STRUCTURE AND PROPERTIES OF NANOCRYSTALLINE SOFT MAGNETIC COMPOSITE MATERIALS WITH POLYMER MATRIX

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Abstract. The paper concerns investigation of nanocrystalline composites technology preparation. The composites in the form of rings with rectangular transverse section, and with polymer matrix and nanocrystalline metallic powders fulfillment were made, for obtaining a good ferromagnetic properties. The nanocrystalline ferromagnetic powders were manufactured by high-energy ball milling of metallic glasses strips (as quenched and after annealing state). Generally for investigation, Co matrix alloys and thermoplastic and hardening polymers including elastomers were used. Magnetic properties in the form of hysteresis loop, by rings method were measured. Magnetic properties of composites materials were compared with properties of winded cores of nanocrystalline strips and powder cores (rings) solidify by pressing and gluing. Generally powder cores showed lower soft ferromagnetic properties than winded cores of nanocrystalline strips, but composites cores showed interesting mechanical properties. Furthermore, the structure of strips and powders on properties of composites were investigated.

Keywords. Nanocrystallisation, Powders, Metalic glasses, Magnetic properties, High-energy ball milling, Composites

1. Introduction

Yoshizawa and his co-workers in the works published in 1988 started the new research on the nanocrystalline composite materials obtained in the metallic glass crystallisation [1]. The unflagging interest in these materials observed nowadays is caused by good magnetic properties typical of nanocrystalline materials [2].

The thermal nanocrystallisation and the mechanical milling of metallic glasses are the most frequent processes of nanocrystallisation. The physical and chemical properties of the product achieved in the process of milling depend foremost on: the process temperature, the size and material of the grinding media (ball milling), the relation of the grinding media mass to the milled material mass, the dynamics of the system depending on the kind of the mill, as well as the chemical composition of the initial material and the atmosphere of the milling process [3-5].

Nanocrystalline metallic materials obtained directly in the process of the metallic glass crystallisation are available only in the form of very thin strips, which results from the production process (planer-flow-casting). The fact that the nanocrystalline composite materials may be obtained and used in the powder (loose) state, seems to be a very interesting issue from the point of view of the production technology, processing and application [7-9].

The production of the powder nanocrystalline materials in the process of the high energy ball milling of metallic glasses, requires long periods of treatment. Furthermore, one process results only in tiny quantities of the powder. The research done proved that the combination of the thermal and mechanical nanocrystallisation of the metallic glass minimises the time of nanocrystallisation, as compared with long mechanical nanocrystallisation [Fig.1].

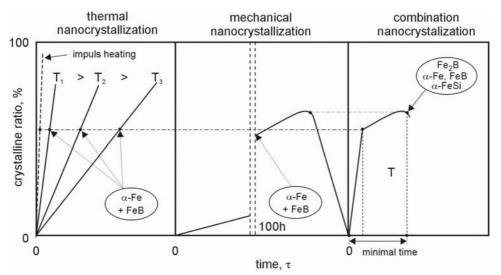


Figure 1. Diagram of minimalization time production nanocrystalline materials from metalic glasses [10]

The production of the nanocrystalline powder materials in the high energy ball milling, enables the scientists to work on the ferromagnetic nanocomposites which dimensions and shape may be formed in various consolidation methods [12-15].

In the case of soft magnetic powder materials, during the magnetisation of the short cylindrical sample, the saturation is achieved at the magnetisation field intensity much grater than in the case when the close ring is used. It is

caused by the fact that the intensity of the magnetic field (H_i) in the short sample is lower than the magnetic field (H) in the whole coil [16, 17].

There is a relation between the magnetisation (J) and the field intensity. When the partial demagnetisation caused by the shape of the sample is taken into account, the relation may be presented in the following way:

$$J = \mu_0 \cdot H \cdot \frac{\kappa}{1 + \kappa N} \tag{1}$$

where:

N- demagnetisation coefficient k- magnetic susceptibility μ_0 – induction constant (magnetic permeability of vacuum)

The N coefficient in the case of, for example, cylindrical samples depends mainly on the relation of sample length (*l*) to its diameter (D_p) [16].

$$p = \frac{l}{D_{\pi}} \tag{2}$$

The demagnetisation effect of sample ends is presented in Fig.2.

