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Field-induced transitions and magnetization reserval in $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$ ($0.0 \leq x \leq 1.0$)

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Magnetic properties of the $Er(Co,Mn)O_3$ solid solution result from a subtle interplay of different interactions between erbium moments and several other magnetic entities present in this system: Mn^{3+}, Mn^{4+}, Co^{2+}, Co^{3+}, as well as double-exchange interactions among them. The special case of the 0.5:0.5 = Co:Mn substitution is particularly important since ferromagnetic domains may reorientate both under low and high external magnetic fields. Magnetic dilution of the erbium sublattice by non-magnetic yttrium (same valence and size) allows separating out the antiferromagnetic 4f-3d interactions leaving just the ferromagnetic Co^{2+}-Mn^{4+} transition metal sublattice.
Magnetic properties of the $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$ ($0.0 \leq x \leq 1.0$) solid solution are presented, going from ferromagnetism (x = 0; $YCo_{0.5}Mn_{0.5}O_3$) to ferrimagnetism (x = 1; $ErCo_{0.5}Mn_{0.5}O_3$). Antiferromagnetic interactions get stronger while the Co/Mn ferromagnetic domains become harder to rotate when the rare-earth sublattice is progressively filled with erbium ions of large magnetic moments. A linear dependence with x(Er) is observed for the critical field H_{crit} related to the reorientation of domains, going from 1.3 T up to 3.5 T, for x = 0 and 1, respectively. At the same time, the compensation temperature T_{comp} increases and the spontaneous magnetization M_{rem} decreases with increasing content of erbium.

Keywords: Field-induced transitions, Spin reversal, Magnetic domains, Dynamical transitions.

Transiciones inducidas por campo e inversión de la imanación en $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$ ($0.0 \leq x \leq 1.0$)

Las propiedades magnéticas de las soluciones sólidas $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$ ($0.0 \leq x \leq 1.0$) son consecuencia de una sutil interrelación de las diferentes interacciones entre los momentos magnéticos del Er^{3+} y de otras entidades magnéticas presentes en el sistema: Mn^{3+}, Mn^{4+}, Co^{2+}, Co^{3+}, así como las interacciones de doble canje entre ellas. El caso especial de la sustitución 0.5:0.5 = Co:Mn es particularmente importante puesto que los dominios ferromagnéticos pueden reorientarse bien con campos externos bajos como con altos. La dilución magnética de la subred de Er^{3+} por el ión no-magnético Y^{3+} (del mismo tamaño y valencia) permite separar las interacciones antiferromagnéticas 4f-3d dejando solamente las interacciones ferromagnéticas Co^{2+}-Mn^{4+} de la subred de los metales de transición.
Las propiedades magnéticas de las soluciones sólidas $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$ ($0.0 \leq x \leq 1.0$) se presentan como variando desde ferromagnetismo (x = 0; $YCo_{0.5}Mn_{0.5}O_3$) a ferrimagnetismo (x = 1; $ErCo_{0.5}Mn_{0.5}O_3$). Las interacciones antiferromagnéticas se hacen más fuertes mientras que los dominios ferromagnéticos se hacen más difíciles de rotar cuando la subred de la tierra rara se va llenando progresivamente con iones Er^{3+}, de momento magnético alto. Se observa una dependencia lineal con x(Er) del campo crítico H_{crit} relacionado con la reorientación de dominios, variando desde 1.3 T hasta 3.5 T, para x = 0 y 1, respectivamente. Al mismo tiempo, la temperatura de compensación, T_{comp} aumenta y la magnetización espontánea, M_{rem} disminuye con el contenido creciente de Er^{3+}.

Palabras clave: Transiciones inducidas por campo, inversión de spin, dominios magnéticos, transiciones dinámicas.

1. INTRODUCTION

The discovery of giant magneto-resistance in manganese oxides has resulted in a renewed interest in mixed-valence manganese perovskites over the last decades (1,2). The key feature is the double-exchange mechanism due to the simultaneous presence of Mn^{3+} and Mn^{4+} ions, which triggers the well-known insulating-conducting phase transitions (3-6). A large number of reports have described the structural, electrical and magnetic properties of doped ABO_3 perovskites, where the A-site is occupied by a rare-earth element RE partially substituted by an alkaline-earth. Substitutions at the B-site occupied by manganese have been studied less frequently in the past, in spite of the fact that similar transformation mechanisms between Mn^{3+} and Mn^{4+} should occur. In this later case, the general formulation would be $RE^{3+}Me^{2+}_xMn^{3+}_{1-2x}Mn^{4+}_xO^{2-}_3$, in which several magnetic entities (Mn^{3+}, Mn^{4+}, Me^{2+}) will coexist with the rare-earth magnetic moment.

