OPEN ACCESS
JOURNAL OF ADVANCED DIELECTRICS
Vol. 13, No. 4 (2023) 2341007 (8 pages)

DOI: 10.1142/S2010135X23410072

History and recent progress in ferroelectrets produced in Brazil

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Received 5 September 2022; Revised 13 March 2023; Accepted 10 April 2023; Published 25 April 2023

More than 30 years ago, a group of researchers in Tampere–Finland developed a thin foamed polymeric material for capacitive sensors. Such soft-voided films exhibited electrical charging characteristics, forming a powerful combination, which resulted in a smart-material with ferroelectric properties. The discovery of the electro-thermo mechanical film (ETMF) has sparked the curiosity of the electret community, leading to the development of several studies. At that time, ETMF became known as cellular electrets and, later, as ferroelectrets or piezoelectrets regarding their electromechanical properties. This paper provides a timeline review of the research on ferroelectrets produced in Brazil, between the years 1990 and 2020, towards demonstrating how the interest in the electret electrical charging mechanism has resulted in the use of ferroelectrets with well-controlled cavities for ultrasound applications.

Keywords: Cellular electrets; piezoelectrets; ferroelectrets; electromechanical films.

1. Introduction

In 1989, Savolainen and Kirjavainen, both researchers from the Institute of Plastic Technology at Tampere University of Technology, Finland, reported a new process for fabricating foamed films with electro-thermo-mechanical properties.[1] The material, called electro-thermo mechanical film (ETMF), was also indicated for some applications as capacitive transducers, electrostatic transducers, and polarized generators, due to its electret properties.

Approximately 10 years earlier, Prof. Ruy Alberto Corrêa Altafim had his first contact with *electrets* during a lecture at the Polymers Group — São Carlos Institute of Physics (IFSC) University of São Paulo (USP), led by Prof. Bernhard Gross and Prof. Guilherme Leal Ferreira, two respected electret researchers. The subject drew Altafim's interest, becoming part of his Ph.D. research, which was closely followed by Prof. Gross and José Alberto Giacometti, who were studying electret charging with corona discharges.[2]

Altafim's thesis concerned an electret charging procedure developed with an upper electrode slightly distanced from a dielectric film through which impulsive voltages were applied. The high electric field formed between the electrode and the grounded insulating foil induced dielectric barrier discharges in the air gap, resulting in a homogeneous electric charging.[3]

At that time, ETMF and Altafim's impulse charging system apparently had no common relation; however, the impulsive electrical charging process on electret films is very similar to the electrical barrier discharges that occur inside the ETMF in the charging process.[4] In fact, this phenomenon was under the investigation of Prof. Reimund Gerhard's research group, who presented his recent developments in electrical charging of cellular electrets under different gas atmospheres at the Conference on Electrical Insulation and Dielectric Phenomena (CEIDP), in 2001.[4]

Attending the same conference, Prof. Altafim envisioned the possibility of electrically charging ETMFs with impulsive discharges. After discussing the topic with Prof. Gerhard, his colleague, Michael Wegener, went to Brazil carrying with him a set of foamed polypropylene (PP) samples from Tampere University. The first experiments with electrical impulsive charging were then conducted and the results were published in the next year's CEIDP.[5]

Further developments of ETMF films performed by the Brazilian research group are presented in the following sections as a temporal review. In Sec. 2, a different approach for fabricating voided structures is presented employing multi-layer electret films. Different methods implemented to enhance performance are also described here. In Sec. 3, the multi-layer system is presented as an electro-mechanical film with extended properties, such as magnetic. Finally, some applications with electro-mechanical films are presented in Sec. 4.

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2. Ferroelectrets Produced in Brazil

After the experiments with PP samples and impulsive charging, a lack of cellular PP fresh samples reduced the intensity of electrical charging research but it also created new opportunities.

2.1. *First nonconventional voided electrets*

Altafim, in a partnership with Prof. Custódio Dias, from the Technological Institute of Porto, Portugal, and Prof. Heitor Cury Basso and Prof. Luiz Gonçalves Neto, from the Electrical Engineering Department at the University of São Paulo, proposed assembling a voided multi-layered electret film with two fluoroethylene propylene (Teflon™-FEP) films separated by a layer of homogeneously distributed shellac drops (Fig. 1).[6] The space between the drops formed air cavities closed by films. Similarly to ETMFss, the voided multi-layered electrets were electrically charged with impulsive voltages with up to −12.5 kV peaks. Measurements performed by a direct quasi-static method indicated piezoelectric coefficient d_{33} of approximately 150 pC/N, i.e., in the same order of magnitude of the one from cellular electrets.

