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Magnetic domains and field-induced transitions in nanosized $ErCo_{0.5}Mn_{0.5}O_3$

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Abstract. In $Er(Co,Mn)O_3$ an avalanche mechanism occurs under the application of moderate magnetic fields, leading to the rotation of the ferromagnetic domains in one or more steps, depending on the rate of variation of the applied field. In order to deeply investigate the influence of the microstructure, we have elaborated nanosized $ErMn_{0.5}Co_{0.5}O_3$ compound by a citrate method, prepared at 700 °C and further calcined at increasing temperatures which assure the formation of very reactive grains of small size (~25 nm) and facilitate their full oxidation. Grains progressively grow from 20-30 nm up to several hundred nm, reaching good percolation at 950 °C (100 nm average size) for which a sudden jump is observed in the magnetization cycles M(H). We have simulated domains rotation by freezing a gel-type solution prepared from nanosized grains, orienting the individual spins on the paramagnetic state and subsequently freezing the gel to form single ferromagnetic domains.

1. Introduction

The research on cooperative phenomena constitutes an important subject because of many applications at the frontier of chemistry, physics and electronics [1-3]. Rare-earth manganites $REMnO_3$ of perovskite structure have received growing attention because of a mixed-valent state Mn^{+3}/Mn^{+4} and the resulting ferromagnetic exchange, at the basis of the magnetoresistance effect [4]. Particularly interesting is the case when the RE cation bears a large magnetic moment since exchange interactions between magnetic sublattices (RE and Mn) may take place [5,6]. In this context, we have previously shown interesting phenomena occurring in $Er(Co,Mn)O_3$ due to an antiferromagnetic interaction between a paramagnetic Er sublattice and the ferromagnetic |Co,Mn| network, producing a reversal of the magnetic moment and the formation of ferromagnetic domains [7]. An avalanche mechanism occurs under the application of moderate (~3-4 tesla) magnetic fields, leading to the rotation of ferromagnetic domains in one or more steps, depending on the rate of variation of the applied field

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[8,9]. In order to deeply investigate the influence of the microstructure, we have elaborated nanosized materials by chemical methods, which assure the formation of very reactive grains of small size (~25 nm) and facilitate their full oxidation.

2. Experimental

$ErMn_{1-x}Co_xO_3$ perovskites were prepared by the citrate method [10]. Stoichiometric amounts of an aqueous solution of the corresponding nitrates were added to an aqueous solution of citric acid with a 10 % excess over the number of ionic equivalents of cations. The resulting solution was stirred at room temperature and slowly evaporated at 70 °C under vacuum in a rotary evaporator until gel formation is reached. This gel was dried in an oven, slowly increasing the temperature up to 250 °C and maintained overnight to yield a solid amorphous citrate precursor. The resulting powder was crushed and sieved to obtain the required particle size (< 200 μm) and calcined at 700 °C in air for 6 h.

Samples were characterized by X-ray diffraction (XRD) both before and after the sintering conditions, confirming the presence of a pure perovskite orthorhombic *Pbnm* structure. Phase purity was checked by energy dispersive analysis (EDX). The microstructure was characterized by using scanning electron microscopy (SEM). Magnetic properties were studied as a function of temperature (ZFC/FC) and field (M-loops), using a Quantum Design MPMS-XL5 SQUID susceptometer.

3. Results and discussion

The as-prepared sample, synthesized at 700 °C, presents a rather amorphous character (Figure 1, bottom), a sponge-like macrostructure (not shown) and some regions composed of nanosized particles, as seen in the upper panel, Figure 2. This powder was then pelletized in as many batches as needed, and each one of them sintered separately at given temperatures. Figure 1 confirms the phase stability and the increasing crystallinity as the sintering temperature increases.

