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Bulk CoNiFe-SiB Amorphous and Nanostructured Alloys Produced by Plasma Spray Deposition and Dynamic Compaction: Formation of Soft Magnetic Properties

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Abstract

The bulk nanostructured $Co_{58}Ni_{10}Fe_5B_{16}Si_{11}$ alloys were prepared by dynamic compaction and plasma spray deposition techniques. The investigation of structure and magnetic properties of bulk samples was carried out by X-ray diffraction, electron microscopy and correlation magnetometry. The bulk samples produced by both methods can be characterized as a heterophase system. The highest value of permeability $20\cdot10^3$ for plasma spraying coating is achieved when volume fraction of nanocrystalline phase with Curie temperature $T_c \sim 640$ K is increased to 30%. The magnetic characteristics, such as the saturation magnetization, the Bloch constant, the local magnetic anisotropy field, the ferromagnetic resonance linewidth, and the coercivity remain unchanged after both compaction techniques. It was shown that the plasma spraying method allows to obtain bulk magnetically soft materials with magnetic parameters that are not inferior to the characteristics of a thermally treated rapidly quenched ribbon with the same composition.

Keywords: bulk nanostructured materials, dynamic compaction, plasma spray deposition, magnetic properties.

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1 Introduction

Nanostructured and amorphous coatings without grain growth can be formed by a variety of methods (1. M. Shapaan, 2004) (M. Cherigui, 2004) (Chen, 2004), but the plasma spray process permits the use of complex geometry substrates. Then its use is extended to a wide range of applications. Plasma spray processing is a droplet deposition method that combines the steps of melting, rapid solidification, and consolidation into a single step. The versatility of the technology enables the processing of freestanding bulk, near-net shapes of a wide range of alloys, intermetallics, ceramics, and composites, while still retaining the benefits of rapid solidification processing (Pawlik, 2010) (K.H. Chen, 2004). The microstructure of the bulk coating can be tuned from full amorphous state to composite with nanocrystalline particles in the amorphous matrix, and finally to nanostructured state by using this synthesis technique. The transformation is accompanied by a change in magnetic properties.

The shock wave compaction is attractive because it can be used as an effective compaction method for powder materials without recrystallization and decomposition including activated microstructural changes. The shock compaction can produce high compressive stresses during short time periods, thereby heavily deforming the particles, melting the surfaces, and producing a fully dense compact without the grain growth.

It was found (A. A. Lepeshev, 1995) that the desired magnetic properties of $Co_{58}Ni_{10}Fe_5B_{16}Si_{11}$ bulk alloy (zero magnetostriction, high initial and maximum magnetic permeability, etc.) can be achieved by a formation of bulk samples with a certain amorphous/crystalline phase ratio. In the present work, the role of the plasma spray deposition and the shock wave compaction regimes in the control of microstructure and magnetic property of the $Co_{58}Ni_{10}Fe_5B_{16}Si_{11}$ bulk samples, on the mechanism of the nanocrystalline and amorphous structure formation, and the relation between the microstructure and magnetic properties are discussed. A comparison between the magnetic properties of the $Co_{58}Ni_{10}Fe_5B_{16}Si_{11}$ alloys prepared by dynamic compaction and a plasma spray deposition techniques is carried out.

2 Experiment

The bulk samples were prepared by two methods using a powder obtained from an amorphous $Co_{58}Ni_{10}Fe_5B_{16}Si_{11}$ ribbon. The first method is dynamic compaction (samples size is 7×12×1.5mm), the second one is plasma spray deposition (thickness of coating is 2 mm). The preparation of bulk samples includes several process steps: obtaining of rapidly quenched amorphous ribbon; annealing of ribbon at 450^0C to embrittlement; ribbon grinding in powder; and finally, plasma spraying or dynamic compaction. The details of the plasma spray deposition and dynamic compaction processes were reported elsewhere (V. N. Saunin, 2012).

The regimes of dynamic compaction and plasma spray deposition were selected so that the basic magnetic characteristics (saturation magnetization, M_0, exchange stiffness constant, D, local magnetic anisotropy field, H_a, FMR linewidth, ΔH, coercive field, H_c) remain unchanged. The morphology and the composition of the investigated materials were analyzed using a scanning electron microscope (S5500 Hitachi and an energy-dispersive spectrometer TM3000). The surface of a metallographic specimen is prepared by grinding, polishing, and etching in a mixture of nitric acid, hydrochloric acid and alcohol. The structure of the bulk samples was determined using a DRON-4 X-ray diffractometer operating with Cu Kα radiation. Magnetization was measured as functions of temperature, field orientation using a vibrating sample magnetometer. The amorphous/crystalline phase ratio was estimated using the technique of magnetic phase analysis. The phases were identified on the basis of their Curie temperatures (T_C) and saturation magnetizations. Information on local anisotropy field is obtained from investigation of approach to saturation magnetization law adapted to heterophase systems in (Ivanov, 1997).

