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Exchange-bias-like effect in $Pr_{0.75}Tb_{0.25}Al_2$ and $Pr_{0.7}Tb_{0.3}Al_2$ samples

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ABSTRACT

The magnetic behavior of pseudobinary $Pr_{0.7}Tb_{0.3}Al_2$ and $Pr_{0.75}Tb_{0.25}Al_2$ compounds was studied, and a predominant ferrimagnetic ordering was observed. Noteworthy characteristics such as negative magnetization, compensation points and exchange-bias-like (EB-like) effect were found. This EB-like effect was observed at temperatures below the compensation points. The effect is somewhat different from the one already studied in similar systems combining light and heavy rare earths. The results indicate that the EB-like effect characteristics are related to the conduction electron magnetic polarization and an induced unidirectional anisotropy present in these compounds.

1. Introduction

In the 1960s, Williams et al. [1] and Swift and Wallace [2] described the magnetic interaction between distinct rare earth elements in intermetallic compounds. Since then, several works on similar systems have been published [3–7]. They assumed that heavy and light rare earths have an antiferromagnetic coupling when immersed in a crystal lattice. Williams and co-workers have studied several $R_{1-x}R'_xAl_2$ systems, where R, R'=rare earths, including the $Pr_{1-x}Gd_xAl_2$ series. Their experimental work indicated that pseudobinary systems present ferromagnetic coupling when either both the lanthanides are light (from La to Eu) or both are heavy (from Gd to Lu). However, the coupling is antiferromagnetic for light–heavy combinations. Moreover, a recent work [8,9] has confirmed a ferrimagnetic ordering in $Pr_{1-x}Gd_xAl_2{}^{\circ}$.

The magnetic ordering diversity in rare earth based intermetallic compounds has become very attractive as it gives rise to several effects with potential applications. Inverse magnetocaloric effect and exchange bias [10,11] are examples of these effects. The exchange bias (EB) is characterized by the displacement of the barycenter of the hysteresis loop along the field axis. The EB effect is generally observed and studied in multilayer thin film systems formed by ferromagnetic-antiferromagnetic (FM–AFM) compounds [12,13]. In general, samples which present EB have the following characteristics: the Néel temperature (T_N) of AFM portion is lower than the Curie temperature (T_C) of the FM portion; the AFM portion presents a uniaxial anisotropy; and there is enough exchange interaction between the spins at the FM/AFM interface. With these characteristics, a unidirectional anisotropy can be induced by application of magnetic field while cooling of the sample. Materials that exhibit this behavior have potential application in information storage technology devices, magnetic random access memory and as magnetic field sensors [14–18].

In the above context, $Pr_{1-x}Tb_xAl_2$ pseudobinary compounds are examples of materials based on light and heavy rare earth elements presenting potentially applicable physical properties. Their magnetic behavior can be "tuned", as to say, by choosing distinct compositions. Ferrimagnetic ordering can be found, as well as a wide range of magnetic transition temperatures. By taking the typical values of the effective magnetic moments ($\mu_{Tb}=9\ \mu_B$ and $\mu_{Pr}=3.2\ \mu_B$) [19,20], and assuming that the moments of Tb ions are coupled in the opposite direction of the Pr ion moments (i.e., antiferromagnetic coupled as predicted by Williams, Swift and Wallace [1,2,21]), one can calculate $x=0.26$ as the composition that favors the appearance of the compensation points. In this sense the compositions close to $x=0.25$ and $x=0.3$ are of particular interest, and, in fact, compensation temperatures (T_{comp}) have been observed below the ferrimagnetic ordering temperature for these two compositions. We have studied samples of this family of compounds prepared with distinct compositions and treated by typical procedures to investigate their magnetic behavior. The present work is focused on the particular compounds $Pr_{0.7}Tb_{0.3}Al_2$ and $Pr_{0.75}Tb_{0.25}Al_2$ for which an exchange-bias-like effect has been observed at low temperatures.

