A comparative study of magnetostrictive strain in $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$-ingot and $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$-melt spun

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Abstract: In this work, the structural and magnetic properties of ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds have been investigated. The structural characterization of the compounds, by X-ray powder diffraction, is evidenced for a monoclinic $Nd_3(Fe,Ti)_{29}$-type structure (A2/m space group). A 2-type FOMP have been observed in the magnetic AC susceptibility curves of the ingot and melt-spun compounds. Magnetostriction and linear thermal expansion measurements have been performed by the standard strain gauge method in magnetic fields up to 1.5 T, and temperature range of 77 to 575 K. The calculated values of the ordering temperature, the room temperature saturation magnetization for the melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compound are several times smaller than the corresponding values obtained in ingot $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compound. The above obtained results have explained the behavior of measured values of spontaneous volume, longitudinal and transverse magnetostriction.
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Introduction

Rare-Earth (R) iron intermetallic compounds based on R_3 (Fe, M)$_{29}$ (R is a Rare Earth or Y, and M are stabilizing elements, Ti, V, Cr, Mo,...) with Nd_3 (Fe, Ti) $_{29}$-type structure and their interstitial compounds have attracted considerable interest. This is because some of them, like Sm_3 (Fe, M)$_{29}N_y$, exhibit excellent hard-magnetic properties [1]. The $Nd_3(Fe,Ti)_{29}$-type compounds (3:29) crystallize in the A2/m space group and consist of rhombohedral Th_2Ni_{17}-type (2:17R) and tetragonal $ThMn_{12}$-type (1:12) segments in a ratio of 1:1 [2]. In this type of compounds, the Rare Earth ions occupy two crystallographic sites (2*a* and 4*i*), and the Fe atoms occupy eleven sites (one 2*c*, one 4*e*, one 4*g*, four 4*i* and four 8*j*) [3].

Partial replacement of Fe by Co in 3:29 influences strongly the local anisotropy, the structure, and magnetic properties of these compounds. Co may substitutes preferentially at Fe_1 (2*c*), Fe_8 (8*j*) and Fe_{11} (4e) for partial replacement of Fe [4]. There, the Fe atoms couple with a negative exchange interaction with the neighboring Fe atoms. The substitution of Co leads to an increase of T_C by reducing the negative contribution of antiferamagnetic coupled sites on the 3d sublattice [4,5]. Ti, Mo and V also occupy Fe_2 (4*i*), Fe_3 (4*i*) and Fe_6 (4*g*) sites where the sites of dumb-bell Fe atoms are found [6]. However, for high Co concentrations, Co atoms are positioned in the dumb-bell sites [7]. For more than 40% Co, when the amount of the stabilizing atom is low, a disordered modification of the hexagonal Th_2Ni_{17}-type structure is formed. It is commonly believed that 3:29 structure exists only for light rare earth based compounds [8]. For heavy rare earth compounds beyond Tb, a different type of structure may be expected [9]. However, the formation of

3:29 phase in $R_3(Fe_{1-x} Co_x)_{29-y}M_y$ system was found for more than 40% substitution of Fe by Co atom for a large amount of stabilizing atom such as R = Gd, and M = Cr with y = 4–7 [10] and R = Sm, M = Cr [11] with y = 4.5–7.

There are some measurements reported on the magnetic properties of ingot [12-15] and melt-spun Pr_3 (Fe, Co, Ti)$_{29}$ ferromagnetic compounds [16, 17]. Shah et al. [12,13] reported the effects of Co substitution for Fe on the magnetocrystalline anisotropy and site occupancies of Pr_3 (Fe$_{1-x}$Co$_x$)$_{27.5}$Ti$_{1.5}$ (x ≤ 0.3) alloys. The Rare Earth sublattice anisotropy is found to dominate over the Fe sublattice anisotropy at room temperature and the easy magnetization direction (EMD) is found to be along the b-axis of the monoclinic structure for $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ and away from the b-axis for other concentrations. The hard magnetization direction (HMD) except for $x = 0$, in all other compounds, is almost in the *a-c* plane [12]. The saturation magnetization increases with Co concentration at both 5 and 300 K. A large increase in Curie temperature has been found [13]. The Curie temperature and saturation magnetization values observed in the melt-spun alloys were lower than those observed in fully crystalline alloys [16].