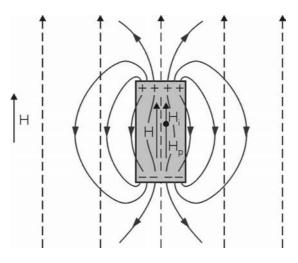


Figure 2. The effect of demagnetisation of sample ends [16]

The shorter and thicker the cylindrical rod is, the smaller value of the p parameter and at the same time the higher value of the (N) parameter is. The weakening demagnetisation field, which is formed inside by the sample introduced into the magnetic field of the (H) intensity is the following:

$$H - H_i = H - H \cdot \frac{1}{1 + \kappa N} = NH \cdot \frac{\kappa}{1 + \kappa N} = N \cdot \frac{J}{\mu_0}$$
(3)

The research proved that the (N) demagnetisation coefficient depends also on the magnetic permeability (μ). The higher the (N), the higher the μ [16].

The characteristics of soft magnetic materials may be improved by means of the magnetic anisotropy. The effect is increased in the thermo-magnetic treatment. In this kind of treatment, the properties are improved to a greater extent than in the case of the classic heat treatment. In amorphous materials, required anisotropic properties are obtained in the process of induction of magnetic anisotropy conducted in a proper direction [18,19].

The aim of this work is to investigate the influence of the high energy ball milling process parameters as well as the isothermal soaking ones on the magnetic properties of the powder nanocrystalline material obtained from the metallic glass in the combination of the high temperature milling and the process of the thermal nanocrystallisation. The research covers also properties of the obtained polymer matrix nanocomposites including the powder material with definite physical properties.

1. Material and methodology

The $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ sections of metallic glass in the form of a strip (0,026 mm thick and 10,2 mm wide) were used as a initial material in the research. The alloy was made in the process of direct casting of a stream of a liquid

alloy on the surface of the spinning metal drum. The speed of milling reached 20-30 m/s. The charge materials were melted in the vacuous induction furnace. The initial alloys were poured in the planar-flow-casting (PFC) method on the spinning cooling cylinder in the securing argon atmosphere.

In the milling of the strips in the "as quenched" form, the high energetic 800 SPEX CertiPrep Mixer/Mill was used. The milling time applied was 1,2,5,15,20 and 25 hours. The isothermal soaking of powder was carried out in the F6020C TERMOLINE resistance furnace in the range of temperature 300÷600°C with the gradation of every 100°C in the atmosphere of argon.

The X-ray tests were done on the DRON-2 X-ray diffractometer with the HZG-3 goniometer and the computer system of secondary radiation registration the DRONEK-2 steering module with the lamp equipped with the Co anode of 40kv voltage and the heater current of 20mA. The diffraction research was done in the range of angels 20 from $40\div120^\circ$, with the measurement step 0,1 and the impulse counting time 3 s.

The magnetic properties were examined on the basis of toroidal samples with the use of the FERROMETER 1 measurement system. The parameters used in the research were the following: AC=5 V, f=50 Hz, the amplitude 1V. The powder was put into the toroidal karkas and then the primary and secondary windings were made. The number of coils was $n_1=n_2=130$. The system consists of the PC computer equipped with the data ackwisition card (PC LAB) of the Advantech company (PC-LAB) and the adequate computer programme.

The dimension measurement of the powder grains was done on the LEICA MAEF4A® light microscope using the LEICA® firm computer programme.

2. Experimental procedure

The magnetic research of the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ powders obtained in the process of milling of the strips in the "as quenched" state proved that the process of the high energy ball milling causes a significant increase in the coercive force. The powder obtained after 1-hour milling of the amorphous strip is characterised by the highest value of the coercive force field (H_C=119,1). The longer the time of milling is, the smaller the value of the parameter is after 10-hour milling H_C=14,1A/m (Tab. 1). Further increase in the milling time causes only slight changes of the H_C with slight increasing tendency (Fig. 3).

