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Magnetoelectric Coupling in PVDF/Nano NaNbO_3 /NanoNi Composites: Influence of Ni Particles Aspect Ratio

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ABSTRACT

In this work, three phase magnetoelectric nanocomposites are successfully prepared by solvent casting from α -PVDF. The extrinsic piezoelectric effect is insured by 20 vol% of NaNbO_3 ferroelectric nanoparticles prepared by the hydrothermal process. Nickel is chosen as the magnetic phase. In order to maintain insulation, electrical percolation is avoided by using nickel contents lower than the percolation threshold. Two different morphologies are employed: spherical nanoparticles (NiNPs) and nanochains (NNCs) with an aspect ratio of 80. NNCs are obtained by a polyol process under a magnetic field. Thin films of nanocomposites are processed by hot pressing. The real part of the dielectric permittivity (ϵ') of composites is governed by Maxwell-Wagner-Sillars polarization at the PVDF/Ni interfaces. An important increase of ϵ' at low frequency is observed for PVDF/ NaNbO_3 /NNCs composites; it favors the magnetoelectric coupling. Piezoelectric properties of composites are dependent upon mechanical modulus so that the most higher d_{33} is obtained for PVDF / NaNbO_3 / NiNPs. Magnetoelectric coupling coefficient (α_{ME}) is determined from magnetoelectric intensity. PVDF / NaNbO_3 / NiNPs composites exhibit enhanced magnetoelectric coupling. The α_{ME} coupling coefficient reaches 4.2V/(mOe). The magnetic properties are significantly improved by the NiNPs

Index Terms — magnetoelectric properties, PVDF / NaNbO_3 / Ni composite, piezoelectric coefficient, dielectric permittivity, magnetization, aspect ratio

1 INTRODUCTION

DURING the last 20 years, many works were carried out on inorganic magnetoelectric (ME) composites [1] that allow to reach strong ME coupling at ambient temperature. Two approaches were developed: laminated structures [2] and dispersed composites [3]. Laminated composites were widely studied because they exhibit the best ME coupling. Dispersed composites [4–6] also exhibit interesting magnetoelectric coupling. Despite important ME response, these inorganic materials exhibit several drawbacks as they are often brittle, and hard to process.

In the opposite, polymeric materials are easily processed and resilient. Accordingly, composites with piezoelectric polymeric matrix were realized and ME polymer-based composites were developed. Several matrices were used: polyurethane [7], but mostly β -PVDF[8] and PVDF-TrFE[9].

Most of these works investigated the influence of the particles nature over the magnetic properties. Thus, several ferrites [10–12] as well as magnetic metals [13] were used. These composites were widely studied and led to applications like magnetic field sensors [14] memories [15] or microwave tunable devices [16]. Despite interesting ME properties, intrinsic piezoelectric PVDF based composites have a huge limitation in temperature since above 90°C, the matrix becomes paraelectric. The other drawback of these two-phase composites is the high electric field needed to get polarization. Alternatively, three-phase composites were also proposed, at first by Nan *et al*[17] by dispersion of terfenol-D and PZT in a α -PVDF matrix. The magnetoelectric coupling was strongly dependent on composition. First, it is mandatory that the composite remains insulating in order to allow its polarization. The introduction of metallic magnetic particles in polymers above the percolation threshold might allow the building of a conductive path responsible for the percolation phenomena. Then, the composite becomes conductive. To avoid

percolation, the content in conductive particles must be lower than the percolation threshold.

This study explores an original way to realize a three-phase ME composite based on α -PVDF. Nickel particles were selected for their good magnetic properties and their ability to reach high aspect ratio with enhanced magnetic properties but with a low fraction in order to keep the electrical conduction as low as possible. Sodium niobate (NaNbO_3) was chosen since it allows us to realize a lead free extrinsic piezoelectric phase with a high Curie temperature. Note that such inorganic piezoelectric ceramics allow mild poling conditions. We present here the route to process composites; then, the influence of the magnetic particles aspect ratio on the ME effect of composites was analysed.

2 EXPERIMENTAL

2.1 MATERIALS

Polyvinylidene fluoride (PVDF) was chosen as a matrix due to its easy processing and good resilience. PVDF in the α phase was provided by Arkema as micronized powder under the trade mark Kynar 500.

Nb_2O_5 and sodium hydroxide were provided by Sigma-Aldrich. Nb_2O_5 was dissolved in concentrated NaOH (aq) and introduced in an autoclave; cubic particles were obtained [18]. NaNbO_3 particles were washed in water and dispersed by sonication in acetone (Figure 1a). NaNbO_3 has been chosen for its lead-free composition and low poling field.