Some reports are available on the magnetic and magnetoelastic properties of the R_3(Fe, T)$_{29}$ compounds (R = Y, Ce, Nd, Pr, Tb, and Gd and T = Ti, and Cr) [18-23]. A large spontaneous magnetostriction and Invar-like behavior in the thermal expansion coefficient have been observed in the vicinity of T_C in these series. This offers the possibility of increasing the ordering temperatures by introducing interstitial atoms that suggest promising properties for hard permanent magnets [24,25]. In this work, we have studied the structure, magnetic properties, and the existence of magnetovolume effect in the ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds. The magnetoelastic effects of these compounds between 77 to near T_C are studied by measuring the magnetostriction and thermal expansion using strain gauge method. To our knowledge, these measurements so far have not been studied.

Experimental

$Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$-ingots were prepared by arc melting of high-purity (at least 99.9%) of the constituent elements. In order to maximize the amount of $R_3Fe_{29-x}M_x$ phase, the ingots were subsequently annealed in sealed quartz tubes under protective argon atmosphere at 1323 K for a period of three days then followed by quenching in water. Pieces of the ingots were then cast into ribbons using the melt-spinning technique, in an argon atmosphere with employing linear wheel speed of 20 m/s. The ribbon samples were annealed for 20 min at 1323 K. The phase purity of the prepared samples was examined by X-ray diffraction analysis that was performed at RT using CuKα radiation. The XRD data were analyzed using a commercial TOPAS package and Fullprof program. The Bragg diffraction patterns were plotted using Fullprof program. The crystallite size was calculated from the XRD peak broadening of the strongest peak (-133) at 42.5° using Scherer's formula. Crystalline density of the samples was obtained from TOPAS package. Magnetization curves of the samples were recorded using a pulsed field magnetometer at room temperature applying external magnetic fields up to 5 T. The temperature dependence of the low-field AC magnetic susceptibility $\chi_{ac}(T)$ was measured between 100 and 300 K at 333 Hz with an AC magnetic field of 50 Am^{-1} peak value using Lake-Shore AC susceptometer model 7000. Magnetostriction and linear thermal expansion (LTE) measurements were performed by the standard strain gauge method on disk-shaped samples of 6 mm diameter and 3 mm thickness in magnetic fields up to 1.5 T, and temperature ranging from 77 to 575 K. The spontaneous volume magnetostriction ω_s was calculated as $\omega_s = 3[(\Delta l/l)_{exp} - (\Delta l/l)_{lat}]$, where $(\Delta l/l)_{exp} (= (l (T) - l (77 K)) / l (77 K))$ and $(\Delta l/l)_{lat}$ is the lattice contribution that can be obtained from the extrapolation of the paramagnetic regime of the LTE curve, where only the non-magnetic inharmonic phonon contribution is expected. The extrapolation has been performed using the Grüneisen–Debye model, with a Debye temperature of T_D = 450 K [10]. The magnetostriction was measured (with the accuracy of 2×10^{-6}) parallel (longitudinal magnetostriction, λ_{Pa}) and normal (transverse magnetostriction, λ_{Pe}) to the applied field direction. It should be emphasized that no significant difference was observed between the strains measured in the plane and perpendicular to the plane of the disc of the samples, suggesting the absence of any preferred orientation effects.

Results and discussion

The XRD patterns for annealed ingot and melt-spun of $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ are presented in Fig. 1.

