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Stability of $PrCo_{5+\delta}$ and $Pr_{1-x}Hf_xCo_{5+\delta}$ alloys with $TbCu_7$-type structure at 900–1150 °C and their magnetic properties

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ABSTRACT

The $TbCu_7$-type structure in bulk Pr–Hf–Co alloys was obtained by high-temperature homogenization of alloys adjacent to the $PrCo_5$ composition. At 1150 °C, the homogeneity range of binary $PrCo_{5+\delta}$ structure stretches to 84.5 at.% Co; addition of up to 1.5 at.% Hf extends this range to 85.3 at.% Co. The lattice constants, Curie temperature and room-temperature saturation magnetization of the $PrCo_{5+\delta}$ phase approximately follow the Vegard's law as if this phase were transitional between the stoichiometric $PrCo_5$ phase and the hypothetical disordered Pr_2Co_{17} phase. Hafnium decreases the saturation magnetization of the $Pr_{1-x}Hf_xCo_{5+\delta}$ phase, but has no significant effect on its Curie temperature. The highest saturation magnetization of 12.9 kG was observed in the off-stoichiometric binary alloy $Pr_{84.5}Co_{15.5}$. The solubility of Co in the $PrCo_5$ structure was found to decrease with decreasing the homogenization temperature; only $Pr_{1-x}Hf_xCo_5$ alloys exhibited the single-phase structure after annealing at 900 °C.

1. Introduction

For more than four decades, rare earth–cobalt compounds continue to attract attention as a base of advanced high-temperature permanent magnets. After a thorough study of the most stable Co-rich R–Co compounds (R = rare earth), RCo_5 and R_2Co_{17}, and the successful commercialization of the compounds with R = Sm, the focus of research shifted to their derivatives. The existence of one such derivative, a high-temperature solid solution based on the $SmCo_5$ compound, is universally accepted and reflected in the Sm–Co binary phase diagram [1]. Its structure, which is formed via random replacement of the Sm atoms by Co atom pairs which line-up along the [0 0 1] direction, is often referred to as the $TbCu_7$ type [2,3]. As early as in 1974, Khan showed [3] that the $RCo_{5+\delta}$ structure was a common feature of many, if not all, R–Co systems; in particular, the Co-rich boundary of the $PrCo_{5+\delta}$ region at 1140 °C was found to lie somewhere between 12.5 and 16.1 at.% Pr. Nevertheless, even the most recent Pr–Co phase diagrams [4,5] present $PrCo_5$ as a stoichiometric compound. Knowing the magnetic properties of the off-stoichiometric $PrCo_{5+\delta}$ alloys not only will result in improved permanent magnet materials, but will help us to understand how the magnetic properties are influenced by both the introduction and ordering of the Co atom pairs (in the stable Pr_2Co_{17} structure, these atom pairs are ordered). It has long been understood that the $RCo_{5+\delta}$ structures are transitional between the RCo_5 and the disordered R_2Co_{17} structures [6,7], and one might reasonably expect a continuous, possibly Vegard's law-type, compositional dependence of the corresponding magnetic properties. However, the experimental data related to such dependence are very limited. One, obvious reason for this shortage is the fact that many $RCo_{5+\delta}$ structures, including the disordered R_2Co_{17}, are not stable. The other reason, which is rather paradoxical, is that too much attention has been focused on the $RCo_{5+\delta}$ structures with $\delta = 2$ (in [8–15] and at least 45 other studies). These RCo_7 structures are typically treated not as representatives of the $RCo_{5+\delta}$ solid solutions, but as unique compounds, even though there is no experimental data favoring the RCo_7 composition over any other in the whole range of the $RCo_{5+\delta}$ structures. This misconception has become so common, that the $SmCo_7$ was recently featured in the Sm–Co phase diagram as a stoichiometric compound [16]. One can argue that the disproportional attention to the RCo_7 structures at the expense of the other $RCo_{5+\delta}$ structures impedes progress in this class of hard magnetic materials.

