Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 24
ISSUE 2
October
2007

Magnetic properties of hot pressed powder $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ alloy

J. Konieczny [a], L. A. Dobrzański [a,*], I. Wnuk [b]

[a] Division of Materials Processing Technology, Management and Computer Techniques in Materials Science, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

[b] Faculty of Materials Processing Technology and Applied Physics, Częstochowa University of Technology, Częstochowa, Armii Krajowej 19 Street, Poland

* Corresponding author: E-mail address: leszek.dobrzanski@polsl.pl

Received 02.04.2007; published in revised form 01.10.2007

Properties

ABSTRACT

Purpose: The aim of the work is to investigate the structure and magnetic properties of the cobalt based hot pressed $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ powder obtained in high-energy ball milling process.

Design/methodology/approach: The nanocrystalline ferromagnetic powders were manufactured by high-energy ball milling of metallic glasses ribbons in as state. The hot pressing process was made on machine "Degussa". Observations of the structure of die stampings were made on the OPTON DSM-940 scanning electron microscope. Graphical analyses of the obtained X-ray diffraction patterns, as well as of the HC=f(TA) relationship were made using the MICROCAL ORIGIN 6.0 program.

Findings: The analysis of the magnetic properties and structure of the die stamping out that compared to the magnetic properties of the amorphous ribbons as their precursor, that hot pressing process deteriorates their magnetically soft properties.

Research limitations/implications: For the metallic Co-based amorphous ribbons, further mechanical and structure examinations are planed.

Practical implications: Structure and magnetic properties analysis of die stampings of powdered amorphous metallic ribbons is helpful to prepare this material by laboratory methods. Feature an alternative to commercial alloys and composite materials are the amorphous and nanocrystalline metal amorphous ribbons obtained by melt spinning technique and make it possible to obtain the new composite materials with best magnetic properties, which dimensions and shape can be freely formed

Originality/value: The paper presents influence of hot pressing parameters process of metallic powdered ribbons on structure and magnetic properties of obtained die stampings.

Keywords: Magnetic properties; Metallic alloys materials; Nanocrystalline materials; Powder Metallurgy

1. Introduction

Amorphous and nanocrystalline alloys based on cobalt produced by melt spinning technique show excellent soft magnetic properties [1÷3]. This properties and mechanical properties can be improve (change) by typical heat treatment (isothermal heating) [4, 5] or by heat treatment in magnetic field [6].

Unfortunately the nanocrystalline metallic materials obtained directly in the process of the metallic glass crystallization are available mainly in the form of very thin ribbons, which results from the production process (melt spinning) [7, 8].

The nanocrystalline composite materials which 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 [9-12].

he production of the soft magnetic powder materials in the high energy ball milling or in the mechanical alloying, enables the scientists to work on the ferromagnetic nanocomposites which dimensions and shape may be formed in various consolidation methods [13-15].

The aim of this work is to investigate the structure and magnetic properties and influence of temperature of hot pressing on the magnetic properties of the powder $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ alloy obtained from the metallic glass in the high energy ball milling process.

2. Material and methods

The investigations were carried out on a $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ metallic glass in form of 0.025 mm thick and 10.2 mm wide ribbons. A 8000 SPEX CertiPrep Mixer/Mill high energy ball mill was applied to mill the ribbons both in „as quenched" state and heat treated. The vibration times were 5 and 20 hours. A THERMOLYNE F6020C resistance furnace was used for isothermal soaking of the powder.

The hot pressing process was made on machine "Degussa" was subjected metallic powder obtained in high energy ball milling amorphous ribbon $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ by 20 hours. Such away prepared powder was compacted in uniaxial press in vacuum (2×10^{-2} Tr), in temperature 800°C as well as 950°C by 20 minutes, with pressure of stamp the P = 15 MPa.

The X-ray tests were realized with the use of the XRD 7 SEIFERT-FPM diffractometer equipped with the lamp of the cobalt anode of 35 kV voltage and 30 mA filament current was used. Diffraction tests were carried out in the 2θ angle range from 40 to 120° (measurement step 0.1°). Pulse counting time was 5 s.