Table 1. Magnetic properties of the powder material obtained from the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy strip, milled for 1, 2,
5, 10, 15, 20 i 25 hours (measurements were made for the frequency of f=50 Hz)

measured	state "as	milling time [h]							
dimension	quenched	1	2	5	10	15	20	25	
$H_C [A/m]$	15,1	119,1	115,4	109,8	14,1	15,2	16,5	19,2	
$B_R[T]$	0,25	0,024	0,035	0,014	≈ 0	0,003	0,001	≈ 0	
B _{max} [T]	0,54	0,13	0,14	0,102	0,062	0,07	0,067	0,066	
H _{max} [A/m]	724	1261	890	990	952	877	735	1059	
μ_{max}	131935	114	151	84	53	69	76	50	
P _{max} [W/kg]	0,0003	0,2	0,15	0,098	0,028	0,02	0,0196	0,0191	

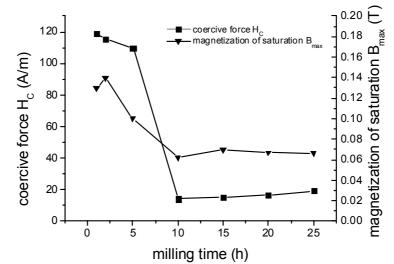


Figure 3. Effect of the milling time for the amoprhus strips made from the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy on the coercion field intensity H_C and magnetization B_{max} for the obtained powders

The longer the milling process, the smaller the value of the saturation of magnetisation, which for the powder obtained after 2-hour milling of the amorphous $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ strip amounts to $B_{max}=0,14$ T. For the powder

obtained in 10-hour milling, the value B_{max} equals only 0,062 T. The value of the saturation of magnetisation maintains at the same level for all samples after 15, 20 and 25 hours of milling.

The maximal permeability value worth μ_{max} = 151 (H_C=115,4 A/m) is reached after 2-hour milling. Along with the further increase in the milling time, the value of maximal permeability decreases. After 10-hour milling the parameter μ_{max} =53 and it is the lowest μ_{max} =50 (H_C=19,2 A/m) when the milling process lasts for 25hours (Tab.1)

The observation of materials in the scanning microscope proved that the high energy ball milling of the amorphous $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ strip lasting 2 hours, results in the production of flakes of strips ("scales"). The shape of powder grains changes together with the time of milling. After 2-hour milling the shape of grains resembles flakes and after 10-hour milling the grains are smaller and more spherical (Fig.4).

The microscope observation proves that the longer the process of milling the smaller the value of the average diameter of powder grains and the smaller the value of the standard deviation of the average diameter of grains.

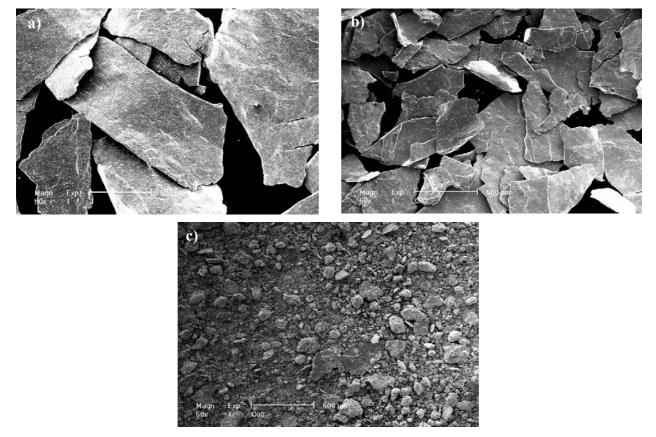


Figure 4. Powder grains image after a) 2 h, b) 5 h, c) 10 h of the high energy miilling, scanning microscope

At the first stage of the milling process the powder obtained was in the form of flakes of the crumbled string of the average dimension 560,5 μ m (standard deviation s=585,3, 2-hour milling). After 10-hour milling, the average diameter of powder grains decreases to 17 μ m (standard deviation s=28,9). At the second stage of the process, the time of milling still rises and the average diameter of powder grains still decreases but with smaller intensity (Tab.2). At the final stage of the tests, after 25 hour high energetic milling, the powder grains are characterised by the equiaxial shape and the distinct size diversity.

