Proceedings of XIX International Scientific Conference "New Technologies and Achievements in Metallurgy, Material Engineering, Production Engineering and Physics", Częstochowa, Poland, June 7–8, 2018

Microstructure and Magnetic Properties of NANOPERM-Type Soft Magnetic Material

M. Hasiak[a,*], A. Łaszcz[a], A. Żak[b] and J. Kaleta[a]

[a]Wrocław University of Science and Technology, Faculty of Mechanical Engineering, Department of Mechanics, Materials Science and Engineering, M. Smoluchowskiego 25, 50-370 Wrocław, Poland

[b]Wrocław University of Science and Technology, Faculty of Mechanical Engineering,Department of Materials Science, Strength of Materials and Welding, M. Smoluchowskiego 25, 50-370 Wrocław, Poland

In recent years, amorphous and nanocrystalline Fe-based alloys, due to their unique soft magnetic properties, have emerged as one of the most promising group of modern materials for various electric applications. This work presents microstructure and AC/DC magnetic properties of the NANOPERM-type material. The as-quenched amorphous $Fe_{76}Mo_8Cu_1B_{15}$ alloy was prepared by rapid quenching method in a form of 10 mm wide and 0.025 mm thick ribbon. Partial nanocrystallization was obtained by subsequent annealing of amorphous precursor at 783 K for 30 min. Microstructure investigation of annealed sample confirmed precipitation of α-Fe nanograins dispersed in amorphous matrix. In order to assess soft magnetic properties of fabricated material the dependences of core losses versus frequency (50 Hz–20 kHz) at room temperature were established, together with eddy currents, hysteresis, and anomalous losses coefficients separation. The frequency dependent correlation between real and imaginary part of permeability was also presented. Moreover, DC hysteresis loops of both as-quenched and annealed alloy were recorded in temperature range from 200 K to 400 K.

DOI: 10.12693/APhysPolA.135.284

PACS/topics: 75.50.Bb, 75.50.Kj, 75.60.–d, 75.60.Ej, 81.40.Rs

1. Introduction

Amorphous and especially nanocrystalline Fe-based alloys, due to their excellent soft magnetic properties, have already become one of the most promising material for many electric applications [1–3]. These types of alloys are usually produced by partial crystallization of metastable, amorphous, rapidly quenched ribbons in which controlled annealing process leads to the nucleation of α-Fe nanograins embedded in amorphous matrix. Nanocrystalline materials are characterized by notably better soft magnetic properties, such as high permeability, low coercivity, high magnetic saturation, and low core losses than their amorphous precursors [4, 5]. Apart from preparation conditions magnetic properties of nanocrystalline materials are also strongly connected with their chemical composition [6, 7]. Considering that fact, there are several groups of modern metallic glasses differing in chemical composition, including FINEMET, NANOPERM, or HITPERM alloys [8–10]. In this study microstructure as well as soft magnetic properties of the amorphous and nanocrystalline NANOPERM-type $Fe_{76}Mo_8Cu_1B_{15}$ alloy were studied. The separation of magnetic core losses was applied in order to provide a clear view on losses characteristics at different frequencies. Moreover, the frequency dependent correlation between real and imaginary component of permeability was also established. The analyses presented in this paper are essential to determine the application possibilities of the $Fe_{76}Mo_8Cu_1B_{15}$ alloy.

2. Experimental procedure

The amorphous NANOPERM-type material with the nominal composition of $Fe_{76}Mo_8Cu_1B_{15}$ (at.%) was prepared in the form of 10 mm wide and 20 μm thick ribbon by rapid solidification using melt-spinning technique. In order to receive nanocrystalline structure fabricated ribbon was annealed at 783 K for 0.5 h, i.e. between primary $T_{x1} = 735$ K and secondary $T_{x2} = 892$ K crystallization temperature (estimated from differential scanning calorimetry with a heating rate of 10 K/min). The structure of the annealed sample and as-spun amorphous precursor was checked with the help of transmission electron microscope (H-800, Hitachi) working at accelerating voltage of 150 kV. AC magnetic characteristics and core losses measurements were carried out by the means of AC/DC hysteresis loop tracer (AMH-50K-S, Laboratorio Elettrofisico Engineering Srl) for toroidal-shaped samples with a mean path of 57.411 mm in a frequency range from 50 Hz to 20 kHz. DC hysteresis loops were recorded with the help of VersaLab System (Quantum Design) at external magnetic field up to $\mu_0 H = 2$ T in the temperature range from 200 K to 400 K.

