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The Influence of $NiZnFe_2O_4$ Content on Magnetic Properties of Supermalloy Type Material

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Soft magnetic composites represent a remarkable kind of materials with wide variety of use. Magnetic properties are dependent on the materials composition and also on the method of preparation. Ni–Fe–Mo alloys (supermalloy) have high complex permeability and low eddy current losses. Soft magnetic $NiZnFe_2O_4$ ferrites have low coercivity and intermediate saturation magnetization. The $Ni_{80}Fe_{14.7}Mo_{4.5}Mn_{0.5}Si_{0.3}$ (wt%) powder sample was prepared by mechanical alloying of the chemical elements for 24 h. $Ni_{0.3}Zn_{0.7}Fe_2O_4$ ferrite is commercially available by Sigma Aldrich. Both powders were mixed at selected ratio and uniaxially compacted at 800 MPa. In this paper, we report the experimental observations of the effects of $Ni_{0.3}Zn_{0.7}Fe_2O_4$ content on the electromagnetic properties of NiFeMoMnSi/$Ni_{0.3}Zn_{0.7}Fe_2O_4$. The samples contained 5, 10, and 15% of $Ni_{0.3}Zn_{0.7}Fe_2O_4$ ferrite and were sintered for 30 min at 800 °C. An addition of $Ni_{0.3}Zn_{0.7}Fe_2O_4$ ferrite caused decrease of complex permeability and increase coercivity of the samples. The 5% of $Ni_{0.3}Zn_{0.7}Fe_2O_4$ sample exhibits the highest value of the real part of complex permeability (48 at 1 kHz). The 10% of $Ni_{0.3}Zn_{0.7}Fe_2O_4$ sample showed the lowest total magnetic losses.

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1. Introduction

Soft magnetic composites (SMCs) are studied for their interesting properties and understanding of magnetization processes leads to improved electromagnetic properties of components for electromagnetic application. SMCs consist of ferromagnetic particles surrounded by thin electrical insulating layer and are compressed into the desired shape using conventional powder metallurgy method. SMCs can be used as ferromagnetic part of high frequency power electronics, transformers, relays, solenoids, sensors, particularly in direct current magnetic field applications and other devices [1]. Because of the wide area of applications of soft magnetic materials and increasing standard on the quality of components and equipments in recent years, there has been progress in their investigation.

The functions of insulating layer are insulation and binder. Type and amount of insulating layer and other preparation conditions of composites affect on resulting properties of SMC [2–4]. Recent studies indicate soft magnetic ferrites as suitable insulating material [5, 6].

In this paper, the base ferromagnetic material is supermalloy powder with nominal composition $Ni_{80}Fe_{14.7}Mo_{4.5}Mn_{0.5}Si_{0.3}$ (in wt%) and as insulating layer of particles is $Ni_{0.3}Zn_{0.7}Fe_2O_4$ ferrite. Supermalloy is characterized by high permeability, low coercivity and nearly zero magnetostriction. Eddy current losses of ferrite are significantly lower than those of ferromagnetic material due to lower electrical conductivity, hence are used in high frequency applications [7]. The prepared samples differed in ferrite content.

2. Experimental

The samples of SMCs were prepared from two materials. The first component was supermalloy powder, this material was prepared by mechanical alloying process. The resulting blend of elements with nominal composition $Ni_{80}Fe_{14.7}Mo_{4.5}Mn_{0.5}Si_{0.3}$ [wt%] was mechanically alloyed. The milling was conducted in hardened steel vial with steel balls in air atmosphere. The ratio of balls to powder was 15:1, the rotational speed of vial was 350 rpm. Resulting density of the powder was 8.282 g/cm^3 [8]. The second component of SMC was $Ni_{0.3}Zn_{0.7}Fe_2O_4$ ferrite, material was commercially distributed by Sigma Aldrich with density 4.427 g/cm^3. The supermalloy particles were covered by $Ni_{0.3}Zn_{0.7}Fe_2O_4$. The sol–gel method was used for $Ni_{0.3}Zn_{0.7}Fe_2O_4$ ferrite preparation. The equimolar ratio of metal nitrates and citric acid was dissolved in distilled water. pH = 6 was adjusted by NH_4OH (26% aq.) after total dissolution of citric acid. The roughly 100 ml of the prepared aqueous solution was heated at 70 °C for 4 h until the viscous gel was created. The prepared gel was mixed with supermalloy particles and heated at 200 °C for 12 h in laboratory dryer.

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The powder was compacted at 800 MPa into the toroidal and cylinder shapes. The samples were heat treated at 800 °C for 30 min in the microwave furnace under nitrogen atmosphere. Dimensions of the toroidal samples were: outer diameter of 24 mm, inner diameter of 17 mm and height of 3 mm. Dimensions of cylinder samples possess height of 3 mm and diameter of 10 mm.

