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# Hard magnetic properties in $Pr_2Fe_{14}B/\alpha$ -Fe nanocomposite magnets with a combined addition of V and C

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#### ABSTRACT

Nanostructured  $Pr_8Fe_{86-x}V_xB_{6-y}C_y$  (x=0,1; y=0,1) ribbons composed of  $Pr_2Fe_{14}B$  and  $\alpha$ -Fe phases with a high coercivity are fabricated by direct melt spinning. The effects of a single addition of V and a combined addition of V and C on the structures and magnetic properties of melt-spun  $Pr_8Fe_{86}VB_{6-x}C_x$  (x=0 and 1) ribbons have been investigated. Compared with addition-free ribbons, 1 at% V addition is found to reduce the grain sizes of the samples and improve their magnetic properties due to a strong exchange coupling between the hard and the soft phase. A remanence ratio of 0.82, a coercive field of 6.2 kOe and a maximum energy product of 23.4 MGOe in melt-spun  $Pr_8Fe_{85}VB_6$  ribbons are obtained at room temperature. The combined addition of V and C is found to lead to the formation of an intermediate phase of VC at grain boundaries, which appears as a pinning barrier during magnetization and results in an increase of the coercivity value to 6.9 kOe for melt-spun  $Pr_8Fe_{85}VB_5C$  ribbons.

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#### 1. Introduction

Nanocomposite magnets comprised of hard and soft phases, including  $R_2Fe_{14}B$  (R denotes Nd or Pr)/ $\alpha$ -Fe [1,2] or  $Fe_3B/R_2Fe_{14}B$ [3,4] systems, have attracted much attention because they have an isotropic remanence ratio larger than 0.5, a potential maximum energy product of up to 50 MGOe [5] and a low cost due to the relatively low contents of rare-earth elements. But the value of room-temperature optimum energy product (BH)<sub>max</sub> obtained in experiment is about 20 MGOe for nanocomposite melt-spun ribbons [6-9], much lower than the theoretical calculations. In the previous studies, the nanocomposite Fe<sub>3</sub>B/R<sub>2</sub>Fe<sub>14</sub>B system containing less rare-earth elements (3.5-4.5 at%) and higher B content (18-20 at%) has an enhancement in remanence due to the exchange-coupling effect between two kinds of phases and exhibits a very high remanence ratio ranging from 0.7 to 0.8 [3,4,10], but a low coercivity less than 5 kOe. The high fraction of soft phase can restrict its further applications. Recently, a new series of R<sub>2</sub>Fe<sub>14</sub>B-based nanocomposite magnets with 8-10 at% Nd or Pr and 8-11 at% B have aroused much interest due to their high coercivity  $_{i}H_{c}$  of 9–13 kOe [11–14]. But the  $B_{r}$  achieved in this kind of magnet decreases to a value less than 10 kG and, consequently, the values of energy product  $(BH)_{max}$  obtained in the magnets are only in a range of 12-18 MGOe. For the further improvement on the magnetic properties of nanocomposite magnets, much effort should be made to achieve a high coercivity at less expense of remanence, or vice versa, which can lead to a high energy product  $(BH)_{\max}$  in nanocomposite magnets.

It is reported that the remarkable magnetic properties with a product (BH)<sub>max</sub> of about 22 MGOe have been obtained in a nanocomposite  $R_2Fe_{14}B/\alpha$ -Fe magnet with an about 8–9 at% rareearth content [15,16]. A high  $B_r$  of 12.7 kG can be obtained in this system [17], but the low coercivity  $_{i}H_{c}$  (<6kOe) still needs improving. Simulation results based on micromagnetics [18] and experimental studies [19] have shown that the coercivity and the energy product can be improved by reducing the grain size in microstructures, which leads to a stronger intergranular exchange coupling between two phases. Microalloving prepared by adding one or two elements with an appropriate quantity into master alloys has been shown to effectively refine the microstructure [20,21], although the saturation magnetization  $J_s$  and remanence  $B_{\rm r}$  decrease simultaneously due to the magnetic dilute effect. Based on the above, the best magnetic properties of nanocomposite magnet with a master alloy composition R<sub>8-9</sub>Fe<sub>87-86</sub>B<sub>5-6</sub> may be achieved by microalloying through balancing the remanence and the coercivity of the magnet.

In the present paper, nanocomposite  $Pr_2Fe_{14}B/\alpha$ -Fe melt-spun ribbons based on  $Pr_8Fe_{86}B$  and  $Pr_8Fe_{86}VB_{6-x}C_x$  (x=0 and 1) alloys are prepared by direct melt spinning at an optimum wheel speed of  $20\,\text{m/s}$ . One percent V addition is found to be able to increase the coercivity at less expense of remanence of V-doped ribbons, and improve their magnetic properties significantly. The combined addition of V and C leads to a remarkable enhancement in coercivity of melt-spun  $Pr_8Fe_{86}VB_5C$  ribbons. The effects of V and

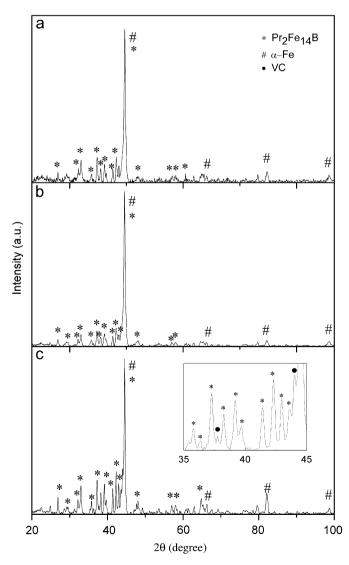
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C additions on structures and magnetic properties of melt-spun  $Pr_8Fe_{86}VB_{6-x}C_x$  (x=0 and 1) ribbons are also examined in this paper.