Interesting features may occur if, instead of a large rare-earth element (e.g., La), the manganite perovskite contains a small rare-earth cation of intrinsically large magnetic moment (e.g., Dy, Er, Gd). Then, the presence of two magnetically strong networks composed of the transition metal and the lanthanide

elements will lead to important co-operative effects. If the exchange interaction between these two networks becomes negative and, in addition, the magnitude of the rare-earth moments is much higher than the one due to the ferromagnetic transition-metal sublattice, then a ferrimagnetic-like situation with inversion of the spin's direction may occur (7).

In recent years we have been studying the magnetic properties of several perovskites of formula $RE(Co,Mn)O_3$ in which the Mn atom has been substituted by cobalt. The most spectacular results were observed for RE = Er, where a subtle interplay develops between different interaction mechanisms leading to unusual phenomena such as a step-like transition and an intersection of the increasing and decreasing branches of the magnetization loops M(H) (8,9). These phenomena are especially evident at the particular composition Co:Mn = 1:1 ($ErCo_{0.50}Mn_{0.50}O_3$) where ferromagnetic Co^{2+}-Mn^{4+} interactions are maximized while, at the same time, an AF exchange interaction between Er and transition metals leads to a spin reversal, the total moment becoming negative below the compensation temperature T_{comp} (10). Similar investigations were done in the past for $Y(Co,Mn)O_3$ perovskites, for which no contribution due to the RE sublattice was expected ; there again, the ferromagnetic interactions are maximized at the 1:1 substitution rate ($YCo_{0.50}Mn_{0.50}O_3$) (11).

In order to better understand the different interactions in $Er(Co,Mn)O_3$ and, in particular, in the $ErCo_{0.50}Mn_{0.50}O_3$ composition, we have prepared the solid solution $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$ ($0.0 \le x \le 1.0$). Thanks to the almost identical ionic radii for Y^{3+} (1.019 Å) and Er^{3+} (1.004 Å) (12), no structural changes are expected. In this way it is possible to "isolate" the effects just due to the magnetic evolution, which goes from ferro (x = 0) to ferri-magnetism (x = 1).

2. EXPERIMENTAL PROCEDURE

Both poles of the solid solution, that is, $ErCo_{0.50}Mn_{0.50}O_3$ and $YCo_{0.50}Mn_{0.50}O_3$, were first prepared by solid state synthesis from the corresponding submicronic powder oxides Er_2O_3, Y_2O_3, MnO and Co_3O_4. The mixtures were thoroughly mixed and homogenized by attrition milling with zirconia balls, using isopropanol as liquid medium. The dried mixtures were then calcined and re-milled three times to assure a total reaction. The thermal cycle consisted of a heating rate of 5 °C/min reaching the reaction temperature of 1150 ºC, held for 6 h and then cooled at 1 °C/min. Corresponding amounts were then thoroughly mixed in order to obtain the solid solution $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$. These mixtures were again calcined at 1150 °C for 6 hours, remilled and sintered under oxygen flow, under strict thermal conditions: 5 °C/min during warming, held 2 hours at 1250 °C, and then cooled at 0.5 °C/min. Samples were characterized by X-ray diffraction techniques both before and after the sintering conditions, confirming the presence of a pure perovskite orthorhombic Pbnm structure. Experimental errors in the lattice parameter determination were of the order of ±0.001-0.002 Å, depending on the acquisition time.

Magnetic measurements were performed in a Quantum Design MPMS-XL5 SQUID susceptometer, between 2 K and 300 K and as a function of the applied field, from -50 kOe up to +50 kOe. All measurements were done in specimens cut from ceramic bulks and glued to calibrated gelatin sample holder, thus avoiding any disorientation due to torque forces exerted on the sample. The ordered regime was investigated by performing magnetization cycles as a function of temperature (ZFC/FC cycles) and applied fields (magnetization loops). The magnetization loops were mostly performed at the lowest temperature (2 K), but some complementary measurements were done at higher temperatures for particular compositions.