For the microstructure and morphology investigation of the final composite samples there was used scanning electron microscope (SEM) JEOL JSM-7000F. The cylinder samples were used for measurement of coercivity by Foerster Koerzimat 1.097 HCJ. The toroidal samples were used for measurement of specific electrical resistivity by four point probe method. The densities were evaluated by dimensions measurements and mass of the sintered bodies. The impedance bridge (HP4194A) was used for complex permeability measurement in the frequency range from 1 kHz to 40 MHz. The Permeameter AMH-1K-S was used for measurement of AC hysteresis loops within frequency range from 290 Hz to 1000 Hz, at maximum induction of 0.1 T. Total hysteresis loss was determined in J/m^3 as an area of the hysteresis loop.

3. Results

Table I shows a comparison of $Ni_{0.3}Zn_{0.7}Fe_2O_4$ content, specific electrical resistivity, density, porosity and coercivity of the prepared samples. Although the specific electrical resistivity of $Ni_{0.3}Zn_{0.7}Fe_2O_4$ is higher than for NiFeMo, the sample with 15% content of $Ni_{0.3}Zn_{0.7}Fe_2O_4$ exhibits lower specific electrical resistivity than the sample with 10% which causes worse coverage of NiFeMo particles due to clustering of ferrite particles. SEM examination of Supermalloy powder coated by 5% $Ni_{0.3}Zn_{0.7}Fe_2O_4$ ferrite is in Fig. 1. The morphology of sample (5% of $Ni_{0.3}Zn_{0.7}Fe_2O_4$) after the compaction is shown in Fig. 2. Supermalloy particles were observed to be of irregular shapes with random orientation.

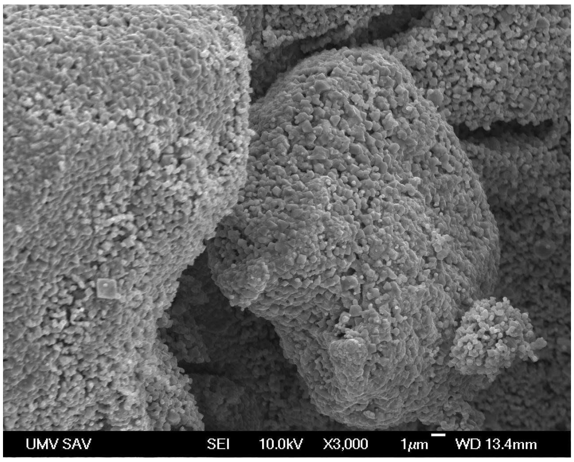

Fig. 1. The particles of supermalloy powder covered by 5% of $Ni_{0.3}Zn_{0.7}Fe_2O_4$ ferrite.

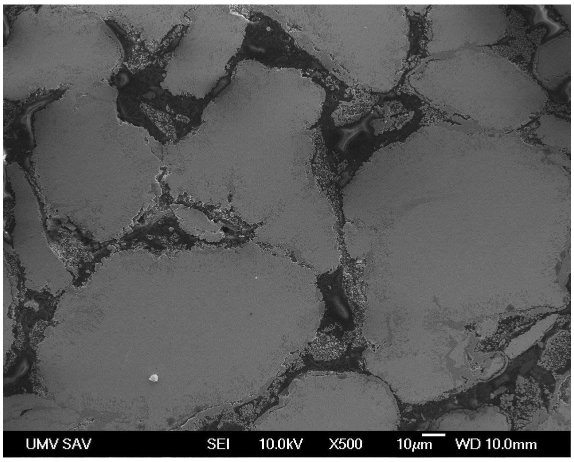

Fig. 2. The surface of the compressed toroidal sample with 5% of $Ni_{0.3}Zn_{0.7}Fe_2O_4$.

TABLE I

The values of $Ni_{0.3}Zn_{0.7}Fe_2O_4$ content [%] (I), specific electrical resistivity [Ωm] (II), density [g/cm^3] (III), porosity [%] (IV) and coercivity [A/m] (V) of prepared samples.

