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Dynamic magnetization on the low temperature magnetoelectric effect in multiferroic composites

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Abstract

The dependence of frequency and applied magnetic field in the magnetoelectric effect (ME) of the composites $0.68\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})-0.32\text{PbTiO}_3/\text{CoFe}_2\text{O}_4$ (PMN-PT/CFO) and $0.68\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})-0.32\text{PbTiO}_3/\text{NiFe}_2\text{O}_4$ (PMN-PT/NFO) are investigated. The results for PMN-PT/CFO composite, show a hysteretic behavior for the ME coefficient, at low temperatures (5 K), for frequencies higher than 1000 Hz. Contrasting with these results, the ME coefficient for the PMN-PT/NFO shows a well-known peak-peak related with the magnetostriction coefficient. Based on energy levels of stabilization for each ferromagnetic phase, it was possible to explain the ME hysteretic distinct behavior of PMN-PT/CFO, because of the degeneracy in the energy levels, due to the spin-orbit coupling causing changes in the dynamic properties of the magnetoelastic interactions.

Keywords: magnetoelectric, multiferroic composite, magnetostriction, dynamic magnetization

(Some figures may appear in colour only in the online journal)

1. Introduction

New developments in multifunctional or smart materials are fundamental for technologies applied to storage, processing information and energy harvesting [1]. Among the class of smart materials, in the last decade, multiferroic composites in which combine ferroelectric and ferromagnetic phases, have shown an increasing interest due to the development of new routes for synthesis, low power consumption and high sensitivity devices, and a larger number of materials that can be used in different temperatures and environments. The emergent magnetoelectric (ME) effects in these composites are induced by an indirect coupling (via strain) between ferroelectric and ferromagnetic and/or ferrimagnetic phase materials [2], enabling each phase to be independently optimized for best performance even at room temperature. In this way, the cross interaction depends simultaneously on the piezoelectric

and on the magnetostrictive effects [2–4]. Some examples of composite materials are PMN-PT/ CoFe_2O_4 , PZT/ CoFe_2O_4 and PZT/ CoFe_2O_4 [5, 6]. The most important characteristic of the direct ME effect is a peak in ME response as a function of a DC magnetic field. This peak is associated with the magnetostrictive behavior of the ferromagnetic and/or ferrimagnetic phase [3]. The literature reports $\text{BaTiO}_3/\text{CoFe}_2\text{O}_4$ (BTO/CFO) samples having the maximum value of ME coefficient of $130 \text{ mV cm}^{-1} \cdot \text{Oe}$ at 5 kOe [7], while for PZT/ CoFe_2O_4 thin film, the maximum value was $287 \text{ mV cm}^{-1} \cdot \text{Oe}$ at 1.5 kOe [5, 8]. Other compounds reached higher values for the ME response near the magnetoelastic resonance, at frequencies of 100 kHz [9, 10]. However, for some applications, lower operational frequencies are required and this can be done using cantilevers [11, 12], heterostructures with different peaks in ME response [13] and multilayers made of different materials [14, 15]. Despite the effort to improve the

Table 1. Lattice parameter, relative density and electric resistivity values for PMN-PT, CFO and NFO phases, PMNPT/CFO and PMN-PT/NFO particulate composites.

