Enhanced piezoelectric performance of ceramic-polymer composite cantilevers with thin metal substrates

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ABSTRACT

In this work the electromechanical properties of lead zirconate titanate-poly(vinylidenefluoride-trifluoroethylene) ceramic-polymer composite on thin brass and steel substrates were investigated. Samples were stencil printed on metal foils and cured at 120 °C. The effective transverse piezoelectric coefficient (d_{31eff}) was calculated by utilizing the converse piezoelectric effect and measuring the displacement of a cantilever sample's tip in an electric field. Interestingly, the results showed improved piezoelectric properties with the stiffer steel substrate samples. The highest d_{31eff} achieved was about -22 pm/V, which was 29 % higher than in samples printed on brass foil (-17 pm/V). Both are substantially higher compared to the coefficients reported with similar ceramic-polymer composites on polymer substrates. The improvement is suggested to originate from the prevention of buckling effects and more effective bending deformation, while the structure remained flexible. Due to the high effective values of d_{31} and g_{31}, the developed material and cantilever structures are feasible for both sensor and energy harvesting applications.

Cantilevers or beams are widely used designs in piezoelectric applications such as energy harvesting, sensors and actuators. They can be categorized by their structure into the following groups: unimorph, bimorph or multimorph.[1–13] The number of layers, their thickness and the Young's modulus ratios between the layers greatly affect the electromechanical behavior of the piezoelectric cantilever which also can easily be seen as a change in the cantilever's stiffness or bendability. This is especially true if the piezoelectric layer is soft polymer or ceramic-polymer composite and the passive layer is bulk ceramic with a high Young's modulus. However, to observe the changes in electromechanical behavior when combining such materials, careful measurements and calculation are required. Many research groups have developed models and equations to describe these properties in multilayer piezoelectric structures and to enable more accurate design of piezoelectric cantilevers for specific applications. Steel et al. reported the quasistatic response of piezoelectric ceramic-metal unimorphs, which they called "heterogenous bimorphs", in 1978[3] and later in 1991 Smits and Choi developed the "constituent equations of piezoelectric heterogeneous bimorphs"[14]. Wang and Cross et al. have furthermore published many equations describing the detailed properties of both bimorph and unimorph cantilevers such as maximum displacement, electromechanical coupling and maximum generated force as a function of layer thickness and Young's modulus ratio[15–18]. They have shown, for example, that with a unimorph structure the blocking force and electromechanical coupling coefficient increase when the Young's modulus ratio of passive and active materials increases[18]. The work of the aforementioned groups is widely referenced when piezoelectric cantilevers have been studied and their calculation models developed further[5, 6, 8,19].

When utilizing any of the developed models or equations, one should bear in mind that the equations might have very different boundary conditions and assumptions about idealized measurements or setups. For example, with unimorph cantilevers it is very often assumed that the thickness of the cantilever is much smaller than its width and length, the electrode layers are so thin that they do not contribute to the mechanical behavior of the cantilever, the applied electric field is evenly distributed along electrode area and the layers of the cantilever do not slip relative to each other during bending.[3, 4, 18, 5–7,13–17] Also, the fixing of the cantilever should be taken into account, for example the unimorphs may be studied by clamping samples from the center (knife edge clamping)[3] or from one end leaving the other free, the latter being more commonly used in cantilever piezoelectrics.

Even though there is a high level of interest in developing piezoelectric cantilevers, most of the reports still concentrate on sintered ceramic piezomaterials[1,6,7,19] and only few can be found for polymer based curable inks or pastes[8,9,11]. Furthermore, even fewer investigations can be found about piezoelectric unimorphs where the active layer of polymer or composite is directly printed onto metallic substrates (or substrates with a much higher modulus than the polymers). The reason might be that the effect of high Young's modulus substrates on a low Young's modulus piezoelectric layer seems to be self-evident; the stiffer substrate material will hinder the effective piezocoefficients. Interestingly, there has been a contrary effect reported in the case of piezoelectric composite unimorph cantilevers in bending mode[20,21]. Evidently, the substrate should not have too low a Young's modulus, because the extensional deformation needs to be suppressed strongly enough to provide the maximum output energy and electromechanical coupling in piezoelectric unimorphs, as shown in[3,18].