Sizes of Co-β crystallites were determined with Scherrer's method [16]:

$$B = \frac{k \cdot \lambda}{d \cdot \cos\theta_B} \tag{1}$$

where:
d – diameter of the crystalline particle,
B – width of the diffraction peak measured at half of its height,
k – coefficient assumed as equal to 1 [16]
λ – X-ray radiation wavelength,
$2\theta_B$ – radiation beam diffraction angle corresponding to the Bragg maximum.

Microscope examinations were made on the OPTON DSM 940 electron scanning microscope and the JEOL JEM 200CX electron transmision one. Tests of magnetic properties were carried out by the use of Lake Shore's Vibrating Sample Magnetometer VSM model 7307.

3. Results and discussion

The obtained powders have the highest portion of the 400÷800 μm fraction at the beginning stage of milling of the $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ amorphous alloy. The most probable sizes in the powder grains population (mode) are 476 μm for the material obtained after 5 hours of milling.

Milling the material for 20 hours causes further size reduction of particles (Fig. 1). The highest portion of ≈ 15% was found out for particles from the range of 13÷18 μm, the arithmetic average of the powders diameter is 14.88 μm.

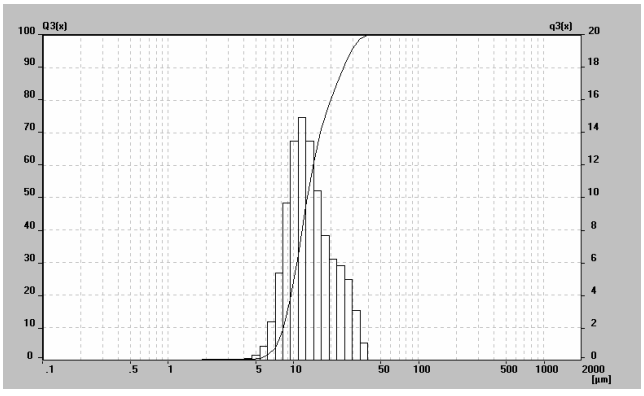

Fig. 1. The cumulative percentage portions curve and the grain size distribution curve for the powder obtained after 20 hours long milling of the $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ amorphous ribbon of the metallic glass

On the basis of the analysis of the electron diffraction pattern (Fig. 2) it may be supposed that apart from the stress relaxation, the hot pressing process results in the structural changes which consists of new phase nucleation in higher temperatures. In the X-ray photograph of die stamping obtained in 800°C of hot pressing process the Co-α (111) and (024) with crystallite size suitably 9 and 39 nm, Co-β (100), (101) and (110) crystallite size suitably 56, 39 and 11 nm, as well as the Co_3B (021) and (022) phases were identified (Table 1).

In the X-ray diffraction pattern of die stamping obtained in 950°C of hot pressing the Co-α (111) and (024) with crystallite size suitably 20 and 21 nm, Co-β (111) and (101) crystallite size suitably 15 and 21 nm, as well as the Co_3B (021) phases were identified (Fig. 2).

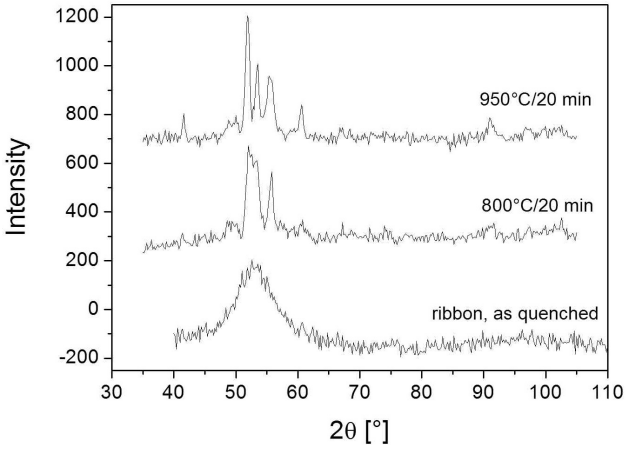

Fig. 2. X-ray diffraction pattern of the $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ ribbon in as quenched state and powder materials hot pressed in temperature 800 and 950°C in argon atmosphere

