# MAGNETIC PROPERTIES OF SINTERED POWDER CORES BASED ON Fe-Ni

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# 1. Introduction

The term "composites" is a simplified way of describing the combining of unique properties of different materials to produce synergistic effects. A combination of materials is needed so that certain properties can be adapted to any area of application. There has been an everlasting desire for composite materials to be made stronger, lighter or more durable than traditional materials [1]. Excellent soft magnetic material based on Fe-Ni can be a basis to create attractive composites.

Fe-Ni alloys belong to the most versatile soft magnetic materials, as the magnetic properties can be controlled within wide limits by suitable processing and choice of composition. The soft magnetic Fe-Ni materials of technical relevance are found in the range of 30-80 wt. % of Ni. These alloys are known with the best soft magnetic properties, such as exceptionally high permeability, lowest coercivity and a variety of hysteresis loop shapes. Materials with a Ni-content of around 50 wt. % (called Permenorm) are characterized by a maximum in the saturation polarization of up to 1.6 T and medium range permeability [2].

## 2. Experimental

As ferromagnetic material thin foil  $Fe_{50}Ni_{50}$  (wt. %) was used, cut into the small pieces appropriate for milling in planetary ball mill with hardened steel vials and balls. The parameters of milling were: BPR 9:1, 200 r.p.m. and 5 h milling time. We prepared two different samples, the first one, metallic, by compaction of pure powder (named as CP) and second one as a mixture of FeNi powder with ATM phenol-formaldehyde resin (23 vol. %), named as COM. The powders were pressed into the ring-shape samples at 800 MPa for 15 sec. Pressed COM sample was cured at 165 °C (below the limit for ATM deterioration) for 1h in air atmosphere. The metallic sample was heat treated as follows: firstly the temperature increased by 1°C/min up to 400°C, where it was held for 30 min. In the second phase the temperature was being increased by 2°C/min up to 1180°C (sintering temperature) and held for 1h.



Fig.1: Sample preparation process (thin foil, cut foil, powder, rings and rings prepared as a coil with turns for ac and dc measurements).

Specific resistivity of samples was determined by four contact method. The coercivity was measured by Foerster Koerzimat 1.097HCJ. The dc hysteresis loops were measured by the fluxmeter based dc-hysteresisgraph. The ac hysteresis loops were measured by three different hysteresisgraphs: in the frequency range 0.1 Hz - 1 Hz by the fluxmeter based achysteresisgraph, in the frequency range 1 Hz-1 kHz by a permeameter Laboratorio Elettrofisico Walker LDJ Scientific AMH-1K-S and in the frequency range 1 kHz - 3.1 kHz by a hysteresisgraph MATS-2010 A. The hysteresis losses  $W_{dc}$  were calculated as the dc hysteresis loop area.

## 3. Results and discussion

Tab. 1 summarizes the parameters and properties of the prepared samples, volume fractions of the constituents and pores, outer diameter D, inner diameter d, height h, mass m, specific resistivity  $\rho$ , the coercivity H<sub>C</sub>, hysteresis losses W<sub>dc</sub> at maximum induction of 0.2 T.

| Sample                                | СР                   | СОМ                  |
|---------------------------------------|----------------------|----------------------|
| FeNi fraction (vol. %)                | 79.86                | 68.55                |
| ATM fraction (vol. %)                 | 0                    | 23                   |
| pores fraction (vol. %)               | 20.14                | 10.98                |
| D (mm)                                | 23.91                | 25.12                |
| d (mm)                                | 17.58                | 17.95                |
| h (mm)                                | 2.44                 | 2.29                 |
| m (g)                                 | 3.36                 | 3.35                 |
| ρ (Ω.m)                               | 7.9×10 <sup>-5</sup> | 1.2×10 <sup>-2</sup> |
| $H_{C}(A/m)$                          | 98.2                 | 1212                 |
| $W_{dc}$ at 0.2 T (J/m <sup>3</sup> ) | 10.20                | 219.89               |

Tab. 1. Parameters and properties of sample.

The structure of samples was investigated by a Carl Zeiss SEMEVO MA15, Fig. 2.



Fig.2: Sample images of CP sample (left) and COM obtained by SEM.

The higher resistance of the CP sample as the resistance of the foil is due to the high porosity and non-homogeneous electrical contacts between the individual powder particles of the sample. The ATM resin separates metallic particles in COM sample and causes significant increase of the resistivity.