Two types of magnetic particles were employed for this study. Commercial Ni spherical particles (NiNPs), provided by Sigma, present an irregular and pseudo spherical aspect with a mean size of 2 μm (Figure 1b).

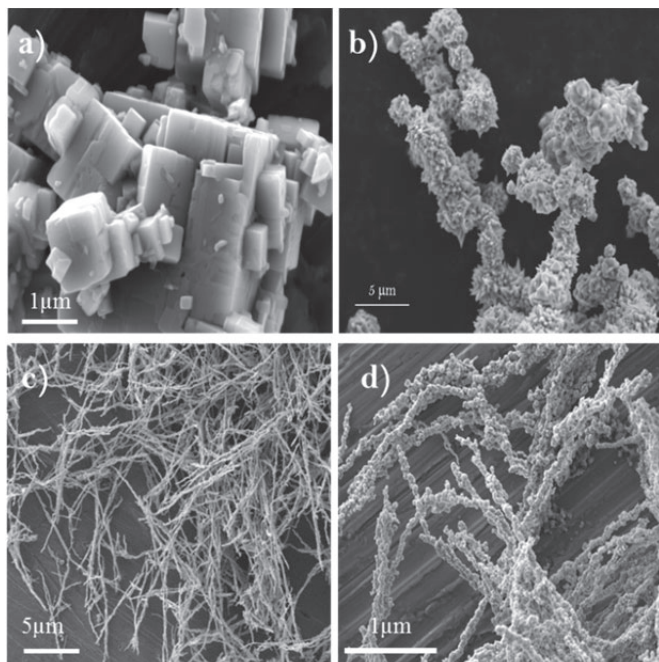


Figure 1. SEM images of a) NaNbO_3 particles; b) Ni spherical particles NiNPs; c) and d) Ni high aspect ratio nanochains NNCs.

High aspect ratio particles were synthesized in the laboratory. High aspect ratio was obtained by polyol reduction of $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ in 1,2 butanediol under 10mT DC magnetic field. These pseudo spherical particles were assembled under the influence of a magnetic field in order to form nickel nanochains (NNC). More details were given in a previous publication [19].

Particles' morphology was characterized by SEM and micrographs are shown in Figure 1. NaNbO_3 particles appear to be cubic with a mean size of 1 μm (Figure 1a).

NNCs present regular length and diameter (Figures 1c and 1d). Statistical analysis was provided in a previous work [19]. Mean aspect ratio ξ Equation (1) is about 80.

$$\xi = \frac{\text{length}}{\text{diameter}} \quad (1)$$

2.2 COMPOSITES PREPARATION

Typical protocol for the composite processing is the following: PVDF powder was dissolved in acetone while nickel and NaNbO_3 particles were also dispersed in acetone in a separated content. Particles suspensions were sonicated to avoid aggregation. The mixture of dissolved matrix, NaNbO_3 and Ni suspensions were mixed together and submitted to ultrasonic agitation for 2 minutes in order to produce a homogeneous mixture. The mixture was then quickly precipitated to obtain the composite.

The precipitate composite was then hot-pressed under 1 MPa at 220°C in order to get films with a thickness of around 200 μm .

The NaNbO_3 content was fixed at 20 vol% for all samples. In order to study the influence of nickel proportion, composites were proceed with 0.3 and 2 vol% of nickel.

2.3 POLING PROTOCOL

Getting macroscopic piezoelectricity in composites requires a poling step. An unidirectional DC electric field was applied to the sample placed in castor oil in order to avoid breakdown. The DC field of 5 kV/mm was maintained for 30 minutes.

The d_{33} piezoelectric coefficient measurements were carried out 24 h after the polarization step in order to allow elimination of electric charges accumulated on the composite's surface.

2.4 SCANNING ELECTRON MICROSCOPY

Scanning electron microscopy (SEM) was performed on particles to examine their morphology on a JEOL 7800 Prime. Images were also made on composites to check the dispersion of particles. Observations were performed under secondary electrons on an aluminium surface.

2.5 DYNAMIC DIELECTRIC SPECTROSCOPY

Dielectric permittivity was measured on composite films thanks to the Novocontrol broadband dielectric spectrometer (BDS). Measurements were performed as a function of frequency between $1 \times 10^2 \text{ Hz}$ and $1 \times 10^6 \text{ Hz}$. Isotherms are recorded at 20 °C.