The multi-layered design containing shellac drops provided an alternative for the production of voided electrets, although some drawbacks were observed during dynamic measurements (e.g., low adherence between the Teflon™-FEP layers and premature electrical discharging caused by the movement of shellac drops during sample compression). The bonding between the layers remained an issue to be taken into consideration in further developments.

2.2. *Vacuum thermo-forming method*

A more consistent multi-layered voided electret was fabricated combining thermal fusing methods with vacuum thermo-forming process. The method, presented at the 2005 CEIDP, uses a hot press and consisted in heating a Teflon™-FEP film at 200°C and molding it with a vacuum pump through a grid, as shown in Fig. 2.[7] The film, shaped with regular domes, was superimposed by another Teflon™-FEP film and compressed in a hot press for a few seconds. The process fused the films together, resulting in a double-layer structure with closed air cavities similar to those in air cushion packages. Gold electrodes were then sputtered on both sides of the thermo-formed structure, through which impulse voltages were applied (−7.5 kV peak). After electrical charging, quasi-static direct measurements were performed and d_{33} coefficients of 500 pC/N were observed.[8]

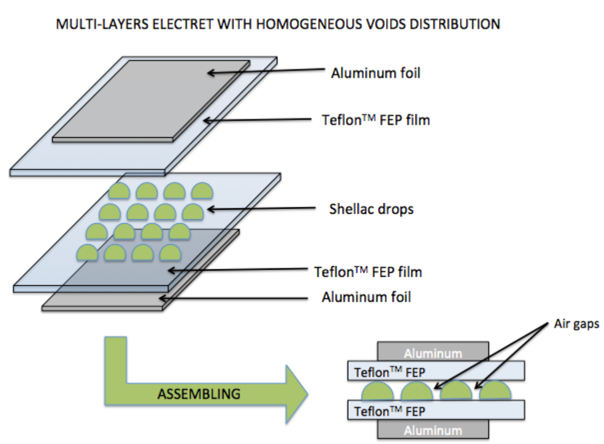

Fig. 1. Assembling process of multi-layer electrets with homogeneous air gaps. Shellac drops are employed as spacers between two Teflon™-FEP films thus forming regular cavities, as indicated in the cross-section representation.

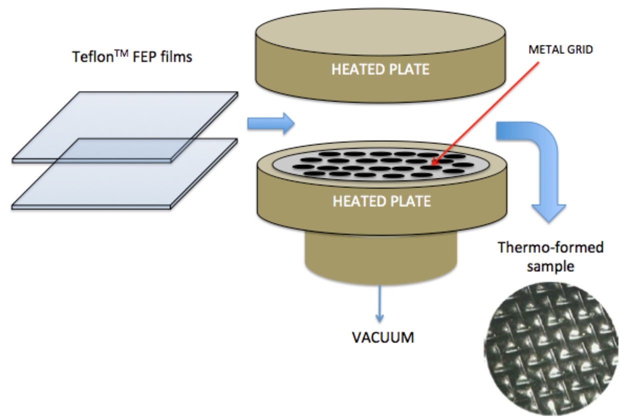

Fig. 2. Representation of the fabrication process for preparing thermo-formed multi-layer electrets. In this drawing, two Teflon™-FEP films are placed over a metal grid mounted in a heated plate. Subjected to a vacuum pump, the lower film is molded into dome shape while the upper film is thermally fused to it.

The cavity pattern obtained from the vacuum thermo-forming process and the stability of the samples motivated researchers to control voids parameters for enhancing piezoelectric performance and encouraged developments in theoretical and practical studies, conducted mainly in samples with a single dome-shape cavity.[9]

Those studies demonstrated the stiffness of the closed dome-shape voids increases with compression, resulting in reduced sensitivity and large pressure dependence.[8] The dome-shape pattern also produced a nonuniform distribution of the electric field in the cavity, inducing electrical discharges.