The Sm–Co and Pr–Co alloys with the $TbCu_7$-type structure are frequently cited as promising hard magnetic materials. It does not seem likely that obtaining the $TbCu_7$-type structure in the Sm–Co alloys can dramatically change the *status quo* in the high-temperature permanent magnet materials; at best, this structure may fill the gap between the two uniaxially anisotropic compounds, Sm_2Co_{17} and $SmCo_5$ (this, of course, is not intended to undermine the importance of the 1:7 "solution" structure in manufacturing the "2:17" Sm–Co–Fe–Cu–Zr magnets!). In contrast, obtaining the $TbCu_7$-type structure in the Pr–Co system, where the Pr_2Co_{17} compound has an unfavorable planar magnetocrystalline anisotropy,

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may indeed substantially increase the Curie temperature and saturation magnetization [and, therefore, the theoretical maximum energy product $(BH)_{max}$] of the uniaxially anisotropic $PrCo_5$ compound. Patra et al. [17] recently reported epitaxial Pr–Co thin films with the $TbCu_7$-type structure and a $(BH)_{max}$ of 43 MGOe, which is 30% higher than $(BH)_{max}$ of the best Sm–Co magnets. Unfortunately, epitaxial stabilization of the $PrCo_{5+\delta}$ structure is not possible in bulk magnets.

Besides epitaxy, the $PrCo_{5+\delta}$ structure can be obtained via rapid solidification [18,19] and high-energy ball milling [9,10]. Also, a number of elements were reported to stabilize this structure in $Pr(Co,M)_7$ ingots (with or without homogenization annealing): 2.5 at.% Zr [8], 2.5 at.% Hf [12], 11 at.% (Ti + Cu) [11], 25 at.% Cu [15]. Since all these non-magnetic stabilizing elements dilute the saturation magnetization M_s, their large percentages diminish the utility of the $PrCo_{5+\delta}$ alloys. In the case of $Pr(Co,M)_7$, zirconium and hafnium appear to be equally (and by far the most) efficient stabilizers; the most numerous and thorough studies of the $Sm(Co,M)_7$ alloys slightly favor Hf over Zr [14]. It is reasonable to expect that stabilizing the $PrCo_{5+\delta}$ structure for smaller δ will require a smaller percentage of the stabilizing element. This principle is well illustrated by the results for the Sm–Zr–Co system reported by Derkaoui et al. [20] and especially by Lefevre et al. [21]. Unfortunately, smaller values of δ also mean fewer Co atoms, which are the major contributors to the high M_s.

In this paper, we report the compositional range and magnetic properties of the $TbCu_7$-type structure in the Pr–Co and Pr–Hf–Co alloys quenched from 1150 °C. In order to be able to utilize in permanent magnets the enhanced (compared to $PrCo_5$) values of M_s, we have also tested the stability of the single-phase $Pr_{1-x}Hf_xCo_{5+\delta}$ alloys at 900 °C; exposure to this temperature range is a necessary part for the manufacturing and/or processing of most R–Co magnets.

2. Experimental

Thirty-two Pr–Hf–Co alloys were prepared from praseodymium (purity 99.9%), hafnium (99.9%) and cobalt (99.8%) by arc-melting under argon. The ingots were re-melted four times to ensure their homogeneity; the subsequent weighting confirmed that the melting was not accompanied by any evaporation losses. The ingots were annealed two times, for 16 h at 1150 °C and subsequently for 24 h at 900 °C. Every annealing treatment was done under argon and was followed by water quenching. Structures of the annealed alloys were characterized by powder X-ray diffraction (XRD) performed with the Cu-Kα radiation; the XRD data were analyzed with a Powder Cell software [22]. Some of the alloys were additionally characterized by scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS) with a JEOL JSM-6335F and IXRF Systems Instruments, respectively. The Curie temperatures were found from differential thermal analysis (DTA) curves recorded with a PerkinElmer Pyris Diamond Instrument using a heating rate of 20 °C/min (the DTA assured a higher accuracy than that of the more conventional thermomagnetic technique). The room-temperature M_s values were determined by extrapolating the $M(H^{-2})$ curves (where H is the applied magnetic field corrected for the self-demagnetizing field of the specimen) measured with a Quantum Design Magnetic Properties Measurement System (magnetic field $H \leq 50$ kOe) on field-oriented powder specimens. Density of the specimens was assumed to be that of the corresponding ingots, measured with a water immersion technique.