Table 1.
The phase analysis results for the hot pressed powder $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ alloy (see Fig. 2)

Hot pressed powder $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ alloy							
950°C/20 minutes				800°C/20 minutes			
2θ [°] calculated	2θ [°] ICDD	phase	(hkl)	2θ [°] calculated	2θ [°] ICDD	phase	(hkl)
41,54	41,24	Co_2Si (Pbnm)	(111)	52,05	52,50	Co-α (Fm3m)	(111)
48,84	48,96	Fe_3B (I-4)	(002)	53,21	53,15	Co-β (P63/mmc)	(100)
49,98	50,28	Co_2B (I4/mcm)	(221)	55,76	56,24	Co_3B (Pbnm)	(021)
52,05	52,50	Co-α (Fm3m)	(111)	60,84	60,94	Co-β (P63/mmc)	(101)
53,65	53,15	Co-β (P63/mmc)	(100)	60,84	60,33	Co-α (Fm3m)	(024)
55,41	56,24	Co_3B (Pbnm)	(021)	67,20	67,90	Co_3B (Pbnm)	(022)
60,58	60,94	Co-β (P63/mmc)	(101)	91,58	91,10	Co_2B (I4/mcm)	(402)
60,84	60,33	Co-α (Fm3m)	(024)	102,64	101,37	Co-β (P63/mmc)	(110)
90,97	89,99	Co_3B (Pbnm)	(130)				

The magnetic research of the $Co_{68}Mo_1Fe_4Si_{13.5}B_{13.5}$ powders obtained in the process of milling of the ribbons in the "as quenched" state proved that the process of the high energy ball milling causes significant increase in the coercive force. The powder obtained after 5-hour milling of the amorphous ribbon is characterized by the highest value of the coercive force (H_C=159,92). The longer the time of milling is, the higher the value of the parameter after 20-hour milling H_C=1286,62 A/m.

The longer the milling process, the smaller the value of the saturation of magnetization, which for the powder obtained after 5-hour milling of the amorphous $Co_{68}Mo_1Fe_4Si_{13,5}B_{13,5}$ ribbon amounts to B_S=0,63 T. For the powder obtained in 20-hour milling, the value B_S equals 0,74 T.

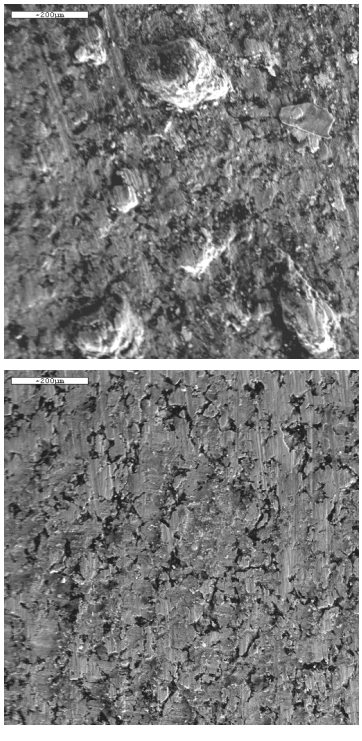

Fig. 3. The image of $Co_{68}Fe_4Mo_1Si_{13.5}B_{13.5}$ surface structure of die stamping hot pressed in a) 950°C, and b) 800°C per 20 minutes

The mass density of material of amorphous $Co_{68}Mo_1Fe_4Si_{13.5}B_{13.5}$ ribbon which was the precursor of die stampings carries out 7.8 g/cm^3. The mass density of powder after hot pressing in vacuum was calculated, which carries out ρ_{800}=3.789 g/cm^3 for temp. 800°C and ρ_{950}=4.042 g/cm^3 for temp. 950°C. The surface structure of die stampings shows on Fig. 3.

