

Magnetic Properties of Sintered Fe₅₀Co₅₀ Powder Cores

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We investigated coercivity, total losses and complex permeability of sintered Fe–Co powder cores to detect magnetization processes performing in ac magnetic field. The Fe–Co solid solution alloy powders with 50:50 wt% ratio were prepared by 1, 15, and 20 h alloyed mixture of pure chemical elements in planetary ball mill. The resulting powder was subsequently sintered into a disk form. The compaction was performed at a pressure of 800 MPa for 5 min at temperatures of 400 °C, 500 °C and 600 °C in vacuum oxidation protective atmosphere, with pressure of 5×10^{-3} Pa. The best magnetic properties exhibit sample prepared from 1 h alloyed powder, compacted at 600 °C.

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1. Introduction

Crystalline Fe-based alloys combine the properties of various soft magnetic materials, such as high permeability and high saturation induction [1]. These properties combinations are the result of the structural correlation length which is much smaller than the ferromagnetic one.

Fe–Co based alloys display interesting magnetic properties very advantageous for high temperature applications [2]. Fe–Co soft magnetic alloys have low coercivity, low hysteresis loss, low eddy current loss, high permeability, high saturation magnetization, and high Curie temperature. In particular Fe₅₀Co₅₀ alloy has a considerably large permeability, low magnetocrystalline anisotropy and low electrical resistivity [3].

The crystalline Fe–Co material prepared by usual methods in the form of a sheet is brittle which limits its application. One of the ways to prepare bulk material suitable for the application is compaction of the powder produced by mechanical milling or mechanical alloying. The mechanical milling was proved to be an effective technique for the synthesis of nanostructured powders of various metal–metal systems. This useful technique can produce a variety of equilibrium and non-equilibrium alloy phases leading to size reduction and particle shape modification. The advantage of this process technology is that the powder can be produced in large quantities and the processing parameters can be easily controlled [4]. Some researches concerning Fe–Co powders system produced by mechanical milling were reported in [3, 5–7].

2. Experimental

Commercial Fe and Co powders with particle size of 1.6 to 10 μm , mixtures in ratio 50:50 (wt%), were milled in a high-energy planetary ball mill (RETSCH PM4000) with hardened steel balls and vials at speed of the milling 180 rpm. The mechanical milling was performed in argon atmosphere with ball-to-powder ratio of 15:1 for time of the milling 20 h.

The crystalline structure of the milled powder was investigated by X-ray diffraction using a Philips PW1050 diffractometer with Fe filtered Co K_{α} radiation and the creation of α -Fe phase was confirmed. After alloying the diffraction peaks of pure elements hcp Co and fcc Co phases completely disappear due to diffusion of Co into bcc Fe structure forming the Fe–Co solid solution [6].

The bulk samples were prepared from these powders in the form of cylinders (with diameter of 10 mm, height of 2.5 mm and weight of approximately 2 g). The compaction was performed at a pressure of 800 MPa for 5 min. During the compaction process the powders were heated with constant rate of 60 K min^{-1} to the final consolidation temperature ranged from 400 °C to 600 °C. In order to prevent oxidation and to remove free gases in the compacted samples, the compaction was performed in vacuum of 5×10^{-3} Pa.

For further measurements and investigations of magnetic properties, bulk samples were cured at a temperature of 1000 °C for 1 h in an electric furnace in argon atmosphere.

The coercivity of bulk samples was measured by a Foerster Koerzimat 1.097 HCJ at room temperature.

An axial hole with diameter of 5 mm was drilled into the cylinders, which produced a ring samples and we have prepared coils with number of turns 15 for mea-

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surement of ac complex permeability. Complex permeability spectra were assigned with an impedance analyzer (HP 4194A) from 200 Hz to 10 MHz with two terminal connection configuration.

Two coils were wound in the ring-shaped samples (number of primary turns 20, number of secondary turns 10) for the measurement of total core losses at maximum flux density of 0.4 T. The total losses were measured in the frequency range 2–400 Hz by AMH-1K-S Permeameter, Laboratorio Elettrofisico.

3. Results

The aim of this work was to study the magnetic properties (coercivity, total energy losses, complex permeability) of ball-milled and subsequently compacted $\text{Fe}_{50}\text{Co}_{50}$ alloys.