Recent works [22–24] have shown similar effects near and below the compensation temperature value of the $Nd_{1-x}Ho_xAl_2$,

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$Pr_{1-x}Gd_xAl_2$ and $Sm_{1-x}Gd_xAl_2$ compounds. These effects are related to the ferrimagnetic ordering and related to particularities of the magnetic polarization of the rare earth conduction electrons. Although similar to what is observed in these compounds, the exchange bias does not occur in $Pr_{0.75}Tb_{0.25}Al_2$ and $Pr_{0.7}Tb_{0.3}Al_2$ around T_{comp}, but only below 20 K for $x=0.3$ and below 10 K for $x=0.25$ samples.

2. Material and methods

Bulk samples were prepared in an arc-melting furnace under argon atmosphere using amounts of pure elements corresponding to the samples nominal stoichiometry. The purity grades of the former materials are 99.99 and 99.9 wt% for aluminum and rare earths, respectively. After the melting process, parts of the samples were thermally treated for 5 h under argon atmosphere at 1273 K. Powder samples have been produced manually by grinding bulk samples down to 20 μm.

X-ray diffraction measurements were performed in a Philips PW1710 diffractometer with a diffracted beam monochromator under Bragg–Brentano geometry and CuKα radiation. Steps of 0.02° and acquisition time of 5 s. per point were employed.

Magnetic measurements were obtained in a superconducting quantum interference device (SQUID—Quantum Design®) for different temperatures and applied magnetic fields. Comparison between different pieces of the bulk samples and powder samples has provided similar results. Successive hysteresis loops have shown no training effects.

3. Results and discussion

3.1. Crystallographic properties

The diffraction patterns confirm the cubic C15 Laves phase, MgCu2-type, Fd3m structure in which the rare earth site is described by the point group T_d [20]. The samples present only one crystallographic phase within the experimental error. The powder diffraction measurements of $Pr_{0.7}Tb_{0.3}Al_2$ is shown in Fig. 1. In this figure, one can observe the good agreement obtained from the Rietveld refinement through the different patterns shown at the bottom of the figure.

Lattice parameters were obtained for the whole series and follow a linear relation respect to 'x' ($Pr_{1-x}Tb_xAl_2$), being $a=7.864$ Å for $TbAl_2$ and $a=8.033$ Å for $PrAl_2$. The $Pr_{0.7}Tb_{0.3}Al_2$ and $Pr_{0.75}Tb_{0.25}Al_2$ samples have provided the values $a=7.986$ Å and 7.992 Å, respectively. An accentuated texture was observed in the Laue measurements (not presented here) for the bulk samples. However, it has caused no effect whatsoever in the magnetic properties.

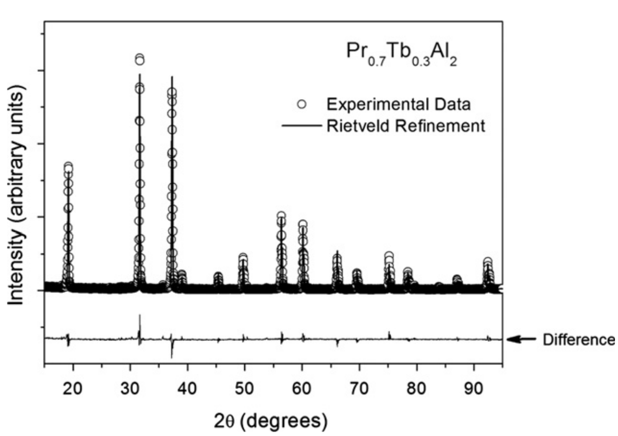

Fig. 1. Powder diffraction pattern of $Pr_{0.7}Tb_{0.3}Al_2$ showing the good agreement between the experimental and the calculated patterns. The difference in pattern between them is also shown.