3. Results and discussion

Figure 1 depicts transmission electron microscope images of the as-spun and annealed $Fe_{76}Mo_8Cu_1B_{15}$ ribbon

*corresponding author

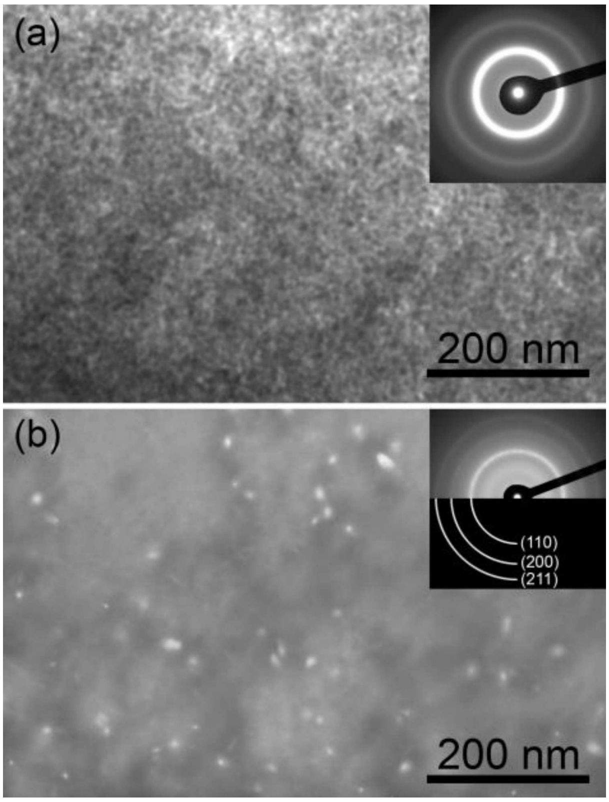

Fig. 1. Transmission electron microscope dark field images and corresponding selected area diffraction patterns for the as-spun (a) and annealed at 783 K for 30 min (b) $Fe_{76}Mo_8Cu_1B_{15}$ alloy.

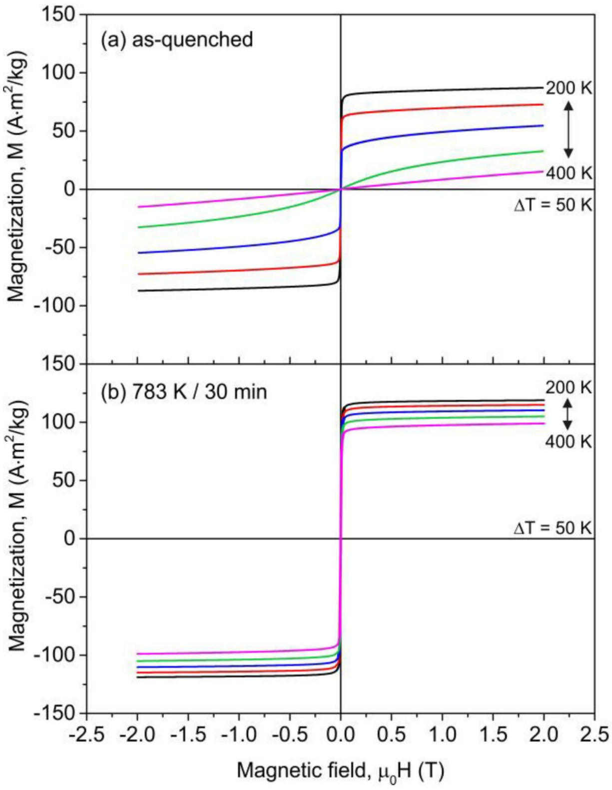

Fig. 2. DC hysteresis loops for the (a) as-spun and (b) annealed at 783 K for 30 min $Fe_{76}Mo_8Cu_1B_{15}$ alloy recorded at indicated temperatures from 200 K to 400 K ($\Delta T = 50$ K).

with corresponding electron diffraction patterns. It can be seen that the as-spun precursor (Fig. 1a) is characterized by uniform amorphous structure. The corresponding selected area in diffraction pattern, presented at the top right-hand corner, reveals broad concentric rings characteristic to amorphous Fe-based structures. On the contrary, Fig. 1b shows the transmission electron microscopy (TEM) image of the annealed sample. The annealing process of the amorphous precursor leads to the nucleation of small nanograins (average grain size of about 8.6 nm) embedded in amorphous matrix. Electron diffraction pattern from analysed area confirmed that apparent nanocrystallites correspond to the bcc-Fe phase identified by (110), (200) and (211) crystallographic planes.