Sample	1	2	3
I	5	10	15
II	7.69×10^{-6}	2.57×10^{-5}	1.14×10^{-5}
III	5.26	5.17	5.37
IV	35.01	34.51	34.48
V	558	698	803

The increasing amount of ferrite as electrically insulating layer between particles of composite is leading to decrease of the real part of complex permeability. The 5% $Ni_{0.3}Zn_{0.7}Fe_2O_4$ sample shows the highest value of the real part of complex permeability (48 at 1 kHz) (Fig. 3). The imaginary part of complex permeability is also illustrated in Fig. 3. This permeability dispersion is mainly

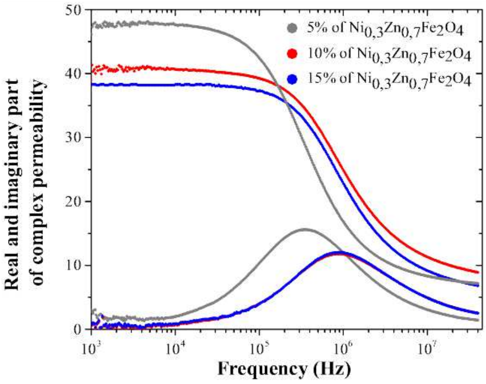

Fig. 3. The dependence of real and imaginary part of complex permeability on frequency for samples with 5, 10, 15% content of $Ni_{0.3}Zn_{0.7}Fe_2O_4$.

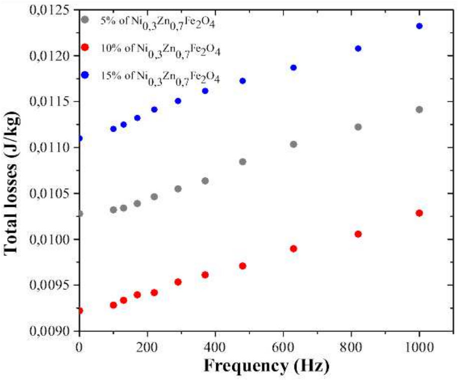

Fig. 4. The dependence of total losses on frequency for samples with 5, 10, 15% content of Ni$_{0.3}$Zn$_{0.7}$Fe$_2$O$_4$ at maximum magnetic induction 0.1 T.

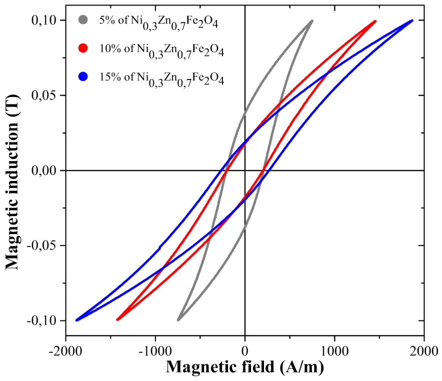

Fig. 5. The comparison of hysteresis loops of the toroidal samples at frequency of 370 Hz at maximum magnetic induction of 0.1 T.

caused by domain walls motion under small magnetic field. The samples with 10% and 15% of Ni$_{0.3}$Zn$_{0.7}$Fe$_2$O$_4$ exhibit the same relaxation frequency, i.e. the frequency at the peak in μ'' (920 kHz). Relaxation frequency in ferromagnetic material should follow the relation:

$$f_r = \frac{4\rho}{\pi\mu_0\mu_i d^2}, \qquad (1)$$

where ρ is specific electrical resistivity, μ_0 is the magnetic constant, μ_i is the static value of initial permeability and d is the average dimension of an area, where eddy currents are flowing [9]. The f_r of the sample containing 10% of Ni$_{0.3}$Zn$_{0.7}$Fe$_2$O$_4$ is expected to be higher than other two samples due to the largest value of specific resistivity (see Table I). The obtained results however report that its value is comparable with 15% Ni$_{0.3}$Zn$_{0.7}$Fe$_2$O$_4$. It can be attributed to the higher value of d as a result of lower ferrite insulating layer, slightly higher μ_i versus more than twice higher resistivity, which creates a balance between the quantities in Eq. (1).

Hysteresis loss is the main part of total magnetic losses at low frequencies for SMC materials. The lowest total magnetic losses are observed for the 10% Ni$_{0.3}$Zn$_{0.7}$Fe$_2$O$_4$ sample due the lowest hysteresis loss, Fig. 4. The comparison of hysteresis loops of prepared samples at frequency of 370 Hz at maximum magnetic induction of 0.1 T is displayed in Fig. 5.

5. Conclusions

In this paper, the basic electromagnetic properties of prepared SMCs samples were studied with different amounts of ferrite used as an insulating layer of the composite. The sample containing the 5% of Ni$_{0.3}$Zn$_{0.7}$Fe$_2$O$_4$ exhibits the highest value of the real part of complex permeability (about 48 at 1 kHz). The increasing content of ferrite results in a decrease of the real part of complex permeability up to approximately 200 kHz. Hysteresis loss influences the total magnetic losses in our studied frequency region. The lowest total losses were found for 10% Ni$_{0.3}$Zn$_{0.7}$Fe$_2$O$_4$ sample.

Acknowledgments

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