In this paper, the active composite layer was directly printed on stainless steel and brass substrates and their electromechanical properties were analyzed. The equations and boundary rules described by[3,14–18] for cantilever unimorphs were used in the calculation and extraction of electromechanical properties from the measurement results. The findings show that by careful selection of the substrate, the effective piezoelectric properties can be substantially enhanced without compromising the flexibility of the unimorph.

Lead zirconate titanate (PZT) ceramic particles (PZ29, Ferroperm Piezoceramics A/S & Meggit, Denmark) were used as the filler material and ferroelectric poly(vinylidene fluoride-trifluoroethylene) copolymer (P(VDF-TrFE) 56/44 mol.%, Solvay-Solexis, Belgium) was used as the matrix in the ceramic-polymer composite ink. The average particle size of the ceramic particles was about 1.3 μm. After drying the filler and matrix material powders, the appropriate amount of PZT was weighed to result in a 48 vol.% filler to matrix ratio in the ink. Prior to the mixing of the ceramic and polymer, PZT particles were coated with a polymeric surfactant (Malialim AAB-0851, NOF Co., Japan). The more detailed formulation of the composite ink can be found in our earlier publication[21]. The composite was stencil printed with a 200 μm thick steel stencil on 50 μm thick metal substrates (stainless steel with 18 % Cr 9 % Ni and brass, 63 % Cu 37 % Zn) and cured at 120 °C for 30 minutes. The top electrode was screen printed with a low temperature curable silver ink and cured according to the manufacturer's instruction (5064H, DuPont, USA). After printing and curing the samples were laser cut into cantilevers having widths of 3 mm or 7.3 mm and a length of 26 mm. Figure 1 show the samples after fabrication and an example of the flexibility of the samples.

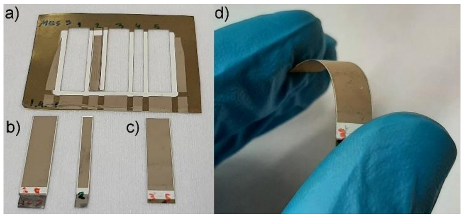

Figure 1. a) Brass foil from which cantilever samples are laser cut, b) laser cut unimorphs fabricated on steel and c) on brass substrates, d) Bent sample with brass substrate showing flexibility of the fabricated cantilevers.

The Young's modulus of each material was measured by using a tensile stress testing stage (Linkam TST350). The strain rate in the measurement was adjusted to 1 % per minute. The sample width and length in the Young's modulus tests were 6 mm and 88 mm, respectively. In the case of the composite ink, samples were first cast on a mylar foil, cured and cut into samples prior to testing. The thickness of the steel and brass foils used was 50 μm. A silver based top electrode with thickness of 8-14 μm, printed on top of the cantilever samples, was not applied for samples in the measurement of Young's modulus, nor considered in the calculation of d_{31eff}. The layer thickness of the printed and cured composite samples was investigated by a polarized light microscope from the cross-section of each sample.

The dielectric properties were measured before poling with a LCR meter (E4980AL, Keysight Technologies, USA) from 100 Hz to 1 MHz with a 1 Vpp signal amplitude. The hysteresis loops and remanent polarization were measured up to 20 V/μm electric field using a 400 ms bipolar triangular signal with a high voltage ferroelectric tester (Precision 10kV HVI-SC, Radiant Tech., USA). Prior to the piezoelectric measurements the samples were poled in a 20 V/μm electric field at room temperature for 1 hour.

The electromechanical properties of the samples were measured utilizing the converse piezoelectric effect. Laser-cut cantilever samples were clamped from one end in the measurement setup. Electric fields with a sinusoidal waveform and frequencies between 15–60 Hz were applied to the sample electrodes with a signal generator and a high voltage amplifier. The displacement of the free sample tip was measured with a Laser Doppler-vibrometer (OFV-552 measuring probe, Polytech GmbH, Waldbronn, Germany) and an oscilloscope using 64 waves averaging. All piezoelectric measurements were done at least 24 hours from poling, but within 48 hours to avoid possible influence of aging on the results. The piezoelectric coefficient d_{31eff} was calculated using constituent equations and notations derived in[14,18]:

$$\delta_0 = \frac{3L^2}{t_p^2} \times \frac{AB(1+B)}{A^2 \times B^4 + 2A(2B+3B^2+2B^3)+1} \times d_{31} V_0 \qquad (1)$$

where L is the tip length, t_p is the tip thickness, V_0 is the input voltage, δ_0 is the tip deflection, and parameters A and B are defined as:

$$A = \frac{E_m}{E_p} \qquad B = \frac{t_m}{t_p} \qquad (2)$$

where E is Young's modulus, t is layer thickness and subscripts m and p correspond to the substrate layer (steel and brass) and the piezoelectric composite layer, respectively.

