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Phase Composition and Magnetic Properties of the Nanocrystalline $Fe_{64.32}Pr_{9.6}B_{22.08}W_4$ Alloy

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The aim of the present work was to study the influence of annealing conditions on magnetic properties and the phase constitution of rapidly solidified $Fe_{64.32}Pr_{9.6}B_{22.08}W_4$ alloy ribbons. The base alloy was prepared by arc-melting of the high purity elements under an Ar atmosphere. Subsequently the ribbon samples were obtained by melt-spinning technique under low pressure of Ar. In order to develop nanocrystalline structure, the samples were annealed at 1003 K for 5, 10, 20 and 30 min. The room temperature magnetic properties were determined from hysteresis loops measured by VSM magnetometry in the external magnetic field up to 2 T. For comparison the influence of annealing temperature on magnetic properties was studied for the same alloy composition. The ribbons were annealed at temperatures from 929 K to 1023 K for 5 min. X-ray diffractometry was used to determine the phase composition of annealed ribbons. Heat treatment resulted in an evolution of the phase constitution, that caused changes in magnetic properties of the alloy.

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1. Introduction

A breakthrough in the development of magnetically hard materials turned out to be the 1984 invention of the $Nd_2Fe_{14}B$ phase [1, 2]. From that moment the RE-Fe-B (RE – rare earth elements) magnetic materials are the subject of intensive research. A numerous work was done in order to improve the properties and to find more favorable manufacturing techniques. To obtain optimal magnetic properties, selection of the alloy composition as well as suitable conditions of technological process are crucial [3-8].

2. Experimental

The ingot samples with the nominal composition $Fe_{64.32}Pr_{9.6}B_{22.08}W_4$ were obtained by arc-melting of high purity elements in an argon atmosphere. The ribbon samples were produced by melt-spinning technique under the Ar atmosphere at linear speed of the copper roll surface of 25 m/s. In order to obtain a nanocrystalline microstructure and induce changes in magnetic parameters the samples were heat-treated. To prevent oxidation during heat treatment, the ribbon samples were sealed off in a quartz tube under low pressure of argon. The evolution of nanocrystallization was followed by annealing at 929, 948, 953 and 966 K for 5 min. The effect of annealing time on the phase structure and magnetic parameters was studied at 1003 K for 5, 10, 20 and 30 min annealing times. The phase analysis of the samples was carried out using Bruker D8 Advance diffractometer with CuK_α radiation. Room temperature hysteresis loops were measured by LakeShore 7307 vibrating sample magnetometer at external magnetic field up to 2 T.

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3. Results and discussion

The XRD patterns measured for the as-cast $Fe_{64.32}Pr_{9.6}B_{22.08}W_4$ alloy ribbons and for samples annealed at 929 K, 948 K, 953 K and 966 K for 5 min are shown in Fig. 1.

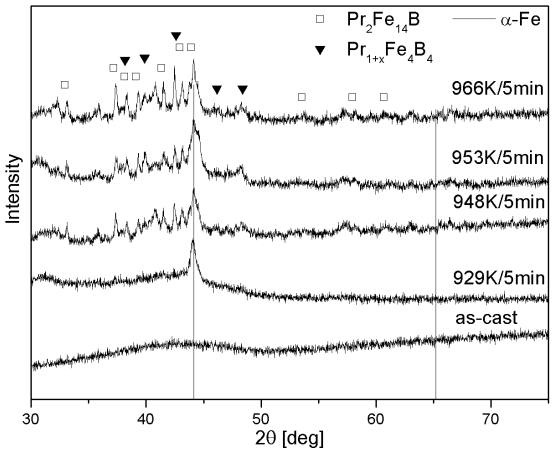

Fig. 1. X-ray diffraction patterns of as-cast $Fe_{64.32}Pr_{9.6}B_{22.08}W_4$ alloy ribbon sample and samples annealed at 929 K, 948 K, 953 K and 966 K for 5 min.

Lack of peaks corresponding to crystalline phases suggest an amorphous structure of as-cast samples.