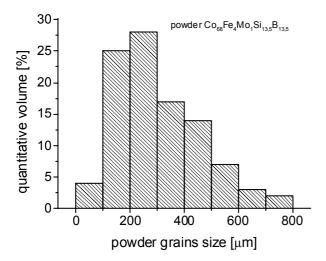
The analysis of powder grain dimensions proved that the value of the average of diameter of powder grains decreases along with the milling time increase. The research proves that there is a process of size homogenisation, even though the range of size of powder grains is still significant (Tab. 2).

Table 2. Measurement results diamete of powder grains obtained after the high-energy milling of the strip made from the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy depending on milling time

measured dimension	milling time [h]								
measured dimension	2	5	10	15	20	25			
average [µm]	560,5	306,1	17	3,82	3,06	2,85			
standard deviation, S	585,3	158	28,9	2,82	1,74	1,1			
max. value [µm]	2230	784,5	207	21,98	12,3	7,26			
min. value [µm]	43,3	67,1	3,39	1,1	0,92	1,2			

Figure 5 presents the bar chart of results of powder grain dimension tests. The powder was obtained from the $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}a_{13.5$

The dimension tests proved that the longer the process of milling is, the more homogeneous the grains are, and after 25 hours their dimensions vary between 1 and 10 um. (Fig. 6).



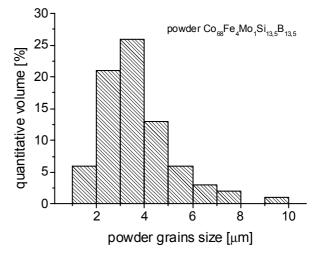


Figure 5. Bar graph of the measurements of powder grain dimension, obtained from the amorphous strip from the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy subjected to the high-energy milling for 5 hours

Figure 6. Bar graph of the measurements of powder grain dimension, obtained from the amorphous strip from the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy subjected to the high-energy milling for 25 hours

In order to compare, strips held at the range of temperature of $200\div500^{\circ}$ C were subjected to the high energy ball milling. The observation of the powder material (obtained in the process) in the scanning microscope proved that the powder obtained after 2-hour milling of the Co₆₈Fe₄Mo₁Si_{13,5}B_{13,5} strips, is characterised by various levels of disintegration. The degree of disintegration depends on the temperature in which the strips were heated.

As the result of the high energy ball milling of strips held in the temperature of 200 and 300°C for 2 hours, the powder obtained was in the form of flakes of strips of the average dimension 446,5 μ m (standard deviation s=172,05) and 534 μ m (standard deviation s =200,1) respectively (Tab.3). Whereas powders obtained in the milling process of strips held in the temperature of 400 and 500°C are characterised by significantly smaller diameter of grains – 237,1 μ m (standard deviation s=138,9) and 63,5 μ m (standard deviation s=32,8) (Fig. 7).

The tests of grain dimension proved that when the temperature of the strip heating rises, the grains become more homogeneous and dimensions after heating in the temperature of 500 C and 2-hour milling oscillate between $1\div160 \,\mu m$ (Fig. 8, Fig. 9)

temperature of heating [°C] measured dimension 200 500 300 400 237,1 63,5 446,3 534,5 average [µm] standard deviation, S 172,05 200,1138,9 32,8 738,4 924,9 539,4 165,6 max. value [µm] 171.3 min. value [µm] 220.7 7.3 10.1

Table 3. Measurement results diamete of powder material obtained after milling for 2 hours and subsequently heated in termal in temperature range 300÷600°C

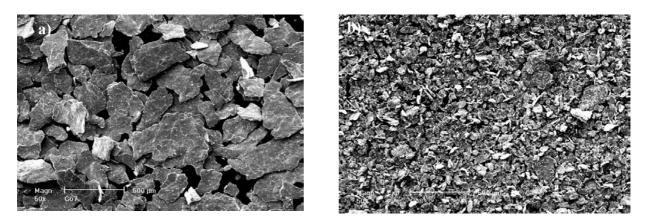
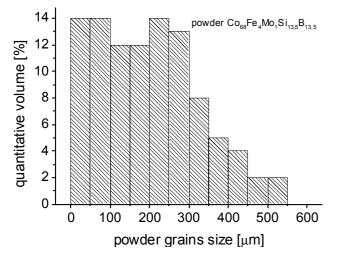