3 Results and Discussion

The atomic structure of the bulk samples was studied by X-ray diffraction and NMR study. In Fig.1 the XRD patterns of the starting ribbon, the dynamic compacted Co-Ni-Fe-Si-B bulk sample and bulk coating are plotted. In all cases we observed an amorphous halo with marginal changes after compaction. The particle sizes of starting powders and the grain sizes of the consolidated samples were estimated using the Debye-Scherer formula. A slight increase in the grain size was observed after both type of consolidation. The average particle size changed from 8 nm to 11 nm. As NMR analysis showed the samples were characterized by the same type of nearest environment of cobalt atom.

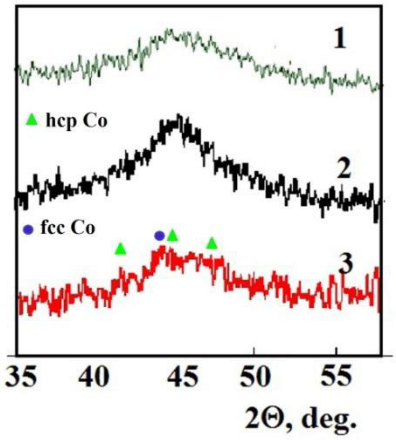

Figure 1. XRD patterns of the starting ribbon (1), the dynamic compacted Co-Ni-Fe-Si-B bulk sample (2) and bulk coating (3).

It is known that the plasma coatings are formed by agglomeration of splats formed by the impact, spread and solidification of individual particles. The particles may be fully or partially melted. The power of the electric arc, *P*, determines the temperature and velocity of sprayed particles, and the substrate temperature, T_s, determines the quenching rate. In dynamic compaction case the defining characteristic is loading pressure. It is found that the structure and magnetic characteristics (the diffraction pattern, heat and temperature of crystallization $T_{crys} \sim 540^oC$, saturation magnetization, $M_0 \sim 640$ Gs, exchange stiffness constant, $D \sim 117$ meV·A^2, local magnetic anisotropy field, $H_a \sim 1$ kOe, coercive field, H_c) of the alloy remain unchanged if power of the electric arc $P < 20$ kW, the substrate temperature substrate temperature $T_s < 150^oC$ and if the loading pressure $P_L < 3.2$ GPa. The SEM images of bulk samples produced by both methods at optimal regimes are presented at Fig.2. EDX analysis shows that such bulk samples are characterized by homogeneous distribution of the elements. Suboptimal spraying conditions ($T_s > 200^oC$) cause quenching rate decrease and some part of splat is characterized by inhomogeneous structure. In figure 2c you can see the inclusions reached by Fe and Si. When the temperature in the spraying spot is optimal, the process of coating deposition is combined with thermal treatment of the earlier sputtered layer with the melted metal of the next layer. In this case, recrystallization of the alloy accompanied by the appearance of nanocrystals in the amorphous matrix takes place. Such microstructure modification can cause a change of the coercive field and the permeability of the bulk Co-Ni-Fe-Si-B sputtered coatings.

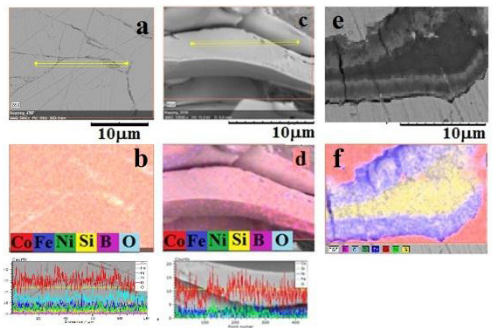

Figure 2. SEM images of bulk Co-Ni-Fe-Si-B samples: a – amorphous compact (the etched longitudinal section) and EDX map (b); amorphous coating (cross-section, fracture) T_s=100^{o}C (c) and EDX map (d); SEM image of the etched longitudinal section of bulk Co-Ni-Fe-Si-B coating T_s=400^{o}C (e) and its EDX map (f).