The magnetic research of the hot pressed powder $Co_{68}Mo_1Fe_4Si_{13.5}B_{13.5}$ obtained in the process of pressing of the powders proved that the process causes significant increase in the coercive force. The die stamping material obtained after hot pressing in 800°C per 20 minutes in vacuum from the metallic powder is characterized by the highest value of the coercive force (H_C=1363,1 A/m). The higher the temperature of pressing is, the higher the value of the parameter after 950°C pressing H_C=3517,0 A/m .

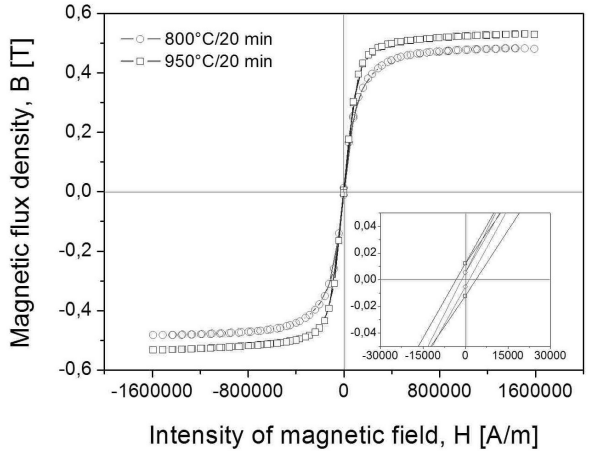

Fig. 4. Histeresis loop of the powder of $Co_{68}Fe_4Mo_1Si_{13,5}B_{13,5}$ alloy hot pressed in 800 and 950°C/20 minutes

A specially significant is the growing value of coercive force H_C with grooving temperature of hot pressing. The coercive force value increases up to 950°C. The saturation magnetization B_S changed too, the value decreases form 0.74 T for powder obtained after 20 hours of high energy ball milling to 0.48 T for die stamping obtained in 800°C per 20 minutes and for 0.52 T for die stamping obtained in 950°C.

The lower the temperature of hot pressing, the smaller the value of the saturation of magnetization, which for the die stamping of the powdered $Co_{68}Mo_1Fe_4Si_{13.5}B_{13.5}$ ribbon amounts to B_S=0,48 T. For the die stamping obtained in temperature 950°C, the value B_S equals 0,52 T. The value of the residual flux density is very low for both pressed materials and B_r is equal 0.0124 T and 0.0055 for die stamping obtained respectively at 800°C and 950°C (Table 2).

Table 2.
Magnetic properties of powder cores obtained in hot pressing process

Materials	B_s [T]	H_c [A/m]	B_r [T]	H_{max} [T]
5h HEBM	0,63	159,9	0,0015	796178,3
20h HEBM	0,74	1286,6	0,0076	
800°C/20'	0,48	1363,1	0,0124	1592356,7
950°C/20'	0,53	3517,0	0,0055	

HEBM – High Energy Ball Milling process

4. Conclusions

On the basis on results of investigations of magnetic properties of the powder material, it was found out that compared to the magnetic properties of the amorphous ribbon and powder obtained from ribbons in high energy ball milling as their both precursor, the hot pressing process deteriorates their magnetically soft properties.

On the basis on analysis of diffraction patterns and using Scherrer's relationship (1) the Co-α and Co-β grains size was calculated, whose dimensions grow in this case along with the hot pressing temperature decrease, but probably the calculated peaks include the background form amorphous phase and the results are not binding.

Acknowledgements

The authors are very grateful to dr. Anna Dolata-Grosz from Department of Metal Alloy and Composites of Technology of Silesian University of Technology for performing hot pressing of metallic powders.