2.3. *Ferroelectrets with fused multi-layers*

In 2007, Altafim and Gerhard proposed an alternative voided multi-layered structure towards overcoming the nonhomogeneous field distribution in the air cavities. The approach consisted in drill holes in a polytetrafluorethylene (PTFE) film and fused it, in a hot press, with two Teflon™-FEP films, thus forming a three-layer system. The holes were mechanical drilled or perforated by Nd:YAG laser. Samples of different

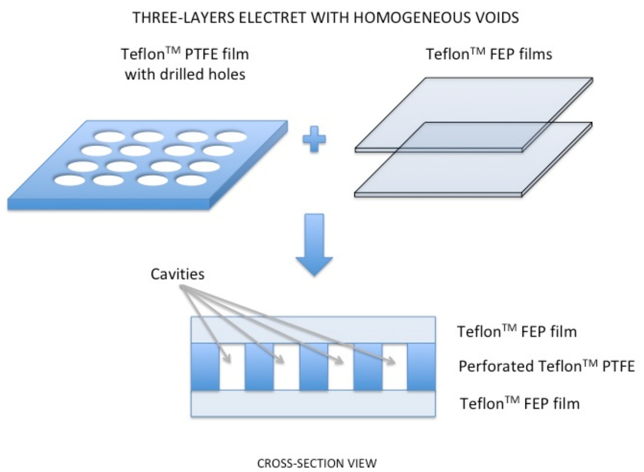

Fig. 3. Schematically representation of the assembling process for producing three-layer electrets with a perforated middle.[10]

cavity sizes and densities were produced and assembled as schematically represented in Fig. 3.[10]

Regardless of the efforts and the fact the three-layer structure provided cavities with well-defined parameters and the thermal fusing process led to voided electrets much more consistent than those initially fabricated with shellac drops, direct quasi-static measurements revealed a piezoelectric activity lower than 20 pC/N.

A better understanding of the ferroelectrets electromechanical behavior indicated the stiffness of the perforated layer as the main responsible factor for the low direct effect, pointing investigations to softer materials with more cavities and also that different measuring methods should be employed in such a sample configuration.

According to the suggestions, a new three-layer ferroelectret was proposed for replacing the perforated layer, with a PP mesh with 1.5 mm × 2.5 mm openings. A thermal-lamination process, instead of a hot press, was adjusted to fuse the mesh with PP films at 140°C for improving layer bonding. However, the newer ferroelectrets continued responding with a direct piezoelectric effect below 20 pC/N.

2.4. *Thermal-lamination with template method*

Motivated by the possibility of creating ferroelectrets with well-defined cavities, Prof. Gerhard invited Ruy Alberto Pisani Altafim, Prof. Altafim's son, to collaborate more closely with his group at the Physics and Astronomy Institute at the University of Potsdam, in Germany. There Altafim developed a method according to which a rectangularly perforated PTFE template of relatively higher melting temperature was thermally laminated with two Teflon^{TM}-FEP films. During the lamination process, the Teflon^{TM}-FEP films were fused through the template openings. After discarding the template a consistent FEP structure was created with open-tubular channels, as shown in Fig. 4.[11]

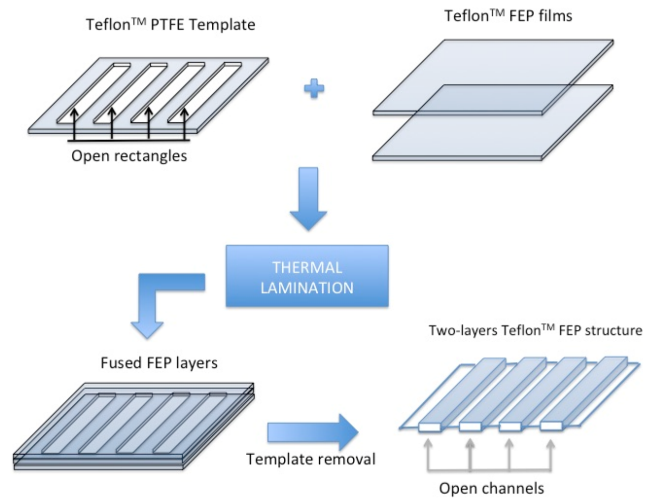

Fig. 4. Schematic representation of the method for producing open-tubular channels FEP structures using thermal lamination and a PTFE template.[11]

The Teflon^{TM}-FEP structure was evaporated with aluminum electrodes and received proper electrical charging. The results were ferroelectrets with well-controlled cavities, high thermal stability, and piezoelectric coefficient as high as those of ETMF.

