

## Magnetic properties of polymer bonded nanocrystalline powder

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### Materials

#### ABSTRACT

**Purpose:** The aim of this paper was to develop a dielectromagnetic based on nanocrystalline  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  powder bonded with organo-silicon polymer and to investigate the powder particle size and content of polymer response of the magnetic properties.

**Design/methodology/approach:** The dynamic magnetic properties at the frequency range from 50 Hz up to 100 kHz of nanocrystalline iron based powder cores were measured using computerized hysteresis loop tracer Remacomp C-100 and Ferrometr device.

**Findings:** It was found from the experimental studies, that nanocrystalline powder cores proved to be suitable for high frequency applications. Their frequency dependences are comparable to that of carbonyl iron powder cores but shows smaller power losses.

**Research limitations/implications:** Further studies should be undertaken in order to produce high density composites with good soft magnetic properties and to find a good compromise between mechanical and magnetic properties for power electronics applications.

**Practical implications:** Developed nanocrystalline powder cores with permeability's below 100 are potential candidates for a variety of industrial applications, such as electromagnetic interference filters, radio frequency coupling devices, filter inductors and radio frequency tuning cores.

**Originality/value:** Soft magnetic materials have recently regained interest as inductive component which is a result of better raw materials, more developed technologies and a need for the materials from electrical micromotors and low power motors for automation, robotics and other equipments. The present study complements and extends earlier investigations of polymer bonded powders.

**Keywords:** Composites; Nanocrystalline materials; Soft magnetic properties; Frequency spectrum

### 1. Introduction

Polymer bonded soft magnetic composites (often named also Soft Magnetic Composites – SMC, Ferromagnetic Composites Iron – FCI or Dielectromagnetics – DM) are manufactured from ferromagnetic powder particles pressed together with a dielectric binder using the powder metallurgy compaction process. In many experiments as ferromagnetic powders Fe, Fe-Ni, Fe-Si,  $\text{Fe}_3\text{P}$ , Fe-Si-Al, Mo-Permalloy,  $\text{Ni}_3\text{Fe}$  were used [1-4]. As dielectric binder one can use organic dielectric (epoxy resin, elastomers, etc.) or

inorganic binder (phosphates, oxides, glass, etc.) [4-9]. Such materials can easily be produced in various shapes with low technological losses at low prices.

The idea of magnetic cores made of a composites and an electrical sheet appeared almost simultaneously in the early 1880s. The dielectromagnetics were proposed by Fritts while laminated thin steel sheets were developed by Edison [10]. Stack laminations were rapidly used in medium power electrical motors while composites found applications in radio engineering and telecommunication. With the introduction of the soft magnetic ferrites into the magnetic materials market, research and production of magnetic cores of

dielectromagnetics were discontinued. However, electrical sheets and ferrites don't cover the full range of technical requirements associated with these applications. As proposed by Węgliński [10] this gap can be filled by dielectromagnetics, as illustrated in Figure 1.

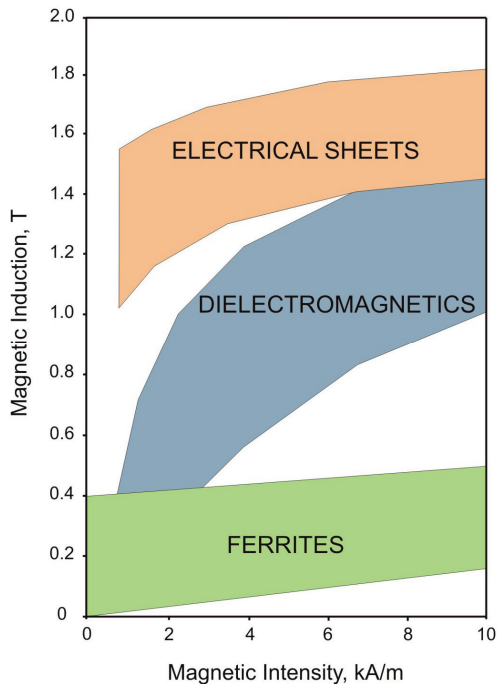


Fig. 1. Magnetization characteristic of electrical sheets, dielectromagnetics and soft ferrites [10]

Soft magnetic materials with various properties are fundamental elements in today's electronic and electrical engineering industry. Recently, increasing the operating frequency in electronic devices and increasing demand to save energy and reduce manufacturing costs has become more and more important. This led to replace, in some cases, the conventional materials with the newly developed amorphous and nanocrystalline based materials [1-4].