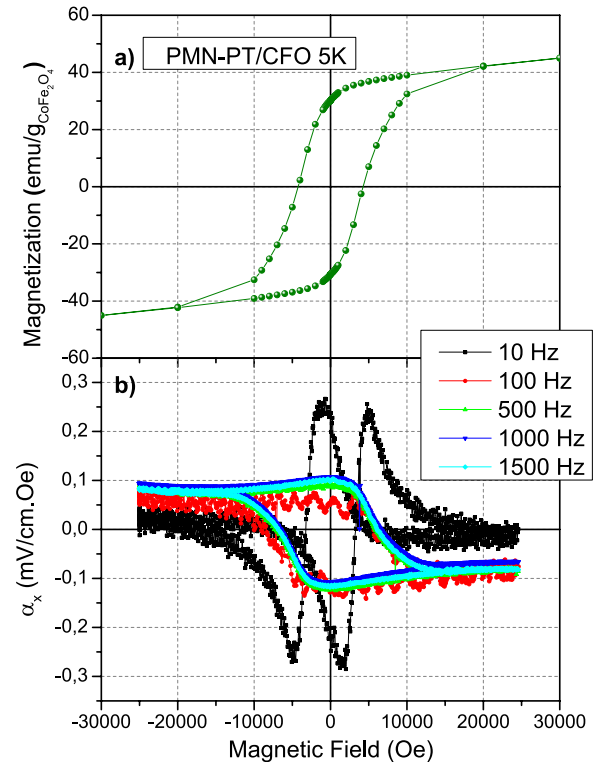
Sample	<i>c/a</i> ratio	Lattice parameter (Å)	Relative density (%)	Electric resistivity ($\Omega \text{ cm}^{-1}$)
PMN-PT	1.005 ± 0.002	—	97.3 ± 0.2	$(7 \pm 2) \times 10^{12}$
CFO	—	8.40 ± 0.03	96.7 ± 0.2	$(8 \pm 3) \times 10^6$
NFO	—	8.35 ± 0.02	95.4 ± 0.4	$(2.2 \pm 0.3) \times 10^6$
PMN-PT/CFO	1.006 ± 0.002	8.40 ± 0.02	97.8 ± 0.1	$(6 \pm 1) \times 10^9$
PMN-PT/NFO	1.003 ± 0.002	8.37 ± 0.04	97.1 ± 0.2	$(1.7 \pm 0.2) \times 10^9$

ME response, the dynamic effects of the AC magnetic field frequency are not fully understood. In this sense, our previous works reported that the ME voltage coefficient for composites based on CoFe_2O_4 shows a dependence on the frequency of the magnetic field, even for low frequency regime ($< 10 \text{ kHz}$). This dynamic response is particularly intense at low temperatures, where the ME effect changes significantly [16], due to the crystalline trigonal field of the ferrite structure, modifying the magnetoelastic contribution, inducing a step-like (pseudo-intrinsic) behavior of the ME coefficient of PMN-PT/CFO [16]. Looking for a better understanding of the ME dynamic response and the role played by the crystalline field of ferrite trigonal structure in PMN-PT composites, we have investigated the frequency dependence of the ME effect of the composites $0.68\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{-}0.32\text{PbTiO}_3/\text{CoFe}_2\text{O}_4$ and $0.68\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{-}0.32\text{PbTiO}_3/\text{NiFe}_2\text{O}_4$ at low temperature (5 K). As shown in the results, a step-like (pseudo-intrinsic) behavior appears in the PMN-PT/CFO, for frequencies higher than 100 Hz, while the same effect is not observed in the PMN-PT/NFO. We have associated this distinct behavior with the t_{2g} energy level stabilization of the ferromagnetic phases.

2. Experimental details

The multiferroic 0–3 composite of $0.68\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{-}0.32\text{PbTiO}_3$ (PMN-PT) with CoFe_2O_4 (CFO) or NiFe_2O_4 (NFO) ferrite were prepared by the conventional solid state reaction method. The specimens were densified in oxygen by uniaxial hot pressing, under maximal pressure of 12 MPa using a hot uniaxial press (HP22-0614-SC; Thermal Inc) at 1050 C, for 0.5 h. Table 1 presents some structural and electric parameter of used samples. The invariance of the *c/a* ratio of PMN-PT matrix and lattice parameter of the ferromagnetic phases in composites suggest that chemical reaction has not significantly occurred between the ferroelectric and ferrite phases during the hot pressing process, at the measurement resolution. The analyses of the apparent density of composites confirm that both composites exhibit high density (values reached 95%). High electrical resistivity ($\sim 10^9 \Omega \text{ cm}^{-1}$), was achieved for all cases, which can be explained based on the additive properties between the electrical properties of PMN-PT electrical resistivity of $\sim 10^{12} \Omega \text{ cm}^{-1}$ and the ferromagnetic phases (electrical resistivity is $\sim 10^6 \Omega \text{ cm}^{-1}$). It means that the adopted processing protocol was suitable to obtain composites with good microstructure and electrical properties, independently of the magnetic phase of the compounds.