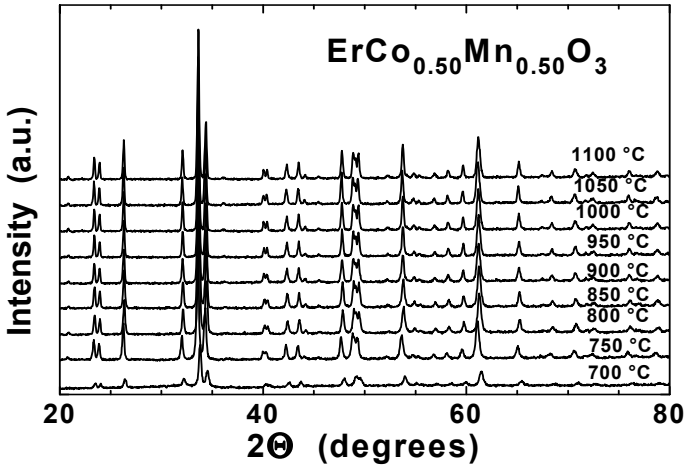


Figure 1. XRD data at increasing calcination temperatures.

Figure 2 shows SEM micrographs for selected samples. The surface is perfectly homogeneous and consists of spherical grains of 20-30 nm diameter, which progressively grow with increasing sintering temperature, attaining 100-200 nm and a very good percolation at 950-1000 °C, that is, when grains fuse together and grain barriers almost disappear (see qualitative changes occurring at 950-1000 °C with respect to micrographs taken at 800-850 °C).

Magnetic measurements were performed on the starting material and sintered pellets. Temperature ZFC/FC cycles (not shown) confirm the reversal phenomenon reported in bulk specimens, which results from the antiferromagnetic (AF) interaction between the Er and Mn/Co sublattices [7]. Such interaction also triggers a rather unusual phenomenon at low field and low temperature, in which the increasing and decreasing branches of the magnetization loop intersect at about 7 kOe. This is partly seen in figure 3, which shows the positive portion of the M(H)-loops for four sintered pellets. In

addition to the AF exchange between magnetic sublattices, a second anomaly occurs at higher fields, characterized by a sudden jump of the magnetization. We have shown in previous reports that this jump obeys to relaxation mechanisms related to a reorientation of magnetic domains [11].

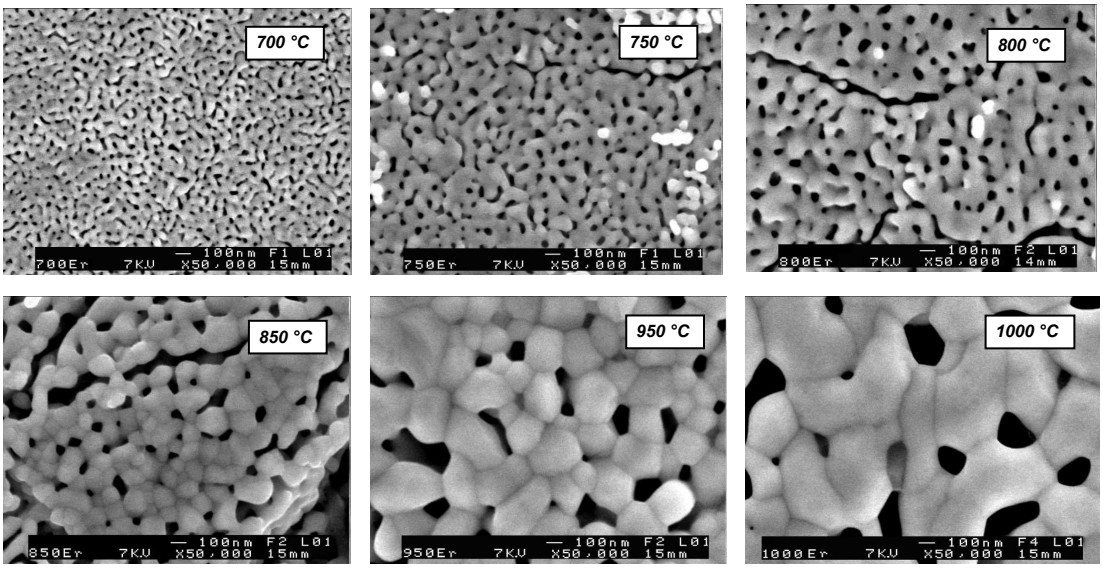


Figure 2. Micrographs at given calcination temperatures (in °C).

