Achievements in Mechanical and Materials Engineering

COMMENT Worldwide Congress on Materials and Manufacturing Engineering and Technology

16th - 19th May 2005
Gliwice-Wisla, Poland

COMMITTEE OF MATERIALS SCIENCE OF THE POLISH ACADEMY OF SCIENCES, KATOWICE, POLAND
INSTITUTE OF ENGINEERING MATERIALS AND BIOMATERIALS OF THE SILESIAN UNIVERSITY OF TECHNOLOGY, GLIWICE, POLAND
ASSOCIATION OF THE ALUMNI OF THE SILESIAN UNIVERSITY OF TECHNOLOGY, MATERIALS ENGINEERING CIRCLE, GLIWICE, POLAND

13th INTERNATIONAL SCIENTIFIC CONFERENCE
ON ACHIEVEMENTS IN MECHANICAL AND MATERIALS ENGINEERING

Ferromagnetic properties of polymer nanocomposites containing $Fe_{78}Si_9B_{13}$ powder particles

R. Nowosielski[a], J.J. Wysłocki[b], I. Wnuk[b], P. Sakiewicz[a], P. Gramatyka[a]

[a]Institute of Engineering Materials and Biomaterials, Silesian University of Technology
ul. Konarskiego 18a, 44-100 Gliwice, Poland, email: pawel.gramatyka@polsl.pl

[b]Institute of Physics, Częstochowa University of Technology
Al. Armii Krajowej 19, 42-200 Częstochowa, Poland

Abstract: Nanocomposites were made from the powders obtained by ball milling of metallic glasses. Influence of the metallic powder fraction on soft magnetic properties was investigated along with the possibility to control these properties with the size and amount of powder fraction. These results showed that magnetic properties of the polymer composites can be controlled in a wide range and depends on mass fraction of the metallic powder $Fe_{78}Si_9B_{13}$ in the composite, on shape and size of the powder particles and their orientation in composite.

Keywords: Magnetic properties, $Fe_{78}Si_9B_{13}$ alloys, Powder cores, Nanocomposites

1. INTRODUCTION

In recent years the range of available soft magnetic materials has been significantly increased by the development of nanocrystalline magnetic materials. The most widely investigated alloys are iron-based metallic glasses e.g. Metglas ($Fe_{78}Si_9B_{13}$) and the Finemet ($Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$) [1]. The first of them are characterized by small initial permeability, high magnetic saturation induction, and are relatively cheap while the Finemet ribbons have very high initial permeability but are very expensive.

The ball milling techniques of metallic glasses ribbons is often used to produce nanocrystalline soft magnetic material which is in powder form, and therefore is suitable for consolidation in a variety of shapes [2, 3]. The proposed consolidation techniques of powders, like sintering, hot isostating pressing, warm compaction, explosive compaction, shock – wave compaction and static high – pressure compaction are costly and complex [4]. That is why the most popular consolidation technique is mixing metallic powders with mineral or polymer binder [5]. The optimization of the magnetic properties of nanocrystalline powder composites can be achieved by adjusting uniform packing density, the powder size, the volume binder rate, the nature of the binder and ideal orientation of powders particles in the cores.

The aim of this study is the preparation of toroidal shape nanostructured composite cores from high energy ball milled $Fe_{78}Si_9B_{13}$ powders and to investigate the powder particle size and content of polymer binder response of the soft magnetic properties.

2. EXPERIMENTAL DETAILS

Amorphous $Fe_{78}Si_9B_{13}$ metallic glasses ribbons were prepared by the melt spinning method. The width and thickness of these ribbons were 11 mm and 0,028 mm, respectively. The ribbons in "as quenched" state were cut into small pieces of about 5 mm transverse dimension. These particles were sealed in a cylindrical stainless steel container under an Ar atmosphere. The milling process reduced the starting material pieces (metallic glasses) to powder form. The metallic powder particles were sieved to obtain different sizes – small with average particles diameter between 25 to 75 µm, medium particles 75÷200 µm and the large ones between 200÷500 µm. The powder particles were annealed to generate the nanocrystalline state and to reduce stresses induced by the high energy ball milling process. Thermal annealing of powder particles were carried out at 773 K for 1 hour in an argon atmosphere. Thermal annealed powders were mixed then with different contents of binder and condensed on mechanical shaker. As a binder, a silicone polymer Dublisil 20 from Dreve-Dentamid GMBH was used. Polymerization process was made under magnetic field H = 500 [A/m] to ensure a preferential orientation of a powder particles. The toroidal cores so obtained had its external diameter of 34 mm, internal diameter of 28 mm and height of 8 mm. Examination of powders and composite cores magnetic properties (hysteresis loops) were carried out on the Lake Shore Cryotronics VSM vibration sample magnetometer with a maximum applied magnetic field of 800 kA/m. The permeability of the powders and composites cores was made on the Ferrometr device.

3. RESULTS AND DISCUSSION

Table 1 comprises the results of examination of the magnetic properties of the $Fe_{78}Si_9B_{13}$ alloy powders with different particles size.