It was expected that precipitation of bcc-Fe nanograins could have significant influence on magnetic properties of the $Fe_{76}Mo_8Cu_1B_{15}$ alloy. Figure 2 presents DC hysteresis loops recorded in a temperature range from 200 K to 400 K for both amorphous and nanocrystalline material. When it comes to amorphous sample, it is seen that ferromagnetic behaviour of the alloy monotonically decrease with temperature and at 400 K the shape of $M(H)$ loop becomes characteristic to the paramagnetic material. The Curie temperature of the amorphous matrix is only slightly above the room temperature. In contrary, hysteresis loops for annealed nanocrystalline sample shows strong ferromagnetic behaviour across the entire temperature range. Moreover, magnetization of the annealed sample is notably higher than magnetization of the amorphous precursor. All these results imply that nucleation of bcc-Fe nanograins during the annealing process increases the Curie point and improves soft magnetic properties of the $Fe_{76}Mo_8Cu_1B_{15}$ alloy.

Figure 3 shows frequency dependent characteristics of magnetic core losses of the as-spun and annealed $Fe_{76}Mo_8Cu_1B_{15}$ alloy measured at indicated maximum inductions. It is difficult to clearly compare the core losses of amorphous and nanocrystalline alloy because of the significant differences between their magnetic inductions. However, the same tendency of core losses increasing with frequency of measurement is visible in both samples. In order to get better understanding of losses characteristics, a separation of core losses was conducted. Statistical magnetic core losses model with three main components was used and fitted to the experimental results, according to the following equation [11]:

$$W = K_h B_m^2 + K_e B_m^2 f + K_a B_m^{3/2} f^{1/2},$$

where W is a total sum of losses per cycle and K_h, K_e, K_a are hysteresis, eddy current, and anomalous losses coefficients, respectively.

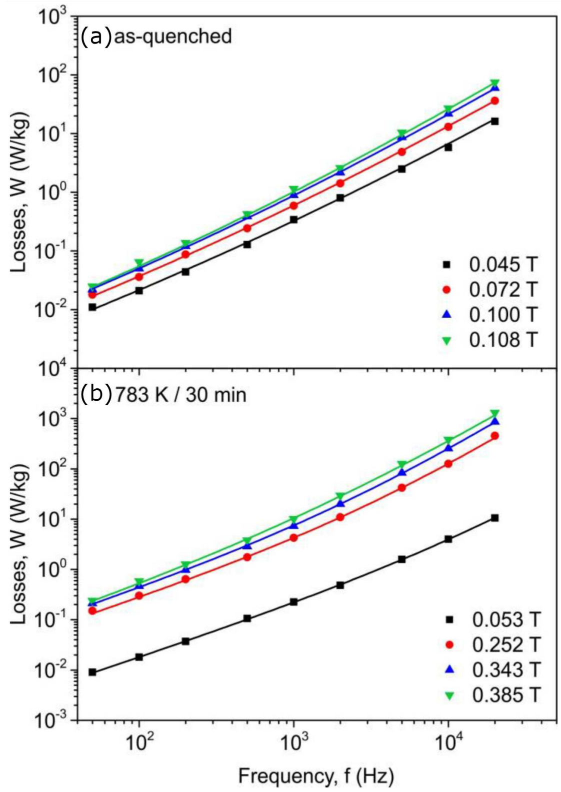

Fig. 3. Core losses versus maximum magnetic induction for the (a) as-spun and (b) annealed at 783 K for 30 min $Fe_{76}Mo_8Cu_1B_{15}$ alloy.

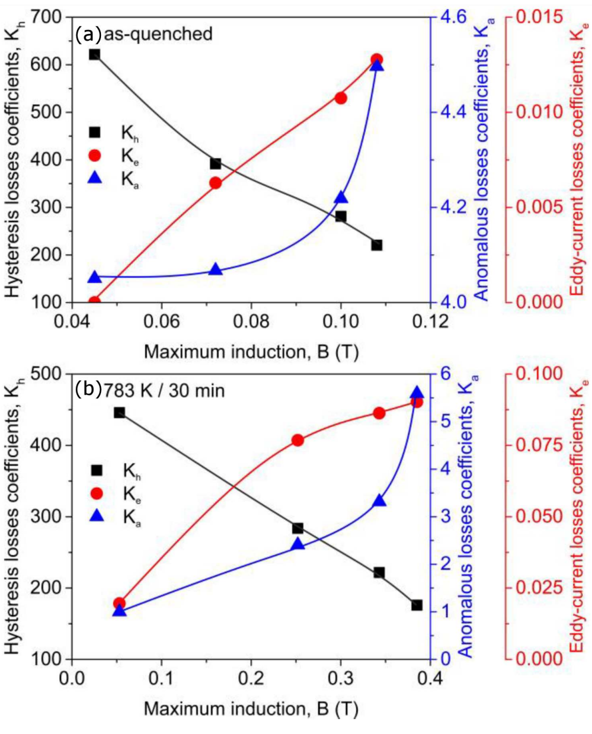

Fig. 4. Hysteresis K_h, eddy currents K_e and anomalous K_a core losses coefficients versus maximum magnetic induction for the (a) as-spun and (b) annealed at 783 K for 30 min $Fe_{76}Mo_8Cu_1B_{15}$ alloy.

