Enhanced magnetoelastic performance in Pr/Mn-doped Laves phase (Tb,Ho)Fe₂ compounds

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Magnetostrictive Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1} (0 ≤ x ≤ 0.20) alloys are prepared by arc-melting and subsequent annealing. The dopant of Pr/Mn introduced into RFe₂ compounds effectively stabilizes the forming of single C15 Laves phase at ambient pressure. The easy magnetization direction (EMD) varies when Tb content increases, which is accompanied by a crystal-structural transition. EMD lies along <1 0 0> axis for x ≤ 0.05, rotating to <1 1 1> axis for x ≤ 0.12, with a tetragonal symmetry changing to a rhombohedral one. Magnetocrystalline-anisotropy compensation is obtained with the optimized composition of x = 0.12, shifting to the Tb-poor side in comparison to Pr/Mn-free counterpart. An enhanced effect on magnetoelastic properties is achieved in Tb_{0.12}Ho_{0.68}Pr_{0.2}Fe_{1.8}Mn_{0.1}, which simultaneously possesses a low anisotropy and high magnetostriction performance, i.e. λ_S ~ 420 ppm, λ₁₁₁ ~ 970 ppm, and a large low-field λ_a ~ 390 ppm/2 kOe, being 30 % higher than that of Pr/Mn-free compound. Combining low-cost light rare earth Pr with the lower Tb content, Tb_{0.12}Ho_{0.68}Pr_{0.2}Fe_{1.8}Mn_{0.1} may make it promising solution in magnetostrictive applications.

Keywords: *intermetallics; magnetostriction; magnetic properties; Laves phase; X-ray technique*

1. Introduction

Joule magnetostriction is considered as a change in length of a material, when it is subjected to an external magnetic field. As a typical magnetostrictive intermetallic compound with C15 Laves structure, TbFe₂ stands out because of its largest magnetostriction to date, exhibiting at the room temperature λ₁₁₁ as large as 2460 ppm. Yet, a huge magnetocrystalline anisotropy energy exists, the first order constant K₁ reaches a large value of -76 × 10⁶ erg/cm³, which has been a long-standing drawback in practical applications, because then large magnetic fields are required to saturate the magnetization and magnetostriction. Indeed, the high anisotropy should be reduced, and simultaneously a large magnetostriction should be retained. Introducing other rare-earth elements (RE) to compensate the anisotropy of Tb³⁺, whilst maintaining the large magnetostriction, is one of the effective methods, and subsequently, the Tb-Dy-Fe alloys (known as Terfenol-D) and Tb-Ho-Fe

alloys, have been developed and widely used for both sensing and actuation [1–3]. However, the raw materials, such as heavy RE metals (e.g. Tb, Ho and Dy), are rare and expensive to obtain. Considering the much richer resources of light rare earth and their low prices, Pr-containing RFe₂ compounds have attracted a lot of research attention in the last decades. Additionally, PrFe₂ is expected to possess a larger magnetic field-induced magnetostriction as compared with TbFe₂, due to its striking spontaneous coefficient λ₁₁₁, whose value calculated theoretically is 5600 ppm at 0 K. On the other hand, the magnetocrystalline anisotropy of PrFe₂ appears to have a low value, e.g. K₁ ~ 7.3 × 10⁶ erg/cm³ at 1.5 K, which is of an order smaller than that of TbFe₂ [4]. Together with the anisotropy nature that the sign of K₁ is positive for PrFe₂, opposite to that of TbFe₂, it is thought that the anisotropy compensation system (Tb,Pr)Fe₂ will be a promising magneto-elastic candidate for applications. In fact, the study and application of Pr-containing RFe₂ Laves phase compounds have been a key issue in the last few years [5–10]. Many efforts have been focused on the synthesis of single

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Laves phase so as to expand the Pr solid solubility, wherein the preparation process was mainly paid to the arc melting with the Fe partial substitution by other transition metals (e.g. Mn, Co and Ni) and the non-equilibrium condition methods (e.g. high pressure and melt-spinning). Up to now, there is still a challenge to synthesize pure PrFe_2 and its relevant Laves compounds at normal pressures because the radius ratio of (Pr,R):Fe, resulting from the big radius for Pr^{3+} , is too large to fit the ideal ratio, i.e. the value 1.225:1 that is essential for the atomic size requirement in Laves phase with C15 cubic structure [11]. Therefore, a key issue in the development of the cheaper light RE compounds is to fabricate the single Laves phase under ambient conditions.