Figure 7. Powder grains image material obtained after milling for 2 hours and subsequently heated in termal in temperature a) 400°C, b) 500°C, scanning microscope



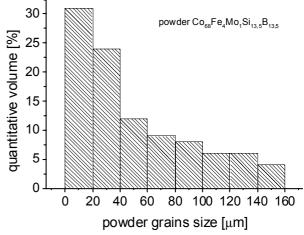


Figure 8. Bar graph of the measurements of powder grain dimension, obtained after milling for 2 hours and subsequently heated in thermal in temperature 400°C the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy

Figure 9. Bar graph of the measurements of powder grain dimension, obtained after milling for 2 hours and subsequently heated in thermal in temperature 500°C the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy

The analysis of the quality and quantity of the chemical constitution was carried out with the use of the EDS attachment. The tests proved that as the result of the amorphous strip milling as well as strips heated in the atmosphere of argon, no oxides which could appear during milling in the atmosphere of surrounding were present (Fig. 10).

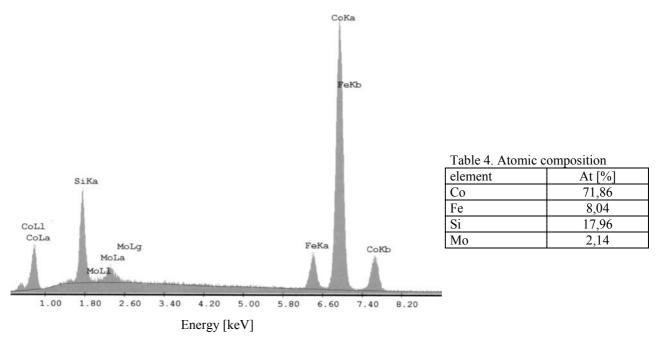


Figure 10. Diagram of energy of dispersed roentgen radiation from powder obtained from the amorphous strip from the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy subjected to the high-energy milling for 25 hours

At the next stage of the experiment, the powder material was subjected to the isothermal heating in order to determine the influence of the temperature on the change of the magnetic properties. Initial powders were obtained as the result of 2 and 5 hour high energetic milling of the amorphous $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy (Tab.5).

Among the powders tested, the lowest value of the coercive field force H_C and the highest maximal permeability μ_{max} are characteristic of the powder material obtained in the process of the amorphous milling lasting for 2 hours. Then the material is heated in the atmosphere of argon in the temperature of 500°C ($H_C=20,5$ A/m, $\mu_{max}=102,5$).

The lowest value of the coercive force and the highest maximal permeability ($H_C=33,4$ A/m, $\mu_{max}=186,1$) belongs to the powder obtained in the amorphous strip milling lasting for 5 hours and then heated in the temperature of 400°C for an hour. The magnetic properties of the powder material heated in the definite temperatures are presented in Fig.11

measured	temperatures of heating [°C]								
dimension	300	400	500	600	300	400	500	600	
$H_C [A/m]$	49	38,4	20,5	70	40,3	33,4	38	50,6	
$B_R[T]$	0,006	0,003	≈ 0	0,001	0,006	0,007	0,004	0,001	
B _{max} [T]	0,101	0,12	0,11	0,04	0,14	0,17	0,14	0,036	
H _{max} [A/m]	960	976	1051	880	1047	880	1090	990	
μ_{max}	98	109,9	102,5	34,9	113,4	186,1	122,8	30,2	
P _{max} [W/kg]	0,147	0,05	0,04	0,014	0,076	0,07	0,067	0,018	
	powder o	btained after	2 -hour mill	ing of the	powder obtained after 5 -hour milling of the				
		amorpho	ous strip		amorphous strip				

Table 5. Magnetic properties of the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ powder materials after milling and termal anniling in temperature range $300\div600^{\circ}C$

