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Manufacturing of hard magnetic composite materials Nd-Fe-B

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Manufacturing and processing

ABSTRACT

Purpose: This paper presents the material and technological solution which makes it possible obtaining of hard magnetic composite materials: nanocrystalline material – polymer.

Design/methodology/approach: For fabrication of composite materials the Nd-Fe-B powder obtained by melt quenching technique was used and for matrix: epoxy resin (EP) or high density polyethylene (HDPE) (2.5 % wt.). Composite materials were compacted by the one-sided uniaxial pressing. The complex relationships among the manufacturing technology of these materials, their microstructure, as well as their properties were evaluated. Materialographic examination of powders morphology and the structure of composite materials were made.

Findings: Composite materials show regular distribution of magnetic powder in polymer matrix. Examination of mechanical properties show that these materials have satisfactory compression strength.

Research limitations/implications: The advantage of the bonded composite materials is their simple technology, possibility of forming their properties, lowering manufacturing costs because of no costly finishing and lowering of material losses resulting from the possibility of forming any shape. The manufacturing of composite materials greatly expand the applicable possibilities of nanocrystalline powders of magnetically hard materials.

Originality/value: Manufacturing processes of hard magnetic composite materials obtaining Nd-Fe-B – polymer matrix.

Keywords: Magnetic materials; Nd-Fe-B; Composite materials

1. Introduction

In the modern and permanently developed world there is the need to find new materials with better and better properties and simultaneously with the lower and lower costs. There is a need of new functional and non-conventional materials with the unique properties. It is connected, among others, with the development of the contemporary electrotechnical and electronic industry employing the modern magnetic materials. The researches try to find new materials or to improve currently known ones by changing their composition, structure and properties or the technology of their manufacturing. [1, 6-8, 12, 13].

In the field of magnetic materials the attention is paid on the intermetalic phases based on the rare earth metals and transition metals Nd-Fe-B nanocrystalline structure showing excellent hard magnetic properties [7, 10]. This properties are connected with nanocrystalline structure of these materials so it is so important to keep that structure during the manufacturing process. One of the way is bonding of Nd-Fe-B powders with the polymer, chemically-setting, thermosetting and thermoplastic materials, low-melting glasses or metals makes to obtain the composite materials. These magnetic materials may be made also by sintering, injection moulding, hot compacting, upseting, casting and explosive consolidation but they can more affect the nanocrystalline structure of magnetic component [2-5, 9, 11, 14, 15].

The mechanical properties of the composite magnets depend mostly on the magnetic powder and binding agent types and on the technology employed. The portion of the polymer matrix affects the mechanical and magnetic properties of the manufactured composite materials. The mechanical properties increase along with the increasing portion of resin in the matrix but this increase has a negative effect on the magnetic properties. Usually the 2.5-3.0% mass portion is assumed, i.e., 15-20% volume portion, and compacting pressure is 350-900 MPa [5, 12, 14, 15].

Magnetic composite materials are characteristic of various magnetic, mechanical, and physical properties depending on the powder manufacturing and compacting technology. This makes it possible to obtain materials for various applications, according to the particular requirements [1, 2, 5, 7, 12, 13, 15].

Bonded composite materials have many advantage mainly simple technology, possibility of forming their properties, lowering manufacturing costs because of no costly finishing and lowering of material losses resulting from the possibility of forming any shape.

The goal of the work is to manufacture hard magnetic composite materials with polymer matrix reinforced with Nd-Fe-B particles by one-sided uniaxial pressing and to investigate the structure and properties.

2. Material and experimental procedure

Hard magnetic nanomaterials are usually produced as powders or thin strips. In this geometrical form their range of application is limited. To extend their potential applications it is useful to connect the nanocrystalline magnetic materials with other materials creating the composite materials. The polymers are used most often to manufacture composites, which volume portion in the composite does not exceed 20 % and the low-melting metals, e.g., zinc in the amount of about 15 % mass. Portion of the matrix and slip agent powders, compacting pressure as well the temperature and curing time of the polymer materials decide the technological conditions of magnets manufacturing. The mechanical properties of the composite magnets depend mostly on the magnetic powder and binding agent types, particles shape and on the technology employed. The portion of the polymer matrix affects the mechanical and magnetic properties of the manufactured composite materials (Fig. 1). The mechanical properties increase along with the increasing portion of resin in the matrix but this increase has a negative effect on the magnetic properties. Usually the 2.5-3.0% mass portion is assumed, i.e., 15-20% volume portion, and compacting pressure is 350-900 MPa.