Recently, some research [12–16] suggested that the substitutions by Mn/Co in the heavy RE-based (Tb,Ho) Fe_2 and (Tb,Dy) Fe_2 systems might circumvent the dilemma of the relationship between magnetic anisotropy and magnetostriction. The change of spin reorientation temperature T_{SR} offers a choice for optimizing the magnetostriction via anisotropy by tailoring the ratio of Tb/Ho. Besides, it is supposed that Pr-containing (Tb,Ho) Fe_2 alloys doped with Mn appear to have a higher potential in the formation of single Laves phase, owing to the nature of the larger atomic radius of Mn than Fe, which gives rise to the value of radius ratio (Pr,R):(Fe,Mn) much closer to the ideal one 1.225:1. In our recent work, the Mn-doped $\text{Tb}_x\text{Ho}_{1-x}\text{Fe}_2$ system was prepared, showing that the prospects in low-field applications are quite promising, which still has a great potential for further progress [15, 16]. In this contribution, the light RE 20 at.% Pr was introduced into Tb-Ho-Fe alloy system to investigate the effect of Pr/Mn on structural and magnetic properties (e.g. magnetocrystalline anisotropy, easy magnetization direction (EMD), and spontaneous magnetostriction coefficient λ_{111}), in order to optimize the resultant magnetic anisotropy and the field-induced magnetostriction to obtain good magnetoelastic properties.

2. Experimental

According to the chemical formula $\text{Tb}_x\text{Ho}_{0.8-x}\text{Pr}_{0.2}\text{Fe}_{1.8}\text{Mn}_{0.1}$ with $0 \leq x \leq 0.20$, the ingots were prepared by arc-melting method under high-purity argon atmosphere [14–16]. The composition was designed with an expected minimum anisotropy based on the opposite sign of K_1 between TbFe_2 and HoFe_2 [1], where the nominal composition x value is about 0.15. The transition metal sublattice was selected as 1.90 instead of 2.0 so as to obtain a purer Laves phase [14]. The homogenization process was performed at annealing temperature of 750 °C for 9 days, followed by furnace cooling to room temperature (RT). The structure was verified by X-ray diffraction (XRD, Bruker D8 Advance) with $\text{CuK}\alpha$ radiation. A high-precision step scanning was carried out for the (4 4 0) XRD peak to investigate the crystal-structural distortion by spontaneous magnetostriction along $\langle 1\ 1\ 1 \rangle$ axis and estimate the coefficient λ_{111} [17].

The ingot was ground to powder particles of $\leq 150\ \mu\text{m}$ and mixed with epoxy resin in a 1:2 weight ratio in a plastic mold. Thereafter, the composite was placed under a magnetic field ($H_{\text{curing}} \sim 12\ \text{kOe}$) for solidification. Thus, the magnetically oriented samples were prepared at RT [2, 18]. To test the EMD [17], an XRD was performed on the sample surface perpendicular to H_{curing} . The magnetic properties were measured by a vibrating sample magnetometer (Lake Shore 7410 model) with the field up to 20 kOe. The room-temperature magnetostriction $\lambda_{\parallel}$ and $\lambda_{\perp}$ denote the measurement direction parallel and perpendicular to the applied field H , respectively) was measured using a standard strain gauge technique. Herein, H is up to 12 kOe with a metal strain gauge of the factor 2.00 and the resistance 120 Ω .