Reinforcement ferromagnetic composite with nanocrystalline  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  particles can lead to improve the magnetic characteristics in the high frequency range, especially power losses as compared to iron powder cores.

Magnetic composites in which ferromagnetic particles are insulated from each other with dielectrics are used in a wide array of applications from low cost electromagnetic interference filters, radio frequency coupling devices, smoothing chokes in switchmode power supplies to filter inductors and radio frequency tuning cores. Soft magnetic composites have yet greater potential to be used as magnetic cores for electrical converters or in an electrical micromotors and low power motors for automation, robotics and other equipment [7, 13-15].

The purpose of the present work is the preparation of toroidal shape nanostructured composite cores from high energy ball milled  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  powders and to investigate the powder particle size, their orientation in composite and content of polymer binder response of the soft magnetic properties (i.e. saturation induction, remanence, coercive field and permeability).

## 2. Experimental

Nanocrystalline  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  powder has been obtained by ball milling of metallic glass ribbons. The detailed synthesis process of nanocrystalline ferromagnetic particles (mechanical milling and thermal annealing) and its structural and magnetic characteristic were reported elsewhere [11,12].

Nanocrystalline powder particles were sieved in fractions ranging from 25 to 1200  $\mu\text{m}$ . Afterwards the Finemet powders were mixed with different contents of binder and cold compacted under applied pressure of 200 MPa. As a binder, an organo-silicon polymer DUBLISIL 20 from Dreve-Dentamid GMBH was used.

To obtain information about an effect of external magnetic field application during the polymerization on magnetic properties of composite cores, polymerization in accordance with two different methods was conducted (with no field and under magnetic field of 500 A/m).

Examination of magnetic properties (hysteresis loops) of polymer bonded nanocrystalline Finemet particles were carried out on the vibration sample magnetometer with a maximum applied magnetic field of 1200kA/m. The permeability of the composites cores and the frequency dependence of the permeability in the range 50Hz-100kHz were carried out on the Ferrometr and Remacomp C-100 devices.

## 3. Results and discussion

In the first series of experiments we investigated the effects of external magnetic field application during the polymerization on magnetic properties of composite cores with average powder particles diameter between 300-500  $\mu\text{m}$ . It was found that the toroidal core polymerized under magnetic field of 500 A/m exhibits better magnetic properties than the core consolidated with no field (Fig. 2). This is a consequence of preferential orientation of a  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  powder particles in a composite polymerized under magnetic field.

In the second series of experiments we investigated the effects of the organo-silicon polymer content (3, 5, 10 wt%) in combination with a fixed particles size 750-1200  $\mu\text{m}$ .

B-H curves of the obtained nanocrystalline cores are shown in Figure 3. The results of experimental studies demonstrated a correlation between the growing powder to polymer ratio and the improvement of the magnetic properties of the composite. The composite core with a polymer content of 3 wt% exhibit the coercive field  $H_c = 83$  A/m, the saturation induction  $B_s = 1.09$  T and the remanence  $B_r = 0.058$  T. Further decrease of powder to polymer ratio leads to deterioration of the magnetic properties, i.e. the coercive field goes considerably high at small values of both the remanence and the saturation magnetic induction: for example, for a composite core with a polymer content of 10 wt% the remanence was as low as 0.049 T, the saturation induction  $B_s = 0.95$  T whilst  $H_c = 214$  A/m. The following phenomenon may be caused by significant decrease of ferromagnetic material concentration in composite core.

Finally, we investigated the magnetic properties of polymer composites depending on the  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  particle size. Nanocrystalline particle size was varied between 25 and 1200  $\mu\text{m}$  mixed with an organo-silicon polymer content of 3 wt%.