Figure 2 shows the dielectric properties of typical samples fabricated on brass and steel foil substrates between 100 Hz and 1 MHz. At 1 kHz the relative permittivity ε_r was about 59 and 58 (± 3 %, grey shadings in Fig. 2) and the dielectric loss tangent 0.036 and 0.028 (± 8 – 11 %, red shadings in Fig. 2). An uncertainty of 1 µm in the determination of thickness of the cured composite layer in the samples caused about 3 % error in the calculations of relative permittivity from the capacitance and in the level of electric field (ferroelectric measurements, poling and d_{31eff}). The decrease of permittivity as a function of frequency is typical in dielectric composites due to the different polarizations present in different frequency ranges. Furthermore, the increase of the dielectric losses, that begins at frequencies higher than about 10 kHz and continues further beyond 1 MHz, is related to the dielectric relaxation of the so-called β_1 phase of the P(VDF-TrFE). The dielectric properties of different P(VDF-TrFE) blends have been comprehensively reported earlier by T. Yagi et al.[22]. Compared to earlier studies with a similar composite, but with no surfactant treatment on filler particles, the results showed slightly lower permittivity and losses, -14 % and -45 %, respectively[20]. This is understood to be the result of interface passivation and smaller interface polarizations on the filler particles due to the surfactant.[21, 23, 24, 25] Thus, the differences between samples were very small, and the substrate material did not affect the dielectric properties.

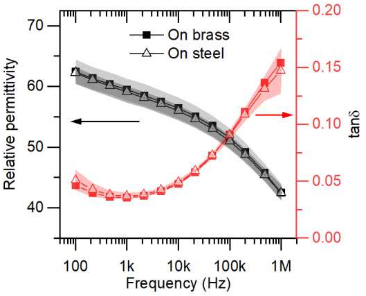

Figure 2. Relative permittivity and dielectric loss tangent of the cantilever samples as a function of measurement frequency (100 Hz to 1 MHz). Typical response for ceramic-polymer composite is observed with copolymer relaxation (peak beyond the measurement range). Shadings represent the standard deviation of the measured samples.

The typical results of P-E measurements (hysteresis loops) are shown in Figure 3. All samples showed a clear ferroelectric response (P-E hysteresis loop) in polarization vs. electric field measurements, but a saturated hysteresis loop was not observed within the maximum electric field (20 V/μm) used. It was also noted that many samples had breakdowns around 20 V/μm, especially with steel substrates, hence higher fields were not measured. The reason for the breakdown field could be in defects in the composite layer thickness. However, there were no visible defects found in the samples and no large deviations in results were observed in small signal measurements, thus the possible defects were not regarded as major. The measured remanent polarization increased nonlinearly with the increasing electric field and showed a rapid increase in higher electric fields. This is a result of two effects. Firstly, the electric field needed to align the dipoles in P(VDF-TrFE) copolymers is rather high (about 60 V/μm), while in the case of PZT less than 2 V/μm is needed[26, 27]. Secondly, the electrical coupling is compromised due to the large differences in the permittivity and conductivity of the matrix and filler materials. Thus, the electric field acting on the filler particles is much lower than the field applied to the sample electrodes.[26, 28] The average remanent polarizations for samples on brass and steel substrates were 1.7 and 1.5 μC/cm² (standard deviation: 0.125 and 0.21 μC/cm²) at 20 and 19 V/μm, respectively.

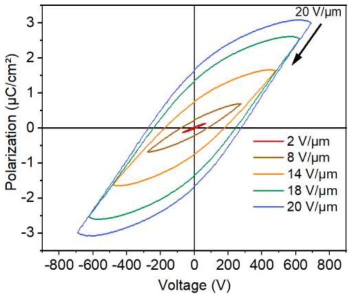

Figure 3. Typical hysteresis loop acquired in P-E measurements with brass and steel substrates in maximum electric field from 2 to 20 V/μm.