3. Results and discussion

The powder XRD patterns of the homogenized $\text{Tb}_x\text{Ho}_{0.8-x}\text{Pr}_{0.2}\text{Fe}_{1.8}\text{Mn}_{0.1}$ alloys at RT are shown in Fig. 1, and the (3 1 1) diffraction peaks around 40° are also present. It can be realized that the single Laves phase has been formed with no trace

of any secondary phase in the recorded data. All diffracted peaks, marked with the indices (h k l), are well matched with the characteristics for the Laves phase with a cubic MgCu₂-type crystal structure. Furthermore, all the peaks slightly move to lower Bragg angles upon increasing the amount of Tb, manifesting the expansion of unit cell volume. This indicates that Mn affects primarily the formation of single phase in the 20 at.% Pr-doped alloy system. As is known, a pure PrFe₂ Laves phase cannot be synthesized at ambient pressure due to the radius ratio of Pr:Fe ions much larger than the ideal ratio (1.225) [1, 6]. The formation in (Tb,Ho,Pr)(Fe,Mn)₂ Laves phase could be explained by the radius ratio of RE versus transition-metals much closer to 1.225, due to the larger radius of Mn than that of Fe. The lattice parameter *a*, calculated by analyzing the XRD peaks, increases from 0.7317 nm for *x* = 0.0 to 0.7330 nm for *x* = 0.20, which is larger compared to the corresponding Mn-free counterpart. The lattice parameter shows a positive correlation with *x*, wherein the dependence on the composition *x* approximately follows the linear Vegard's Law. In other word, this linear regularity also suggests that the light RE 20 at.% Pr substitution does not show a mixed-valence behavior [15, 16].

Fig. 1. XRD patterns of the Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1} alloys at room temperature.

XRD patterns at RT for the magnetically oriented Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1} powders are shown in Fig. 2. The random powder without the

curing field, for *x* = 0.0, is shown at the bottom. It is taken as a reference to compare and verify the relative intensities of the diffraction peaks of other compounds. The variation of peak intensity occurs, that is, the (8 0 0) peak around 114° and the (3 3 3) peak around 66° are the strongest for the Ho-rich and Tb-rich side, respectively. Obviously, the strongest peak around 41° corresponding to (3 1 1) plane appears for random H_{curing}-free powders. This infers that the particle rotates to orientation along its $\langle uvw \rangle$ EMD axis due to the magnetic torque supplied by curing field, which mainly depends on the anisotropy of magnetic susceptibility, particle size and magnetic flux density [19]. In general, the evolution of XRD relative intensity as a function of composition *x* and the variation for $I_{(800)}/I_{(311)}$ or $I_{(222)}/I_{(311)}$, indicate that the magnetocrystalline alignment results from the change in magnetocrystalline anisotropy for different Tb/Ho ratios [17]. The $\langle 1\ 0\ 0 \rangle$ -type reflection peaks, e.g. (8 0 0) and (4 0 0), become more intense for *x* ≤ 0.05 in the aligned samples. In comparison, a trace of (8 0 0) reflection appears in the random powder, but the (4 0 0) is not observed. Thus, it is concluded that the EMD occurs along the $\langle 1\ 0\ 0 \rangle$ axis, in agreement with the $\langle 1\ 0\ 0 \rangle$ EMD for the end-member HoFe₂. The value of $I_{(800)}/I_{(311)}$ decreases with decreasing Ho content, approaching a zero value when *x* increases to 0.10, indicating that the EMD deviates from $\langle 1\ 0\ 0 \rangle$ direction. With further increasing *x* to = 0.12 and above, the $\langle 1\ 1\ 1 \rangle$ -type peaks become more intense, wherein the ratio $I_{(222)}/I_{(311)}$ continues to increase, while the remaining non- $\langle 1\ 1\ 1 \rangle$ reflections weaken accordingly. Considering the $\langle 1\ 1\ 1 \rangle$ EMD for the end-member TbFe₂ at RT, the EMD for Tb-rich sides of *x* ≥ 0.12 can be established as $\langle 1\ 1\ 1 \rangle$ axis. As is known, the variation of the Tb/Ho ratio affects the change in spin reorientation temperature, simultaneously changing the EMD at a certain temperature. The anisotropy constant *K*₁ of TbFe₂ has a negative value, opposite to HoFe₂, corresponding to the $\langle 1\ 1\ 1 \rangle$ EMD for TbFe₂ and $\langle 1\ 0\ 0 \rangle$ EMD for HoFe₂ [1]. Consequently, it is concluded that the magnetocrystalline anisotropy can be compensated by changing Tb/Ho ratio, followed by the critical composition point occurring near *x* = 0.12.