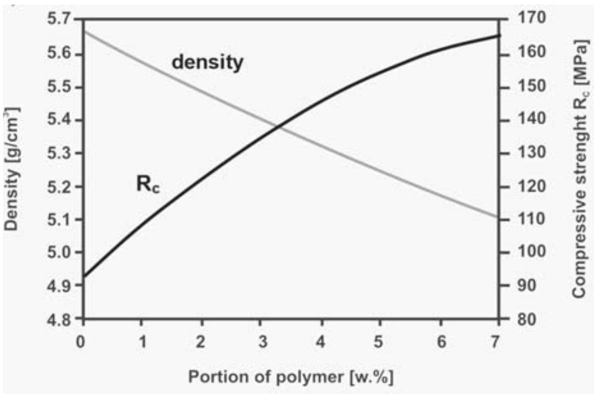
2.1. Material and technology

The experiments were made with the polymer matrix magnetic composite materials reinforced with particles of the powdered rapid quenched Nd-Fe-B ($Nd_{14.8}Fe_{76}Co_{4.95}B_{4.25}$) strip bonded with thermosetting epoxy resin (EP) and with the high density pressureless polyethylene (PEHD) (Table 1.). The amount of polymer matrix was 2.5% wt.. Advanced composite materials were compacted by the one-sided uniaxial pressing. Figure 2 shows examples of manufactured magnetic composite materials.

The following compacting process parameters were used:

- Cold pressing Nd-Fe-B – EP:
  - weighing of powders,
  - mixing of powders in „Shaker" 800 SPEX mixer 15 minutes,
  - pressing under the pressure 900 MPa at room temperature (5 minutes) (Fig. 3),
  - curing of the polymer matrix 180°C for 2 hours after compacting,
- Hot pressing Nd-Fe-B – PEHD:
  - weighing of powders,
  - mixing of powders in „Shaker" 800 SPEX mixer 15 minutes,
  - prepressing at room temperature under the pressure of 50MPa (1 minute) (Fig. 3),
  - heating up to the 170°C (temperature of HDPE processing),
  - hot pressing – 170°C, under the pressure 350 MPa (3 minutes).

a)



b)

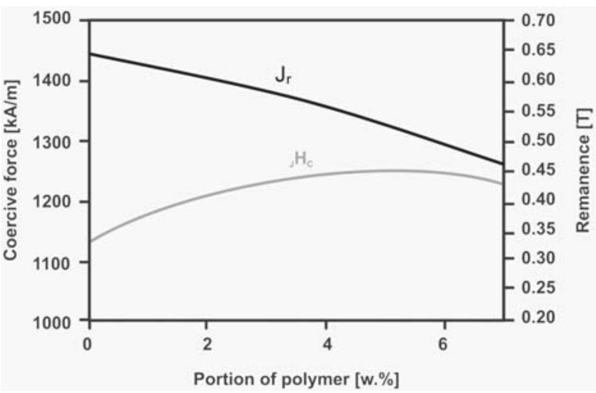


Fig. 1. Influence of the portion of polymer matrix on: a) mechanical properties of composite materials, b) magnetic properties of composite materials

Table 1.
Characteristic of binders used to manufacturing composite materials

Characteristic	Epoxy resin	High density polyethylene
Geometrical form	powder	powder
Grain size [μm]	50-500	50-650
Density [g/cm^3]	1.18	0.94
Tensile strength R_m [MPa]	27	20
Processing temperature [°C]	130-180	160-250

a)

b)

Fig. 2. Composite materials: a) Nd-Fe-B – EP b) Nd-Fe-B - PEHD

Fig. 3. Press for composite materials manufacturing

2.2. Methodology

Observations of morphology of powder were made in the SUPRA25 scanning electron microscope using the secondary electron detection at the 20 kV accelerating voltage.

The density of the composite materials was evaluated by determining the test piece mass using the analytical balance with the accuracy of $\pm 10^{-4}$ g and its volume basing on the apparent mass loss by immersing in water.

Resistivity of the composite materials was calculated from the following formula:

$$\rho = \frac{Rs}{l} = \frac{Us}{I \cdot l} = \frac{U\pi D^2}{4I \cdot l} \qquad (1)$$

where: U- power unit voltage [V], s– transverse section of the test piece [m^2], R- test piece resistance [mΩ], I – current passing through the test piece [A], D – test piece diameter [m], l – test piece length [m].