In Figure 4a there are results from vibrometer measurements, showing the results for typical sample cantilevers with brass and steel substrates (peak to peak displacement and electric field). Furthermore, the maximum and minimum values of all samples are marked with shaded colored areas. For all samples on steel and brass, the tip deflection changes linearly with increasing electric field indicating typical piezoelectric behavior (linear fitting for visible curves showed R^2 >0.9998). However, there is a clear difference in the levels and slopes of the cantilevers with brass and steel substrates in Figure 4. This is a consequence of the different stiffnesses of the cantilevers derived from Young's modulus of the substrate materials (brass vs. steel). The bending stiffness of steel is about 63 % higher in samples with similar length and width and layer thickness (Supplementary Information, Section 3 and Table S2). Within similar sample structures standard deviations of 8 % and 14 % in tip displacement were observed in samples on brass and steel, respectively. This deviation is partially caused by the slight variations in composite layer thicknesses (inertia of the cantilever in bending) and the length of the free end of the cantilever (accuracy ±0.1 mm) between individual samples. However, these effects are taken into account while calculating with equation 1, thus not causing such high deviations in the calculation of the piezoelectric coefficient. In Figure 4b the d_{31eff} calculated from the displacement measurement is plotted with the critical stress needed for buckling to emerge in these cantilevers. The calculation of critical stress for buckling of unimorph cantilevers can be found in Supplementary Information (Section 3 and Table S3). Along with the results acquired with the steel and brass substrates, there are also results from our previous investigation with a similar piezoelectric composite but with polymer and reinforced polymer substrates[21]. The critical stresses have been calculated from the actual dimensions of the measured samples, which best represented the average results of similar samples, thus the dimensions of the cantilevers varied between different cantilever structures. As can be seen in the figure, the higher piezoelectricity follows the increasing substrate stiffness in these cantilevers. The effective piezoelectricity in these printed composite cantilevers was deduced to originate from the ceramic particles by observing the phase of the cantilevers' tip displacement and the applied electric field. Thus, the d_{31eff} is negative in the samples. The highest d_{31eff} achieved with a steel substrate was -22 pm/V (1.6 pm/V standard deviation), while with a brass substrate it was found to be -17 pm/V (0.8 pm/V standard deviation).

Both show substantially higher d_{31eff} (by factors of 4.3 and 3.3, for steel and brass substrates, respectively), than that achieved with similar ceramic-polymer composites on polymer substrates. Compared to a steel reinforced cantilever (-56 pm/V), the results obtained here with single metal substrates were lower. However, the change in piezoelectric coefficient is similar when comparing the PET substrate cantilevers, as it follows the increasing bending stiffness.[21] Interestingly, there is only a small difference in critical stress between the PET and brass substrate samples regardless of a large difference in the substrates' Young's modulus. This is due to the slightly different dimensions of the cantilevers, but the increase in d_{31eff} is over 200 %. With PET substrates, the stress caused by shrinking of the composite layer in curing can stress the substrate close to buckling and interfere with the bending when actuated with an electric field[21]. Now the relatively small difference in critical stress between the PET and brass substrates seems to be enough to prevent buckling from enhancing the bending motion. The reinforced PET, however, possesses remarkably higher critical stress. Thus, the bending stiffness for this structure is much higher when compared to others. Yet, in this case it is likely that preventing the buckling is not the only effect to result in such high piezoelectricity. Instead, the straightening of the cantilever might also have an influence when the PET substrate is being reinforced with steel foil. This effect, however, needs more investigation.