Later, in Brazil, samples with open-tubular channels were investigated by a Michelson interferometer regarding their mechanical resonances.[12]

2.5. *Pressure-molding and thermal-lamination*

The Brazilian group continued working towards improvements in the method for the production of dome-shaped ferroelectrets. The research conducted by Prof. Altafim and Daniel Rodrigo Falconi concerned the pre-molding of a FEP film into dome shapes without the use of vacuum pump or hot press.[13] Instead, a soft rubber and a Teflon^{TM}-FEP film were compressed against a metal grid in a hydraulic press, as shown in the diagram in Fig. 5. The metallic matrix containing circular holes spaced 5.5 mm from each other determined the bubbles diameter and the pressure applied on the press allowed control the dome height. The pre-molded Teflon^{TM}-FEP film resulted from the process was assembled with another Teflon^{TM}-FEP film and fed into the thermal-lamination machine. After this process a Teflon^{TM}-FEP structure with cavities molded into dome-shapes regularly distributed was obtained, as schematically represented in cross-sectional view as shown in Fig. 5.

Pre-molded compressed samples showed significant improvements in cavities homogeneity when compared with those produced by the vacuum thermo fusing process. However, the heat dissipation during thermal-lamination occurs differently on both Teflon^{TM}-FEP films and the aluminum matrix, causing sample distortions after lamination.

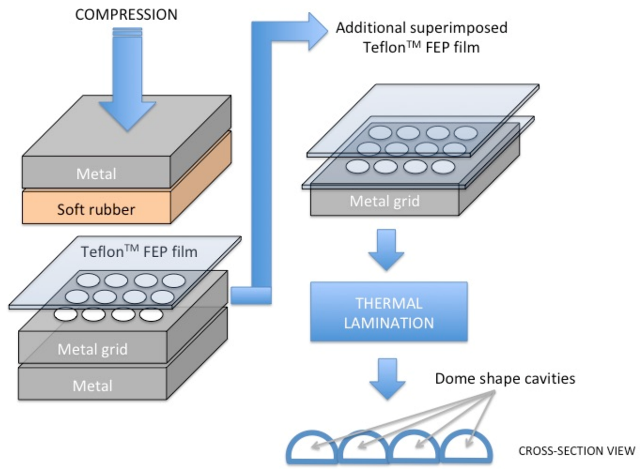

Fig. 5. Schematic representation of the process for pre-molding FEP films with a dome-shape pattern followed by the double-layer assembling, the thermal-lamination method and a cross-sectional view of the dome-shape cavities.[13]

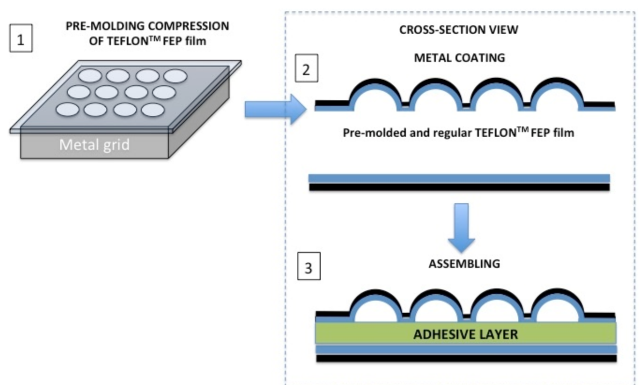

Fig. 6. Schematic process for producing ferroelectrets with an adhesive middle layer, consisting in: Step [1] pre-molding, Step [2] metal coating and Step [3] assembling.[14]

Towards overcoming those deformations, in 2011, an adhesive layer placed between the pre-molded and nonmolded film was used instead of thermal-lamination (Fig. 6).[14] The ferroelectrets were highly effective for most applications, however the lower thermal resistance of the adhesive layer was inconsistent with applications that required temperatures above 100°C. Therefore, the thermal-lamination method with templates remained the Brazilian group's choice for studies and development of ferroelectret applications.