#### 2. Experiment

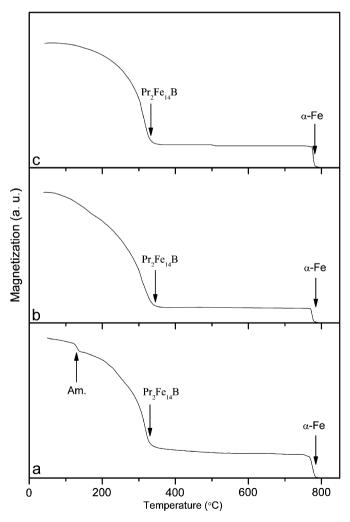
The alloy ingots with nominal compositions of Pr<sub>8</sub>Fe<sub>86</sub>B<sub>6</sub> and  $Pr_8Fe_{85}VB_{6-x}C_x$  (x = 0 and 1) were prepared by electric arc melting of the constituent materials Pr, Fe, V and C (each with a purity of at least 99.99%) and a FeB pre-alloy in a high-purity argon atmosphere. In total, 5 wt% Pr, more than the stoichiometric value, was added into the alloys to compensate for the loss of Pr. The ribbons of these alloys were obtained by melt spinning in an argon atmosphere at a wheel speed of 18-25 m/s. The phase compositions and the structures of melt-spun ribbons were examined by X-ray diffraction (XRD) with Cu K<sub>x</sub> radiation. The microstructures of the ribbons were observed by an H-800 transmission electron microscope (TEM). Thermomagnetic analysis was performed by using a vibrating sample magnetometer (VSM) in a temperature range of 25–850 °C under an applied field of 1 kOe. Demagnetization curves of the samples were measured by the same VSM with a maximum applied field of 24 kOe.



**Fig. 1.** XRD patterns of melt-spun  $Pr_8Fe_{86}B_6$  (a),  $Pr_8Fe_{85}VB_6$  (b) and  $Pr_8Fe_{85}VB_5C$  (c) ribbons spun at an optimum wheel speed of  $20 \, \text{m/s}$ .

#### 3. Results and discussion

Fig. 1 shows the XRD patterns of melt-spun Pr<sub>8</sub>Fe<sub>86</sub>B<sub>6</sub>, Pr<sub>8</sub>Fe<sub>85</sub>VB<sub>6</sub> and Pr<sub>8</sub>Fe<sub>85</sub>VB<sub>5</sub>C ribbons spun at an optimum wheel speed of 20 m/s. As shown in Fig. 1, the Pr<sub>8</sub>Fe<sub>86</sub>B<sub>6</sub> ribbons and the Pr<sub>8</sub>Fe<sub>85</sub>VB<sub>6</sub> ribbons are composed of a magnetic hard 2:14:1 phase and an  $\alpha$ -Fe phase. In the ribbons with a combined addition V and C, besides the 2:14:1 and  $\alpha$ -Fe phases, the diffraction peaks of VC phase observed in the patterns indicate that the VC phase is formed directly during melt spinning. A similar carbon compound of TiC precipitated at the Nd<sub>2</sub>Fe<sub>14</sub>B grain boundary is also observed in melt-spun nanocomposite Fe<sub>3</sub>B/Nd<sub>2</sub>Fe<sub>14</sub>B magnets with a combined addition Ti and C, but after a heat treatment [22]. The lattice constants of 2:14:1 phase ( $a = 0.8802 \,\mathrm{nm}$ ,  $c = 1.2227 \,\mathrm{nm}$ ) in V-doped samples are 0.1% larger than those  $(a = 0.8792 \,\mathrm{nm}, \, c = 1.2200 \,\mathrm{nm})$  in addition-free samples. Meanwhile, the lattice constants for  $\alpha$ -Fe phases (a = 0.2872 nm) in Vdoped samples are also larger than those of  $\alpha$ -Fe phases  $(a = 0.2866 \,\mathrm{nm})$  in addition-free samples. These suggest that V atoms do not form the V-rich intergranular phase as the case of Nb addition does, in which the Nb-rich phase is found to form in nanocomposite magnets in the melt-spinning process [23,24], and may distribute uniformly in the magnets as the interstitial or substitutional atoms. Similar results about V atom distribution detected by a three-dimensional atom probe are also observed in



**Fig. 2.** Temperature dependences of magnetization of melt-spun  $Pr_8Fe_{86}B_6$  (a),  $Pr_8Fe_{85}VB_6$  (b) and  $Pr_8Fe_{85}VB_5C$  (c) ribbons spun at an optimum wheel speed of 20 m/s.

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