Thermomagnetic curves for the $Co_{58}Ni_{10}Fe_5B_{16}Si_{11}$ coating annealed at different temperature are presented in figure 3. Thermomagnetic curves of initial bulk samples produced by both investigated technique coincide. The results of thermomagnetic analysis of the annealed coating indicate that the initial alloy can be characterized as a heterophase system in which phase 1 with T_C~550 K comprises 90% of the volume and the phase 2 with T_C~840 K comprises 10%. The Curie temperatures of these phases are different and consequently chemical short-range order in these phases is different. It is found that the appearance of dispersed inclusions of phase 2 in phase 1 was stimulated by an increase of substrate temperature. At T_{an}~300°C the nanocrystalline phase inclusions with T_C~640 K arise. Figure 3b represents the dependencies of permeability and volume fraction of nanocrystalline phase on T_{ann}. You can see that they commove. So it is likely that the maximum of permeability was caused by the appearance of the nanocrystalline phase inclusions. The highest value of permeability $20 \cdot 10^3$ is achieved when volume fraction of the amorphous phase 1 is decreased to 50% and nanocrystalline phase volume fraction (Y_3) is increased to 30%.

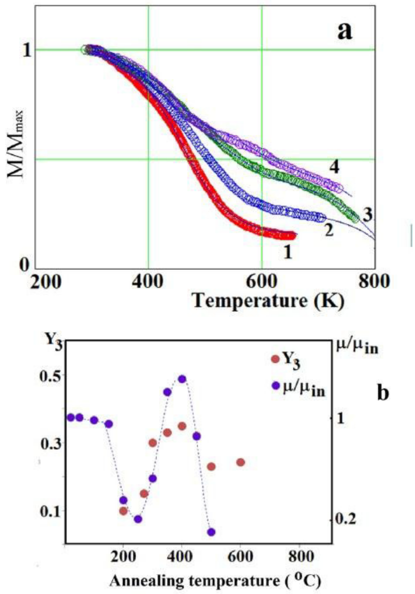

Figure 3. Thermomagnetic curves for the $Co_{58}Ni_{10}Fe_5B_{16}Si_{11}$ coating annealed at different temperature: 1- Tan~100°C; 2- Tan~400°C; 3- Tan~600°C;4-Tan~800°C (a); The dependencies of Y_3 (the volume fraction of phases with Curie temperature 640 K) and permeability on annealing temperature (b).

The values of Bloch constant, B, saturation magnetization, M_0, and spin-wave stiffness, D were determined from low temperature measurement of magnetization M(T). The variation of magnetization with temperature of both type of bulk samples (compacts and coatings) obeys Bloch's law $M(T) = M_0(1 - BT^{3/2})$. Using this equation we determined the value of Bloch constant, *B*, and saturation magnetization, M_0. The initial CoFeNi-SiB amorphous ribbon has $B = 2.4 \cdot 10^{-5}$ $K^{-3/2}$. The *B* value practically does not change during both compaction processes. The saturation magnetization, M_0, is equal to 625 G for the initial ribbon. Neither shock compaction process, no plasma spraying process changed the value of M_0. This indicates that both compaction processes do not cause an oxidation of the powder particles.

The magnetization curves for the $Co_{58}Ni_{10}Fe_5B_{16}Si_{11}$ coating and compact are presented in figure 4. The analysis of the magnetic hysteresis loops curves shows that plasma spraying does not cause a change of H_c value (0.3 Oe). The coercive field of shock wave compacted sample is slightly bigger than that of the initial ribbon (0.5 Oe). Information on local magnetic anisotropy field H_a (contain a contribution of internal stresses) was obtained from investigation of approach magnetization to saturation law (R. S. Iskhakov and S. V Komogortsev, 2011). The magnetic saturation curves of all the nanocomposites obey Akulov's law $M(H) \sim (H)^{-2}$ for applied fields up to 4 ÷ 11kOe. This allowed us to determine the values of mean square fluctuations of local magnetic anisotropy field, aH_a. The value of aH_a changes during the formation of bulk samples (for the initial amorphous ribbon aH_a = 1kOe, for grinding ribbon aH_a = 14 kOe, for compacted sample is 1.3 kOe, and for coating is 1.1 kOe). The magnetic saturation curves obey the law $M(H) \sim (H)^{-0.5}$ for applied fields less than 2.5 kOe. Such *M(H)* behavior reveals that the bulk

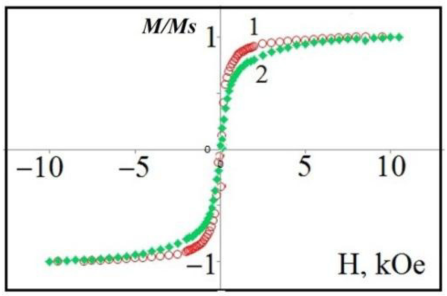

Figure 4. The magnetization curves for the $Co_{58}Ni_{10}Fe_5B_{16}Si_{11}$ coating (1) and compact (2)

materials produced by shock compaction technique as well as plasma spraying technique are nanostructured.