To further characterize this phenomenon, we have correlated the microstructure and the qualitative change in the grains distribution (percolation threshold) to the magnetization jump. Figure 3 zooms part of the M(H)-loops for some selected pellets. Below an annealing temperature of about 950 °C, the magnetization increases smoothly with increasing field, until an inflexion point occurs at about 40 kOe. This inflexion point transforms into a sudden jump at H_c, for sintering temperatures of 950 °C and above, that is, when grain barriers tend to disappear. We correlate this sudden jump with the reorientation of ferromagnetic domains. At the same time, the critical field H_c decreases with increasing annealing temperature suggesting that ferromagnetic domains rotate more easily inside a large homogeneous grain since no barriers are present.

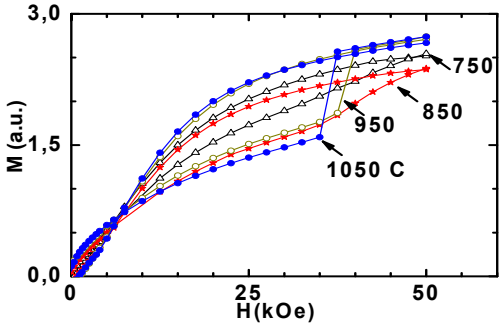


Figure 3. Positive part of magnetization loops at T = 2 K for given calcination temperatures in °C.

To confirm the reorientation phenomenon, we have performed subsequent runs at 2 K, measuring the full M(H) loop, as shown in Figure 4. The starting bulk corresponds to a 22-mg piece of the pellet sintered at 950 °C. This piece was then crushed into fine powder (sieved at 80 μm) and let free to rotate under the action of the applied field. In a third run, this same powder was homogeneously dispersed inside a drop of liquefied vacuum grease, then frozen at 2 K in order to block any rotation of the fine particles, and performed a M(H) loop. Finally, the gel-type solution was heated at 350 K inside the cryostat, and the maximum available field (50 kOe) was applied. The gel was then cooled under the same static field down to 2 K, with all domains oriented parallel to the applied field. Figure

4 shows the full M(H) loop under these 4 different experimental set-ups. It can immediately be noticed that the most significant change in the magnetization loop concerns the height of the jump, which increases by about 60 % in the oriented powder with respect to the bulk ceramics. It is also evident that, when the particles are homogeneously dispersed and not allowed to rotate (run n° 3), the magnetization jump is smoothed out and becomes just an inflexion point.

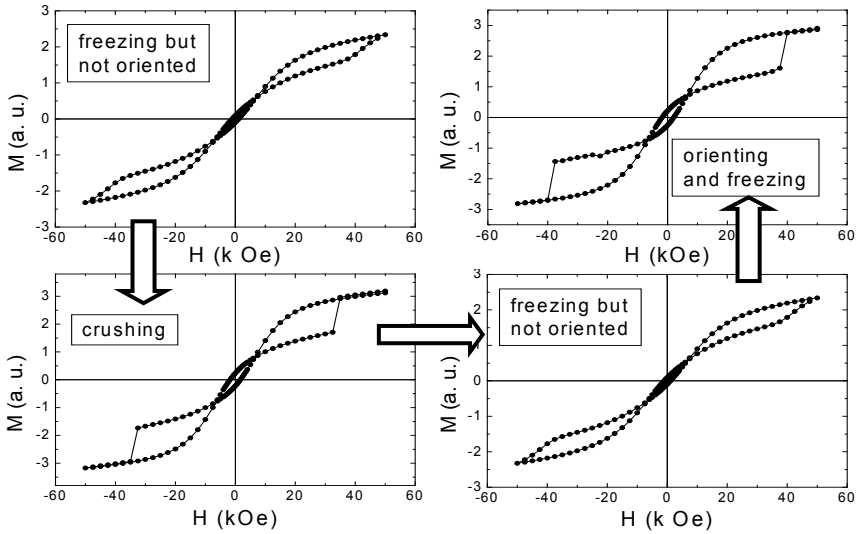


Figure 4. M(H)-loops measured under 4 different conditions (see text) for sample sintered at 950 °C

4. Conclusions

We have shown the intimate correlation between the microstructure and the rotation of the ferromagnetic domains in the ferrimagnetic compound $ErMn_{0.5}Co_{0.5}O_3$. Thanks to the experimental simulation using a gel-type nanomaterial, we have unambiguously shown that grains size and percolation are the most important mechanisms in the rotation of magnetic domains in this material. Next step will be to perform *in-situ* sintering at 950 °C under an applied external field.

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