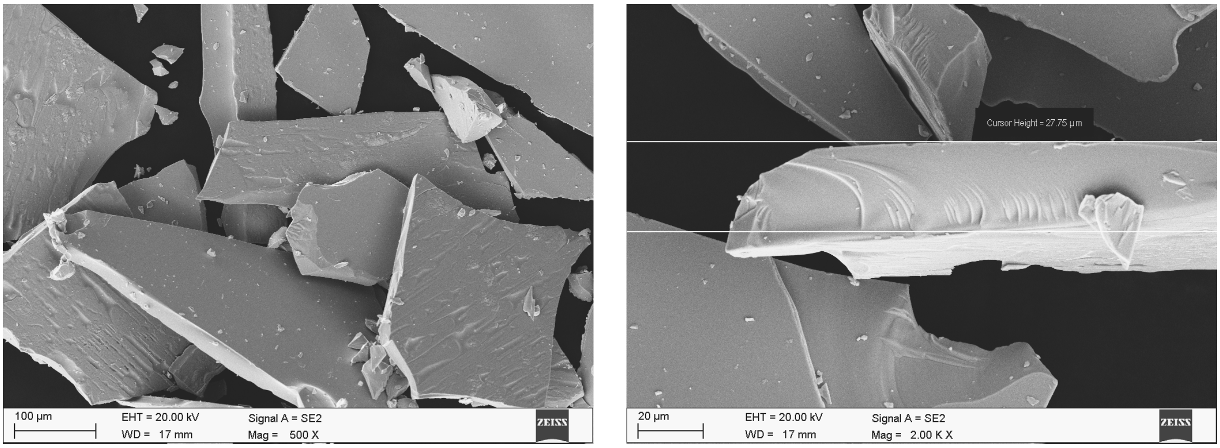
Compression tests were made on the ZWICK Z100 all-purpose testing machine at room temperature according to the standard PN-EN 10002-1:2004. Hardness tests of the composite materials were carried out with the Brinnell method using the 2.5 mm ball and load of 31.25 N.

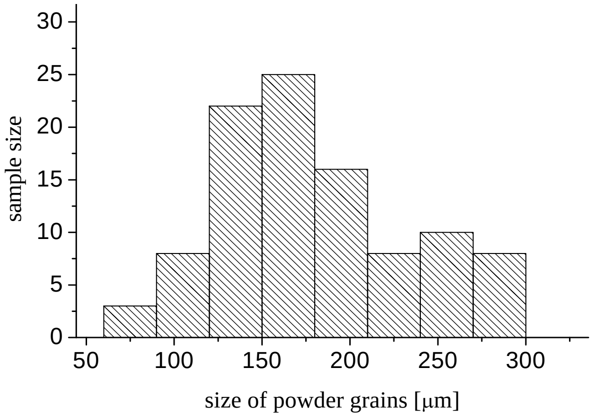
3. Results and discussion

The selection of the method of obtaining composites determines the following criteria:

- the process of compacting has been executed at the temperature which take into account the metastable structure of powders,
- this method enables to make elements with sophisticated shapes,
- during the compacting process the powder particles get closer to each other what increases their contact area and causes their mechanical meshing. That is the reason why the shape of particles has great influence on mechanical properties of obtained composite materials because the connection of their elements is made by the adhesion forces.

Manufacturing of composite materials by cold or hot one-side uniaxial pressing with polymer materials is a simple and not costly method taking into consideration low cost of polymer material and simplicity of their manufacturing. The technology of cold pressing of composite materials with epoxy resin in comparison with hot pressing with high density pressureless polyethylene does not need the preheating up to the polymer processing temperature but needs





Maximum	Minimum	Mean
275µm	70µm	180µm

Fig. 4. Morphology, distribution and particle size of $Nd_{14.8}Fe_{76}Co_{4.95}B_{4,25}$ powder

a) b)