2.6 PIEZOELECTRIC MEASUREMENTS

The piezoelectric coefficient was measured by a PM 200 piezometer supplied by Piezotest UK.

Frequency and stress were set at 110 Hz and 0.25 N, respectively. The stress was applied to the sample in the same direction as the poling field; the polarization was measured in order to get the d_{33} coefficient.

2.7 MAGNETIC PROPERTIES MEASUREMENTS

Magnetization was measured thanks to SQUID magnetometer. Hysteresis loops were recorded between -5 and 5 T in order to reach saturation. These measurements were carried out on raw NiNPs, NNCs and PVDF / nickel composite in order to determine the influence of the processing method over the NNCs orientation.

2.8 MAGNETOELECTRIC MEASUREMENTS

ME properties were measured as a function of AC magnetic field (H_{AC}) and DC magnetic field (H_{DC}). Samples were thin film shaped with a thickness of 200 μm . Their surfaces were metalized to avoid contact resistivity.

3 RESULTS AND DISCUSSION

3.1 ELECTRICAL PROPERTIES

Real part of dielectric permittivity (ϵ') is plotted as a function of frequency at 300K (Figure 2). Experiments were carried out on the two types of composites i.e with NiNPs and NNCs. For PVDF / NiNPs composites and PVDF / NNCs composites at 0.3 vol%, permittivity values are close from each other in the whole frequency range. For the 2 vol% PVDF / NNCs composite, a significant increase of ϵ' is observed below 1 Hz. This increase is characteristic of the proximity of the percolation threshold and caused by the Maxwell-Wagner-Sillars (MWS) polarisation. This polarisation is due to charge accumulation at the interfaces.

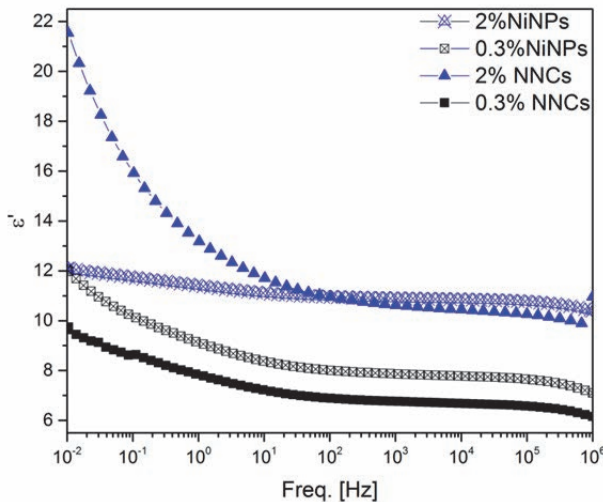


Figure 2. Dielectric permittivity versus frequency at ambient temperature for PVDF / NaNbO₃ / Ni composites.

Many studies [20] have reported the increase of permittivity towards lower frequencies upon the introduction of conductive

particles in polymeric matrices. Electrical percolation corresponding to the insulator / conductor transition is responsible for this evolution. Then, the electrical conductivity (σ_{DC}) is described by the Stauffer law [21],

$$\sigma_{DC} = \sigma_0(p - p_c)^t \quad (2)$$

where σ_0 is the theoretical conductivity of 100% NNCs, p is the volume fraction, p_c is the volume fraction at the percolation threshold and t is the critical exponent.

We showed in a previous work that $p_c = 2.3$ vol% for PVDF / NNCs[19]. The proximity of the percolation threshold explains why ϵ' reaches such a high value. Moreover, it has been shown that p_c strongly depends on the aspect ratio of particles [22]. Thus, for spherical particles, p_c is much higher than 2 vol%.

3.2 PIEZOELECTRIC PROPERTIES

Electro-mechanical coupling is characterized by the d_{33} coefficient. The influence of Ni particles morphology is presented on Figure 3 where d_{33} is plotted as a function of Ni volume content.

