Size effects on the magnetoelectric response on PVDF/Vitrovac 4040 laminate composites

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ABSTRACT: Tri-layered and bi-layered magnetoelectric (ME) flexible composite structures of varying geometries and sizes consisting on magnetostrictive Vitrovac and piezoelectric poly(vinylidene fluoride) (PVDF) layers were fabricated by direct bonding. From the ME measurements it was determined that tri-layered composites structures (magnetostrictive-piezoelectric-magnetostrictive type), show a higher ME response ($75 \text{ V.cm}^{-1}.\text{Oe}^{-1}$) than the bi-layer structure ($66 \text{ V.cm}^{-1}.\text{Oe}^{-1}$).

The ME voltage coefficient decreased with increasing longitudinal size aspect ratio between PVDF and Vitrovac layers (from 1.1 to 4.3), being observed a maximum ME voltage coefficient of $66 \text{ V.cm}^{-1}.\text{Oe}^{-1}$. It was also observed that the composite with the lowest transversal

aspect ratio between PVDF and Vitrovac layers resulted in better ME performance than the structures with higher transversal size aspect ratios.

It was further determined an intimate relation between the $\frac{Area_{PVDF}}{Area_{Vitrovac}}$ ratio and the ME response of the composites. When such ratio values approach 1, the ME response is the largest.

Additionally the ME output value and magnetic field response was controlled by changing the number of Vitrovac layers, which allows the development of magnetic sensors and energy harvesting devices.

Keywords: Magnetoelectric; piezoelectric; magnetostrictive, laminates; aspect ratio

1. Introduction

Magnetoelectric materials (ME) are characterized by the variation of the electric polarization (P) in the presence of an applied magnetic field (H) (equation 1),

$$\Delta P = \alpha \Delta H \quad (1)$$

where α is the ME coupling coefficient [1-4]. In single-phase materials this effect is intrinsic and corresponds to the coupling between magnetic moments and electric dipoles [1-3]. Low Curie temperatures and weak ME coupling at room temperature hinder their technological applications [4-6].

Due to those limitations, the research interest began to focus in the ME effect of multiple-phase materials, in which such effect is a result of the product of the

magnetostrictive effect (magnetic/mechanical) within the magnetostrictive phase and the piezoelectric effect (mechanical/electrical) within the piezoelectric phase (equation 2).

$$ME_{\text{H effect}} = \frac{\text{electric}}{\text{mechanical}} \times \frac{\text{mechanical}}{\text{magnetic}} = \frac{\partial E}{\partial \lambda} \times \frac{\partial \lambda}{\partial B} \quad (2)$$

where $\frac{\partial \lambda}{\partial B}$ and $\frac{\partial E}{\partial \lambda}$ are the piezomagnetic coefficient and piezoelectric coefficients respectively.

Thus, the ME effect in multiple-phase materials is extrinsic, strongly depending on the microstructure and coupling interaction across the magnetic-piezoelectric interfaces [6-8].

This ME-product property leads to output signals at room temperature that are orders of magnitude higher than the ones in single phase materials, being therefore very attractive for innovative applications in areas such as such as brain activity sensors and magnetic sensors (Figure 1), among others [1, 6, 7, 9].

Additionally, the multiferroic composite approach offers a large potential for technological optimization due to the degrees of freedom in sensor design, including the choice of the piezoelectric and magnetostrictive materials, their deposition process and/or binding and their three dimensional arrangement [10]. In the last decade, different approaches for the development of ME-sensors have been reported, all of them based on one of two fundamental type of piezoelectric materials: either are constituted

by piezoelectric ceramics or piezoelectric polymers [6], together with the magnetostrictive element.

Piezoelectric ceramics have low electrical resistivity, high dielectric losses and are dense and brittle, which can lead to fatigue and failure.[6, 11, 12] In order to improve and solve some problems related to ceramic composites, piezoelectric polymers such as poly(vinylidene fluoride), PVDF, and its copolymers have been used since they are flexible, show large electrical resistivity and low losses, and can be processed in different shapes at low processing temperatures [6, 12-14]. Three main types of polymer-based ME composites can be found in the literature: (a) nanocomposites, (b) polymer “*as a binder*”, and (c) laminated composites [6]. Within those materials, the highest ME response is obtained in laminated polymer based ME materials, being the highest value ($21.46 \text{ V.cm}^{-1}.\text{Oe}^{-1}$) at non resonance frequencies reported for a three layer laminate comprising PVDF, Metglas and a Devcon epoxy [15]. On the other hand, the highest ME value reported at electromechanical resonance was $383 \text{ V.cm}^{-1}.\text{Oe}^{-1}$ using P(VDF-TrFE)/Metglas 2605SA1 bonded with an epoxy resin, [16].

