

# Soft magnetic composite based on nanocrystalline Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> and Fe powders

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### **ABSTRACT**

**Purpose:** The main aim of this study is the producing of toroidal shape soft magnetic composite cores from nanocrystalline  $Fe_{73,5}Si_{13,5}Nb_3Cu_1B_9$  and Fe powders insulated with epoxy resin and to investigate the density and content of powder response of the soft magnetic properties.

**Design/methodology/approach:** Examination of magnetic properties of SMC was made on the Ferrometr device. The magnetic parameters were determined from hysteresis loops, permeability as a function of applied magnetic field and core losses as a function of magnetic induction characteristics.

**Findings:** The results of experimental studies demonstrated a correlation between the increase density and the improvement of the magnetic properties of the composite.

**Research limitations/implications:** The measurements of magnetic permeability should be conducted in a wide range of frequency.

**Practical implications:** The usability of composite cores as inductive component in electronic industry depends upon further investigations.

**Originality/value:** Soft magnetic composites based on iron powders are essential elements in today's electronic world. As far as we know, composites materials based on nanocrystalline material as soft magnetic materials have not been examined yet.

Keywords: Composites; Powder metallurgy; Magnetic properties; Powder cores; Nanocrystalline structure

#### **1. Introduction**

The endless efforts to improve the performance of electrical devices and to create the new high quality applications lead to the optimization of their design and application of existing magnetic materials such as soft magnetic composites (SMC). These materials are manufactured from iron and iron-based powder particles pressed together with a dielectric binder using the powder metallurgy compaction process, as shown in Fig.1 [1÷3]. In many experiments as ferromagnetic powders Fe, Fe-Ni, Fe-Si,

Fe<sub>3</sub>P, Fe-Si-Al, Mo-Permalloy, Ni<sub>3</sub>Fe were used [4]. As dielectric binder one can use organic dielectric (epoxy resin, elastomers, etc.) or inorganic binder (phosphates, oxides, glass, etc.) [4÷7].

The iron-based nanocrystalline alloys such as Finemet  $(Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9)$  exhibit very good properties as soft magnetic materials being the combination of advantages of conventional crystalline and amorphous competitors (e.g. high saturation induction, high permeability, low coercive field, low losses, low magnetostriction) [8÷10].

It can be suppose that by using  $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$  powder particles, it could be possible to obtain soft magnetic composite



Fig. 1. Composition and processing of soft magnetic composite [2]



Fig. 2. Factors affecting on final SMC properties [1]



Fig 3. Soft magnetic composites based on nanocrystalline  $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$  and Fe powders

with better magnetic properties when compared with the usual composite based on iron particles.

Usually, SMC have worse magnetic properties then ferromagnetic sintered ones, but they are characterized by high

electrical resisitivity and very small core losses, being from this point of view useful for high-frequency applications [4, 11]. Some technical applications of the soft magnetic composite materials as magnetic cores are presented in Refs.  $[12\div14]$ .

It is generally known that by varying the composition of an iron and iron-based powder composite material (e.g. purity of powder, particle size, microstructure, the amount of binder) and controlling the fabrication process (e.g. the compaction pressure and heat treatment) a material with suitable electromagnetic and mechanical properties can be achieved (Fig. 2).

As noticed in Ref. [11], besides the possibility of controlling material properties another advantage of SMC materials are isotropic behaviour. The thermal isotropy makes the thermal conductivity the same in all directions while the magnetic isotropy makes three-dimensional flux paths possible. A laminated material suffers from high anisotropy that makes only two-dimensional fluxpaths possible and also gives a big difference in thermal conductivity in a direction parallel to the laminations and a direction perpendicular to the lamination.

The pressing process also makes it possible to fabricate components with a complex geometry not possible by stacked laminations. The two steps of stamping and stacking in the manufacturing of laminated cores are replaced by a single step of pressing in a powder core.

The main aim of this study is the producing of toroidal shape soft magnetic composite cores from nanocrystalline  $Fe_{73.5}Si_{13.5}Nb_3Cu_1B_9$  and Fe powders insulated with epoxy resin and to investigate the density and content of powder response of the soft magnetic properties.