3.2. General magnetic behavior

Fig. 2 shows the magnetization curves as a function of the temperature for $Pr_{0.75}Tb_{0.25}Al_2$ and $Pr_{0.7}Tb_{0.3}Al_2$ samples. These pseudobinary materials present the ferrimagnetic characteristic behavior. The magnetization curves present a maximum value followed by a significant reduction as the temperature is lowered below the magnetic ordering value. At low temperatures, one can observe that the behavior of both samples is severely influenced by the application of the magnetic field. A thermal hysteresis is clearly observed between the zero field cooling (ZFC) and field cooling curves (FC) obtained at lower fields. But the field heating (FH) curves (not presented here) are practically identical to the FC curves, showing no thermal hysteresis no matter which field is applied. Nevertheless, the magnetic general behavior is rather distinct for each sample.

In Fig. 2(a), one can see that the magnetization curves of the $Pr_{0.75}Tb_{0.25}Al_2$ compound present thermal hysteresis for $H=200$ Oe and for $H=3$ kOe. The 200 Oe curve exhibits the Curie temperature (T_C) at 48 K. This temperature is between 32 K (transition temperature for $PrAl_2$ [20]) and 107 K (T_C for $TbAl_2$ [20]). For $Pr_{0.75}Tb_{0.25}Al_2$, it is possible to clearly distinguish a compensation point at 26 K (T_{comp}) in the 200 Oe FC curve. For temperatures below T_{comp}, the magnetization values become negative. In this case, the magnetization modulus increases up to 0.15 μ_B/f.u. and remains at this value in the lowest temperature range. This behavior only occurs at low magnetic fields. It is believed that negative values of magnetization are related to a strong induced unidirectional anisotropy. The system formed by the Tb and Pr magnetic sublattices probably exhibits uniaxial anisotropy [23]. However, the polycrystalline structure hinders this anisotropy in the magnetic bulk results. Nevertheless, a unidirectional anisotropy is induced when a magnetic field is applied during the sample cooling, similarly to what occurs in FM–AFM thin films systems. One can also observe a minimum for

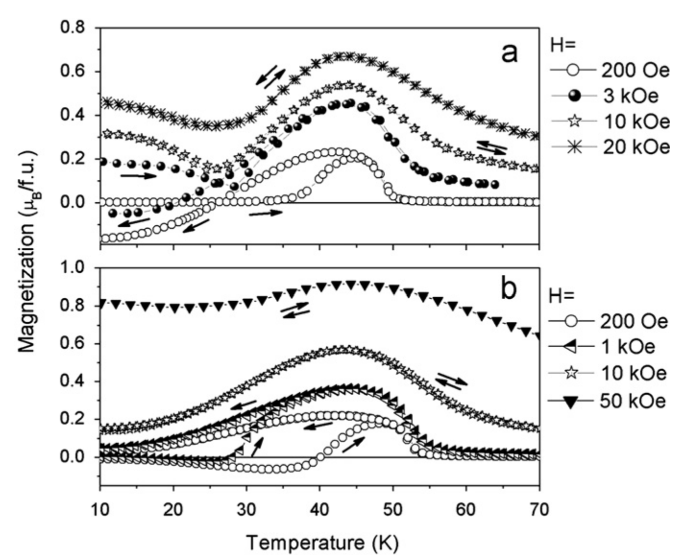

Fig. 2. Magnetization as a function of temperature obtained with several applied fields for the two samples: (a) $Pr_{0.75}Tb_{0.25}Al_2$ and (b) $Pr_{0.7}Tb_{0.3}Al_2$. The arrows indicate the measurements done increasing (ZFC) or decreasing (FC) the temperature.

temperatures next to 26 K in the curves obtained with higher fields, characterizing the T_{comp}.