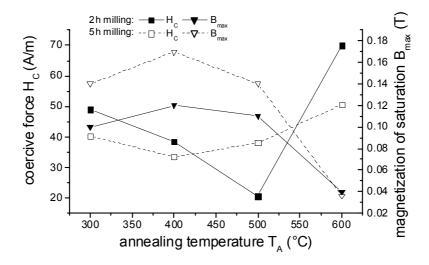


Figure 11. Effect of the isothermal heating of the T_A powder on the coercive field intensity H_C and magnetization B_{max} . The powder was obtained by the high-energy milling the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ amorphous alloy for 2 and 5 hours

For both, powders obtained after 2 and 5 hours of milling and at the following stage – heated, similar effects as in the case of amorphous strips heating were achieved. Similar results were obtained in the research [20] where the $Co_{68}Fe_4Mo_{1.5}Si_{13.5}B_{13}$ amorphous alloy strips were needed. In this case, the relation of the coercive field to the temperature of heating was similar to the one presented in Fig. 11. It is probable that the isothermal heating of powders provoked the relaxation of stress appeared in the process of high energetic milling. All this is responsible form the change of the magnetic properties of powders.

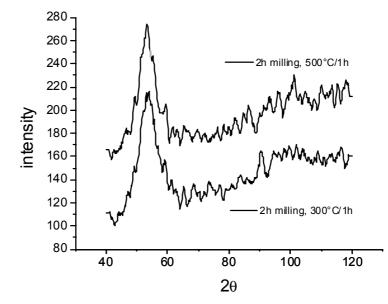


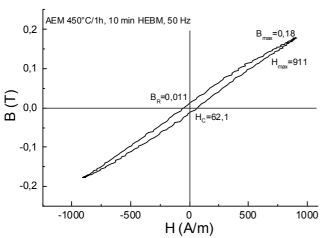
Figure 12. X-ray phase diagram of the powder material obtained after milling for 2 hours and subsequently heated in the temperatures of 300 and 500°C

On the basis of the analysis of the electron diffraction pattern (Fig. 12) it may be supposed that apart from the stress relaxation, the process of heating results in the structural changes which consists in new phase nucleation in higher temperatures.

In order to create polymer composites with silicon matrix, powder material obtained in the process of 10-minute high energy ball milling of the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy strip heated for an hour in the temperature of 450°C in the atmosphere of argon was used as a filler. In another version of the test, powder obtained by 1-hour milling of the strip isothermally heated in the same temperature was the filler. The magnetic properties of powder materials obtained in these processes are presented in Table 6 and Figures 13 and 14.

measured dimension	milling time of ribbons annealing in temperature 450°C in 1 hours [min]								
		10		60					
unitension	50 Hz	500 Hz	1 kHz	50 Hz	500 Hz	1 kHz			
$H_C [A/m]$	62,1	15,2	72,2	114,7	≈0	15,1			
$B_{R}[T]$	0,011	≈ 0	0,012	0,014	0,002	0,003			
B _{max} [T]	0,18	0,177	0,155	0,13	0,13	0,11			
H _{max} [A/m]	911	893	786	978	978	850			
μ_{max}	163,4	163,5	163	109,6	109,7	109,9			
P _{max} [W/kg]	0,13	_	_	0,097	_	_			

Table 6. Magnetic properties of powders



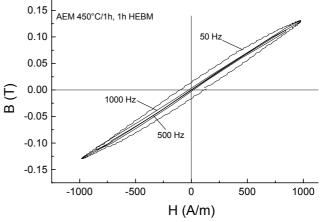


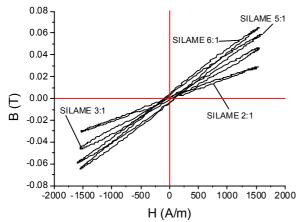
Figure 13. Histeresis loops for the powder obtained by milling for 10 min of the strip made from the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy heated at 450°C/1h

Figure 14. Histeresis loops for the powder obtained by milling for 1 hour of the strip made from the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy heated at temperature 450°C/1h (variant II)

The powder obtained as the result of the 1-hour heating in the temperature of 450°C and the 10-minute and 1-hour milling was mixed with silicon in the mass ratio of the powder to the rasin 6:1, 5:1, 3:1, i 2:1. From the suspended matter (paste) obtained, toroidal cores were formed. The hardening of the nanocomposite cores was carried out in the room temperature and lasted for 48 hours. The magnetic properties of toroidal cores of the SILAME® composite are presented in Table 7 and Figures 15 and 16.

