamical development of the technical civilisation causes greater and greater progress in magnetic materials. It is the result of the fact that modern civilisation is based on converting

electric current devices and there is the need to extent the possibilities of their application.

Obtained results of corrosion resistance allows to evaluated corrosion wear of composite materials FINEMET ($Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$) – PEHD in acidic solutions of 0.1M HCl and 0.1M H_2SO_4. It was found that the composite materials with 7.5% wt. of polyethylene portion show best corrosion resistance. Results allow to complete data concerning composite materials nanocrystalline powder – polymer type which are an attractive alternative for traditional materials with specific magnetic properties.

Modern magnetic materials, with excellent magnetic properties allow to miniaturize machines and devices. That influence also on our common life because most of devices, till now are considered to be stationary and they can be now replaced in any place. The development in magnetic materials is still in progress. Now not only magnetic properties are taken into consideration, but also others like mechanical, physical properties or corrosion resistance are coming to be important.

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