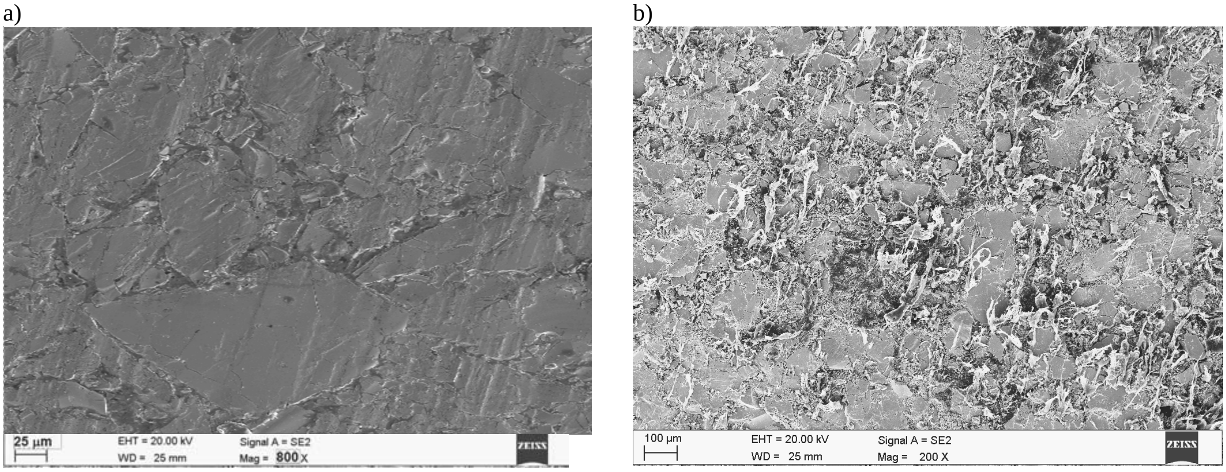


Fig. 5. Topography of the surface of composite materials: a) Nd-Fe-B – EP b) Nd-Fe-B– PEHD

Table 2.
Comparison of composite materials properties

Properties	Nd-Fe-B-EP	Nd-Fe-B-HDPE
Theoretical density [g/cm^3]	7.44	7.23
Density of composite materials [g/cm^3]	5.58	4.81
Ratio of density to theoretical density [%]	75.00	66.52
Resistivity [mΩ m]	323	392
Hardness [HBW]	35.0	25.0
Compressive strength [MPa]	112.1	50.2

higher pressure to consolidate the powders. There is also an additional operation – curing of the epoxy resin after the process.

Figure 4 shows morphology, distribution and powder size of $Nd_{14.8}Fe_{76}Co_{4.95}B_{4.25}$, observed in scanning electron microscope. The powder is characteristic of flaky shape grains from 70 µm to 275 µm. Figure 5 shows structure of composite materials observed in scanning electron microscopy. The distribution of magnetic material in polymer matrix were evaluated. It was found that the magnetic powders particles are uniformly distributed in the polymer matrix. During the compaction by the one-sided uniaxial pressing occurs the approaching of powders particles and causes the increase of their contact surface and mechanical meshing of powder's particles what allows acting of the adhesion forces. Occurrence of the small portion of pores was observed in the manufactured composite material, which attests to the good compacting of powders.

Density measurement results confirm it (Table 2). The density of composite material Nd-Fe-B – PEHD is equal to 66.5% of theoretical density while for composite material Nd-Fe-B – EP is observed even higher value – 75% of theoretical density. The density of composite materials depends on the pressing pressure, the higher pressure the higher ratio of density to theoretical density.

Examinations of mechanical properties of composite materials show that Nd-Fe-B –EP is characteristic of compressive strength – 112.1 MPa and hardness of about 35.0 HBW while Nd-Fe-B - PEHD shows 50.2 MPa and 25.0 HBW, respectively (Table 2). Chosen flaky shape of magnetic powder particles causes that during pressing, powder particles are meshing, that results with greater adhesion forces between powder particles than in the case of another shape of particles. The improvement of composite materials properties can be made by modification of technological process. The modification can be made both by choosing the component materials and changing the technological operation and treatment:

- selection of magnetic component,
- selecting of heat treatment making nanocrystalline structure,
- choice of tape's milling (kind of mill, time of milling),
- heat treatment and heat-magnetic treatment of nanocrystalline powders,
- selection of powder size,
- selection of binder,
- blending of components – choice of component portion in composite material,
- choice of compacting technology,
- optimization of geometrical form.

4. Conclusions

The research allows to develop the general technological process that makes possible to manufacture composite materials, consisting of the nanocrystalline powders of hard magnetic materials bounded by thermosetting or thermoplastic polymers, with required properties. This results from the high density of composite materials caused by the homogenous distribution of Nd-Fe-B powder in the polymer matrix and by the occurrence of the low portion of pores.

Manufacturing of composite materials by cold or hot one-side uniaxial pressing with polymer materials is a simple and not costly method taking into consideration low cost of polymer material and simplicity of their manufacturing. Bonded composite materials manufacturing technology is very simple and allows to form the properties of materials, lowering manufacturing costs because of no costly finishing and lowering of material losses resulting from the possibility of forming any shape. This method greatly expand the applicable possibilities of nanocrystalline powders of magnetically hard materials.

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