The data were analyzed using commercial TOPAS package and Fullprof program. Results of the Rietveld analysis indicate that the main phase of each sample has the $Nd_3Fe_{27.5}Ti_{1.5}$-type structure with monoclinic symmetry (A2/m space group). In addition, there is some amount of α-Fe (S. G. Im3m) phase. The strongest impurity peak of α-Fe (2θ ∼ 44.65°) has been marked on X-ray diffraction patterns of Fig. 1. Rietveld analysis gave a phase distribution of 95.5 Pr_3 (Fe, Co, $Ti)_{29}$, and 4.5 wt.% α-Fe for the ingot and 74.3 Pr_3 (Fe, Co, $Ti)_{29}$ and 25.7 wt.% α-Fe for the melt-spun sample, respectively. Phase composition, unit cell parameters, crystalline size and crystalline density of the monoclinic ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ are summarized in Table 1. A trace of α-Fe phase is also reported in the other ingot Pr-based compounds [26]. Gjoka *et al.* [17] in the melt-spun $Nd_3Fe_{27.5}Ti_{1.5}$ and $Pr_3Fe_{27.5}Ti_{1.5}$ alloys and K. Sirisha *et al.* [16] in the melt-spun Pr_3 $(Fe_{1-x}Co_{0.x})_{27.5}Ti_{1.5}$ annealed alloys found the 3:29 phase and strong presence of α-Fe phase. A significant decrease of the cell volume is observed from 886.383 to 843.955 Å^3 with respect to the bulk sample, which represents a lattice contraction of about 5%. This difference was observed in other melt-spun alloys that have smaller crystalline size with respect to the bulk sample [16,17].
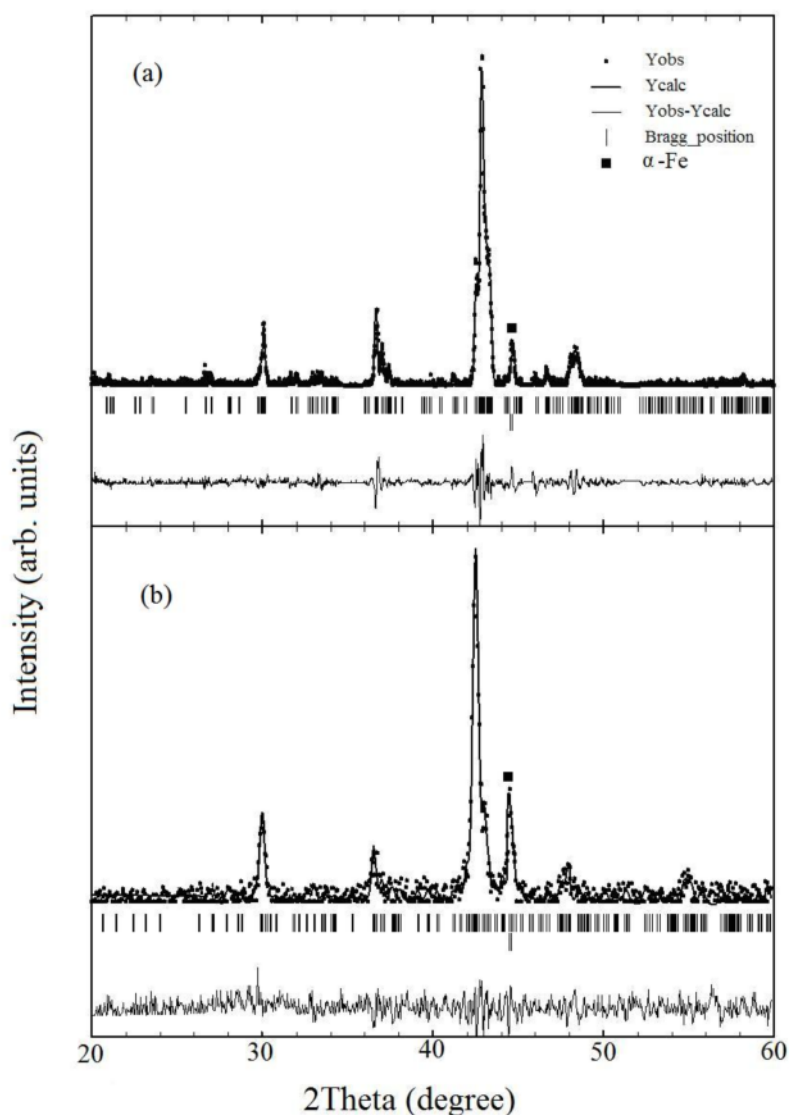

Fig. 1 X-ray diffraction of (a) ingot and (b) melt spun of $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compound. The circles represent the raw data. The solid line represents the calculated profile. Vertical bars indicate the position of Bragg peaks for the 3:29, 1:12, and α-Fe structures, respectively. The lowest curve is the difference between the observed and the calculated patterns. The strongest impurity peaks are marked with (■) for α-Fe phase.