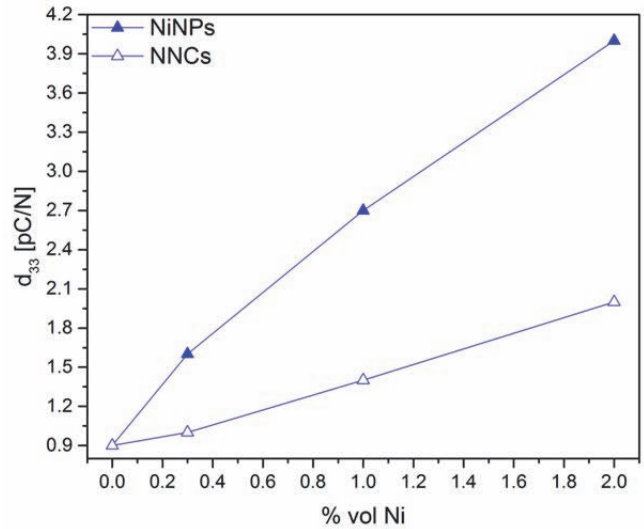


Figure 3. Piezoelectric coefficient d_{33} (pC/N) in PVDF / NaNbO₃ 20 vol % / Ni particles versus volume content in Ni.

Composites were poled under the same macroscopic field i.e. 5 kV/mm. Despite the proximity of the percolation threshold, NNCs composites were successfully poled.

It is important to note that all three-phase composites exhibit a higher piezoelectric coefficient than the two-phase composites without conducting particles. It has already been shown that adding conductive particles in a piezoelectric composite leads to a higher d_{33} [23].

Differences recorded between composites with NiNPs and NNCs respectively have been associated with the variation induced on the mechanical storage modulus that increases with the aspect ratio of particles [24]. It explains the decrease on d_{33} upon increasing of the aspect ratio.

3.2 MAGNETIC PROPERTIES

Magnetic properties of NiNPs, NNCs, PVDF / NiNPs and PVDF / NNCs have been determined in order to underline the

influence of morphology over the magnetic behaviour. We have reported in Table 1 remnant magnetization in the parallel direction ($M_{r//}$) and remnant magnetization in the perpendicular direction ($M_{r\perp}$), coercive field in the parallel direction ($H_{C//}$) and coercive field in the perpendicular direction ($H_{C\perp}$).

Table 1. Magnetic properties of NPS; NNCs, PVDF / NiNPs and PVDF / NNCs composites.

Sample	$M_{r//}$ (emu/g)	$M_{r\perp}$ (emu/g)	M_r / M_s	$H_{C//}$ (Oe)	$H_{C\perp}$ (Oe)
NiNPs	3.81	3.81	18	148	148
NNCs	10.80	10.80	20	163	163
PVDF/NiNPs	0.24	0.24	5.03	89	89
PVDF/NNCs	0.41	0.14	4.3	210	180

Spherical particles show an isotropic magnetization, as expected. $M_{r//}$ and $M_{r\perp}$ reach the value of 3.81 emu/g and a ratio M_r / M_s of 18%. As for the magnetization, coercive field (H_C), has the same value of 148 Oe in the transverse and in the longitudinal direction.

Figure 4 represents the magnetization of composites as magnetic hysteresis loops. We notice that despite a low content of Ni (2 vol%), composites present a ferromagnetic behaviour. As for the NiNPs, PVDF / NiNPs composite shows isotropic properties. The good dispersion and the isotropic properties of particles lead to an isotropic magnetic composite.

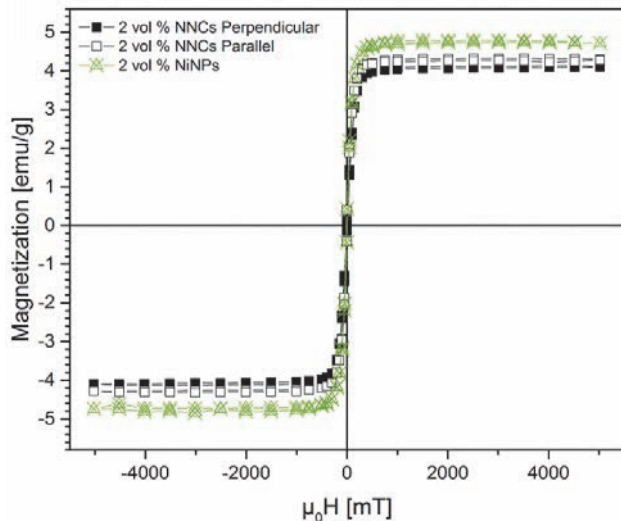


Figure 4. Magnetization versus field hysteresis loop for PVDF / Ni composites in perpendicular and parallel direction at 300K.