For sensor applications, the optimization of the element responsible for the coupling between magnetostrictive and piezoelectric components plays a crucial role [17-19]. A study on PVDF bonded to Vitrovac 4040 with epoxies with different elastic moduli show a decrease in the ME voltage coefficient from 53 to $6 \text{ V.cm}^{-1}.\text{Oe}^{-1}$ with increasing epoxy Young Modulus from 2.7×10^8 to 9.0×10^9 Pa, the highest ME response of $53 \text{ V.cm}^{-1}.\text{Oe}^{-1}$ being obtained for a PVDF/M-Bond epoxy/Vitrovac laminate. [17]

A different approach for the coupling of the piezoelectric and magnetostrictive elements was recently presented by direct spin coating of a PVDF layer onto a Metglas substrate. By eliminating the use of an adhesive epoxy for the mechanical coupling between the layers and taking advantage of a clamping effect, a giant ME voltage coefficient of

$850 \text{ V.cm}^{-1}.\text{Oe}^{-1}$ was obtained at the bending mode resonance frequency of 27.8 Hz [10].

However, coupling between the different phases is not the only parameter that requires optimization prior to their incorporation into technological applications: characteristics such as size, structure and relative geometry of the components may allow tailoring the applicability of ME composite materials [20].

In this way, this work shows the influence of the relative size of the magnetostrictive and piezoelectric elements on the ME response. Additionally the effect of distinct geometries in the ME response will be also addressed. Vitrovac ($\text{Fe}_{39}\text{Ni}_{39}\text{Mo}_4\text{Si}_6\text{B}_{12}$) 4040 was used as the magnetostrictive element due to its large piezomagnetic coefficient (1.3 ppm.Oe^{-1}) at low magnetic fields, [21] and PVDF was chosen as the piezoelectric element, due to its highest piezoelectric response among polymers [13, 22]. M-Bond epoxy was used as coupling agent since it has proved obtain higher ME coefficients in ME laminates when compared to other epoxy resins [17].

2. Materials and Methods

The magnetostrictive samples from Vitrovac ($\text{Fe}_{39}\text{Ni}_{39}\text{Mo}_4\text{Si}_6\text{B}_{12}$) 4040 (25 μm thick) were cut into rectangular shapes using a clean and sharp ceramic scalpel. The rectangular samples of Vitrovac 4040 exhibit a magnetostrictive coefficient λ_{11} of 8ppm [23].

Poled β -PVDF films 28 μm thick with Cu–Ni electrodes deposited on both sides were purchased from Measurement Specialties, USA, and used as provided ($d_{33} = -33 \text{ pC.N}^{-1}$ and $d_{31} = 23 \text{ pC.N}^{-1}$) [13]. All piezoelectric samples were cut into rectangular shapes using a clean and sharp scalpel. The PVDF piezoelectric response (d_{33}) was verified

with a wide range d₃₃-meter (model 8000, APC Int. Ltd.) to ensure that the sample preparation process has no effect on the piezoelectric response of the polymer.

To study the effect of the materials size, structure and geometry on the ME response, various samples were produced as represented on Figure 2.

In this way, in samples A-C the longitudinal size aspect ratio (LAR) between the PVDF and the Vitrovac layers was changed between 1.1 and 4.3, in the samples A, D-F the transversal size aspect ratio (TAR) between the PVDF and the Vitrovac layers was changed between 2.0 and 4.0. Samples A-F allowed also to vary the $\frac{Area_{PVDF}}{Area_{Vitrovac}} (= \frac{A_P}{A_M})$ relation between 2.2 and 8.6.