In Fig. 2(b), the M vs. T curves for $Pr_{0.7}Tb_{0.3}Al_2$ compound are presented. The Curie temperature is $T_C=52$ K and a thermal hysteresis is also present in the low field curves. For this sample, compensation points are only seen in ZFC curves at low fields and are highly field dependent. For 200 Oe M vs. T curve, $T_{comp}=40$ K. For 1 kOe magnetization curve, T_{comp} is less than 30 K. At higher fields, the compensation point shifts to lower temperatures and the thermal hysteresis vanishes. For all magnetization curves, below the compensation point, the modulus of the magnetization decreases smoothly, being very small (<0.1 μ_B/f.u.) at 10 K, when the applied field is relatively low ($H=200$ Oe and $H=1$ kOe).

These field sensitive characteristics manifest themselves in the ordered phase only at very low temperatures and relatively low field. They are probably related to the conduction electrons, more susceptible to fields under those circumstances. In fact, in the exchange-bias-like effect, the role of the conduction electrons in the magnetic behavior becomes more important below 10 K, as it will be discussed in the following section.

Assuming the model with two magnetic sublattices [9,25] for ferrimagnetic systems, one can justify most of the properties described above. Naturally, the first guess would be to take each one of these sublattices as related to each one of the rare earths. The main Pr and Tb contribution for the atomic magnetic moments is due to the core electrons [19]. This aspect is not expected to be significantly influenced by the condensed matter state of these compounds. Then, the association of a sublattice to each rare earth element is enough to promote compensation points depending upon the specific composition. Concentrations next to $x=0.26$ favor the appearance of the compensation points in $Pr_{1-x}Tb_xAl_2$ pseudobinary family compounds, as commented in the introduction.

The variations observed in the M vs. T curves are related to the distinct changes of the effective magnetic moments of the Pr and Tb sublattices along with the temperature variation, which also includes the contribution from the conduction electrons polarization. Both the polarization and its role in the interaction between the spins of the rare earth ions can be interpreted in the context of the RKKY interaction. The RKKY is the usual base model for the magnetic interactions in rare earth metals and alloys, and it depends on the intensity of the interaction between the ion spin and the conduction electrons, as well as, the extension of the magnetic polarization of these electrons. The conduction electrons wave functions of Tb extend over a shorter distance than the ones of Pr [19], therefore it should be expected to have significant distinction between the behavior of the moments associated to each rare earth as a function of the temperature.

3.3. Exchange-bias-like effect

Fig. 3 shows hysteresis loops for $Pr_{0.75}Tb_{0.25}Al_2$ and $Pr_{0.7}Tb_{0.3}Al_2$ compounds obtained after cooling with an applied field of 50 kOe. The insets present almost the linear portions of the loops for two different temperatures.

Above the ordering temperatures, the curves have the Langevin-like shape. These shapes are typical of the paramagnetic state, but saturation does not occur. The linear increase of the magnetization as a function of the field shown in Fig. 3 is related to a significant contribution from the conduction electrons (Pauli paramagnetism). In the ferrimagnetic temperature range, the samples present relatively large hysteresis and the Pauli contribution is even clearer. The magnetization reversion is smoother in $Pr_{0.7}Tb_{0.3}Al_2$ than in $Pr_{0.75}Tb_{0.25}Al_2$, reflecting a harder ferrimagnetic ordering in the last compound.

Below 20 K, and therefore below T_{comp}, the Pauli contribution to the magnetization is the most important. The Pauli susceptibility can be estimated from the curves as $\chi_P=1.2\times10^{-4}$ emu/mol for $Pr_{0.7}Tb_{0.3}Al_2$ and $\chi_P=1.1\times10^{-4}$ emu/mol for $Pr_{0.75}Tb_{0.25}Al_2$. It should be noted that such values are of the same order of the ones obtained for the nonmagnetic compounds YAl_2 (1.76×10^{-4}) and $LaAl_2$ (2.25×10^{-4} emu/mol) [26], and smaller than the obtained for $GdAl_2$ (6×10^{-4} emu/mol [27]). Nevertheless, the coercivity at this temperature range cannot be disregarded. One can also observe the clear displacements of the hysteresis with respect to the zero field position, which is a signature of the exchange bias effect.