References

[1] C. Miguel, A. Zhukov, J.J. del Val, J. Gonzalez, Coercivity and induced magnetic anisotropy by stress and/or field annealing in Fe- and Co- based (Finemet-type) amorphous alloys, Journal of Magnetism and Magnetic Materials 294 (2005) 245–251.

[2] R. Nowosielski, S. Griner, Shielding of electromagnetic fields by mono- and multi-layer fabrics made of metallic glasses with Fe and Co matrix, Journal of Achievements in Materials and Manufacturing Engineering 20 (2007) 45-54.

[3] S. Lesz, R. Nowosielski, A. Zajdel, B. Kostrubiec, Z. Stokłosa, Structure and magnetic properties of the amorphous $Co_{80}Si_9B_{11}$ alloy, Journal of Achievements in Materials and Manufacturing Engineering 18 (2006) 155-158.

[4] J. Moya, V. Cremaschi, F.C.S. Silva, M. Knobel, H. Sirkin, Influence of the heat treatment method on magnetic and mechanical properties of the $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$ alloy, Journal of Magnetism and Magnetic Materials 226-230 (2001) 522-1523.

[5] J. Gutiérrez, J.M. Barandiarán, P. Mínguez, Z. Kaczkowski, P. Ruuskanen, G. Vlasák, P. Svec, P. Duhaj, Influence of heat treatment on the magnetic and piezomagnetic properties of amorphous and nanocrystalline $Fe_{64}Ni_{10}Nb_3Cu_1Si_{13}B_9$ alloy strips, Sensors and Actuators A 106 (2003) 69–72.

[6] Y. Iwami, Y. Okazaki, A. Saito, Unique stress-magnetization change of Co-based amorphous ribbon due to magnetic field heat treatment, Journal of Magnetism and Magnetic Materials 254–255 (2003) 127–129.

[7] G. Saage, S. Roth, J. Eckert, L. Schultz, Low magneto-striction crystalline ribbons prepared by melt spinning and reactive annealing, Journal of Magnetism and Magnetic Materials 254–255 (2003) 26–28.

[8] R. Yapp, B.E. Watts, F. Leccabue, Rapid solidification of Fe-Cr-Si-B alloys by melt spinning, Materials Science and Engineering A304–306 (2001) 1008–1010.

[9] J. Degmová, I. Tóth, J. Bednarčík, P. Kollár, The influence of ball-milling on structural and magnetic properties of Co-based powders, Czechoslovak Journal of Physics 55 (2005) 791-801

[10] P. Gramatyka, A. Kolano-Burian, R. Kolano, M. Polak, Nanocrystalline iron based powder cores for high frequency applications, Journal of Achievements in Materials and Manufacturing Engineering 16 (2006) 99-102.

[11] H. Moumeni, S. Alleg, J.M. Greneche, Structural properties of $Fe_{50}Co_{50}$ nanostructured powder prepared by mechanical alloying, Journal of Alloys and Compounds 386 (2005) 12-19.

[12] J. Konieczny, L.A. Dobrzański, J. E. Frąckowiak, Structure and magnetic properties of powder HITPERM material, Archives of Materials Science and Engineering 28/3 (2007) 156-164.

[13] Y.B. Kim, D.H. Jang, H.K. Seok, K.Y. Kim, Fabrication of Fe–Si–B based amorphous powder cores by cold pressing and their magnetic properties, Materials Science and Engineering A 449–451 (2007) 389–393.

[14] P. Gramatyka, R. Nowosielski, P. Sakiewicz, Magnetic properties of polymer bonded nanocrystalline powder, Journal of Achievements in Materials and Manufacturing Engineering 20 (2007) 115-118.

[15] P. Gramatyka, A. Kolano-Burian, R. Kolano, M. Polak, Nanocrystalline iron based powder cores for high frequency applications, Journal of Achievements in Materials and Manufacturing Engineering 18 (2006) 99-102.

[16] Z. Bojarski, E. Łągiewka, Structural X-ray analysis, Universsity of Silesia Publishing House, Katowice 1995.