3. Results

3.1. Stability of the $Pr_{1-x}Hf_xCo_{5+\delta}$ structure

Compositions for the single-phase and multiphase alloys annealed at 1150 °C are shown in Fig. 1(a). In the binary Pr–Co alloys, the confirmed single-phase $PrCo_{5+\delta}$ region stretches from 83.3 to 84.5 at.% Co; alloys having the lower and higher Co contents contained the rhombohedral Pr_5Co_{19} and rhombohedral Pr_2Co_{17} structures, respectively. In the ternary Pr–Hf–Co alloys, the Co-rich boundary of the $Pr_{1-x}Hf_xCo_{5+\delta}$ region extends to 85.3 at.% (note, that we associate Hf with Pr rather than with Co based of the finding by Gupta et al. [24] that in the $PrCo_5$ structure, the Hf atoms can only substitute for the Pr atoms). This Co-rich boundary was verified for x = 0, 0.5, 1.5 by plotting the intensity ratio of the (3 0 0) and (1 1 0) peaks for the Pr_2Co_{17} and $Pr_{1-x}Hf_xCo_{5+\delta}$ phases, respectively, as a function of the alloy composition (using the XRD peak intensity method); the result is shown in Fig. 1(a) with a dotted line. In Fig. 2(a), the XRD spectra of the Co-richest $PrCo_{5+\delta}$ and $Pr_{1-x}Hf_xCo_{5+\delta}$ structures ($Pr_{15.5}Co_{84.5}$ and $Pr_{13.2}Hf_{1.5}Co_{85.3}$, respectively) are compared with the spectrum of the stoichiometric $PrCo_5$ structure.

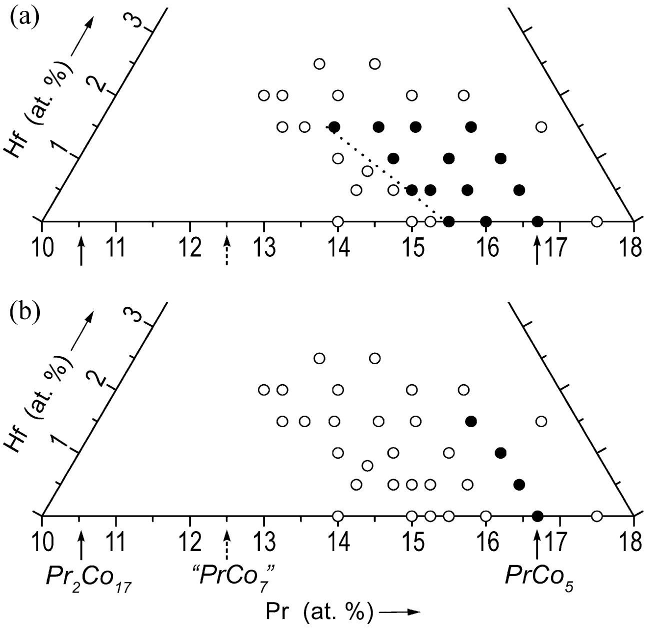

Fig. 1. Pr–Hf–Co alloys exhibiting single-phase $Pr_{1-x}Hf_xCo_{5+\delta}$ structure (filled circles) and multiphase structure (open circles) after (a) annealing at 1150 °C and (b) subsequent annealing at 900 °C. Dotted line marks the single phase region boundary found by XRD peak intensity method. The $PrCo_7$ composition is sometimes incorrectly treated as that of a stoichiometric compound.

The Hf-rich boundary of the $Pr_{1-x}Hf_xCo_{5+\delta}$ region was found to be between 1.5 and 2 at.% Hf. The Hf_6Co_{23} phase, which emerges beyond this boundary, could not be definitively recognized based on the XRD spectra (where it is manifested by the only stand alone peak at 40.73°), and it had to be confirmed by the microstructure characterization presented in Fig. 3. According to the EDS analysis, the 6:23 phase is Pr-free.