As the main contribution to the magnetization at this temperature range is apparently related to the conduction electron, it cannot be associated with the conventional exchange bias effect. In analogy with FM–AFM thin films systems, polarized conduction electrons play the role of the FM layer, and the lattice of spins formed by the magnetic Pr–Tb ions acts as the AFM layer, resulting in an induced unidirectional anisotropy, as mentioned before. The characteristic direction of this anisotropy is set by the magnetic field orientation adopted during the cooling procedure.

The remanent magnetizations (M_r) as a function of temperature, collected from the M vs. H curves, are presented in Fig. 4.

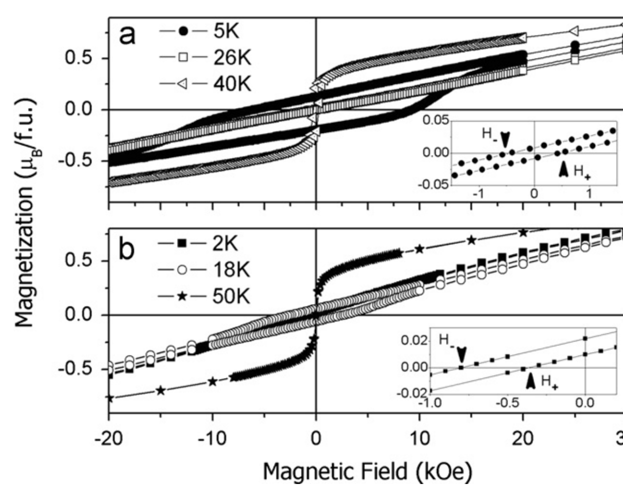

Fig. 3. Hysteresis loops for (a) $Pr_{0.75}Tb_{0.25}Al_2$ and (b) $Pr_{0.7}Tb_{0.3}Al_2$ samples at different temperatures. The insets show the almost linear regions of the loops around zero applied fields at (a) 26 K and (b) 2 K.

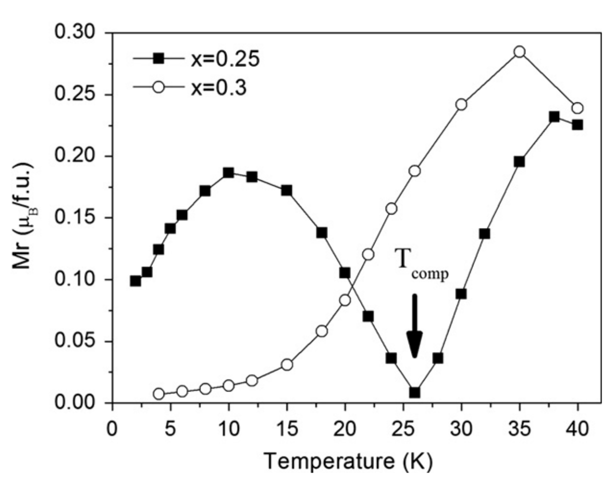

Fig. 4. Remanent magnetization (Mr) behavior with respect to the temperature for $Pr_{0.75}Tb_{0.25}Al_2$ and $Pr_{0.7}Tb_{0.3}Al_2$ bulk samples.

The curve for the $x=0.25$ sample shows a global minimum at about $T=26$ K, which indicates the previously mentioned compensation temperature. As expected, this compensation point behavior is characteristic of ferrimagnetic systems [28]. In the case of the $x=0.3$ sample, its compensation temperature is highly dependent on the applied magnetic field, and the M_r vs. T curve does not present a global minimum. However, it is possible to observe that the value of M_r decreases while the temperature is lowered, which is in agreement with previously observed behavior for T_{comp}.

Fig. 5 presents the exchange bias field (HE) and the coercive field (HC) as a function of temperature for both samples. The HC curves present peaks at 6 K and 21 K, for $x=0.25$ and 0.3 samples, respectively. However, although $Pr_{0.75}Tb_{0.25}Al_2$ sample exhibits a negative minimum for HE in the 2.5 K and 12 K temperature range, it does not change the effect global behavior.