Table 1 The phase composition and unit cell parameters of the monoclinic ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds; space group A2/m.

sample	a (Å)	b (Å)	c (Å)	β (°)	V (Å³)	Crys. Size (nm)	Crys. Density (g/cm³)	R_{wp}	R_{exp}
ingot	10.650	8.6291	9.721	97.174	886.383	26.912	7.939	12.5	7.8
melt-spun	10.250	8.609	9.631	96.774	843.955	21.958	7.645	15.9	8.6

Figure 2, illustrates the magnetic isotherms measured at room temperature. The room temperature saturation magnetization for ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds deduced from the law of approach to saturation are 142.4 and 132.5 emu/g, respectively. It is known that metallic iron presents a saturation magnetization of 220 emu/g at RT. Therefore, the saturation magnetization of 3:29 phase for melt-spun sample decreases from 132.5 to 102.2 emu/g compared to the bulk sample. In the inset of Fig. 2, we display the room temperature hysteresis loops for ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds. The coercivity often increases in alloys that are melt-spun to form nanocrystalline alloys due to smaller crystalline size [16].

Figure 3 shows the real part of the magnetic AC susceptibility versus temperature for ingot and melt-spun compounds. Peaks are observed at 190 K and 210 K, in the magnetic AC susceptibility of ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds, respectively. Markandeyulu et al. [27] reported the presence of a type-2 FOMP (first order magnetization processes) in the temperature range between 5 K and 200 K for $Pr_3(Fe_{1-x}Co_x)_{27.5}Ti_{1.5}$ (x = 0- 0.4). Gholizadeh et al. [28], observed the FOMP of type 2 using the singular point detection (SPD) technique from 5 to 300 K, in $Tb_3 (Fe_{28-x}Co_x) V_{1.0}$ (x = 0, 3, 6) polycrystalline compounds. The observed maximum in AC susceptibility can be attributed to type-2 FOMP transition in ingot and melt-spun compounds.
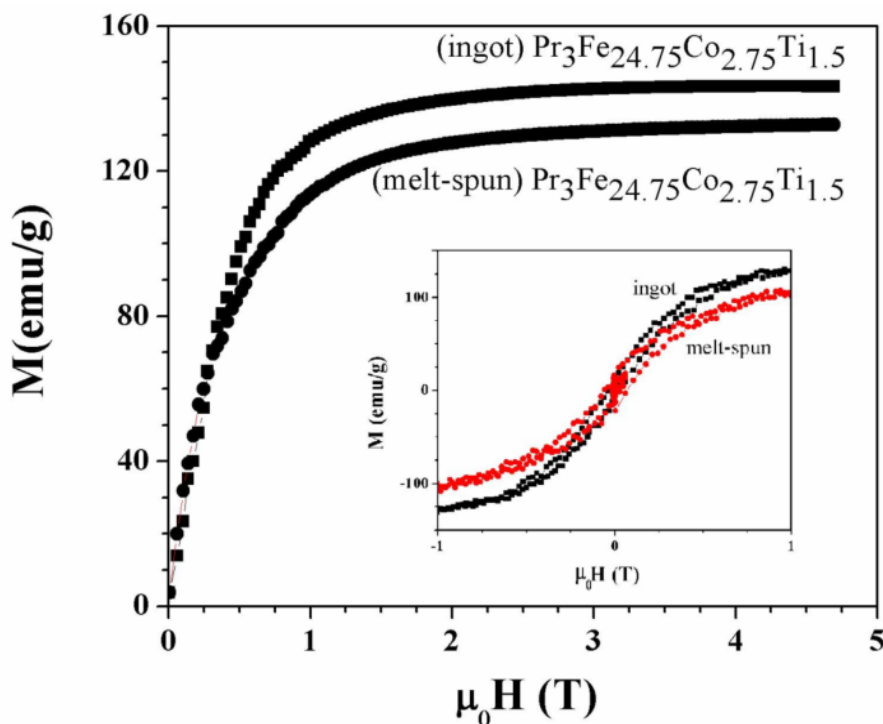
Fig. 2 The magnetic isotherms measured at room temperature for ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds. Inset: hysteresis loops at 300 K of ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds.