Further details of structural, dielectric, magnetic and magnetolectric properties at room temperature of these samples

**Figure 1.** (a) Magnetization and (b) frequency dependence of the magnetolectric response for composite PMN-PT/CFO at 5 K.

can be found in [17]. These composites were electrically poled in the presence of an electric field with an intensity of 25 kV cm^{-1} , for 30 min, at room temperature. The magnetization measurements were carried out using the Physical Properties Measurement System (PPMS; Quantum Design) extraction magnetometer. The low temperature magnetolectric voltage (α) measurements were performed using a homemade customized PPMS, up to 1500 Hz and AC magnetic field of 1 Oe. The measurements were carried out in the sample out-plane direction and in the 3–3 configuration (magnetic field in the same direction of the electric polarization). The dynamic magnetization measurements were performed using the MPMS3 magnetometer by quantum design, where the magnetic field was changed at different rates.

3. Results and discussion

Figures 1 and 2 show the magnetization and ME (α) coefficient of the composites PMN-PT/CFO and PMN-PT/NFO at 5 K. It was not possible to reach the saturation magnetization of these samples. In this way, the analysis of the magnetic

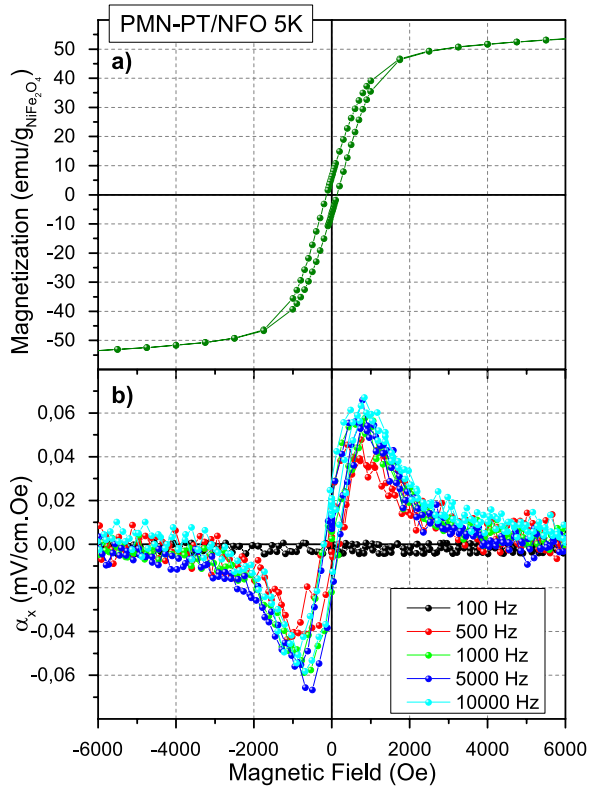


Figure 2. (a) Magnetization and (b) frequency dependence of the magnetolectric response for composite PMN-PT/NFO at 5 K.

properties was done using the data for magnetic field up to 30 kOe. The highest magnetization reached near saturation and the remanent magnetization, normalized by mass of CFO and NFO, respectively, and the coercive field are shown in table 2.