$Co_{58}Ni_{10}Fe_5B_{16}Si_{11}$	M_0, G	D, mev·A^2	aH_a, kOe	Hc, Oe	μ Eff.	ΔH kOe
Ribbon	625	117	1	0.3	$20 \cdot 10^3$	0.9
Coating	625	117	1.1	0.3	$20 \cdot 10^3$	1.1
Compact	625	117	1.3	0.5	-	1.1

Table 1 : Magnetic measurements results

Magnetic property measurements were performed on samples obtained from several regions of the same compact. We revealed uniformity in magnetic properties that indicates the uniform shock pressure and density distribution throughout the samples. The ferromagnetic resonance spectra of the initial ribbon and bulk samples were studied as well. The FMR spectra consist of a single absorption line for all types of samples. The FMR linewidth of grinding ribbon ($\Delta H \approx 3.5$kOe) is significantly larger than that of initial amorphous ribbon ($\Delta H \approx 0.9$ kOe). The ΔH value decreases after both consolidation processes ($\Delta H \approx 1.1$ kOe). Magnetic measurements results are summarized in Table 1. It is seen that plasma spraying is prefer for producing bulk soft magnetic materials.

4 Conclusions

The bulk nanostructured CoFeNi-SiB alloys were prepared by dynamic compaction and plasma spray deposition techniques. For plasma spraying coating the appearance of nanocrystalline phase with Curie temperature $T_c \sim 640$ K during relaxation heat treatment leads to a decrease of the coercivity (0.3 Oe) and to an increase of the permeability up to $20 \cdot 10^3$. The results of magnetic measurements lead to the conclusion that the dynamic compaction and the plasma spray deposition techniques conducted under the optimal regime allow obtaining bulk nanostructured CoNiFe-BSi alloys with the same diffraction pattern, heat and temperature of crystallization, saturation magnetization (Mo - 600 G), spin-wave stiffness (D – 117 meV·A^2), and Curie temperature than those of the rapidly quenched $Co_{58}Fe_5Ni_{10}B_{16}Si_{11}$ ribbon. It was shown that the plasma spraying method allows to obtain bulk magnetically soft materials with magnetic parameters that are not inferior to the characteristics of a thermally treated rapidly quenched ribbon with the same composition.

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References

1. M. Shapaan, A. B. (2004). Thermal stability and glass forming ability of cast iron based CiPxB4.35 bulk amorphous alloys. *Phys. status solid*, 476-481.

A. A. Lepeshev, R. S. (1995). Plasma deposition of amorphous Co-Ni-Fe-B-Si alloys: Structure and magnetic properties. *Tech. Phys. Lett, 21*(8), 641-643.

Chen, S. C. (2004). Nanocrystalline materials and coatings. *Mater. Sci. Eng. R Reports, 45*(1-2), 1-88.

Ivanov, G. V. (1997). Study of nanocrystal FINMET alloy . *Fiz. Met. Met., 84*(1), 55-61.

K.H. Chen, Z. J. (2004). Bulk nanocomposite magnets produced by dynamic shock compaction. *J. Appl. Phys, 96*, 1276-1280.

M. Cherigui, H. .. (2004). Structure of amorphous iron-based coatings processed by HVOF and APS thermally spraying. *Mater. Chem. Phys, 85*(1), 113-119.

Pawlik, J. J. (2010). Nanocrystalline materials and coatings. *J. Achievments Mater. Manuf. Eng, 43*(1), 463-468.

R. S. Iskhakov and S. V Komogortsev. (2011). “Magnetic microstructure of amorphous, nanocrystalline, and nanophase ferromagnets. ” *Phys. Met. Metallogr, 112*(7), 666-681.

V. N. Saunin, S. V. (2012). Formation of bulk magnetically soft materials with nano- and amorphous structure using plasma spraying. *Inorg. Mater. Appl. Res, 3*(3), 201-209.