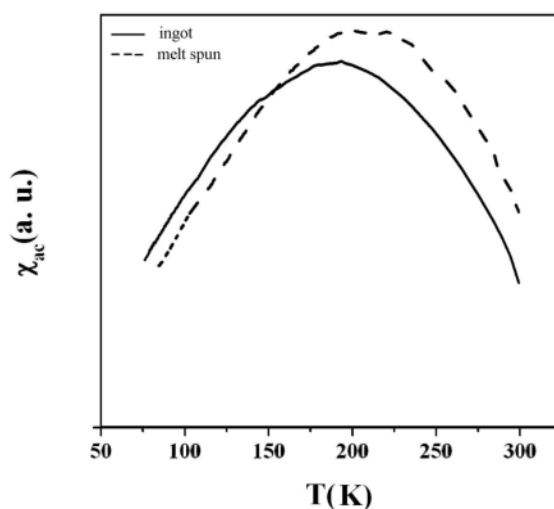**Fig. 3** Ac-magnetic susceptibilities versus temperature for ingot and melt spun of $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compound.

The measured linear thermal expansion (LTE), the LTE coefficient, α, and the spontaneous volume magnetostricton, ω_s (T), for ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds are displayed from 77 to 575 K in Fig. 4. The magnetic ordering temperature (T_C) can be obtained accurately from the LTE coefficient data showing a well-defined anomaly at T_C. The values of T_C are found to be 502 K and 331 K for ingot and melt-spun samples, respectively. The results indicate a reduction in the T_C values for the melt-spun compound compared to ingot. This reduction was also reported by Sirisha et al. [16] in melt-spun Pr_3 (Fe, Co, Ti) $_{29}$ compounds that has been attributed to the increasing negative contribution of antiferamagnetic coupled sites on 3d sublattice. The average LTE coefficients below magnetic ordering temperatures are almost 8×10^{-6} and 6.5×10^{-6} K^{-1} for ingot and melt-spun, respectively. Fig. 4 (c) shows the spontaneous volume magnetostricton, ω_s (T), for two samples. The large magnetovolume effect observed in ingot compound is expected to originate from the dependence of the exchange interaction on distance and also the effect of volume on the 3d sublattice magnetic moment [20]. It can be seen that as the temperature increases from 77 K to T_C, ω_s decreases from 8.3×10^{-3} and 0.5×10^{-3} to near zero for ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$, respectively. The non-zero value of ω_s beyond T_C indicates the existence of strong short-range magnetic correlations similar to those found in other $R_3(Fe,Ti)_{29}$ compounds [18, 19]. However, the magnitude of ω_s (77 K) obtained for the ingot is much bigger than the melt-spun value.

It is well known that the major contribution to ω_s of intermetallic compounds originates from the spontaneous volume magnetostriction of the iron sublattice arising from the exchange interactions between the magnetic moments of the Fe atoms. The unit cell volume of ingot and melt-spun is 886.383 and 843.955 $Å^3$, respectively (see Table 1). The values of the T_C and the saturation magnetization observed for the melt-spun are smaller than the ingot. In the ingot sample, the substitution of Co leads to an increase of T_C and saturation magnetization [5]. However, the latter values are found to be smaller for the melt-spun samples of the same composition having lower unit cell volume and higher impurity α-Fe phase [16]. It is worth mentioning that the value of unit cell volume of these compounds influence on different values and signs of the Fe-Fe exchange interactions on the 3d sublattice magnetic moments [29]. The Fe atoms couple with a negative exchange interaction at Fe_1 ($2c$), Fe_8 ($8j$) and Fe_{11} ($4e$) sites with their neighboring atoms in monoclinic structure where there are two Fe atoms in a distance closer than 2.45 Å [29]. The decrease of the unit cell volume observed in the melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compound decreases net exchange interactions by increasing the negative contribution of antiferromagnetically coupled sites on the 3d sublattice. Therefore, the lower values of T_C, M_S and ω_s determined for the melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ with respect to the ingot sample can be attributed to the much lower value of the unit cell volume. In addition, free α-Fe atoms as well as other impurity phases found may cause a variation of the actual Fe, or Co concentrations in