Table 7. Magnetic properties of composite materials SILAME®

wielkość	kompozyt SILAME® (wariant I)				Kompozyt SILAME® (wariant II)			
wielkose	6:1	5:1	3:1	2:1	6:1	5:1	3:1	2:1
$H_C [A/m]$	88,2	85,8	34,5	71,7	145,2	145,2	52,8	33,2
$B_R[T]$	0,004	0,002	≈0	≈0	0,012	0,013	0,008	0,01
B _{max} [T]	0,065	0,059	0,046	0,003	0,11	0,11	0,066	0,043
H _{max} [A/m]	1557	1611	1557	1557	1428	1440	946	946
μ_{max}	34,4	30,8	24,3	16,1	61,7	64,5	59,1	38,1
P _{max} [W/kg]	0,1	0,09	0,04	0,02	0,2	0,19	0,095	0,06



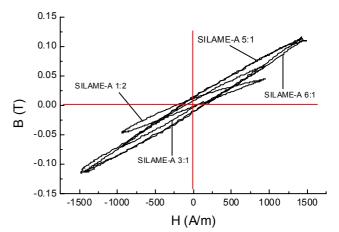


Figure 15. Histeresis loops for the nanocomposite from the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ powder with the silicon matrix

magnetization of saturation Bmax [T]

a)

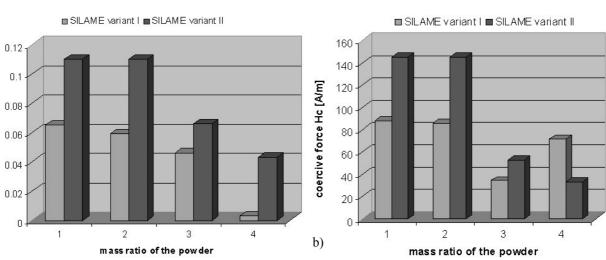


Figure 16. Histeresis loops for the nanocomposite from the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ powder with the silicon matrix (variant II)

Figure 17. The ifluence of powder mass fracton on a) magnetization, b) corecivity H_C of composite SILAME 6:1, the powder mass fracton 1 - 6:1, 2 - 5:1, 3 - 3:1, 4 - 2:1

Nanocomposites, as compared with the powder material used to produce them, are characterised by the very low saturation of magnetisation and very low maximal permeability in the intensity range of field which amounts to around 0,08 T. The SILAME® composites (variant I) as compared with the SILAME® composites (variant II) are characterised by the higher value of the coercive field and the higher maximal permeability.

The obtained properties of nanocomposites (Tab. 7) prove that there is a relation of the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy powder share to the magnetic properties of the composite. The properties are reduced together with the decrease in the share of the powder (in the composite). The structure of the SILAME® composite material is presented in Fig.18.

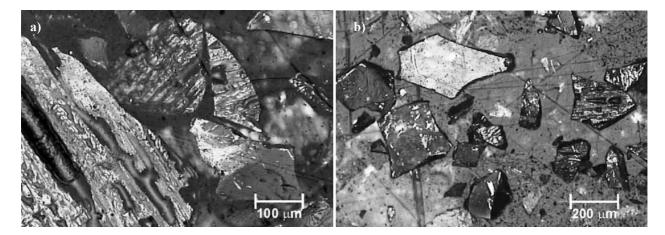


Figure 18. Structure of composite material a) SILAME 6:1, b) SILAME 6:1, light microscopy

4. Summary

The magnetic tests of the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ powders obtained in the high energy ball of milling process proved that the process causes a significant decrease in the magnetic properties.

On the basis of the research done, it was stated that the process of the high energy ball milling combined with thermal crystallisation of the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy, results in the production of the nanocrystalline powder material. The structure and magnetic properties of this material may be improved by means of a proper choice of parameters of this process as well as the final thermal treatment.

The composite material obtained in the process of solidification of the $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ nanocrystalline powder with silicon, demonstrates magnetic properties which increase along with the volume share increase in the metallic powder.

5. Acknowledgements

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