#### 2. Experimental details

In these experiments as a raw material nanocrystalline  $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$  (also known as Finemet) powder was used. Nanocrystalline powder has been obtained by ball milling of metallic glass ribbons. The synthesis process of nanocrystalline ferromagnetic particles (mechanical milling and thermal annealing) and its structural and magnetic characteristic were reported elsewhere [15, 16].

To improve compressibility of soft magnetic composites high purity iron powder (by Höganäs AB) was added with different

The characteristic of the	SMC cores used in experiments			
Material	Composition [%vol]		Pesin [%vol]	Processing
	$Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$	Fe		Trocessing
Core P1	70	30	5	cold compaction 350 MPa
Core P2				warm compaction 350 MPa + 443 K
Core P3	60	40		cold compaction 350 MPa
Core P4	00			warm compaction 350 MPa + 443 K
Core P5	50	50		cold compaction 350 MPa
Core P6				warm compaction 350 MPa + 443 K

Table 1 The characteristic of the SMC energy used in experimenta



Fig. 4. The average densities of soft magnetic composites with different volume fraction of  $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$  and Fe powders.

content. To obtain similar particle size  $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$  and Fe particles were sieved and then mixed in a high energy ball mill (8000 Spex). The average powders particle size in the particle size distribution curve (d=34.5  $\mu$ m) was made using a laser particle analyzer Analysette 22 (Fritsch).

As a dielectric binder an epoxy resin Epidian 100 was used. The resin was dissolved in an organic solvent. Through a covering process each individual Fe and  $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$  particles were covered with a thin layer of polymer.

The obtained mixtures were filled into the die and compacted using a hydraulic press at a surface pressure of 350 MPa. The composite powders were compacted in a toroidal shape with dimension of  $ø34 \times ø28 \times 6$  mm in order to determine magnetic parameters (Fig. 3). For some of the composites, curing of the resin was done under pressure at 443K, 1 hour, within the die in a hot press. Table 1 comprises the detailed characteristic of the manufactured SMC cores.

Examination of magnetic properties of SMC based on nanocrystalline  $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$  and Fe powders were made on the Ferrometr device. The magnetic parameters were determined from hysteresis loops, permeability as a function of applied magnetic field and core losses as a function of magnetic induction characteristics.



Fig. 5. Hysteresis loop for soft magnetic composite P6 obtained by compaction under pressure of 350MPa at 443 K.

#### **3. Results and discussion**

In the processing of soft magnetic composites, the green compact is sintered to decrease the porosity and to increase the density, which depends on the kind of powder materials, kind of insulation/binder and the sintering conditions. Sintering temperature of nanocrystalline powder mixed with epoxy resin is limited due to the melting point of resin and the metastable state of nanocrystals. Annealing at high temperatures causes grain growth and soft magnetic properties decrease. However for a green compact made from mixed composite powders, the density change is affected not only by the sintering conditions but also by the combination and volume fraction of the composite powders.

Fig. 4 shows the results of density measurements of the obtained soft magnetic composites. The SMC average densities are in the range  $5.88 \div 6.74$  g/cm<sup>3</sup>. It was noticed, that density increases with increasing volume fraction of iron powder.

This behaviour could be explained by the low compressibility of the  $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$  powder produced by mechanical milling.

The saturation induction  $B_s$  and coercive field  $H_c$  were determined from magnetic hysteresis loops of composites measured on the Ferrometr device (Fig. 5). The coercive field



Fig. 6. The coercive field of soft magnetic composites with different volume fraction of  $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$  and Fe powders.



Fig. 8. The permeability as a function of applied magnetic field of soft magnetic composites with different powder fraction.

(Fig. 6) and the saturation induction (Fig. 7) of SMC cores in dependence on volume fraction of powders and processing methods are compared.

It can be seen that the coercive field decrease and the saturation induction increase with increasing volume fraction of iron powder. This improvement of soft magnetic properties is result of the highest densities of the soft magnetic composite cores.

The results of experimental studies also demonstrated differences in magnetic properties of core samples with the same powders compositions. The composite core P5 with Fe particles content of 50 %vol. exhibit  $H_c = 863$  A/m and  $B_s = 0.73$  T.