3. RESULTS AND DISCUSSION

3.1. Lattice parameters

As it is well known, the ferromagnetic interactions in the $REMnO_3$ compounds greatly depend on the angle between the spin carriers orbitals along the Mn^{3+}-O-Mn^{4+} chain (13). In the case of the solid solution presented in this work, the yttrium ion has the same valence and ionic size as erbium, and thus no significant modifications are expected either in the crystal structure and lattice parameters or on the ordering temperature. In this way, this appropriate choice of the substitute allows us to get rid of any extrinsic parameter which could modify the magnetic properties of the overall system. Figure 1 shows the X-ray diffraction patterns (XRD) of several compositions of $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$ with no systematic variations on the whole range [$0.0 \le x \le 1.0$]. The lattice parameters were almost identical for more than 20 samples prepared in different sets, with average values of 5.210 (±0.005), 5.570 (±0.005) and 7.450 (±0.005) Å, for a, b and c, respectively, space group Pbnm.

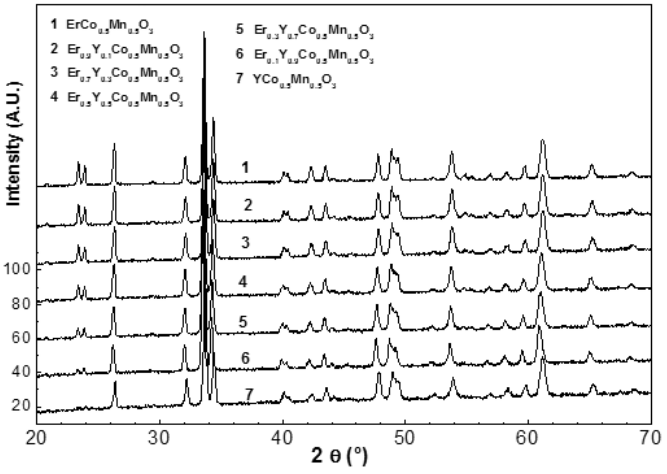

Fig. 1-X-ray diffraction patterns (CuK_α radiation, λ = 1.54186 Å) for the solid solution $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$. Diagramas de difracción de rayos X (radiación CuK_α, λ = 1.54186 Å) de las soluciones sólidas $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$

3.2. ZFC/FC magnetization cycles

Figure 2 shows the ZFC/FC cycles measured under an applied field of 50 Oe, for the solid solution $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$. For non-magnetic yttrium (x = 0), a ferromagnetic behavior is observed, characterized by an almost constant value of the M^{FC} magnetization when cooling the sample below the ordering temperature of 80 K. The corresponding M^{ZFC} magnetization is typical of an antiferromagnetic-like system, where antiferromagnetic inter-plane interactions compete with ferromagnetic in-plane exchange interactions (14,15). The

observed behavior for this particular sample ($YCo_{0.50}Mn_{0.50}O_3$) confirms our previous studies on the $YCo_xMn_{1-x}O_3$ system (11).

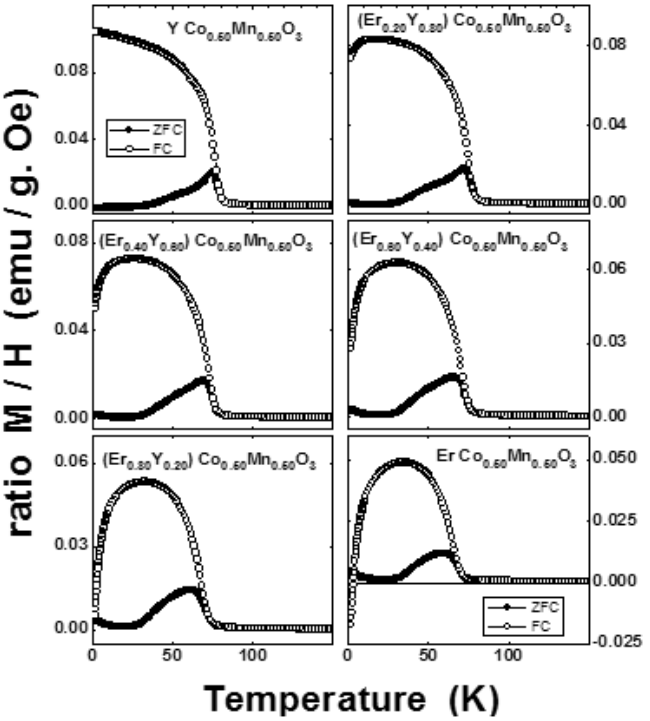

Fig. 2- Magnetization cycles ZFC/FC measured at 50 Oe, for given samples of $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$. Ciclos de magnetización ZFC/FC, medidos a 50 Oe para muestras $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$