When NNCs are introduced in a composite, anisotropy appears clearly. In the parallel direction, M_r (PVDF / NNCs) becomes almost twice as high as PVDF / NiNPs. In the perpendicular direction, M_r (PVDF / NNCs) is almost twice lower than for PVDF / NiNPs. This anisotropy reflects the morphology of Ni particles but also their orientation due to the

composite processing. H_C exhibits different values in the longitudinal and in the transverse direction of the composite (Figure 5). It is interesting to note that, in the parallel direction H_C is also analogous for PVDF / NNCs and PVDF / NiNPs.

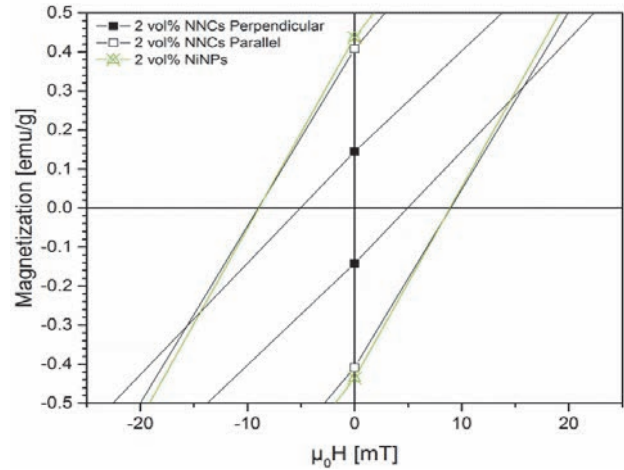


Figure 5. Zoom on the hysteresis loop between -25 and 25mT for PVDF / Ni composites in the perpendicular and parallel direction at 300K.

3.4 MAGNETOELECTRIC COUPLING MEASUREMENTS

Magnetoelectric response of PVDF / NaNbO_3 / Ni composites was registered in the intensity mode (I_{ME}), on a homemade set-up.

Composites were metalized with silver paint in order to minimize the contact resistivity between sample and probe. Intensity was measured thanks to a lock-in amplifier in order to extract signal from noise. Samples were placed in the middle of the coil where a homogenous and unidirectional field prevailed.

Figure 6 shows I_{ME} as a function of AC field intensity (H_{AC}) at 1 kHz under a DC field of 4000 Oe. I_{ME} values exhibit a linear increase with H_{AC} regardless from the nickel particles content or morphology. For 0.3 vol % Ni, I_{ME} has practically the same value for NiNPs or NNCs. The slope is also similar for both. In NiNPs composites, d_{33} was found to be higher than in NNCs composites. Nevertheless, it has been shown that NNCs exhibit enhanced magnetic properties. In these composites with low nickel content, magnetic properties are preeminent for the magnetoelectric coupling.

For 2 vol% nickel composites, magnetic effects are more important. I_{ME} is higher for both morphologies than for 0.3 vol% composites. I_{ME} becomes much higher for NiNPs composite than for the NNCs one. The slope is also increased. These strong differences can be explained by the important evolution of d_{33} . As shown in Figure 3, d_{33} is twice as high as in NiNPs composite, which plays an important role over the coupling. Here, magnetic properties enhancement induces by the aspect ratio of NNCs is not strong enough to set I_{ME} off. Two causes appear to explain this phenomenon. Firstly, the d_{33} is much more important than previously, more than double for NiNPs when it was only 30% higher in 0.3 vol% Ni composites. The other reason is the proximity of percolation

threshold in the 2 vol% NNCs composite. As shown in Figure 2, permittivity rises strongly inducing more dielectric losses.

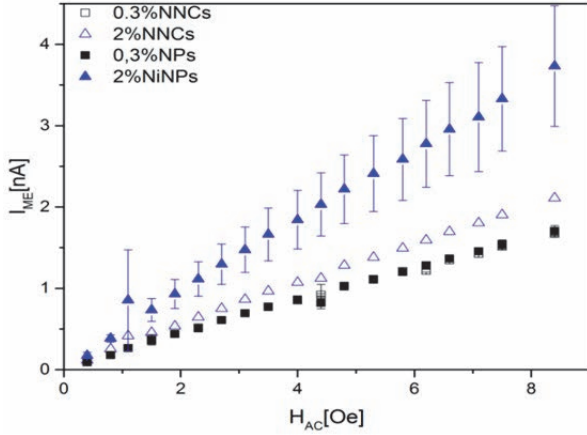


Figure 6. Magnetolectric intensity for PVDF/NaNbO₃/Ni composites.