The dc magnetization measurements (figures 1(a) and 2(a)) show higher saturation for PMN-PT/NFO with a much lower coercive field $H_C = 78$ Oe, when compared with $H_C = 4.4$ kOe for PMN-PT/CFO. These differences are likely due to the magnetic anisotropy constant features [18, 19]. The ME voltage coefficients (α), as a function of applied magnetic field for different frequencies at 5 K, for the PMN-PT/CFO are shown in figure 1(b). For low frequency, at 10 Hz, the ME voltage coefficient exhibit a maximum value at 5 kOe and vanishes for high magnetic fields. As the frequency is increased, there is a change in the curve trend, that becomes hysteric, presenting a step-like appearance. However, for frequencies above 100 Hz, the ME voltage coefficient does not present significant frequency dependence. The α coefficient for PMN-PT/NFO (figure 2(b)) increases with frequency. This increasing in the ME response can be associated to the electric permittivity of the sample in which decreases with the frequency [17] since the ME voltage coefficient is inversely proportional to the electrical permittivity $\alpha_x = \alpha_p/\epsilon$ [3] where α_p is the of electric polarization with respect of magnetic field. Below 100 Hz the response is very weak and within the measurement sensibility, contrasting with PMN-PT/CFO results (figure 1(b)). For PMN-PT/NFO composite α coefficient presents a maximum near 1 kOe and α vanishes for magnetic field higher than 5 kOe.

Table 2. Highest reached magnetization (M_h), remanent magnetization (M_R) and coercive field (H_C) at 5 K for composites PMN-PT/CFO and PMN-PT/NFO.

Composite	M_h (emu g ⁻¹)	M_R (emu g ⁻¹)	H_C (Oe)
PMN-PT/CFO	48.5 (30 kOe)	34.0	4400
PMN-PT/NFO	52.2 (6 kOe)	3.9	78

It is known that ferrites have a spinel structure with formula AB_2O_4 , where the B is the iron ion and A is another magnetic ion [20]. The two magnetic ions (A in the octahedral site and B in the tetrahedral site) have different orientation creating two ferrimagnetic sublattices. In particular, nickel ferrite $Fe^{3+}(Ni^{2+}Fe^{3+})O_4$ is an inverted spinel with the ions Ni^{2+} and Fe^{3+} sharing the octahedral B sublattice. However, a small amount (<5%) of the Ni^{2+} ion can be found in the tetrahedral A site [20]. Furthermore, the Ni^{2+} (d8) ion has a singlet ground state in the octahedral site ($L = 0$) due to a cubic field and cannot be stabilized by a spontaneous distortion. On the other hand, for the ion in the tetrahedral site, the $L = \pm 1$ doublet stabilizes the structure due to a spin-orbit (SO) compression. As a consequence of this stabilization, the magnetoelasticity, the magnetic anisotropy and the spin/lattice relaxation effects of the $Fe^{3+}(Ni^{2+}Fe^{3+})O_4$ can be associated with the small amount of Ni^{2+} ion in the tetrahedral A sites [20]. Cobalt ferrite $Fe^{3+}(Co^{2+}Fe^{3+})O_4$ is also a ferrimagnetic material with spinel inverse structure, where the ion Co^{2+} is in the octahedral B site and the ion Fe^{3+} is both in A and B sites [20]. In the cobalt ferrite, a small amount of Co^{2+} ion can be found on the tetrahedral site, as the same as that observed in the nickel ferrite. However, for cobalt ferrites, the T_{2g} is stabilized by SO contraction for the Co^{2+} in the octahedral site [20, 21]. This splitting in the ground state of the cobalt ferrite can be associated with the trigonal field (direction [1 1 1]) [22–24]. Due to the difference in the electronic stabilization, the effects of cation distribution are stronger in Co-ferrite than in Ni-ferrite [25]. In terms of magnetoelastic response, the cation distribution for Co^{2+} leads to a stronger SO effect when compared with the Ni^{2+} . Furthermore, an important result suggested by Dionne [26] shows that the small fraction of the ion in the tetrahedral sublattice provides faster relaxation rates (consistent with $MgFe_2O_4$ and $CuFe_2O_4$ ferrites). Therefore, the dynamic effects of Ni-ferrites should occur at higher frequencies, as compared with Co-ferrites, since the magnetoelastic effects in Ni-ferrites are due to the ions in the tetrahedral site. Thus, the magnetostrictive effect of cobalt ferrites is more sensitive to low frequencies AC magnetic field or to temperature changes than nickel ferrites.