One can observe that the decrease of the HE curve for $Pr_{0.75}Tb_{0.25}Al_2$ from its maximum is more abrupt than the one for $Pr_{0.7}Tb_{0.3}Al_2$. For both compounds, HE values vanishes near to the global maximum in the HC vs. T curves, what is a common feature in systems presenting exchange bias. The temperature at which the exchange-bias effect disappears is called blocking temperature, and above it, HC decreases, as expected [12,28].

At lower temperatures, the unidirectional anisotropy is stronger. This fact implies in an increase in the HE modulus. When the temperature is raised the HE modulus decreases and HC modulus increases. When the blocking temperature is reached, whereas HE vanishes, HC shows a maximum. For higher temperatures, HC has its value reduced. The HC curve for $Pr_{0.75}Tb_{0.25}Al_2$ exhibits a global minimum at 26 K, also indicating T_{comp}.

Considering the ordering model discussed in the previous section and the relevance of the Pauli contributions to the magnetization, we are led to the conclusion that the bias in these loops are due to the behavior of the polarized conduction electrons. This behavior is similarly to what has been observed in $Nd_{1-x}Ho_xAl_2$ [23]. By observing the ZFC curve (Fig. 2), it can be seen that the smaller values of magnetization are found below 40 K, in the ferrimagnetic ordering for both samples. At this temperature range (<40 K), the Pauli paramagnetism contribution from the conduction electrons is already relatively important as compared to the contribution from the ionic spins to the total magnetization. But the bias effect is only seen at lower temperatures, in which the magnetic polarization of the conduction electrons becomes important (in the same way as

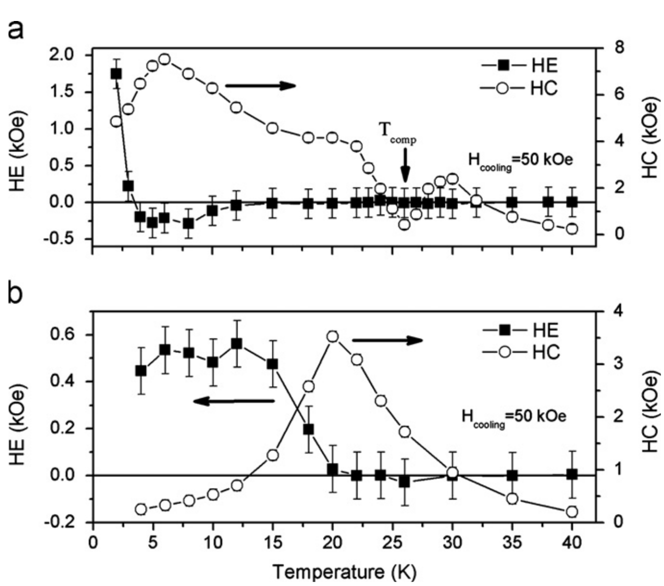

Fig. 5. Exchange bias and coercive fields as a function of temperature for the two samples: (a) $Pr_{0.75}Tb_{0.25}Al_2$ and (b) $Pr_{0.7}Tb_{0.3}Al_2$.

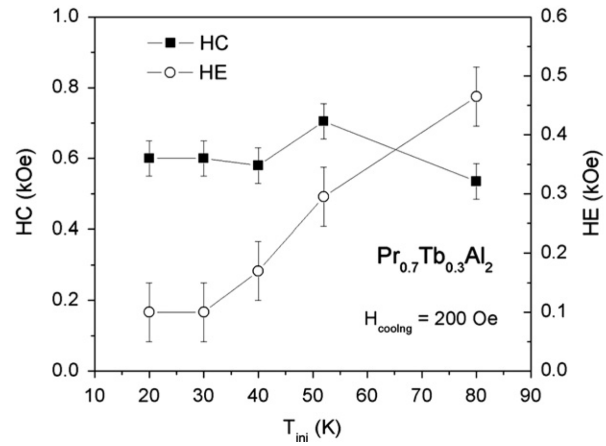

Fig. 6. Values of HE and HC at 10 K as a function of the initial temperature of field cooling.

observed in Kondo materials [29,30]) and in which the induced unidirectional anisotropy is stronger.