Since previous studies have shown the large importance of the bonding layer in the ME response of ME laminates [17], special has to be taken in the bonding process with the M-Bond epoxy in order to ensure a reproducible bonding and that the inner structure of the interface is the same in all the samples. In this way, it is guaranteed that the differences observed in the ME response of the samples is due to the distinct configurations and phase sizes and not a result of the heterogeneity the bonding process.

ME measurements were performed by simultaneously applying a continuous H_{DC} magnetic field ranging from 0 to 50 Oe and an alternating 0.13 Oe H_{AC} field at magnetoelastic resonance frequencies ranging from 25 kHz to 160 kHz.

The ME response of the laminate is then determined as:

$$\alpha_{ME} = \frac{dE}{dH} = \frac{1}{t} \left(\frac{\delta V}{\delta H_{AC}} \right) \quad (3)$$

where δH_{AC} is the applied AC magnetic field amplitude; δV is the induced magnetolectric voltage and t is the thickness of the piezoelectric element.

Each sample was measured at the magnetoelastic resonant frequency and at the DC field at which the magnetoelastic response was the highest. δV was accurately measured using a SR830 DSP lock-in amplifier.

3. Results and discussion

The influence of the magnetostrictive layer length on the ME response of the laminate was studied by varying the LAR between 1.1 mm and 4.3 (Figure 3).

Results reveal the strong influence of the LAR on the ME response of the composite. The highest ME response has been obtained for the Vitrovac with the LAR value close to 1 (Laminate A), and the lowest response was obtained for the highest LAR (Laminate C) (Figure 3a). It can be observed on Figure 3 that the ME response varies almost linearly with LAR, since with higher magnetostrictive area, more strain will be transmitted to the piezoelectric layer, thereby producing a higher voltage.

Figure 3 also shows that the magnetic field at which the maximum ME response is obtained increases with increasing LAR, behavior which is related to the demagnetizing field, which is stronger for smaller samples [24].

Figure 4 demonstrates the behavior of ME laminate composites with the variation of TAR.

The obtained results indicate the strong influence of TAR on the ME response. The highest response was obtained for the composite with the TAR closest to 1 (2.0), and the lowest response was obtained for the sample with the highest TAR (4.0). With increasing PVDF surface area not directly bonded to the magnetostrictive Vitrovac, A_{NB} , relative to the PVDF area directly bonded to Vitrovac, A_B , clamping effects will arise, since the A_{NB} will attenuate the strain variations of the A_B . In this way, stress

clamping of the A_{NB} hinders polarization switching on the A_B , reducing the piezoelectric response [25] and, as a consequence, decreasing the ME response. Such effect is further evidenced when the shape of the ME peaks of Figure 1a is analyzed together with Figure 1b. When the width of PVDF is close to the width of Vitrovac, A_B is much higher than A_{NB} and the PVDF layer is free to strain as a response of the deformation of the Vitrovac layer. When the magnetic field increases, the magnetostrictive strain variation of Vitrovac ranges from zero to its maximum value and consequently the piezomagnetic coefficient achieves its maximum value. In this situation, the deformation variation increases with increasing DC magnetic field and, as a result, the ME coefficient also increases and reaches its maximum when the magnetostrictive strain variation also reaches the maximum value. As the external magnetic field continues to increase, the magnetostrictive strain variation starts to decrease together with the deformation variation values leading to a decrease of the ME field coefficient [26]. When A_{NB} is much higher than A_B , the PVDF not directly bonded to Vitrovac will mitigate the strain of PVDF directly bonded to Vitrovac when the applied DC magnetic field is between 1 and 5 Oe. In this situation, the increase of the magnetostrictive strain is not accompanied by an increase in the ME response [25].

Additionally the $\frac{A_P}{A_M}$ ratio was changed between 2.2 and 8.6 and the effect of this variation in the ME response of the composites has been studied (Figure 4c). It is verified that when the $\frac{A_P}{A_M}$ ratio approach 1, the ME response is optimized. This

behaviour is consistent with the results shown in Figures 3 and 4.

Finally, the effect of the laminate configuration in the ME response was analyzed by measuring a by-layer composite (a), a three-layer magnetostrictive-piezoelectric-

magnetostrictive (MPM) composite (g) and a three-layer piezoelectric-magnetostrictive-piezoelectric (PMP) composite (Figure 5).