The ZFC behavior stays almost unchanged in the rest of the series in spite of the large difference in their magnetic moments, with canted-type antiferromagnetism and similar ordering temperatures, confirming that the perovskite structure is not modified since the rare-earth elements have similar ionic radii (13). However, strong modifications are observed when cooling the samples under the applied field: a slight decrease of M^{FC} appears at low temperature, getting stronger when increasing x(Er) (figure 3), and eventually becoming negative when x(Er) equals or is higher than 80 at.%. At the far-end of the solid solution (x = 1 ; $ErCo_{0.50}Mn_{0.50}O_3$), the low temperature magnetization reaches a negative sign, as in ferrimagnetic systems. This original behavior is due to the simultaneous presence of three important conditions : firstly, the rare-earth cation (erbium, in this case, but also gadolinium in other cases (7,16)) has a strong magnetic moment (μ_{eff} = 9.58 μ_B), much higher than the moment of $|Co_{0.50}Mn_{0.50}|$; secondly, the erbium moments behave as independent non-correlated spins, with a Curie-Weiss temperature dependence of their magnetic contribution ($M \sim T^{-1}$) ; and finally, a negative exchange interaction between the erbium and the transition-metal sublattices occurs when the erbium spins are subjected to the internal field imposed by the |Co,Mn| network. Then, the resulting magnetization will be the superposition of both sublattices, their relative weights depending on their thermal behaviors: one which is almost constant at $T < T_c$, the other varying as T^{-1}, over the whole range of temperature. When the contributions of both sublattices are identical (but pointing in opposite directions) then, they are fully compensated (crossing the Y = 0 axis at T_{comp}), becoming negative if the rare-earth moment is sufficiently high compared to the transition-metal moments (10). A full description of this process in terms of a mean-field model of interacting sublattices can be found in ref. (8) for the $Er(Me,Mn)O_3$ (Me = Ni, Co) solid solution.

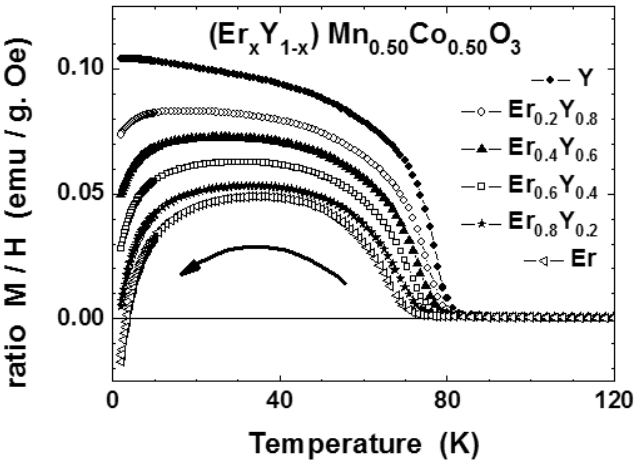

Fig. 3- Field-cooled magnetization measured at 50 Oe, for the solid solution $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$ (x = 0, 0.2, 0.4, 0.6, 0.8 and 1.0). Magnetización FC medida a 50 Oe para las soluciones sólidas $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$ (x = 0, 0.2, 0.4, 0.6, 0.8 y 1.0).

3.3. Magnetization loops

Magnetization loops were performed at 2 K, for all samples of the $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$ solid solution. Figure 4 shows typical data recorded between -50 kOe and +50 kOe, while figure 5 superposes all data in order to highlight the most relevant features. Again, for non-magnetic yttrium (x = 0), a classical ferromagnetic loop is observed, with a coercive field of 12.5 kOe, in perfect agreement with our old reports on the $YCo_xMn_{1-x}O_3$ system (11). At the other end of the solid solution, $ErCo_{0.50}Mn_{0.50}O_3$ presents interesting and original features, characterized by two well-defined anomalies, fully symmetrical with respect to the orientation of the applied external field : the first anomaly occurs at low fields where the decreasing and increasing branches of the magnetization clearly intersect ; the second one occurs at high fields (at approximately 35 kOe), and corresponds to a sudden increase of the magnetization when the applied magnetic field increases. Both anomalies are also seen, although less pronounced, in some other compositions, all of them situated close to the 0.50/0.50 = Mn/Co substitution rate (8). By analogy to the $YCo_{0.50}Mn_{0.50}O_3$ composition, the sudden jump at high fields can be related to the reorientation of the ferromagnetic domains, which are probably harder to rotate since the critical field is more than twice the value observed for the simple ferromagnetic loop represented by the $YCo_{0.50}Mn_{0.50}O_3$ sample. Figure 5, indeed, shows the progressive (and linear) evolution of the critical field, with the Er/Y ratio.