In Fig. 4, the lattice constants of the $Pr_{1-x}Hf_xCo_{5+\delta}$ structure are plotted as a function of the Co concentration. As the composition deviates from $PrCo_5$ with increasing either x or δ, the a lattice constant decreases and the c constant increases. We assume that the hypothetical Pr_2Co_{17} structure with disordered Co atom pairs (in other words, the $PrCo_{5+\delta}$ structure with δ = 3.5 or 89.5 at.% Co) has $a_{2:17D} = a_{2:17R}/\sqrt{3}$ and $c_{2:17D} = c_{2:17R}/3$, where $a_{2:17R}$ and $c_{2:17R}$ are the lattice constants of the rhombohedral Pr_2Co_{17} structure. The broken lines in Fig. 4 present the Vegard's law for the lattice constants of the binary $PrCo_{5+\delta}$ solid solution between $\delta = 0$ and $\delta = 3.5$. The experimentally observed a values are perfectly consistent with the Vegard's law, whereas the c values exhibit a small positive deviation. The data on the effect of Hf in $Pr_{1-x}Hf_xCo_5$ are in agreement with an earlier report [24].

The data on phases observed after the second annealing at 900 °C are shown in Fig. 1(b). At this lower temperature, the Co atom pairs are no longer soluble in the $PrCo_5$ structure, but the solubility of the Hf atoms remains the same. It is clear that the shrinkage of the $Pr_{1-x}Hf_xCo_{5+\delta}$ region at 900 °C, when it reduces to $Pr_{1-x}Hf_xCo_5$, must strongly limit technological options of exploring the off-stoichiometric $PrCo_5$ alloys.

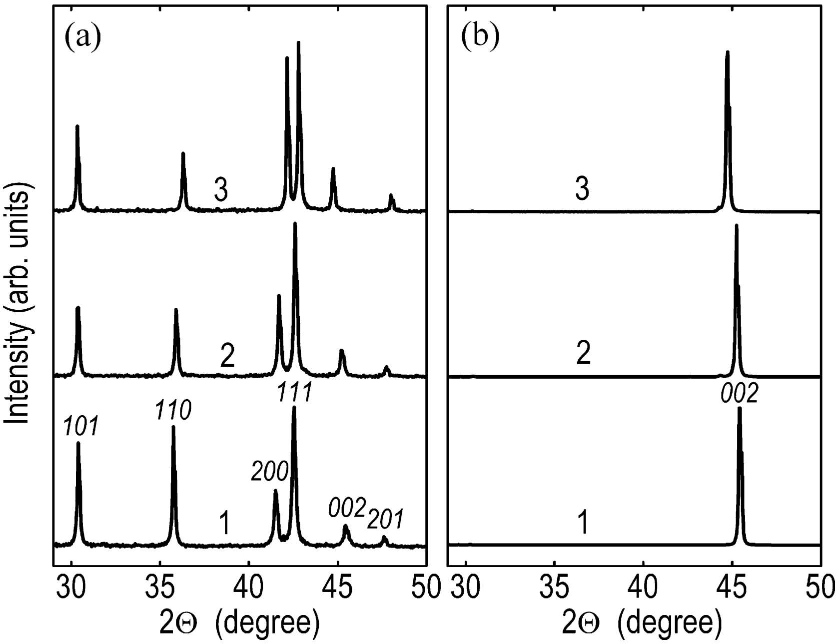

Fig. 2. XRD patterns of (a) randomly oriented and (b) field-oriented powders of alloys annealed at 1150 °C: (1) $Pr_{16.7}Co_{83.3}$, (2) $Pr_{15.5}Co_{84.5}$, (3) $Pr_{13.2}Hf_{1.5}Co_{85.3}$. All alloys exhibit single-phase $Pr_{1-x}Hf_xCo_{5+\delta}$ structure.

3.2. Magnetic properties

Curie temperature, T_C, measured for the single-phase $Pr_{1-x}Hf_xCo_{5+\delta}$ alloys is presented in Fig. 5 (since DTA is rarely used for this characterization, we included the examples of raw data including those for Ni and Fe reference materials). We assume that due to the relatively fast heating rate the $TbCu_7$-type structure did not decompose even in the alloys where it is not stable at 900 °C and, presumably, at the lower temperatures. The T_C of the $Pr_{1-x}Hf_xCo_{5+\delta}$ compound determined under such assumption depends strongly on the Co concentration increasing at the rate of 50 °C/at.% Co. On the other hand, the effect of Hf on the T_C is very weak. This result is consistent with the well-established fact that T_C of the RCo_5 compounds is determined by the exchange interactions within the Co sublattice, although in the $Pr_{1-x}Zr_xCo_5$ alloys, Gupta et al. [24] observed a considerable increase of T_C with x. Similarly to the lattice parameters, the Curie temperature of the $PrCo_{5+\delta}$ structure appears to follow the Vegard's law as if this structure were a solid solution between the $PrCo_5$ and Pr_2Co_{17} compounds (assuming that the ordered and disordered Pr_2Co_{17} structures have the same T_C).