The formal origin of magnetostriction lies in the fact that the anisotropy energy varies with strain and if the strain is such as to decrease the anisotropy energy, then the lattice will strain spontaneously [20]. The distortion may occur spontaneously at the cation site because of the orbit/lattice or the spin/orbit/lattice interactions, in which depends on the electronic configuration of the transition ion [20]. As discussed before, the ME is the coupling between the piezoelectric and magnetostrictive materials and the magnetic behavior of the ME is due only to the magnetostrictive material. Furthermore, the ME effect is

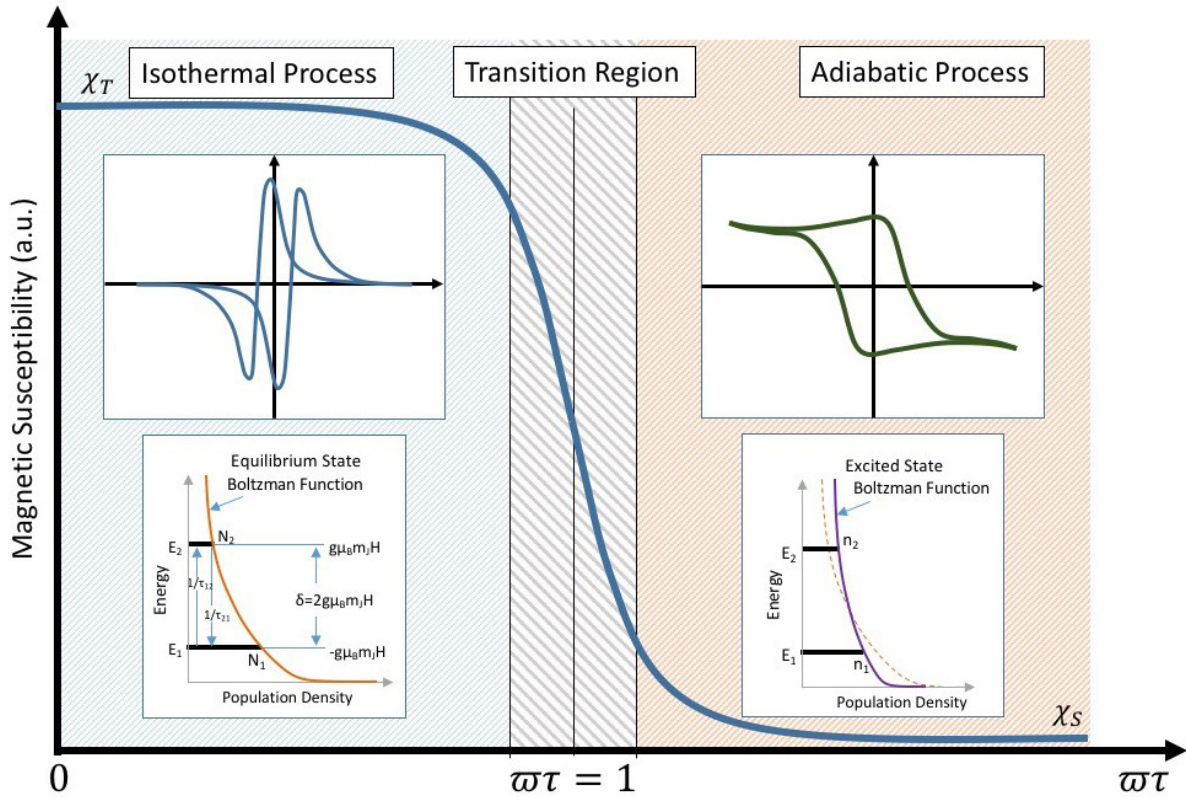


Figure 3. Schematic representation of frequency effects of multiferroic composites. In the isothermal process ($\omega\tau < 1$) there is an equilibrium state between the energy levels. In this case, the rate variation in the population for the E_1 level is the same for the E_2 level and the total population in each level is constant. In the adiabatic process ($\omega\tau > 1$), the E_2 population rate variation is different than the E_1 , causing an excited state. The magnetoelastic effect will be different since the E_2 level has a null total angular momentum (SO coupling is null).