the alloy composition of the samples. The α-Fe phase can produce short-range ferromagnetic interaction originated from magnetic phase separation in samples [30]. The existence of strong short-range ferromagnetic interaction has been found in the non-zero value of ω_s beyond T_C of samples. The higher value of α-Fe phase in the melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ with respect to the

ingot sample may extremely decrease the long range ferromagnetic interaction and increase the short range ferromagnetic interaction in Fe sites. Therefore, the increase of short range ferromagnetic interaction may explain the lower values of T_C, M_S and ω_s determined for the melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ sample with respect to the ingot sample.
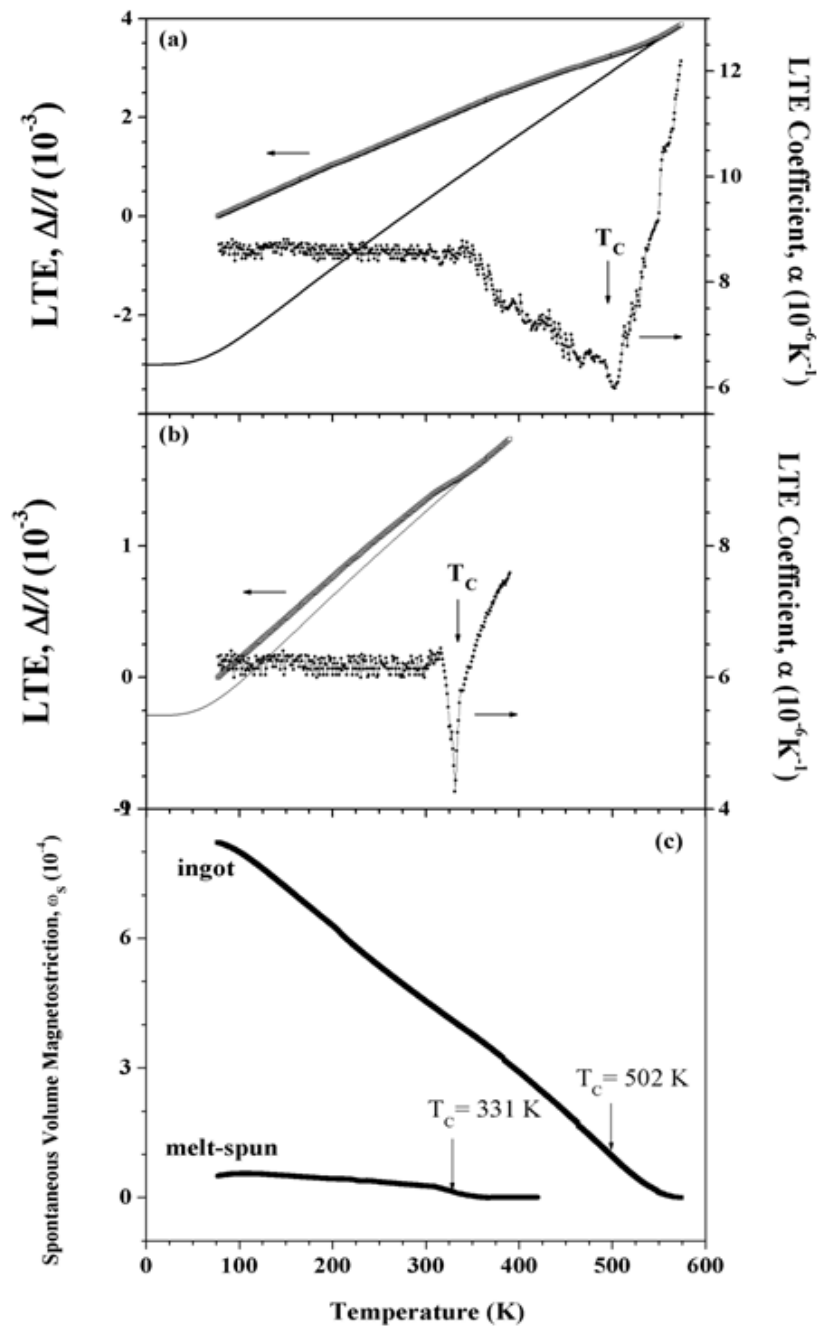

Fig. 4 Temperature dependence of the measured linear thermal expansion (LTE) and the LTE coefficient, α, for (a) ingot and (b) melt spun of $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$. (c) Display the spontaneous volume magnetostricton, ωs (T), of samples.