The values of the coercivity for bulk samples $\text{Fe}_{50}\text{Co}_{50}$, prepared from the powder milled from 1 to 20 h and subsequently compacted at temperatures 400 °C, 500 °C, and 600 °C, are shown in Table I. We suppose that stresses introduced to the material during mechanical milling causes an additional anisotropy, through large enough value of saturation magnetostriction [8]. The higher temperature of the compaction of bulk samples causes relaxation of these stresses and the coercivity decreases with the decrease of the material porosity [9]. Further tendency of the coercivity as a function of milling time (the particle becomes smaller) indicates that the process of magnetization rotation dominates over domain wall displacement [9].

TABLE I

The values of the coercivity [A m^{-1}] of bulk $\text{Fe}_{50}\text{Co}_{50}$ prepared by the mechanical milling of powder from 1 h to 20 h and compacted at the temperature range from 400 °C to 600 °C.

Compacting temperature	Time of the milling		
	1 h	15 h	20 h
400 °C	4040	4040	4150
500 °C	3720	3800	3860
600 °C	780	1160	1180

We decided to anneal these bulk samples at temperature of 1000 °C for 1 h in an electric furnace in argon atmosphere, because Fe–Co based material are used for high temperature applications, too. The values of the coercivity decrease with the increase of the annealing temperature, Table II.

The coercivity of the compacted samples decreases after annealing reaching minimum value of 130, 140, 370 A m^{-1} for samples prepared from the powder milled 1, 15, and 20 h and compacted at the highest temperature 600 °C, respectively. The value of the coercivity depends mostly on the surface and volume pinning of magnetic domain walls displacement [9]. The annealing at higher temperature (1000 °C) causes relaxation of residual stresses (which were introduced to the samples du-

ring mechanical milling and compacting) and it improves contact between powder particles and causes lowering of the coercivity.

TABLE II

The values of the coercivity [A m^{-1}] of bulk $\text{Fe}_{50}\text{Co}_{50}$ prepared by the mechanical milling of powder from 1 h to 20 h, compacted at the temperature range from 400 °C to 600 °C and annealed at 1000 °C for 1 h.

Compacting temperature	Time of the milling		
	1 h	15 h	20 h
400 °C	150	180	530
500 °C	140	150	460
600 °C	130	140	370

The magnetic permeability was characterized by the measuring of the inductance L_s and the resistance R_s of the bulk ring samples $\text{Fe}_{50}\text{Co}_{50}$. For this measurement each toroidal sample was modelled as an ideal resistor in series with an ideal inductor. The complex permeability consists of two parts: real part (μ') and imaginary part (μ''). These parts of complex permeability are determined from the inductance and resistance of the coil on the ring sample with turns of the coil 15. The formulae for determining of the inductance and the resistance and their measurements are reported in [10, 11].

Figure 1 displays the frequency spectra of real part of complex permeability in cores prepared from different powders (milled for 1, 15, and 20 h) and compacted at 600 °C. It can be seen that real part of complex permeability for all three samples keeps stable at low frequency region (200–500 Hz), after that the permeability rapidly decreases to the frequency approximately 10 kHz and then we can see slow decent. The real part of complex permeability depends on the magnetocrystalline anisotropy, non-magnetic phase, density, and number of pores [12]. The presence of pores (air gaps) between the magnetic powders is a source of demagnetizing field. The pores act as pinning sites for the domain wall movement. Consequently, domain wall movement is restricted and this limits the growth of real part of permeability. The highest value of initial permeability is noted for the sample prepared by the powder milled for the shortest time (1 h) and the lowest one is noted for sample prepared by the compacting of powder milled 15 h.

We can see the frequency spectra of imaginary part of complex permeability in cores prepared from different powders (milled for 1, 15, and 20 h) and compacted at 600 °C, displayed in Fig. 1.

Imaginary part of complex permeability for samples prepared from the powder milled for 1 and 20 h has a peak at 1000 kHz, so it is the same value where the real part of complex permeability rapidly decreases.

Figure 2 shows the total energy losses as a function of frequency of all three samples in the frequency range from 2 Hz to 400 Hz measured at $B_m = 0.4$ T. The total core losses consist of three components: hysteresis losses, eddy current losses, and anomalous losses, described

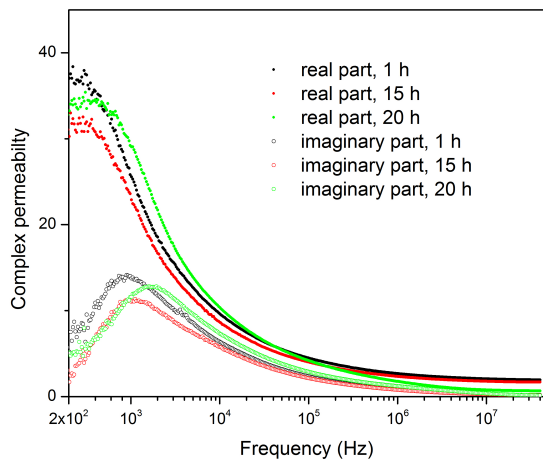


Fig. 1. The real and imaginary part of complex permeability of bulk Fe₅₀Co₅₀ prepared by the compaction of powder milled for 1, 15, and 20 h.