According to the XRD characterization of selected powder samples after they had been oriented by a magnetic field [see Fig. 2(b)], the $Pr_{1-x}Hf_xCo_{5+\delta}$ alloys maintain the uniaxial magnetocrystalline anisotropy with the [0 0 1] easy magnetization direction throughout the whole homogeneity range (this observation agrees with the earlier studies [12,17]). Data presented in Fig. 6 demonstrate that the saturation magnetization $4\pi M_s$ of these alloys increases with

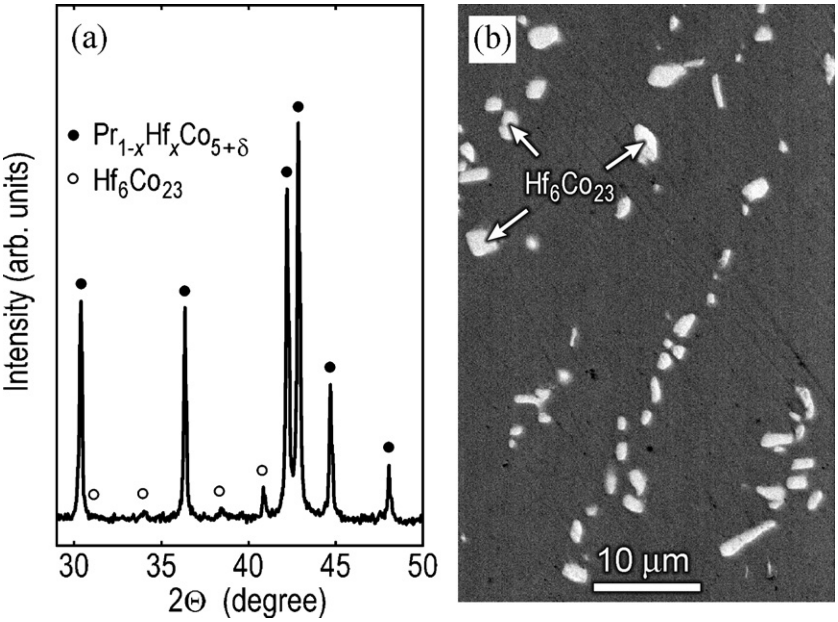

Fig. 3. (a) Powder XRD pattern and (b) BSE SEM microstructure of $Pr_{12.5}Hf_{2.5}Co_{85}$ alloy annealed at 1150 °C.