A similar phenomenon has been observed in the (Tb,Ho)(Fe,Mn)₂ system, and particularly, the critical point was moved to the Tb-poor part as compared to the Pr/Mn-free (Tb,Ho)Fe₂ system [1, 16].

Fig. 2. XRD patterns of the magnetically aligned Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1} powders and the random Ho_{0.8}Pr_{0.2}Fe_{1.8}Mn_{0.1} powder.

Fig. 3. Profiles of the step-scanned (4 4 0) reflection of the Laves phase in Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1} alloys.

As is known, the variation in EMD corresponds to crystal-structural transition/distortion in Laves compounds, caused by the spontaneous magnetostriction, when cooling down below its Curie

temperature [20]. A spontaneous magnetostriction λ_{111} occurs contributing to a rhombohedral distortion with $\langle 1\ 1\ 1 \rangle$ EMD, while λ_{100} contributes to a tetragonal distortion with $\langle 1\ 0\ 0 \rangle$ EMD. This means that the crystal-structural transition takes place from tetragonal symmetry for $x \leq 0.05$ to rhombohedral one for $x \geq 0.12$ with increasing Tb content. Similar behaviors have been observed in other Laves compounds (Tb_{0.2}Pr_{0.8})_xDy_{1-x}Fe_{1.93} and Tb_{1-x}Nd_xCo₂ using different methodology, i.e. the step-scanned and the synchrotron XRD, respectively [8, 21]. Thus, the EMD and the spontaneous magnetostriction coefficient (λ_{111} or λ_{100}) can also be evaluated by X-ray crystallographic study. For this case, as shown in Fig. 3, the λ_{111} value can be calculated by the step-scanned XRD as [5]:

$$\lambda_{111} = 2 \frac{d_{440} - d_{\bar{4}40}}{d_{440} + d_{\bar{4}40}} \quad (1)$$

d_{440} and $d_{\bar{4}40}$ denote the crystallographic plane distances of (4 4 0) and (4 $\bar{4}$ 0), respectively. The double splitting of the $\langle 1\ 1\ 0 \rangle$ -type reflection is observed for $x \geq 0.12$, indicating the EMD lying along $\langle 1\ 1\ 1 \rangle$ axis and a large rhombohedral distortion caused by the spontaneous magnetostriction (λ_{111}). Based on equation 1, λ_{111} for the critical point of $x = 0.12$ is calculated to be as large as 970 ppm. The splitting cannot be detected for $x \leq 0.05$, which can be attributed to the small structural distortion, when its EMD is lying along $\langle 1\ 0\ 0 \rangle$ axis, in consistency with its tetragonal distortion [20].

The initial magnetization curves of the Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1} alloys at RT are shown in Fig. 4. One can see that the magnetization is close to being saturated at the available maximum field of 20 kOe. Saturation magnetization M_S can be estimated according to the law of approach to saturation, expressed as [22]:

$$M = M_S \left(1 - \frac{a}{H} - \frac{b}{H^2} \right) + \chi_p \cdot H \quad (2)$$

where a and b are constants, χ_p is the susceptibility of the paramagnetic (parallel) magnetization process. As shown in the inset of Fig. 4, M_S increases monotonously with the increase of Tb content x .

The magnetic moment μ of Laves phase can be described by the formula 3 based on molecular-field theory [22]:

$$\mu = x\mu_{Tb} + (0.8 - x)\mu_{Ho} - 0.2\mu_{Pr} - 1.9\mu_{Fe/Mn} \quad (3)$$

where μ is the magnetic moment of the Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1} Laves phase, μ_{Tb} , μ_{Ho} , μ_{Pr} and $\mu_{Fe/Mn}$ are the magnetic moments of Tb, Ho, Pr and Fe/Mn atom, respectively.

Fig. 4. Magnetic field dependence of magnetization M for different compositions x . The inset shows the saturation magnetization M_S .