As the major amount of these conduction electrons is from Pr, their magnetic polarization should dominate the observed effects. Such polarization is changing rapidly in the temperature range below 30 K and stabilizes only below 10 K, temperature of saturation of the magnetization of $PrAl_2$ [31,32]. This temperature range is the same in which the bias effect has been observed. Although it is not the conventional exchange bias effect (an interface phenomenon), the effect observed here is indeed an "exchange" effect. It is due to the exchange interaction among the 4f electrons and the conduction electrons. This interaction is the central feature of the RKKY coupling and can be characterized by a positive or negative j_0 exchange constant. Therefore, the magnetic polarization close to the ion will be positive or negative relative to the 4f magnetic moment. The polarization becomes less intense and oscillates as the distance from the ion core increases. But the fact that the more intense polarization has a definite sign indicates the major role of the Pauli paramagnetic contribution, which by its turn provides the occurrence of the exchange-bias-like effect and its sign. The j_0 exchange constant is better seen as a parameter that depends on the specific aspects of the Fermi surface. Concerning its sign, it can be either negative, as for $GdAl_2$, or positive, as for $EuAl_2$ [33]. In $GdAl_2$, if Gd is partially substituted by the rare earths Dy, Ho, Er, La, Lu e Y, j_0 is also negative [26,34].

The bias effect is not affected by the magnitude of the magnetic field applied during the cooling process. This observation endorses that the exchange-bias-like effect observed here is related to the conduction electrons. Measurements performed with cooling fields of 0.2, 1, 2 and 50 kOe do not present significant alterations in the hysteresis loops obtained at low temperatures. On the other hand, the initial temperature from which the field cooling procedure begins affects the exchange bias field. This last aspect can be seen in Fig. 6.

This property is related to the polycrystalline structure of the samples. High initial temperature for field cooling implies in a large amount of spins oriented in the field direction. A more uniform orientation of the spins leads to a clearer and more intense exchange-bias-like effect. In fact, the largest HE values are obtained for those initial temperatures which are above the ordering temperature.

4. Conclusions

Ferrimagnetic order has been observed in pseudobinary $Pr_{1-x}Tb_xAl_2$ compounds with two different compositions, $x=0.3$

and 0.25. In the $Pr_{0.7}Tb_{0.3}Al_2$ compound, negative magnetization below 40 K has been observed in the ZFC curve. This behavior also happens in FC curve for $Pr_{0.75}Tb_{0.25}Al_2$ below 26 K. Compensation points for the magnetization were observed for both compounds. For $Pr_{0.75}Tb_{0.25}Al_2$, T_{comp} has no significant field dependence. But for $Pr_{0.7}Tb_{0.5}Al_2$, T_{comp} is highly field dependent: its value decreases while the field increases. Those negative magnetization values are mainly due to a strong unidirectional anisotropy. This characteristic is also important to explain the other observed phenomenon: the exchange-bias-like effect.

This effect has been observed for both compositions $x=0.3$ and 0.25. It should be noticed that the bias effect is only seen at lower temperatures, below the compensation points. Several observed characteristics lead us to believe that the EB-like effect is related to the significant contribution from the polarized conduction electrons to the magnetization. The combination of the AFM arrangement between Tb and Pr spins with the expressive magnetic polarization of the conduction electrons at low temperatures provides this unidirectional anisotropy, which is induced by cooling under magnetic field application.

The use of neutron scattering techniques is a possible complementary experimental study to this work, as well as the possibility of growing and studying $Pr_{1-x}Tb_xAl_2$ single crystals. They could provide further information on these compounds and, so, clarify several physical properties.

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