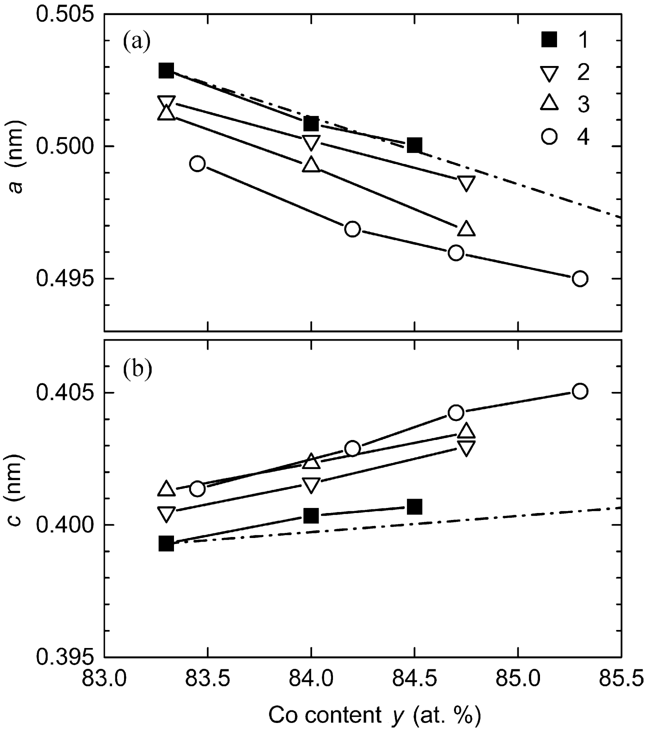

Fig. 4. Lattice constants of $Pr_{1-x}Hf_xCo_{5+\delta}$ structure in single-phase alloys annealed at 1150 °C: (1) $Pr_{100-y}Co_y$, (2) $Pr_{99.5-y}Hf_{0.5}Co_y$, (3) $Pr_{99-y}HfCo_y$, (4) $Pr_{98.5-y}Hf_{1.5}Co_y$. Broken lines are linear interpolations between *a* and *c* values for $PrCo_5$ structure (this work) and those for disordered hexagonal Pr_2Co_{17} structure defined as $a_{2:17R}/\sqrt{3}$ and $c_{2:17R}/3$ ($a_{2:17R}$ and $c_{2:17R}$ are taken from [4]).

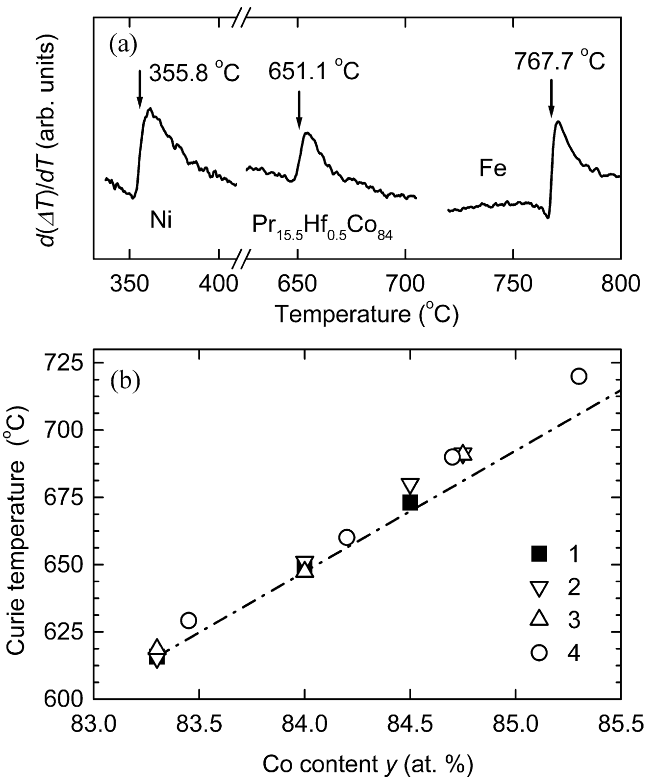

Fig. 5. (a) Parts of derivative DTA curves for Ni, Fe and $Pr_{15.5}Hf_{0.5}Co_{84}$ samples showing examples of T_C determination and (b) Curie temperatures of single-phase $Pr_{1-x}Hf_xCo_{5+\delta}$ alloys annealed at 1150 °C: (1) $Pr_{100-y}Co_y$, (2) $Pr_{99.5-y}Hf_{0.5}Co_y$, (3) $Pr_{99-y}HfCo_y$, and (4) $Pr_{98.5-y}Hf_{1.5}Co_y$. Broken line is a linear interpolation between T_C values for $PrCo_5$ (this work) and Pr_2Co_{17} [23].

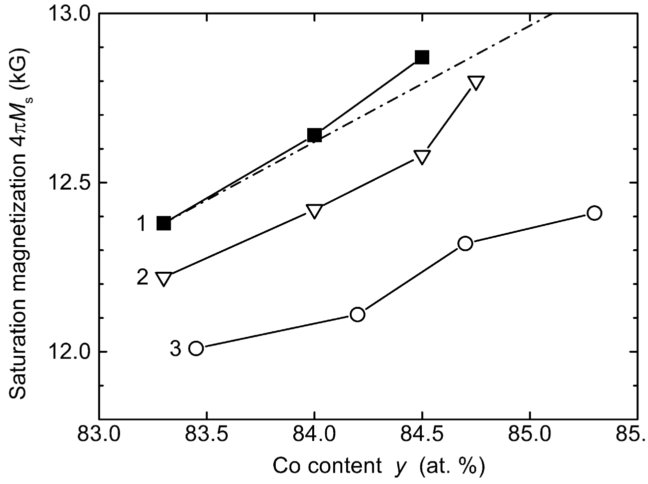

Fig. 6. Room-temperature saturation magnetization of single-phase $Pr_{1-x}Hf_xCo_{5+\delta}$ alloys annealed at 1150 °C: (1) $Pr_{100-y}Co_y$, (2) $Pr_{99.5-y}Hf_{0.5}Co_y$, and (3) $Pr_{98.5-y}Hf_{1.5}Co_y$. Broken line is a linear interpolation between $4\pi M_s$ values for $PrCo_5$ (this work) and Pr_2Co_{17} [25].