2.6. *Patterned hot press and water-pads*

Since the thermal-lamination with template method was more frequently adopted, the use of PTFE templates clearly became a concern due to the amount of discarded material.

Therefore, instead of templates, Teflon™-FEP water-pads molded and patterned in a hot press were used. The method

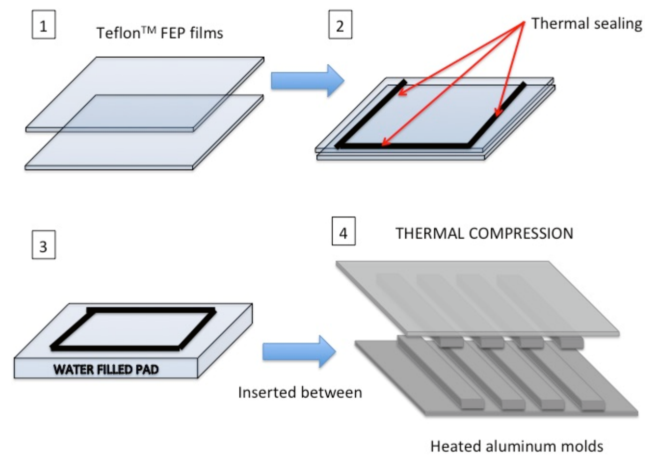

Fig. 7. Schematic representation of the water-template process with the pad fabrication (steps [1], [2], and [3]) and thermal compression between heated aluminum molds (step [4]).[15]

consisted in compressing two heated stamped metal plates over a prefabricated water-filled pad made of Teflon™-FEP films. The pads were produced cutting 4 cm × 4 cm films and sealing their edges for creating a Teflon™-FEP bag. Distilled water was injected over the side open, which was later sealed, thus forming a water cushion. The water-pad was placed between two patterned plates compressed by a preheated press. During compression at elevated temperature, the heating water increased the pad's internal pressure, pushing the FEP film against stamped metal plates. The increased pressure inside the pad unsealed it, which expelled water vapor, thus helping the creation of a Teflon™-FEP structure with open-tubular channels.[15] A schematic representation of the process is shown in Fig. 7.

The method with water-pads provided an efficient solution for the problem of discarded templates, producing multilayer ferroelectrets with well-defined and regular cavities. However, samples with different patterns are required for investigations on influences of the cavity geometric parameter of the piezoelectric activity.

Although the method can produce different sets of samples by simply replacing the molded plates, it requires fine machining, which is costly and time-consuming and may not always be available.

Layered manufacturing, also known as 3D printing, is a manufacturing process that reduces prototype timing and costs.

2.7. *Printing ferroelectrets*

Searching for alternative methods to create electret structures with well-defined cavities and exploring the benefits of layered manufacturing, the Brazilian group modeled a voided double-layered ferroelectret to be printed by a standard 3D-printer. The structure, similar to those founded in perforated middle layer electrets, was designed with two parts as

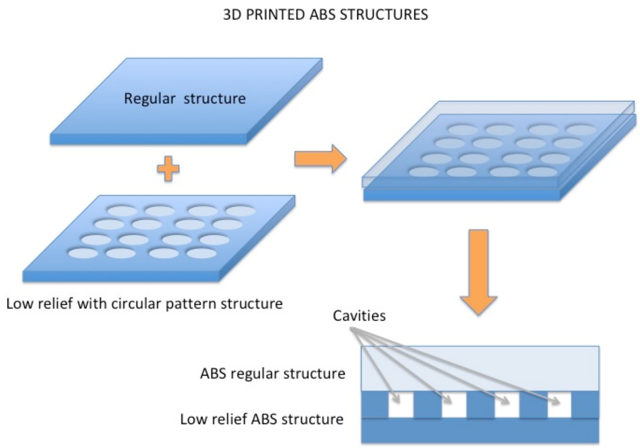

Fig. 8. Representation of the two-part designed model for 3D printing. One part consisting of a nonvoided film and the other in a film with a low relief pattern. In the sequence the printed layers are assembled and a cross-sectional view of the regular cavities is presented.[16]