It is well established that in the rare earth-transition metal compounds, the μ of Fe/Mn sublattice arranges in parallel with that of light RE, but antiparallel with heavy RE. In addition, the μ of TbFe₂ is larger than that of HoFe₂ at RT (e.g. under 300 K, the μ is 88 emu/g and 62 emu/g for TbFe₂ and HoFe₂, respectively [1]). Thus, it would be not difficult to understand why magnetization increases with increasing x in the studied composition range of $x \leq 0.20$. Furthermore, the dependence of M_S on composition x almost follows the linear tendency, which also suggests that these compounds maintain single-phase structure and accord well with the XRD results (Fig. 1).

Fig. 5 represents the field-induced magnetostriction $\lambda_a = \lambda_{\parallel} - \lambda_{\perp}$ as a function of external magnetic field H . It can be seen that all the

Fig. 5. Magnetic field dependence of the anisotropic magnetostriction ($\lambda_a = \lambda_{\parallel} - \lambda_{\perp}$) for the Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1} alloys.

λ_a approach the saturation at different rates with H increasing up to be 12 kOe. Here, the approach to saturation for $x = 0.12$ emerges more pronounced than the others, presenting a large low-field $\lambda_a \sim 390$ ppm/2 kOe. The tendency to saturation is different for different compositions x , depending mostly on the ratio of magnetostriction to magnetization λ/M , which could be attributed to the anisotropy variation between Tb³⁺ and Ho³⁺ ions [18, 23]. To further study this magnetoelastic behavior, the $\lambda_a/\lambda_{\max} - H$ and $M/M_S - H$ curves for different compositions x are plotted in Fig. 6, where $\lambda_{\max}$ denotes the λ_a value at the maximum measured field of 12 kOe. One can see that the curve of $x = 0.12$ lies on the uppermost one, indicating its most easy saturation. It is obvious that the sample $x = 0.12$ has the enhanced low-field saturation, inferring that it possesses the lowest anisotropy among all the studied compounds, in accordance with the EMD analysis, as a result of the anisotropy compensation between Tb³⁺ and Ho³⁺ ions.

To better understand the anisotropy variation effect on magnetostriction process originating from the competition between Tb³⁺ and Ho³⁺ ions, $\lambda_a = \lambda_{\parallel} - \lambda_{\perp}$ as a function of composition x at various H has been plotted in Fig. 7a. As expected, a peak

Fig. 6. Magnetic field dependence of (a) normalized magnetostriction λ_a/λ_{0s} and (b) normalized magnetization M/M_s for the $Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1}$ alloys.

emerges near $x = 0.12$, which is prominent especially at the lower field, reflecting the minimum magnetic anisotropy among these concentrations. Similar behavior was found in the $Tb_xDy_{1-x}Fe_2$ [1] and Co-doped $Tb_{1-x}Ho_xFe_2$ alloys [14], demonstrating that the $Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1}$ is also an anisotropy compensation system, in accordance with the previous analysis of EMD, step-scanned (4 4 0) XRD and magnetization. $\lambda_{\max}$ increases with increasing Tb content x , which can be attributed to the larger spontaneous magnetostriction λ_{111} of $TbFe_2$ compared to $HoFe_2$ [1]. For example, the coefficients λ_{111} at 0 K for $TbFe_2$ and $HoFe_2$ are 4400 ppm and 1600 ppm, respectively.

To characterize the available magnetostriction, the saturation magnetostriction λ_s was estimated with the law of approach to saturation, described phenomenologically as [24, 25]:

$$\lambda_a = \lambda_{0s} \left(1 - \frac{a_0}{H} \right) \quad (4)$$

where a_0 is a magnetostrictive parameter, the measure of the approach rate to saturation.

The saturation magnetostriction λ_s was obtained with the relation $\lambda_s = 2/3\lambda_{0s}$, shown in Fig. 7b. It is worth pointing out that around the anisotropy compensation point, λ_s of the polycrystalline $Tb_{0.12}Ho_{0.68}Pr_{0.2}Fe_{1.8}Mn_{0.1}$ alloy is calculated to be as large as 420 ppm.