δ and decreases with x; its increase with Co is consistent with the assumed Vegard's law. Even though the Hf addition extends the homogeneity range of this structure at 1150 °C, the highest $4\pi M_s$ of 12.9 kG was measured for the binary alloy, $Pr_{84.5}Co_{15.5}$.

4. Discussion

The variations of the a, c, T_C and $4\pi M_s$ values observed in the binary $PrCo_{5+\delta}$ alloys are consistent with the model of $PrCo_{5+\delta}$ as a structure transitional between $PrCo_5$ and hexagonal (disordered) Pr_2Co_{17} structures. Moreover, with the assumption that the disordered and ordered R_2Co_{17} structures have the same values of T_C and $4\pi M_s$, the structure and magnetic parameters of $PrCo_{5+\delta}$ appear to follow the Vegard's law. Since the $PrCo_{5+\delta}$ single-phase region which we were able to study by stabilizing the $TbCu_7$-type structure at 1150 °C was quite limited, it would be highly desirable to include in the analysis the alloys with more than 84.5 at.% Co before claiming that the pattern of the properties variation is established. The published data on Co-rich Pr–Co alloys with $TbCu_7$-type structure do not exactly qualify for that. The a, c and T_C values reported for Co-rich $TbCu_7$-type alloys obtained via high-energy ball milling [9,10] cannot be directly compared to our data, since those alloys were modified with Ti or Zr. Also, the a, c and $4\pi M_s$ values reported for $Pr_{13}Co_{87}$ epitaxial films [26] (strongly deviating from the Vegard's law) may be incompatible to our data on bulk alloys.

The $4\pi M_s$ of 12.9 kG which we observed in $Pr_{84.5}Co_{15.5}$ translates into a theoretical maximum energy product of 41.6 MGOe, an 8% increase compared to 38.4 MGOe for $PrCo_5$ ($4\pi M_s = 12.4$ kG). However, those $Pr_{1-x}Hf_xCo_{5+\delta}$ structures which exhibit $4\pi M_s$ higher than that of $Pr_{1-x}Hf_xCo_5$ are not stable at 900 °C, and it seems most likely that they are equally unstable at the lower temperatures. Out of the several techniques used for manufacturing $PrCo_5$ permanent magnets, only powder sintering (without a follow-up heat treatment!) may take advantage of this enhanced magnetization. The $PrCo_5$ magnets sintered at temperature as high as 1140 °C exhibited reasonably high values of the remanence, but their coercivity was rather low [27]. The latter may be further decreased in the off-stoichiometric $PrCo_{5+\delta}$ magnets because of their (presumably) lower magnetocrystalline anisotropy. It seems highly desirable, therefore, to supplement any attempts of high-temperatures sintering of the high-magnetization $PrCo_{5+\delta}$ magnets by steps intended to inhibit grain growth.

5. Conclusions

(1) Addition of up to 1.5 at.% Hf increases the range of the $Pr_{1-x}Hf_xCo_{5+\delta}$ structures stable at 1150 °C from 84.5 to 85.3 at.% Co. However, this increase does not appear to be useful for the manufacturing of permanent magnets, since the higher room-temperature $4\pi M_s$ values were observed in the binary $PrCo_{5+\delta}$ alloys.

(2) The $Pr_{1-x}Hf_xCo_{5+\delta}$ structure with $\delta > 0$ becomes unstable at 900 °C, thus narrowing the options of utilizing the enhanced $4\pi M_s$ values to sintered permanent magnets and imposing restrictions on the conditions of their manufacturing.

(3) Lattice constants, T_C and $4\pi M_s$ values of the binary $PrCo_{5+\delta}$ alloys vary continuously and appear to be transitional between those of the stoichiometric $PrCo_5$ structure and hypothetical disordered Pr_2Co_{17} structure.

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