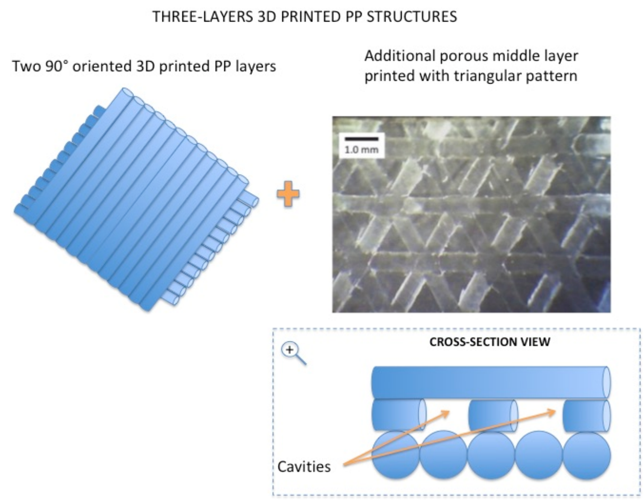

Fig. 10. 3D-printed model for a three-layer design, with a magnified view from the triangular pattern formed by increasing the layer porosity. And a cross-sectional representation of the cavities.[18]

shown in Fig. 8. One designed as a regular base film, and the other with a low relief pattern with circular shapes.[16]

A 3D-printer model Wanhao-Duplicator 4X of 0.1–0.5 mm layer resolution was chosen. After printing the frames with acrylonitrile-butadiene-styrene (ABS) in a single step, they were assembled towards creating a two layers structure with closed circular voids. The result was ferroelectrets with a piezoelectric coefficient d_{33} of approximately 100 pC/N.

The assembling step was considered a disadvantage of the process causing reproducibility issues; therefore, a more direct approach was proposed. Instead of ABS, PP was used as the printed filament. And two orthogonally superimposed layers were printed in a single step, as shown in Fig. 9. According to the printing process, the layers are formed by parallelly extruded filaments providing a corrugated surface; consequently, by printing two perpendicular layers air cavities were formed between them.[17]

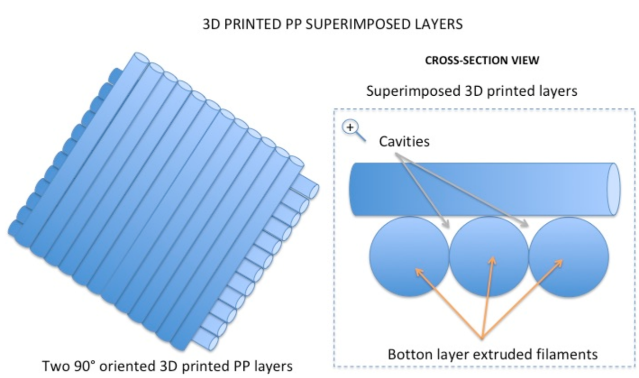

Fig. 9. Representation of the 3D-printing process with two orthogonally oriented layers and a zoom view on how the cavities were formed.[17]

Eventually, a regular printing pattern was obtained in PP samples; however, the process did not consider the possibility for controlling geometrical void parameters.

Cavity control was obtained by printing three-layer structures. The first and third layers were printed as previously described, with parallel extruded filaments. The middle layer, however, was set with a different porosity, as shown in Fig. 10. Such a procedure is normally employed in 3D-printing for reducing the amount of material. Another advantage of this is to adjust the porosity pattern with different geometrical holes. Here, triangular pores were chosen to construct the middle layer. The resulting three-layer ferroelectret, exhibited piezoelectric coefficients d_{33} of approximately 200 pC/N and a thermal stability similar to those of previously described PP ferroelectrets.[18]

The vast possibilities of the 3D-printing inspired other research groups, and three-layer printed ferroelectrets have been continuously investigated.[19]

3. Ferroelectrets with Magnetic Properties

Focused on exploring other ferroelectret possibilities, Altafim *et al.* covered the open-tubular channel ferroelectret with a magnetic layer towards creating a ferroelectret that was also sensitive to magnetic fields. According to his conception, a magnetic layer would be attracted by a magnetic force, causing mechanical deformations in the tubular channels, hence an electrical response. Therefore, samples with magnetic stripes placed above the open-channels were prepared for demonstrating the concept. The fabrication process is schematically represented in Fig. 11, and is very similar to the lamination method employed on the fabrication of open-tubular channels.[20] The difference regards on the additional magnetic stripes placed above the channels and the third