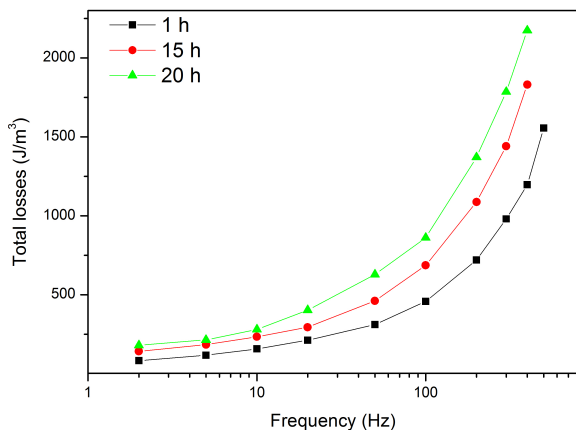


Fig. 2. The total energy losses (measured at 0.4 T) of bulk Fe₅₀Co₅₀ prepared by the compaction of powder milled for 1, 15, and 20 h.

in [13]. The frequency dependence of the total losses of sample milled for the shortest time (1 h) exhibits the lowest values. This sample consists of the largest particles in which there are likely present relatively large and good movable domain walls (lower hysteresis losses) and these all parameters can cause the lowest value of total losses [13]. The higher values of total losses for sample prepared by powder milled higher time (15 and 20 h) can be explained by the influence of the defects introduced during the milling process, thereby creating pinning centers for domain wall displacement.

4. Conclusion

Investigating magnetic properties of compacted crystalline Fe–Co (50:50 wt%) powder material we obtained that coercivity of compacts is strongly influenced by particle size of precursor powder, temperature of compaction and heat treatment after compaction. The lowest values

of coercivity were achieved for samples prepared by milling of the material for 1 h in planetary ball mill. The same sample exhibits the highest permeability and the lowest total energy losses in frequency range up to 1 kHz.

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References

- [1] A. Guittoum, A. Layadi, A. Bourzami, H. Tafat, N. Souami, S. Boutarfaia, D. Lacour, *J. Magn. Magn. Mater.* **320**, 1385 (2008).
- [2] Qi Zeng, I. Baker, V. McCreary, Zhicheng Yan, *J. Magn. Magn. Mater.* **318**, 28 (2007).
- [3] K. Akkouche, A. Guittoum, N. Boukherroub, N. Souami, *J. Magn. Magn. Mater.* **323**, 2542 (2011).
- [4] C. Suryanarayana, *Progr. Mater. Sci.* **46**, 1 (2001).
- [5] T. Szumiata, M. Gzik-Szumiata, K. Brzózka, M. Gawroński, B. Górka, P. Kollár, D. Olekšáková, K. Polański, W. Szmaja, *Acta Phys. Pol. A* **115**, 403 (2009).
- [6] A. Zelenáková, D. Olekšáková, J. Degmová, J. Kováč, P. Kollár, M. Kusý, P. Sovák, *J. Magn. Magn. Mater.* **316**, e519 (2007).
- [7] M. Hasiak, M. Migliorini, A. Łukiewska, J. Kaleta, J. Zbroszczyk, *Acta Phys. Pol. A* **126**, 118 (2014).
- [8] J.M.D. Coey, *Magnetism and Magnetic Materials*, Cambridge University Press, Cambridge 2010.
- [9] P. Kollár, J. Bednarcik, S. Roth, H. Grahl, J. Eckert, *J. Magn. Magn. Mater.* **278**, 373 (2004).
- [10] J. Füzervová, J. Füzerv, P. Kollár, R. Bureš, M. Fábervová, *J. Magn. Magn. Mater.* **345**, 77 (2013).
- [11] J. Füzerv, S. Dobák, J. Füzervová, *Acta Phys. Pol. A* **126**, 88 (2014).
- [12] G. Q. Lin, Z.W. Li, L.F. Chen, Y.P. Wu, C.K. Ong, *J. Magn. Magn. Mater.* **305**, 291 (2006).
- [13] D. Olekšáková, J. Füzerv, P. Kollár, S. Roth, *J. Magn. Magn. Mater.* **333**, 18 (2013).