Isothermal curves of the longitudinal magnetostriction (λ_{pa}) for ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ at selected temperatures are shown in Fig. 5. λ_{Pa} of both samples shows parabolic behavior at all temperatures tending to saturate near T_C. In addition, saturation trend of λ_{Pa} is consistent with the fact that the anisotropy field of $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compound is determined to be 1.2 T at room temperature [12]. It can be noted that λ_{Pa} values versus field strength for the ingot is several times larger than the values for the melt-spun. In accordance with the above results, the lower values of λ_{Pa} observed for the melt-spun relative to the values obtained for the ingot sample is attributed to the higher value of the unit cell volume of the latter sample. In addition, the value of λ_{Pa} at maximum applied field decreases with temperature. The non-zero values of λ_{Pa} beyond the ordering temperature for melt-spun sample can be due to the presence of α-Fe in the compound.

In Figure 6; the transverse magnetostriction (λ_{pe}) of the ingot and melt-spun samples are plotted versus the applied fields at selected temperatures. λ_{pe} passes through a minimum at different applied fields and higher temperatures for the ingot and the melt-spun samples. The EMD for the $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compound is along the b-axis and there is a anisotropy field of 1.2 T at RT in the a-c plane of the monoclinic structure [12]. The analysis of the present data may the of follows; low temperatures, where the maximum applied field (H) is smaller than anisotropy field (H_a) and consequently the magnetisation vector rotates in the a-c plane, λ_{pe} for the ingot sample shows a decrease. Finally, when $H > H_a$, the magnetization vector rotates toward the applied field and then increases with the applied field and reaches to a small value at T_C. In the case of melt-spun sample, λ_{Pe} behaviour is nearly the same, except that the minimum of the curves occur at lower applied fields. The results and behavior of λ_{pa} and λ_{pe} versus magnetic field at different temperatures for the ingot and melt-spun samples can be related to their different microstructure, grain size and their crystallographic texture. Future systematic experimental investigations should be carried out to elucidate the anisotropic λ behavior in these compounds.
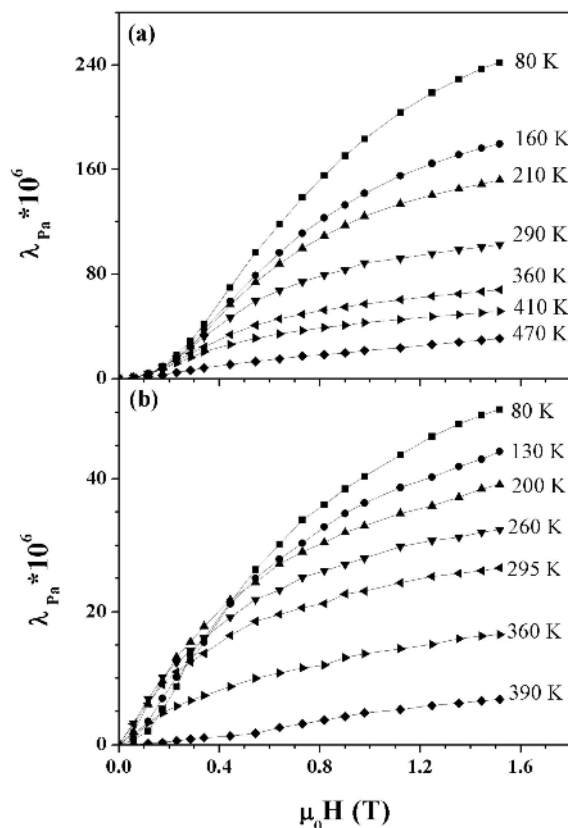

Fig. 5 Isothermal curves of the longitudinal magnetostriction of (a) ingot and (b) melt spun of $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compound as a function of applied field at selected temperatures.