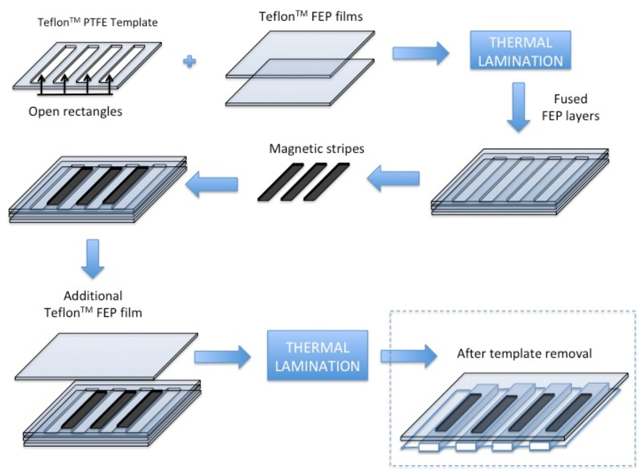

Fig. 11. Schematic representation of the process for creating ferroelectrets with magnetic sensitivity.[20]

layer of FEP film that covers the final structures to ensure a regular surface for metal coating.

Ferroelectrets with magnetic layers were exposed to different external magnetic fields, and electrical responses observed resulting in a novel piezoelectric-magnetic behavior. Since then, studies on the sensitivity of "piezoelectric-magnetic electret (PME)" and applications for detecting magnetic fields from electrical current have been developed.

4. Applications with Ferroelectrets

4.1. *Acoustic transducer for partial discharges*

In the late 90s, Hillenbrand and Sessler presented a theoretical study relating ferroelectret electromechanical behavior to the electret microphone theory[21] and, since then, different ferroelectret applications of acoustic transducers have been published.[22–24]

Ferroelectrets were investigated as ultrasonic transducers for the detection of electric partial discharges in air. A ferroelectret with open-tubular channels was mounted on a shielded aluminum case with a built-in high-impedance amplifier. The transducer was calibrated according to ASTM E976-10, and partial discharges were produced by means of electrical pulses applied to a 2 mm long point-to-point spark gap placed inside a cylindrical mesh coated with foam for the avoidance of electromagnetic noise and acoustical echoes. The spark gap device was initially mounted inside an anechoic chamber and later placed in a metal box for simulating a discharge inside a distribution transformer. Results showed the high efficiency of the transducer in detecting partial discharge in both cases.[25]

Other ferroelectrets configurations were employed for the acoustic detection of partial discharges in a comparative analysis[26] and further used in partial discharges produced from a single electrode and from multiple point-to-point electrodes arranged with different air gaps.[27]

4.2. *Hydrophone for vibro-acoustography*

The close acoustic impedance of ferroelectrets to air and water and the high piezoelectric coefficients have made them a suitable transducer for ultrasonic underwater applications. Such characteristics have influenced the development of a ferroelectret hydrophone to be employed in ultrasonic imaging techniques, such as vibro-acoustography, which is used for medical diagnoses or material analyses.[28] In this study, a vibro-acoustography setup was set according to the representation in Fig. 12(a). The ferroelectret transducer (TRU) was initially placed in front of a vibrating object (target) and later displaced in the *XY* directions in small steps. The vibration signal amplitude is recorded for each new transducer position and this data is processed later into images. Figure 12(b) shows the resulting image from a small metallic sphere used as a target while the TRU was used as a hydrophone.