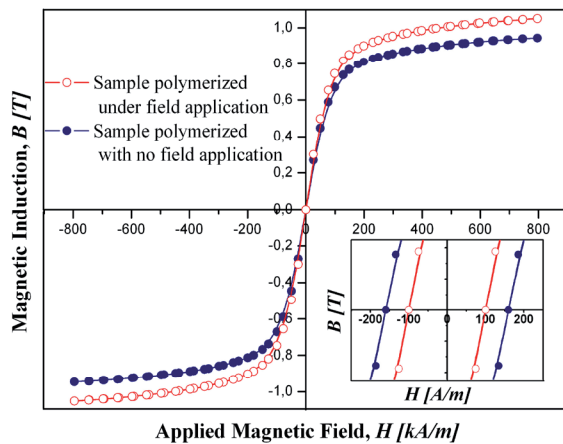


Fig. 2. Hysteresis loops for composite cores polymerized with no field and under magnetic field of 500 A/m

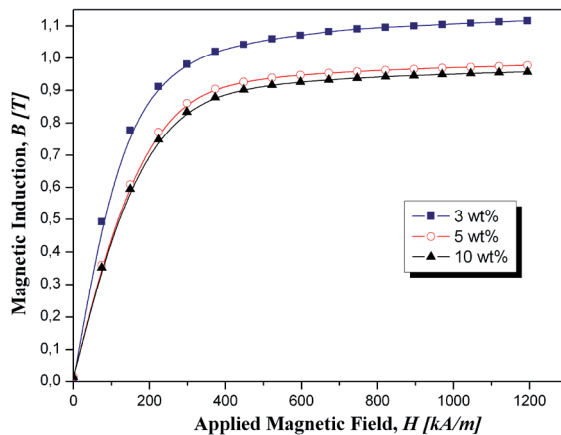


Fig. 3. The influence of dielectric content on the B-H curves of  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  composites

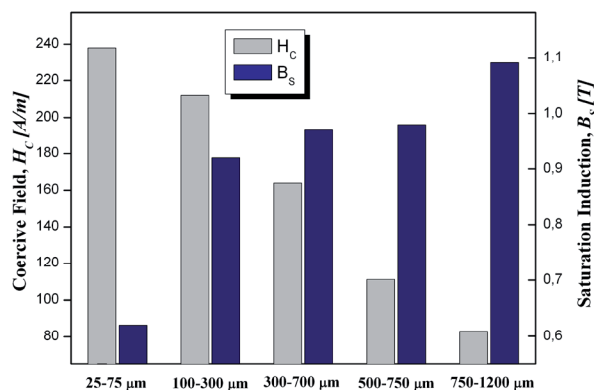


Fig. 4. Effect of  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  particles size on the saturation induction and coercive field of polymer composites

The composite core with powder particles between  $750 \div 1200 \mu\text{m}$  exhibited the coercive field  $H_c = 83 \text{ A/m}$ , the saturation induction  $B_s = 1.09 \text{ T}$  and the remanence  $B_r = 0.058 \text{ T}$ . By decreasing the particles size fraction to  $500 \div 750 \mu\text{m}$  the coercive field of ferromagnetic composite core is increased at low values of both the remanence and the saturation magnetic induction. For medium particle size ( $300 \div 500 \mu\text{m}$ ), composite core showed  $H_c = 164 \text{ A/m}$ , the value of  $B_s = 0.97 \text{ T}$  and the remanence  $B_r = 0.054 \text{ T}$ . Toroidal core filled with powder particles between  $100$  and  $300 \mu\text{m}$  demonstrated the coercive field  $H_c$  equal to  $212 \text{ A/m}$ , the saturation induction  $B_s = 0.92 \text{ T}$  and the remanence  $B_r$  value of  $0.052 \text{ T}$ . Further decrease particle size leads to deterioration of the magnetic properties of composite, i.e. the coercive field goes considerably high ( $H_c = 238 \text{ A/m}$ ) at small values of both the remanence ( $B_r = 0.032 \text{ T}$ ) and the saturation magnetic induction ( $B_s = 0.62 \text{ T}$ ).

The dependence of the nanocrystalline  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  particles size on coercive field and the saturation induction of polymer bonded powder particles is shown in Fig. 4.