Calculated values of core losses coefficients versus maximum induction are presented in Fig. 4. It is seen that losses coefficients in the amorphous and nanocrystalline $Fe_{76}Mo_8Cu_1B_{15}$ alloy change with the same tendencies. Static hysteresis losses K_h play dominant role at low value of induction and frequency. For higher values of magnetic induction eddy current K_e and anomalous K_a losses start increasing. What is important, anomalous losses coefficient considerably increases for the higher values of induction.

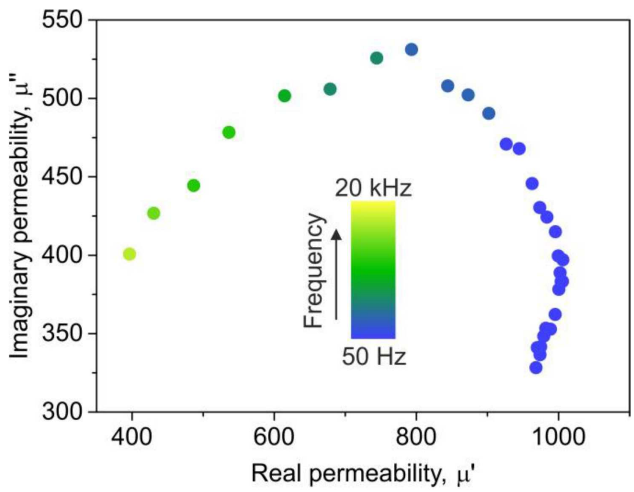

Fig. 5. Correlation between the real μ' and imaginary μ'' part of magnetic permeability for the $Fe_{76}Mo_8Cu_1B_{15}$ alloy annealed at 783 K for 30 min measured at magnetic field of 65 A/m.

Presented results show that investigated nanocrystalline material reveals notably better soft magnetic properties than its amorphous precursor. Considering that fact, Fig. 5 depicts the frequency dependent correlation between the real μ' and imaginary μ'' component of magnetic permeability for the annealed $Fe_{76}Mo_8Cu_1B_{15}$ alloy measured at magnetic field of 65 A/m. It is seen that μ' slightly increase from 970 to 1005 in frequency range 50 Hz–1 kHz and then starts decreasing. At the same time μ'' component rises from 330 to 530 in frequency range 50 Hz–5 kHz and then it monotonically decreases to 400 at 20 kHz. Additionally, at 14 kHz the same value of 430 for μ' and μ'' were measured.

4. Conclusions

The paper shows the difference between soft magnetic properties of the amorphous and annealed NANOPERM-type $Fe_{76}Mo_8Cu_1B_{15}$ alloy. TEM investigation confirmed amorphicity of the as-spun precursor and partial nanocrystallization of the sample annealed at 783 K for 30 min. Detailed AC magnetic characteristics measured in wide range of frequency have contributed to the core loss estimation, together with subsequent losses parameters separation. Frequency stability of magnetic permeability were also discussed in this study. Presented losses separation provides invaluable input data to losses prediction and material optimization for low power electrical inductive devices based on the nanocrystalline $Fe_{76}Mo_8Cu_1B_{15}$ alloy.

References

[1] M.E. McHenry, M.A. Willard, D.E. Laughlin, *Prog. Mater. Sci.* **44**, 291 (1999).

[2] O. Gutfleisch, M.A. Willard, E. Brück, C.H. Chen, S.G. Sankar, J. Ping Liu, *Adv. Mater.* **23**, 821 (2011).

[3] M. Hasiak, M. Miglierini, M. Łukiewski, *IEEE Trans. Magn.* **50**, 2004104 (2014).

[4] G. Herzer, *IEEE Trans. Magn.* **26**, 1397 (1990).

[5] K. Suzuki, *Mater. Sci. Forum* **312–314**, 521 (1999).

[6] M. Miglierini, I. Tóth, M. Seberíni, E. Illeková, B. Idzikowski, *J. Phys. Condens. Matter* **14**, 1249 (2002).

[7] F. Li, B. Shen, A. Makino, A. Inoue, *Appl. Phys. Lett.* **91**, 234101 (2007).

[8] T. Gheiratmand, H.R. Madaah Hosseini, *J. Magn. Magn. Mater.* **408**, 177 (2016).

[9] A. Makino, T. Hatanai, Y. Naitoh, T. Bitoh, A. Inoue, T. Masumoto, *IEEE Trans. Magn.* **33**, 9216 (1997).

[10] M. Hasiak, M. Miglierini, M. Łukiewski, J. Kaleta, *IEEE Trans. Magn.* **48**, 1665 (2012).

[11] G. Bertotti, *IEEE Trans. Magn.* **24**, 621 (1988).