Figure 5a shows that the laminate configuration with the highest ME response is the Vitrovac-PVDF-Vitrovac (MPM) since the two magnetostrictive layers deliver more deformation to the piezoelectric layer and, as result, a higher ME response is obtained. On the other hand, the PMP configuration achieves the lowest ME response due to larger clamping effect between the two layers of PVDF and the Vitrovac layer.

In terms of the inner structure of the bilayer laminates and under the H_{AC} field excitation applied along the length axis, the Vitrovac layer will suffer strain variations determined by the magnetostrictive response (Vitrovac 4040, $\lambda_{11}=8\text{ppm}$ [23]). The strain variations will be transmitted through the epoxy binder to the piezoelectric PVDF film which will undergo a longitudinal strain, inducing dielectric polarization variations in its transverse direction as determined mainly by the piezoelectric $d_{31}=23 \text{ pC.N}^{-1}$ [13] coefficient. Such transfer process is optimized in the MPM configuration.

Figure 5b allows to study the effect of the $\frac{Thickness_{PVDF}}{Thickness_{Vitrovac}}$ ratio in the ME response of the composites. The decrease in the ME coefficient with increasing $\frac{Thickness_{PVDF}}{Thickness_{Vitrovac}}$ ratio being explained in terms of the decreased compressive stress in the piezoelectric layer [27].

Note that the ME peak of the sample G, is slightly shifted to higher ME fields as a result of the demagnetizing effect of the Vitrovac layers [28, 29]. These results indicate that as the number of Vitrovac layers increases, a higher DC magnetic field is required to reach

the maximum ME coefficient. This implies that it is possible to control the ME output value and saturation point by changing the number of Vitrovac layers [30].

4. Conclusions

Thin, flexible, low-weight and low-cost ME laminates with simple fabrication and tunable properties, consisting of Metglas/PVDF unimorph and three-layer sandwich configurations have been produced with the objective to study the influence of the size of magnetostrictive and piezoelectric elements on the ME response as well as the effect of distinct geometries in the ME response will be also addressed.

It is concluded that the ME voltage coefficient increases with decreasing LAR (from 4.3 to 1.1), reaching a maximum ME voltage coefficient of $66 \text{ V.cm}^{-1}.\text{Oe}^{-1}$. Multiferroic laminates with lowest TAR resulted in better ME performance when compared with higher TAR. It was further demonstrated an intimate relation between the $\frac{A_p}{A_M}$ ratio and the ME response of the composites. When such ratio values approach 1, the ME response is optimized.

Tri-layered composites configurations (magnetostrictive-piezoelectric-magnetostrictive type), have a higher ME response ($75 \text{ V.cm}^{-1}.\text{Oe}^{-1}$) than the bi-layer configuration ($66 \text{ V.cm}^{-1}.\text{Oe}^{-1}$).

Additionally it was observed a decrease in the ME coefficient with increasing

$\frac{\text{Thickness}_{\text{PVDF}}}{\text{Thickness}_{\text{Vitrovac}}}$ ratio.

Furthermore the ME output voltage and optimum magnetic field can be controlled by changing the number of Vitrovac layers, which makes this composite a promising candidate for magnetic sensors and energy harvesting applications.

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Figures and Figure Captions

Figure 1. Possible applications of ME materials: Monitoring brain activity and magnetic sensors.

Figure 2. Top view of the Vitrovac/PVDF by-layer composites produced in this study (A-F). Front view of the three-layer composites produced in this study (G-H).

Figure 3. a) ME response of laminates with different LAR; b) Variation of the ME response with increasing LAR.

Figure 4. a) ME response obtained with different TAR; b) Variation of the ME response with increasing TAR; c) ME response obtained with distinct $\frac{A_P}{A_M}$ ratios.

Figure 5. a) ME response obtained on laminates with bilayer composite (Sample A), three-layer magnetostrictive-piezoelectric-magnetostrictive (MPM) (Sample G) composite, and three-layer piezoelectric-magnetostrictive-piezoelectric composite (PMP) (Sample H) configurations. b) ME response obtained with distinct $\frac{\text{Thickness}_{\text{PVDF}}}{\text{Thickness}_{\text{Vitrovac}}}$ ratios.

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