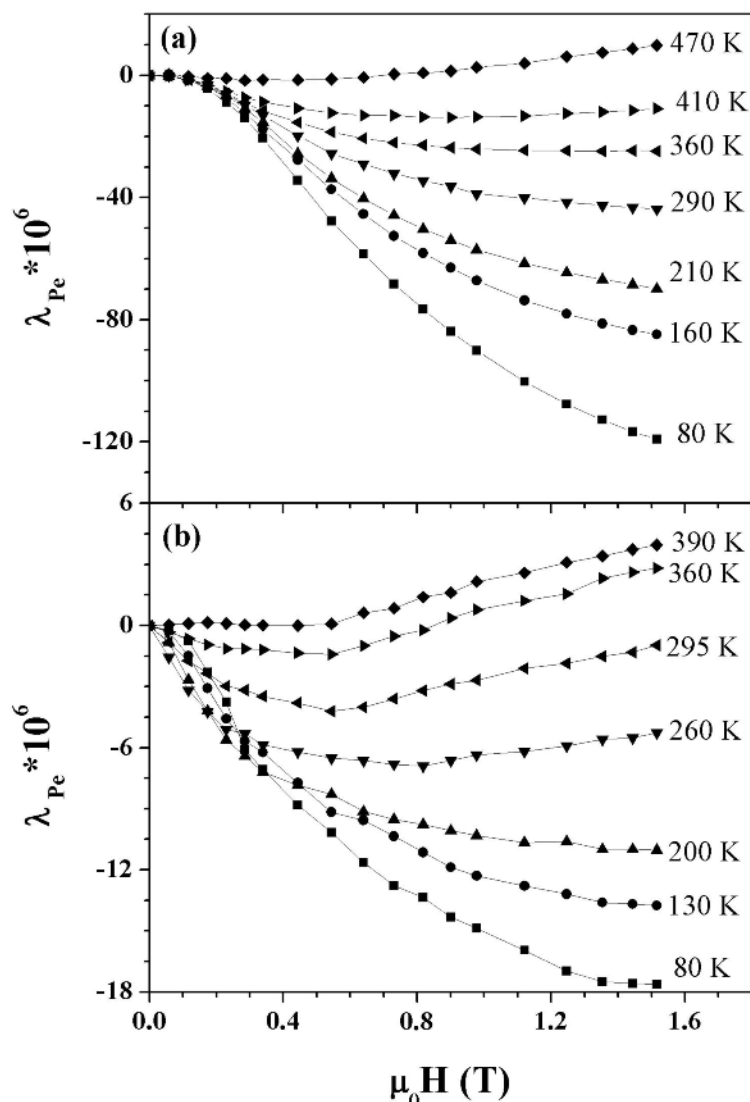**Fig. 6:** Temperature dependence of the transverse magnetostriction of ingot and melt spun of $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compound as a function of applied field at selected temperatures.

Conclusion

The type of the results observed for the linear and volume magnetostriction for both studied samples are believed to belong to the main $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ phase. The lattice parameters refined show a reduction in the melt-spun compound with respect to ingot compound. The room temperature saturation magnetization of 3:29 phase for ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds deduced from the law of approach to saturation (LAS) are 132.5 and 77.7 emu/g, respectively. The 2-type FOMP has been observed at 190 K and 210 K, in the magnetic AC susceptibility of ingot and melt-spun compounds, respectively. The magnetic ordering temperatures obtained from the LTE coefficient, are 502 K and 331 K for ingot and melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds, respectively, indicating a reduction in the T_C values for the melt-spun compound. The spontaneous volume magnetostriction obtained from the extrapolation of the paramagnetic regime of the LTE curve and the longitudinal and transverse magnetostriction values measured for ingot $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compound are several times larger than the melt-spun. The observed magnetic behaviors in melt-spun $Pr_3Fe_{24.75}Co_{2.75}Ti_{1.5}$ compounds are related to the different values and signs of the Fe-Fe exchange interactions on the 3d sublattice magnetic moments originated from the volume effect. The values of the longitudinal and transverse magnetostriction at the maximum applied field decreases and reaches

to a small value at T_C. Future systematic experimental investigations should be carried out to elucidate the magnetostriction behavior in these compounds.

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