The values of coercive field of polymer composites are mainly influenced by the structural defects induced by the ball milling process. Small  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  particles have a higher content of such structural defects in relation to their size than larger ones leading to an enhanced domain wall pinning. This is the main reason for the increase of the coercive field of polymer bonded small particles. Obviously these structural defects cannot be completely recovered by annealing. Additionally, higher demagnetization factor of small powder particles also caused a deterioration of magnetic properties.

The results of examination of the magnetic permeability dependence of frequency in the range  $50 \div 100 \text{ kHz}$  confirmed that permeability of the composite cores increases if the particle size increases (see Fig. 5). The permeability value of composite core with  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  particles between  $750 \div 1200 \mu\text{m}$  at frequency  $f = 50 \text{ Hz}$  was equal to 124. For medium particle size ( $300 \div 500 \mu\text{m}$ ), composite core showed  $\mu = 98$  (at  $f = 50 \text{ Hz}$ ) and for particles size between  $25 \div 75 \mu\text{m}$ , permeability of composite was equal 48. It was also noticed that the values of permeability of powder cores with a polymer addition of 3 wt% is nearly constant in a frequency range between 50 Hz and 10 kHz. At higher frequencies the eddy current losses reduce the permeability, but even at 100 kHz the composites show permeability between 100 and 40 depending on the particle size. This behaviour is interesting for technical use in high-frequency inductive components of the power electronic.

Comparison of power losses versus frequency showed that the losses increase with frequency as expected. The total power losses of composite core with  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  particles between  $750 \div 1200 \mu\text{m}$  at the magnetic induction  $B = 0.6 \text{ T}$  increased from 2.3 to 220 W/kg when the frequency was increased from 50 Hz to 10 kHz.

Summarizing the results of magnetic examination it has been concluded that magnetic permeability of  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  powder cores are comparable with those obtained on carbonyl iron or permalloy powder core and from this point of view, Finemet powder has no relevant advantage. On the other hand, nanocrystalline  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  powder cores exhibits lower power losses at the frequency range from 50 Hz to 100 kHz than any other material.

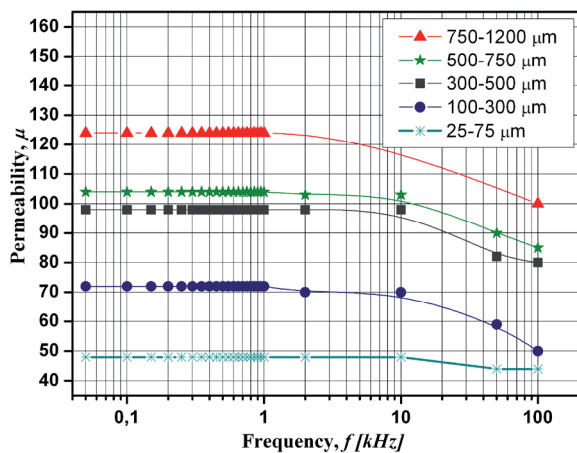


Fig. 5. Magnetic permeability (at  $H=3\text{kA/m}$ ) of the composite cores with different particles sizes as a function of frequency

## 4. Conclusions

Investigating the magnetic properties of organo-silicon polymer bonded nanocrystalline  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  powder particles we obtained that:

- Polymerization process made under magnetic field  $H = 500 \text{ A/m}$  ensured a preferential orientation of a powder particles in composites and caused the improvement of soft magnetic properties compared with composites polymerized without existence of magnetic field.
- The results of experimental studies demonstrated a correlation between the growing powder to polymer ratio and the improvement of the magnetic properties of the composite.
- The size and shape of the powder particles were responsible for soft magnetic properties of powder cores. Decreasing size of powder particles led to magnetic hardening of nanocrystalline  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  alloy. The main reasons for the worse magnetic properties of small particles were structural defects induced by the ball milling process and high demagnetization factor.
- The control of the soft magnetic properties of composite cores consolidated from nanocrystalline  $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  particles and organo-silicon polymer binder can be achieved by adjusting powder particle size and powder-to-silicon polymer ratio.
- The values of permeability of polymer bonded nanocrystalline powder particles are nearly constant in a frequency range between 50 Hz and 10 kHz.

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