Table 1.
Magnetic properties of $Fe_{78}Si_9B_{13}$ powders

Powder particles size [µm]		B_S [T]	B_r [T]	H_c [A/m]	H_{max} [kA/m]	µ at H=3 [kA/m]
200 ÷ 500	as milled	1.06	0.068	1341	800	39
	after annealed at 773K	1.22	0.090	237	800	106
75 ÷ 200	as milled	0.98	0.061	1920	800	18
	after annealed at 773K	1.09	0.075	863	800	63
25 ÷ 75	as milled	0.72	0.048	3215	800	12
	after annealed at 773K	1.02	0.062	1240	800	38

It was found that the larger powder particles (200 ÷ 500 µm) exhibits better soft magnetic properties than the small ones i.e. the coercive field H_c = 1341 [A/m], the saturation induction B_s = 1.06 [T] and the remanence B_r = 0.068 [T]. For small particle size (25 ÷ 75 µm) powder sample demonstrated the value of B_s = 0.72 [T] and the remanence B_r = 0.048 [T], whilst coercive field H_c = 3215 [A/m]. The permeability value of large and medium powder particles (measured at magnetic field H = 3 [kA/m] and at frequency f = 50 Hz) are µ = 39 and µ = 18 respectively. Annealing powders particles at 773 K produces the effect of a considerable increase of their magnetic properties. For large particles the value of coercive field was equal to 237 [A/m], the saturation induction B_s = 1.22 [T], the remanence B_r = 0.09 [T] and the permeability exhibited value of 106.

The results of examination of the magnetic properties of the nanocomposite cores with different powder-to-silicone polymer ratio are shown in Table 2.

Table 2.
Magnetic properties of polymer composites containing $Fe_{78}Si_9B_{13}$ powder particles.

Composite cores			B_S [T]	B_r [T]	H_c [A/m]	H_{max} [kA/m]	μ at H=3 [kA/m]
Powder particles size [μm]	Powder to silicone ratio	Content of powder [wt%]					
200÷500	6 : 1	85.7	1.06	0.084	312	800	98
	5 : 1	83.3	0.99	0.080	344	800	93
	4 : 1	80.0	0.97	0.080	338	800	79
	3 : 1	75.0	0.95	0.068	580	800	56
	2 : 1	66.6	0.91	0.062	692	800	40
	1 : 1	50.0	0.78	0.044	667	800	22
75÷200	6 : 1	85.7	0.94	0.070	1420	800	56
	5 : 1	83.3	0.94	0.071	1563	800	54
	4 : 1	80.0	0.94	0.070	1542	800	54
	3 : 1	75.0	0.86	0.061	1933	800	33
	2 : 1	66.6	0.78	0.048	2359	800	25
	1 : 1	50.0	0.73	0.045	2765	800	17
25÷75	6 : 1	85.7	0.86	0.054	2015	800	33
	5 : 1	83.3	0.87	0.056	1973	800	36
	4 : 1	80.0	0.83	0.048	2262	800	30
	3 : 1	75.0	0.79	0.048	2459	800	24
	2 : 1	66.6	0.76	0.041	3327	800	20
	1 : 1	50.0	0.68	0.040	3823	800	13

The nanocrystalline cores with powder particles between 200 ÷ 500 μm exhibited high value of saturation induction and coercive field was comparable for the powders obtained from ball milled ribbon and annealed in argon atmosphere for 1h at temperature of 773 K. The results of experimental studies demonstrated a correlation between the growing powder to silicon polymer ratio and the improvement of the magnetic properties of the composite. The composite core with a powder particle content of 85.7 wt% exhibit the coercive field H_c = 312 [A/m], the saturation induction B_s = 1.06 [T] and the remanence B_r = 0.084 [T]. For comparison, the composite polymerized without existence of magnetic field exhibit the coercive field H_c = 370 [A/m], the saturation induction B_s = 0.98 [T] and the remanence B_r = 0.07 [T]. Further decrease powder to silicon polymer ratio leads to deterioration of the magnetic properties, i.e. the coercive field goes considerably high at small values of both the remanence and the saturation magnetic induction.

By decreasing the particles size fraction to 75 ÷ 200 μm the coercive field of composite core with powder particle content of 75 wt% is increased at low values of both the remanence and the saturation magnetic induction. Toroidal cores filled with small powder particles (25÷75 μm) demonstrated worse magnetic properties than the other samples. At 85.7 wt% powder the coercive field H_c was equal 2015 [A/m], the saturation induction B_s = 0.86 [T] and the remanence B_r exhibited value of 0.054 [T]. The magnetic permeability measurements confirmed better magnetic properties of nanocomposite cores with powder particles between 200 ÷ 500 μm. These results represent the highest reported permeability (measured at magnetic field H = 3 [kA/m] and at frequency f = 50 Hz) μ = 98 for core with $Fe_{78}Si_9B_{13}$ powder particle content of 85.7 wt%. The medium powder particles (75÷200 μm) in the composite cores showed permeability between 17 and 56 depending on the powder to silicon polymer ratio. It was also found that for powder particles size between 25 ÷ 75 μm, composite cores with different content of polymer binder showed permeability in the range 13 ÷ 33 (measured at magnetic field H = 3 [kA/m] and at frequency f = 50 Hz).

4. CONCLUSION

The experimental studies showed that size and shape of the powder particles ware responsible for soft magnetic properties of powder cores. Decreasing size of powder particles led to magnetic hardening of nanocrystalline $Fe_{78}Si_9B_{13}$ alloy. The main reason for the worse magnetic properties of small particles was high demagnetization factor. Additionally, structural defects induced during ball milling process of small particles also caused deterioration of magnetic properties. The control of the soft magnetic properties of composite cores consolidated from nanocrystalline powders and silicone polymer binder can be achieved by adjusting powder particle size and powder to silicon polymer ratio. It was noticed that polymerization process made under magnetic field H = 500 [A/m] ensured a preferential orientation of a powder particles in composites and caused the improvement of soft magnetic properties compared with composites polymerized without existence of magnetic field. It was found that composite cores fabricated from silicon polymer-solidified $Fe_{78}Si_9B_{13}$ powder particles exhibits magnetic properties that strengthen with the growing metallic powder-to-silicon weight ratio.

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