a dynamic property and depends on an applied AC magnetic field. Therefore, to understand the ME effect it is necessary to understand the dynamic response of the magnetostriction of the magnetic phase. To understand the dynamic properties of magnetic systems, we assume that the switching rate of magnetic moments between equilibrium states is limited by the relaxation time τ . It is known that the orbital angular momentum is the key of the coupling between the spin and lattice systems [20]. In this way, the spin/orbit stabilization of the $l_z = \pm 1$ doublet, as in the octahedral site in Co-ferrite, would be a prerequisite for anisotropy and spin/lattice relaxation rate, contributing to magnetostrictive extension or compression, depending on the sign of the stabilization [20]. On the other hand, the stabilizations of the $l_z = 0$ singlet by Jahn/Teller distortions would be expected to contribute to magnetostriction once they become cooperative, with anisotropy and relaxation effects appearing as lower order phenomena.

In previous work [16] we have shown that PMN-PT/CFO composite presents strong dependence on frequency of the AC magnetic field. In this model, to analyse the magnetostriction using the magnetization data, it was subtract from the total magnetization (measured magnetization) the contribution of electrons with null angular momentum, once only electrons involved in spin-lattice coupling can contribute to the magnetostriction. For this reason, the dynamic magnetostriction, and consequently the ME, can be related to the frequency (ω) of the magnetic field, the AC magnetic susceptibility (isothermal χT and in-phase AC χ' components) and the spin/lattice

relaxation time τ . In this way, for low values of $\omega\tau$, the magnetostriction is proportional to the isothermal susceptibility and a peak-peak behavior on the α coefficient is observed. On the other hand, for high values of $\omega\tau$, the magnetostriction is proportional to the in-phase AC susceptibility and a step-like behavior on the α coefficient is observed. For intermediate values of $\omega\tau$, a mixed behavior is observed. Figure 3 shows a schematic for the α behavior as a function of $\omega\tau$. As discussed before, the SO stabilization may induce a splitting in the ground state energy level. In a presence of an AC magnetic field, the population density at each T_{2g} doublet (E_1 and E_2) depends on temperature and frequency and can be described by a Boltzmann function (figure 3). In an equilibrium state (low frequency), the amount of decreasing population in level E_1 is the same as the decreasing population in the excited E_2 level. At this condition, χ' tends to the isothermal χT magnetic susceptibility (DC magnetic susceptibility) and peaks appear in the ME coefficient. On the other hand, increasing the frequency of the AC magnetic field, the population in E_1 decreases faster than that in E_2 , causing a relative increase in the population density of E_2 . Since the E_2 is not coupled with the lattice ($L = 0$), the process is considered adiabatic. At this condition, χ' tends to the adiabatic susceptibility regime and peaks in the ME coefficient disappears, giving rise to the step-like behavior. Using the Landau-Zener adiabatic approach [22–24], it is possible to identify the adiabatic state, comparing the dynamic magnetization measurement with the equilibrium measurement. For the adiabatic condition, the