In order to compare our data with literature, the α_{ME} magnetolectric coefficient of polarisation has been calculated thanks to Equation (3):

$$\alpha_P = \frac{I_{ME}}{S\omega h_{AC}} \quad (3)$$

where I_{ME} the ME response of material, S is the surface of the sample, ω is the angular frequency and h_{AC} is the intensity of the ac magnetic field.

Then, we obtain the magneto electric coupling coefficient α_{ME} from Equation (4):

$$\alpha_{ME} = \frac{\alpha_P}{\epsilon\epsilon_0} \quad (4)$$

where ϵ is the relative dielectric permittivity of the sample and ϵ_0 the vacuum dielectric permittivity.

The α_{ME} values give us a more precise understanding of the phenomenon. The histogram of Figure 7 reports the coupling coefficient for the studied composites.

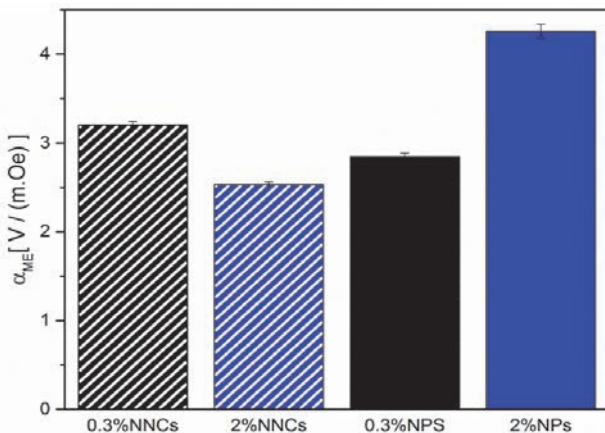


Figure 7. Magnetolectric coupling coefficient α_{ME} for PVDF / NaNbO₃ / Ni composites.

First, let us consider PVDF / NNCs composites. We observe that for 0.3 vol% Ni, PVDF / NNCs exhibit a higher value of α_{ME} than PVDF / NiNPs. Based on the previous experiences, it confirms that the enhancement of magnetic properties plays an important role while the permittivity of composite remains close and d_{33} does not change in a drastic manner. For the 2 vol% NNCs composite, we observe a decrease of d_{33} . Despite the slightly higher I_{ME} , ϵ' increases due to the proximity of the percolation threshold. Consequently, α_{ME} decreases as predicted by Equation 4.

Second, let us discuss data on PVDF / NiNPs composites. α_{ME} rises from 2.8 V/(mOe) for 0.3vol% Ni to 4.2 V/(mOe) for 2 vol% Ni. As show by Figure 6, I_{ME} increases strongly with NiNPs. This increase reflects the increase of d_{33} by a factor 2 in this composite. Since the percolation threshold is far away from 2 vol% for spherical particles [20], the evolution of the piezoelectric transduction governs the magnetolectric coupling for PVDF / NiNPs.

4 CONCLUSIONS

Three-phase polymer based composites PVDF / NaNbO₃ / Ni have been elaborated in order to reach satisfactory magnetolectric coupling; extrinsic piezoelectric effect is insured by 20 vol% of NaNbO₃ particles (1 μ m) and magnetic properties are due to Ni particles with different aspect ratios. Ni quasi-spherical particles (2 μ m) NiNPs have been compared to Ni nano chains (NNCs) obtained by a polyol reduction method under a magnetic field leading to particles with a mean aspect ratio of 80. Two contents of nickel 0.3 and 2 vol% have been used. Dielectric permittivity exhibits an important increase for the 2 vol% NNCs composite due to its proximity with the electrical percolation threshold. Electro-mechanical coupling has shown that adding Ni in the composite increases the piezoelectric response. This effect is more important in PVDF / NaNbO₃ / NiNPs composite, which give a d_{33} of 4.1 pC/N, i.e. four times higher than in PVDF / NaNbO₃ composite. Magnetic measurements confirm the ferromagnetic behavior of the composites despite the low amount of Ni. It also shows that NNCs allow an enhancement of magnetic properties such as M_r and H_C . Magnetolectric measurements prove the existence of the ME coupling by producing a ME signal as a function of H_{AC} . It shows that for low amount of nickel, NNCs allow better coupling while when the permittivity increases, it is more interesting to use Ni NPs.

Magnetolectric properties of PVDF / NaNbO₃ / Ni composites open the possibility of new applications in the field of smart materials. Thanks to NaNbO₃'s high T_C , we might propose magnetolectric composites with a non-electroactive matrix such as high performance thermoplastic. These materials would allow us to envision the processing of high temperature sensors.

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