Fig. 7. Composition dependence of (a) anisotropic magnetostriction ($\lambda_a = \lambda_{\parallel} - \lambda_{\perp}$), (b) saturation magnetostriction λ_s , and (c) magnetostrictive parameter a_0 for the $Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1}$ alloys.

To further quantitatively characterize the rate of approach to saturation, the dependence of parameter a_0 on x is displayed in Fig. 7c. As is known, a_0 is a factor characterizing the magnetocrystalline anisotropy associated with the first order anisotropy constant K_1 [25]. As expected, the $a_0 - x$ curve shows a significant minimum around $x = 0.12$, indicating the lowest K_1 , caused by the anisotropy compensation. In addition, this phenomenon coincides with the magnetization

process, as shown in Fig. 4, for which the magnetization curve for $x = 0.12$ tends to saturation faster than others. Therefore, the compensation point is achieved to be near $x = 0.12$ for the Pr/Mn-doped (Tb,Ho)Fe₂ system, shifting to the Tb-poor side at RT in comparison with the Co-doped and Pr/Mn-free counterparts [1, 25, 26]. In other words, similar to the behavior of Tb element, the Mn contribution in resultant anisotropy is opposite to that of Ho. An optimized effect on both magnetic anisotropy and magnetostriction was obtained in the Tb_{0.12}Ho_{0.68}Pr_{0.2}Fe_{1.8}Mn_{0.1} alloy. It means that the minimum anisotropy as well as the large λ_S (~420 ppm) have been achieved, and simultaneously, the low-field induced λ_a at 2 kOe and λ_{111} are as large as 420 ppm and 970 ppm, respectively. Therefore, an excellent comprehensive performance via the 20 at.% Pr introduction into (Tb,Ho)(Fe,Mn)₂ system has been realized, wherein both the λ_S and low-field λ_a are improved with the minimum in anisotropy. Particularly, the λ value is much larger than that of the Pr-free counterparts, such as the well-known Tb_xHo_{1-x}Fe₂ ($\lambda_S \sim 325$ ppm in Tb_{0.15}Ho_{0.85}Fe₂) and the Mn-doped one ($\lambda_S \sim 330$ ppm in Tb_{0.14}Ho_{0.86}Fe_{1.9}Mn_{0.1}, $\lambda_a \sim 340$ ppm at 2 kOe in Tb_{0.12}Ho_{0.88}Fe_{1.9}Mn_{0.1}) [16, 25]. Containing the low-cost light rare-earth Pr of 20 at.%, along with the relatively low Tb content of $x = 0.12$ for anisotropy compensation, Tb_{0.12}Ho_{0.68}Pr_{0.2}Fe_{1.8}Mn_{0.1} seems to be a promising candidate from the applications point of view.

4. Conclusions

In summary, the structural, magnetoelastic properties of Tb_xHo_{0.8-x}Pr_{0.2}Fe_{1.8}Mn_{0.1} alloys have been investigated. The dopant of Pr/Mn in RFe₂ compounds effectively stabilizes the forming of single C15 Laves phase at ambient pressure. It was demonstrated that this alloy system is a magnetocrystalline-anisotropy compensation system. At room temperature, the EMD lies along $\langle 1\ 0\ 0 \rangle$ axis for $x \leq 0.05$, changing to $\langle 1\ 1\ 1 \rangle$ axis for $x \geq 0.12$, and simultaneously, crystal-structural distortion transforms from tetragonal symmetry to rhombohedral one

with increasing Tb content. The critical composition point of anisotropy compensation is tailored to be near $x = 0.12$, shifting to the Tb-poor side in comparison to Pr/Mn-free counterparts. An enhanced effect on magnetoelastic performance has been obtained in Tb_{0.12}Ho_{0.68}Pr_{0.2}Fe_{1.8}Mn_{0.1} alloy, i.e. the high magnetostriction with a low magnetocrystalline anisotropy (the large low-field-induced $\lambda_a \sim 390$ ppm/2 kOe, $\lambda_S \sim 420$ ppm and $\lambda_{111} \sim 970$ ppm). The excellent magnetoelastic properties and the combination with 20 at.% cheap-Pr, together with the critical point shifting to Tb-poor part, suggest the promising prospects for applications.

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