For comparison, the composite P6 curried at 443 K exhibit coercive field  $H_c = 780$  A/m and saturation induction  $B_s = 0.83$  T. This fact may be a consequence of the removal the compaction induced internal stresses during heat treatment of the composite.

The magnetic permeability versus magnetic field (Fig. 8) has been measured at maximum applied magnetic field of 5800 A/m and the obtained values are shown in Fig. 9 as a function of powders compositions and processing methods.

The permeability values of composites P1 and P2 with Fe<sub>73,5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13,5</sub>B<sub>9</sub> particles content of 70 %vol. (measured at



Fig. 7. The saturation induction of soft magnetic composites with different volume fraction of  $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$  and Fe powders.



Fig. 9. The permeability of soft magnetic composites with different volume fraction of  $Fe_{73}$  <sub>5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13</sub> <sub>5</sub>B<sub>9</sub> and Fe powders.

frequency f = 50 Hz) are  $\mu = 83$  and  $\mu = 106$  respectively. By increasing the Fe particles content in composite, the maximum permeability is increased as a consequence of the high density of the composite core. The sample P6 with Fe<sub>73,5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13,5</sub>B<sub>9</sub> powder particles content of 50 %vol. exhibit magnetic permeability  $\mu = 167$  (measured at f = 50 Hz).

Likewise to Ref. [4] the significant increase of the magnetic permeability versus the composites density suggests that an improvement of the composites density could produce an important increase of the permeability. This could be obtained by an improvement of the  $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$  nanocrystalline powder and Fe particles compressibility.

According to Ref. [1] magnetic permeability of SMC depends not only on a range of microstructural features of the materials but especially on the additional distributed air-gaps introduced by the insulating powder particles. It was noticed [1] that certain ferromagnetic powder compacted to a certain density should have constant permeability conditioned by the coating thickness in combination with the total surface area of the powder. It can be observed, in Fig. 9, some differences in permeability values of core samples (e.g. P5 and P6) with the same powders compositions and densities.

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Fig. 10. The core losses as a function of magnetic induction of soft magnetic composites with different powder fraction.

The permeability is higher for cores compacted at 443 K. This phenomenon could be explained by the fact that during warm compaction (at 443 K) the internal lubrication was improved leading to that ferromagnetic particles come closer to each other. The result will be decreased distributed air-gaps and effected on permeability increase.

The core loss versus magnetic induction (Fig. 10) has been measured at frequency of  $\overline{50}$  Hz and the obtained values at induction B = 0.5 T are shown in Fig. 11 as a function of powders compositions and processing methods.

It can be seen that the core losses of soft magnetic composites decrease with the increasing volume fraction of iron powder. The highest core loss (measured at frequency of 50 Hz and at induction B = 0.5 T)  $P_w = 15.5$  W/kg was reported for core P1 with Fe73,5Cu1Nb3Si13,5B9 particles content of 70 %vol. and compacted under pressure of 350 MPa. The lowest core loss  $P_w = 6.1$  W/kg was reported for core P6 with Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> particles content of 50 %vol. and warm compacted under pressure of 350 MPa at 443 K.

According to Ref. [4] the low core losses could be explained by the high electrical resistivity of obtained cores.

#### 4.Conclusions

Investigating the magnetic properties the SMC cores based on nanocrystalline Fe73.5Si13.5Nb3Cu1B9 and Fe powders insulated with epoxy resin we obtained that:

- SMC density increases with increasing volume fraction of iron powder, which could be explained by the low compressibility of the Fe<sub>73.5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13.5</sub>B<sub>9</sub> powder produced by mechanical milling.
- The results of experimental studies demonstrated a correlation between the increase density and the improvement of the magnetic properties of the composite.
- Warm compaction process made under pressure of 350 MPa at temperature 443 K caused the improvement of soft magnetic properties compared with core samples obtained in cold compaction process.



Fig. 11. The core losses of soft magnetic composites with different volume fraction of Fe<sub>73 5</sub>Cu<sub>1</sub>Nb<sub>3</sub>Si<sub>13 5</sub>B<sub>9</sub> and Fe powders.

The usability of composite cores as inductive component in electronic industry depends upon further investigations (especially in a wide range of frequency) and improvement of magnetic properties.

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