The higher value of the coercivity of COM sample is mainly caused to the limitation of the magnetic interaction between well insulated sample particles.

The total energy losses  $W_{tot}$  can be divided (according to the Bertotti's statistical model) into following components, dc losses (hysteresis losses)  $W_{dc}$ , interparticle classical losses (losses due to eddy currents flowing in the cross section perpendicular to magnetic flux)  $W_C^{inter}$ , intraparticle classical losses (losses due to eddy currents inside the particles)  $W_C^{intra}$  and excess losses coming from eddy currents due to domain walls displacements (known also as anomalous losses)  $W_e$  [3]:

$$W_{tot} = W_{dc} + W_c^{inter} + W_c^{intra} + W_e$$
 Eq. (1).

The total energy losses  $W_{tot}$  of both samples were measured at maximum induction of 0.2 T and in the frequency range from 1 Hz to 3100 Hz and are plotted in Fig. 3.



Fig.3: Total losses  $W_{tot}$  of both samples as a function of frequency. The symbols denote experimental results and lines are fits according to Eq. (2) and Eq. (3).

As expected, total losses of both samples increase with the frequency. The core losses for the sample CP are lower at lowest frequencies, but increase steeper than that for sample COM The crossing point of the core losses dependences of the samples is approx. at 3100 Hz, when the COM sample becomes to be more suitable as soft magnetic material for medium frequency application.

We expect that the eddy currents flow in the whole cross section of the CP sample, the intra-particle eddy current can be neglected and the formula for total energy losses can we written as follows

$$W_{totCP} = A_{CP} + B_{CP}f + C_{CP}f^{1/2}$$
 Eq. (2)

The anomalous losses component proportional to  $f^{1/2}$  is typical for homogenous materials as FeSi sheets.

The total energy losses for SMCs usually has anomalous losses component proportional linearly to frequency

$$W_{totCOM} = A_{COM} + B_{COM} f$$
 Eq. (3)

The experimental results for CP sample were fitted using Eq. (2) and COM were fitted using Eq. (3). Fitted values for coefficients and are in Tab. 2.

| parameter | A <sub>CP</sub> | B <sub>CP</sub> | C <sub>CP</sub> | A <sub>COM</sub> | B <sub>COM</sub> |
|-----------|-----------------|-----------------|-----------------|------------------|------------------|
| value     | 11.38           | 0.147           | 1.21            | 254.44           | 0.097            |

Tab. 2. The values for the parameters of fitted functions.

We can see, by the comparing of the values of parameters  $B_{CP}$  and  $B_{COM}$  that the addition of the ATM resin to the ferromagnetic samples leads to the decreasing of the eddy current losses in the samples. Parameter  $B_{COM}$  also includes the component of anomalous energy losses in the composite sample COM. The values of the parameters  $A_{CP}$  and  $A_{COM}$  are dc losses and it is clear that the higher value is reached for sample COM with higher coercivity.

The total energy losses  $W_{tot}$  of both samples as a function of the maximum induction  $B_m$  in the range to 0.6 T are plotted in Fig. 4 for two different frequencies (20 Hz and 800 Hz).



Fig.4: Total losses  $W_{tot}$  both samples as a function of maximum induction to 1.2 T. The symbols denote experimental results and lines are fits according to Eq. (4).

The range of validity of the Steinmetz law

$$W_{tot} = L_S (B_m)^{B_S}$$
 Eq. (4),

where  $L_s$  and  $B_s$  are parameters was detected for both samples [4]. The calculated parameters are in Tab. 3.

| Sample | L <sub>s</sub> (20 Hz) | B <sub>s</sub> (20 Hz) | L <sub>S</sub> (800 Hz) | B <sub>s</sub> (800 Hz) |
|--------|------------------------|------------------------|-------------------------|-------------------------|
| СР     | 643.71                 | 2.17                   | 9380.51                 | 2.82                    |
| COM    | 3523.21                | 1.61                   | 6519.30                 | 1.94                    |

Tab. 3. The parameters in Steinmetz law for sample CP and COM.

The exponent  $B_s$  in the Steinmetz law is usually expected in the range of 1.5 to 3. The value of 2.17 for metallic sample CP is close to values for exponents valid in Fe-rich alloys (as FeSi non-oriented and grain –oriented steels) [5]. The  $B_s$  constant for COM sample equal to 1.61 is very close to the value of 1.5 obtained for Fe-based composites [6].

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