Concerning the low field anomaly, it must be definitely associated with the spin reversal of the FC magnetization shown in figs. 2 and 3. At temperatures slightly above the compensation temperature T_{comp} (where M = 0), the increasing and decreasing branches do not intersect, although they approach each other (a full description of this process is

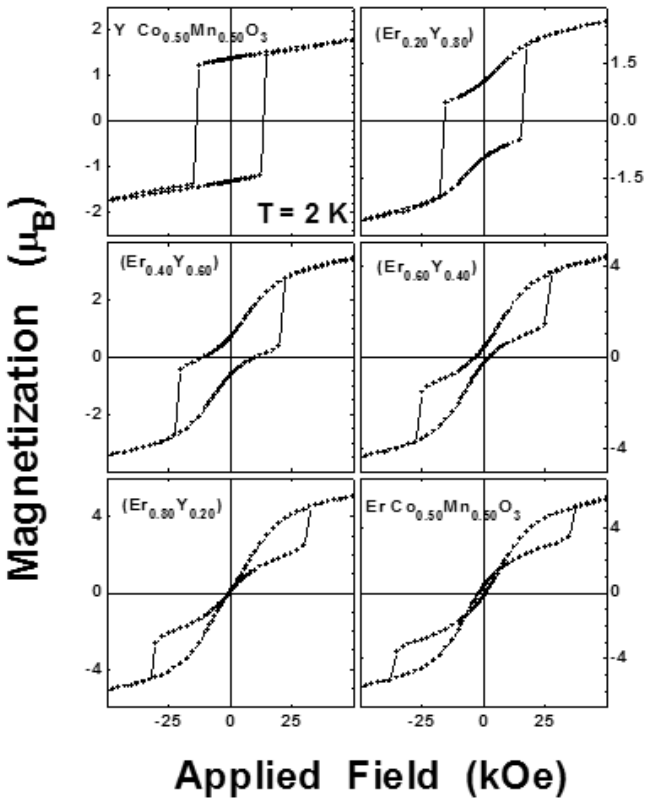

Fig. 4- Magnetization loops as a function of the applied field, measured at T = 2 K, for given samples of $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$. Curvas de magnetización en función del campo aplicado, medidas a T=2 K para algunas muestras de $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$

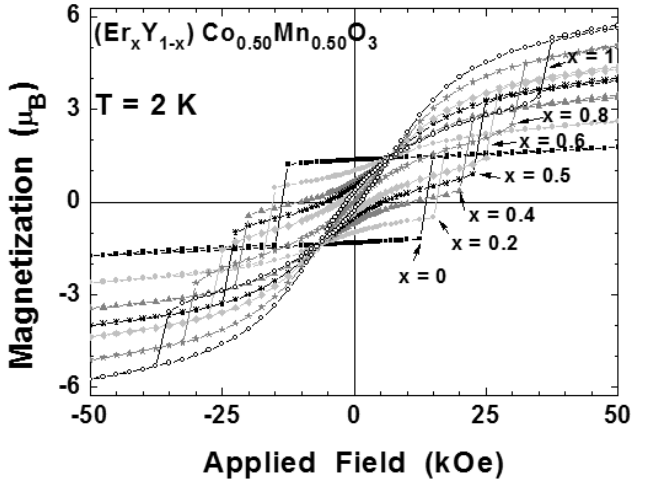

Fig. 5- Magnetization loops as a function of the applied field, for several samples of $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$ (x = 0, 0.2, 0.4, 0.6, 0.8 and 1.0). Curvas de magnetización en función del campo aplicado para algunas muestras de $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$ (x = 0, 0.2, 0.4, 0.6, 0.8 y 1.0).

reported in ref. (17) for $ErCo_{0.50}Mn_{0.50}O_3$). In the present solid solution, such behavior is clearly evident, as shown in figure 6 for a few examples.