It was shown that for samples annealed at 929 K for 5 min a crystallization of α-Fe phase takes place, however the samples are partially amorphous. Annealing at

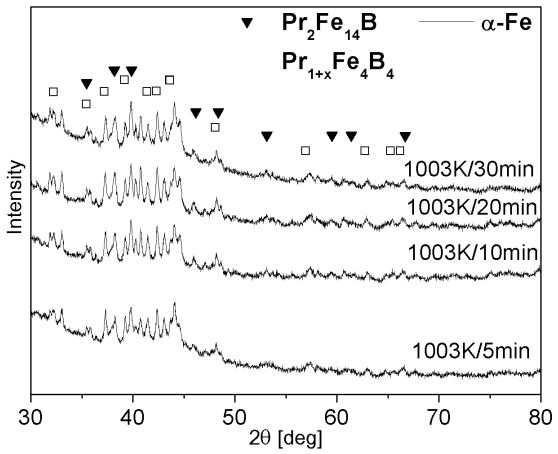

Fig. 2. X-ray diffraction patterns of $Fe_{64.32}Pr_{9.6}B_{22.08}W_4$ alloy ribbon samples annealed at 1003 K for 5, 10, 20 and 30 min.

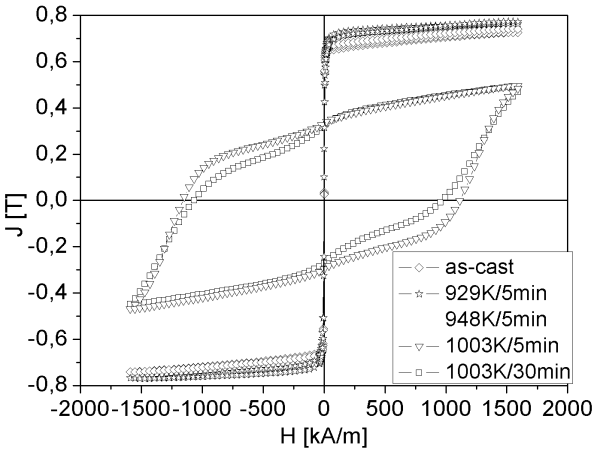

Fig. 3. The hysteresis loops of $Fe_{64.32}Pr_{9.6}B_{22.08}W_4$ alloy ribbon samples annealed at 929 K, 948 K and 1003 K for 5 min and at 1003 K for 30 min.

a temperatures of 948 K and higher for 5 min allows to obtain a nanocrystalline structure and led to nucleation and growth of the crystalline phase. The main crystalline phase observed in the investigated material is the hard magnetic $Pr_2Fe_{14}B$. The analysis indicates also a presence of the $Pr_{1+x}Fe_4B_4$ paramagnetic and α-Fe soft phases.

With the increase of annealing time, no significant changes in the phase structure, related to the contribution of hard magnetic $Pr_2Fe_{14}B$ phase and the paramagnetic $Pr_{1+x}Fe_4B_4$ were observed (Fig. 2).

The selected hysteresis loops of ribbon subjected to annealing at 929 K, 948 K and 1003 K for 5 min and at 1003 K for 30 min are shown in Fig. 3.

Soft magnetic properties of as-cast ribbons confirm their amorphous microstructure. Annealing at 948 K, and above, resulted in a change of crystalline phases. Magnetic hysteresis loops measured for these samples are characteristic for hard magnetic materials. With the increase of annealing temperature a change of remanence and maximum magnetic energy density were observed. Changes in the shape of magnetic hysteresis loops are related to different microstructure of samples annealed at various temperatures. The maximum value of coercivity $H_c = 1135$ kA/m, remanence $J_R = 0.31$ T, saturation polarization $J_S = 0.50$ T and maximum magnetic energy density $(BH)_{max} = 18$ kJ/m^3 were measured for the ribbon annealed at 1003 K for 5 min. The shape of the magnetic hysteresis loop and magnetic parameters in case of ribbons annealed at constant temperature 1003 K for 5, 10, 20 or 30 min do not shown greater dependence on annealing time.

4. Conclusions

The $Fe_{64.32}Pr_{9.6}B_{22.08}W_4$ alloy in as-cast state had amorphous structure and soft magnetic properties. Heat treatment of these ribbons led to nucleation and growth of the $Pr_2Fe_{14}B$ hard magnetic phase. X-ray diffraction patterns shown presence of additional peaks originating from the $Pr_{1+x}Fe_4B_4$ paramagnetic and α-Fe soft phases. With the increase of annealing temperature an increase of magnetic parameters was observed. The maximum values of these parameters were obtained in the sample annealed at 1003 K for 5 min. Studies have also shown that the increase of the annealing time from 5 to 30 min at a constant temperature of 1003 K results in no improvement of the magnetic properties of investigated alloy.

Acknowledgments

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