4.3. *Acoustic transducer for the detection of oil contamination*

Ferroelectret hydrophones were also used for investigations on a novel method that could determine the quality of mineral

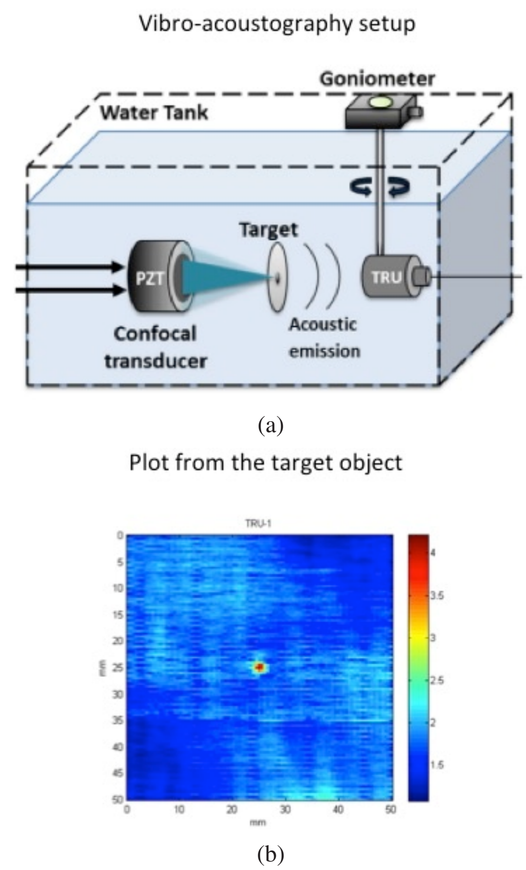

Fig. 12. (a) Vibro-acoustography setup assembled with a hydrophone built with a ferroelectret transducer (TRU) for capturing the target vibrations. (b) Resulting image of a small spherical object produced from the scanning with the TRU.[28]

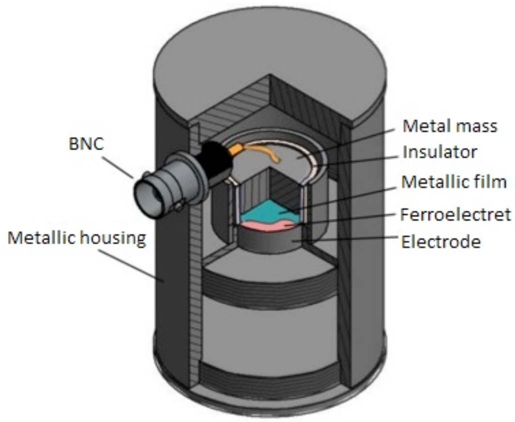

Fig. 13. Schematic assembling of the mass-sensor built with an open-channel ferroelectret.[31]

oil used in electrical power transformers.[29] Such oils are constantly contaminated with moisture, residual insulating material, etc. and, depending on the amount of contaminant, their density modifies their ultrasound characteristics. In this study, the recorded ultrasonic signals from the ferroelectret hydrophone were supervised according to a machine learning technique that evaluated their differences. The results showed the oils were successfully classified by the method.

4.4. *Accelerometer of thermo-formed ferroelectrets*

Sessler *et al.* showed the viability of producing accelerometers using ferroelectrets.[30] A device that stimulated the Brazilian group to develop a similar prototype using open-channel ferroelectrets. The mass-sensor, built in aluminum housing, is schematically represented in Fig. 13. The performance test was conducted on a standard shaker driven with frequencies up to 4.5 kHz. Results indicate a linear behavior for frequencies between 1.1 kHz and 4 kHz, corroborating with those from commercial accelerometers.[31]

5. Conclusions

This paper provided a historic background on how two unrelated research studies became complementary. A history initiated with studies on electret charging methods led by Prof. Bernhard Gross evolved into an impulsive charging system developed by Prof. Altafim and met the investigation on dielectric barrier discharges inside cellular electrets, conducted by Prof. Gerhard. A lack of samples motivated Prof. Altafim's group to create alternatives for producing ferroelectrets, which resulted in consistent methods for the production of thermally stable samples with well-defined cavities. Difficulties faced by each system were addressed together with the knowledge accumulated in the process. Descriptions of the most important ferroelectret applications developed by the Brazilian group were highlighted and demonstrated the benefits of a great partnership.

Acknowledgments

The fundamentals for the electret research developed at São Carlos Institute of Physics were crucial for the start of ferroelectret research in Brazil. However, its development was possible only after the approval of a Probral CAPES-DAAD project, coordinated by Prof. Reimund Gerhard and Prof. Ruy A. C. Altafim. The program enabled the exchange of researchers from both countries, fully collaborating to the creation of basic and high-standard science, as illustrated here. Therefore, the authors acknowledge FAPESP, CNPq, and CAPES for funding the present research.

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