It is interesting to notice that the high-field transition also depends on temperature. Figure 6 shows, for instance, that the sudden jump which occurs at about 30 kOe for composition $Er_{0.80}Y_{0.20}Co_{0.50}Mn_{0.50}O_3$ at T = 2 K, becomes just an inflexion point at T = 3 and 4 K. Similarly, for $Er_{0.50}Y_{0.50}Co_{0.50}Mn_{0.50}O_3$, the jump still occurs at 2, 3 and 3.5 K, but it smoothes out at T = 4 K. Finally, as it is shown in figure 6, left-hand panels, the high-field transition for $Er_{0.20}Y_{0.80}Co_{0.50}Mn_{0.50}O_3$ still occurs at 4 K, but it is smeared out at 6 K. This fact should be related with the fact that ferromagnetic domains are easier to rotate for yttrium-rich samples, since reorientation occurs at higher temperatures than those necessary for the erbium-rich samples, as we pointed out above with respect to the progressive decrease of H_{crit} with increasing yttrium content (fig. 5).

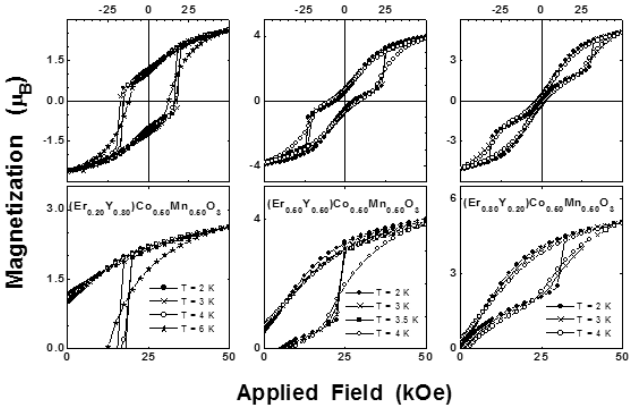

Fig. 6- Magnetization loops at given temperatures for three $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$ compositions. Notice that the abrupt jump (lower panels) occurs at lower temperatures with increasing erbium concentration (below 6 K, below 4 K, below 3 K, for x(Er) = 0.2, 0.5 and 0.8, respectively). Curvas de magnetización a ciertas temperaturas de tres composiciones $Er_xY_{1-x}Co_{0.5}Mn_{0.5}O_3$. Hay que señalar que el salto brusco, (recuadros bajos) sucede a menores temperaturas cuando aumenta la concentración de Er (inferior a 6 K, inferior a 4 K, inferior a 3 K, para x(Er) = 0.2, 0.5 y 0.8, respectivamente).

We should remark at this point that, as we have already reported for $ErCo_{0.50}Mn_{0.50}O_3$ (18), the high-field transition has a dynamical character. This means that, when the applied field varies rapidly, the sudden jump is well defined and the magnetization increases suddenly by 65 % of its actual value (fig. 4). On the other hand, if the applied field varies much slowly (for instance, by taking more frequent data points), the transition is smoothed out (18). Such feature was also pointed out by other authors in perovskites-related manganites and intermetallic compounds, and attributed to a competition between the magnetic energy necessary to promote the ferromagnetic phase and the elastic energy associated with the strains at the interfaces (19). It could also be a manifestation of phase separation or spinodal decomposition (20). Anyhow, all data presented in figs. 4-6 have been taken in strictly similar conditions, and any variation on the position of the high-field transition or in the abruptness of such a jump, is not due to any difference of the field sweep-rate from one sample to another. These phenomena are intrinsic to the materials and depend just on the relative ratio between Er and Y. Further work is already planned in order to have a better control of the magnetic-field sweep-rate.

4. CONCLUSION

Our previous studies on the $Er(Co,Mn)O_3$ and $Y(Co,Mn)O_3$ solid solutions have been enlarged in this report to the inter-system $(Er,Y)Co_{0.50}Mn_{0.50}O_3$, in which Mn has been substituted by an equal amount of cobalt. The subtle interplay which exists between the different interaction mechanisms lead to unusual phenomena as for instance, a step-like transition at high fields due to a reorientation of magnetic domains, or an intersection of the increasing and decreasing branches. Our results showed that the $Er_xY_{1-x}Co_{0.50}Mn_{0.50}O_3$ solid solution goes from ferromagnetism (x=0; $YCo_{0.50}Mn_{0.50}O_3$) to ferrimagnetism (x=1; $ErCo_{0.50}Mn_{0.50}O_3$). The critical field H_{crit}, related to the reorientation of domains, varies linearly with x(Er). The compensation temperature T_{comp} increases and the spontaneous magnetization M_{rem} decreases with increasing content of erbium. All these observations indicate that the antiferromagnetic interactions get stronger while the Co/Mn ferromagnetic domains are harder to rotate when the rare-earth sublattice is progressively filled with erbium spins of large magnetic moments.

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