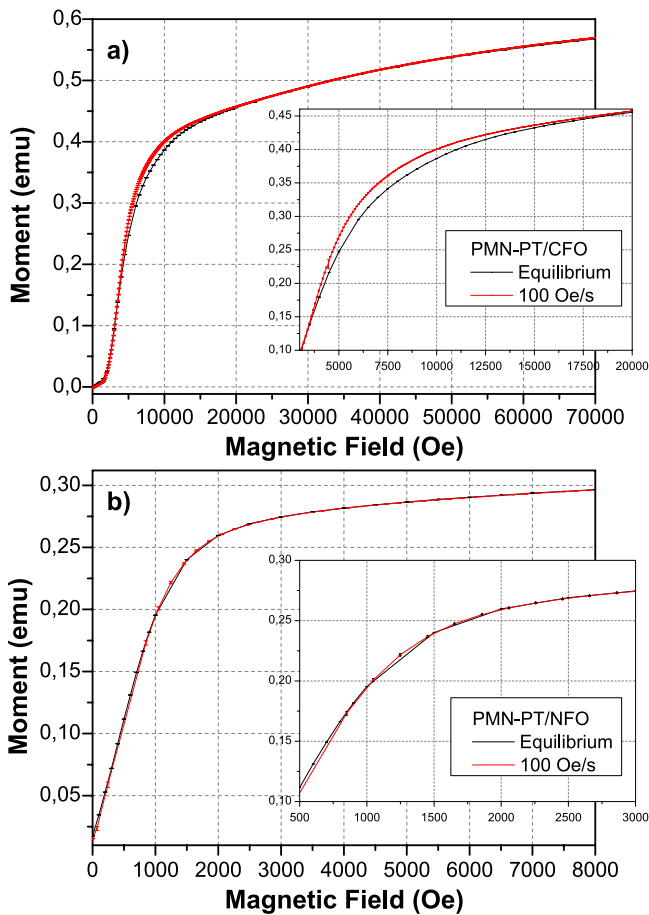


Figure 4. Dynamic magnetization and equilibrium magnetization of the composite (a) PMN-PT/CFO and (b) PMN-PT/NFO at 5 K.

dynamic magnetization must be higher than the equilibrium magnetization.

Figure 4 shows the comparison between the dynamic magnetization and the equilibrium magnetization at 5 K. These measurements were carried out using a magnetometer MPMS3-VSM by Quantum Design. Assuming that equilibrium was reached 100 s after the magnetic field stabilizes, data points were collected. This procedure was chosen based on the slow variation of magnetization compared with the changes during the initial 100 s. The dynamic magnetization was obtained with the rate of 100 Oe s⁻¹ field changes. For the PMN-PT/CFO composite, dynamic magnetization from 2500 to 25 000 Oe increases with respect to the equilibrium measurement, indicating an adiabatic process [22–24]. For the PMN-PT/NFO, there is no significant difference between the dynamic and equilibrium measurements indicating that there is no adiabatic process at these conditions [22–24]. The occurrence of an adiabatic process for the PMN-PT/CFO, implies that part of the spin system does not dissipate energy during the magnetization process. Therefore, there is no energy transferred to the ferrimagnetic phonon system. To understand this condition, we have assumed a two level system, where a null orbital momentum has higher energy level than a non-null orbital momentum. In the presence of an AC magnetic field, the electrons that relax to the higher energy level do not contribute to the magnetostriction.

However, this condition is meta-stable and the electrons return to the initial condition after a characteristic spin/lattice relaxation time. Thus, for long enough relaxation process compared with the measurement time, these electrons will not contribute to the in-phase component of the ME induced voltage. Therefore, considering one period of the AC magnetic field as the measurement time, if the frequency is high enough (period smaller than the relaxation time) only part of the spin system can contribute to the ME effect. The absence of time dependence for the ME effect in Ni-ferrite composite allow us to consider that it correspond to a DC ME response.

4. Conclusions

Summarizing, we suggest that for the appearance of the step-like ME effect, two conditions must be satisfied. First, the ferrimagnetic phase must present splitting in the energy level configuration with a null orbital momentum at the higher energy. This condition is necessary since the spin/orbit coupling depends on the orbital momentum value $\mathcal{H} = \lambda \mathbf{L} \cdot \mathbf{S}$. Second, the spin/lattice relaxation time must be long enough, so that the product $\omega\tau > 1$ and the process to be considered adiabatic with respect to the population of the energy levels. Our results give evidence that the dynamic nature of the ME response can be tuned by the choice of the ferrite used to prepare multiferroic composites. The dependence of the dynamic ME response, considering isothermal process ($\omega\tau < 1$) and the adiabatic process ($\omega\tau > 1$), where the measurement frequencies ω can be controlled experimentally and the relaxation time (τ) is a material feature, let one consider these parameters on the process of materials engineering or even for especial interest on understanding the dynamic behavior and its relation with electronic structure.

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