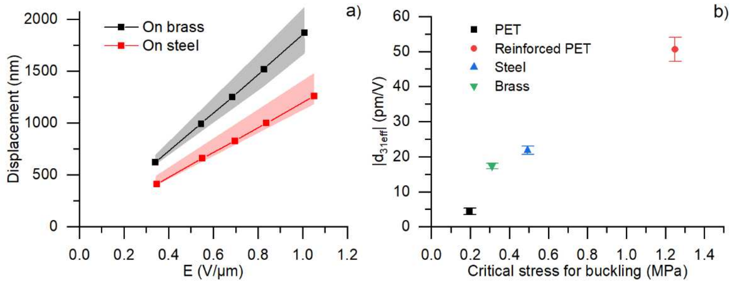

Figure 4. a) Displacement (average of 64 waves, peak to peak) of typical composite unimorph cantilever tips with brass (black) and steel (red) substrates when excited with electric field from about 0.3 to 1 V/µm (peak to peak) at 10 Hz showing linearity and effect of substrate stiffness. Grey and light red shadings present the maximum and minimum values in brass and steel substrate cantilevers, respectively. **b)** Effective piezoelectric d_{31} and critical stress for buckling in unimorph cantilevers with the same ceramic-polymer composite, but with different substrates: PET[21] and metallic.

Table1 gathers the results obtained here, from our earlier study with polymer substrates, and similar ceramic-polymer composites, P(VDF-TrFE) and PVDF[21, 26, 29, 30]. The piezoelectric polymer films are used here as reference materials due to the lack of published investigations on d_{31} in printed piezoelectric cantilever structures. It should be kept in mind that comparison of the properties of cantilevers with different structures (such as without a substrate or with different stiffnesses) or by using a different measurement method (converse vs. direct effect) is not as straightforward as comparison of the intrinsic properties of materials. The structure has a great impact by, for example,

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mechanics and bending stiffness in cantilevers as seen also in this study. Nevertheless, these materials in Table 1 provide a broader point of view to the results obtained with the printed bendable cantilevers. As can be seen in Table 1, the composites without a substrate have a d_{31} comparable to the printed composite with a PET substrate. It is much smaller than that achieved with stiffer substrates. When the d_{31eff} of cantilevers with metal substrates and d_{31} of PVDF and P(VDF-TrFE) films are compared, it is seen that the oriented or stretched films possess a similar level of piezoelectricity to that the cantilevers with steel substrates. In polymers~~,~~ this is highly dependent on the crystallization, which in turn can be enhanced greatly by orienting the film[29, 30]. However, with printable piezoelectrics orientation by stretching is very difficult to implement in fabrication.

The piezoelectric voltage coefficient (g_{31}) is often used to evaluate the suitability of the cantilevers for sensor or harvester applications, and it can be calculated by dividing the d_{31eff} with the permittivity. The calculated g_{31} values are also shown in Table 1. Since the permittivity of the composite is low and the d_{31eff} high, g_{31eff} for the cantilevers on metals becomes very high: -40 mV/mN and -31 mV/mN, respectively for steel and brass (ε_r at 100 Hz). These are substantially larger than the coefficients calculated for cantilevers with a polymer substrate. This is mainly due to the differences in charge coefficient, since the permittivities between these are close to each other. The piezoelectric polymers possess higher voltage coefficients than the studied cantilevers because they have very low permittivity, but again the structures of these samples are different. From the application point of view it is also good to mention that metallic substrates enable more freedom to design component dimensions and resonance frequencies, which is central, for example, in wideband energy harvesters[31].

Table 1. Electromechanical properties of the ceramic-polymer unimorphs with steel and brass substrates

Material	Substrate	Filler (vol%)	ε_r (1kHz)	tan δ (1kHz)	P_r (μC/cm^2)	d_{31} (pm/V)	g_{31} (mV/Nm)	Ref.
PZT-P(VDF-TrFE)	Steel	48	59.1	0.032	1.58	-22	-42	This study
PZT-P(VDF-TrFE)	Brass	48	58.7	0.032	1.62	-17	-32	This study
PZT-P(VDF-TrFE)	PET	48	58	0.03	1.6	-5.1	-10 ^{1}	[21]
PZT-P(VDF-TrFE)	PET reinf.	48	58	0.03	1.6	-56	-109	[21]
PZT-P(VDF-TrFE)^1	no sub.	45	52	-	-	-3.7	-8 ^{3}	[26]
PZT-P(VDF-TrFE)^1	no sub.	51	72	-	-	-4.6	-7 ^{3}	[26]
PZT-P(VDF-TrFE)^1	no sub.	51	72	-	-	-8.0 ^{4}	13 ^{3}	[26]
P(VDF-TrFE)^2 unstretched	no sub.	-	14	-	-	8.0	75 ^{3}	[29]
P(VDF-TrFE)^2 stretched	no sub.	-	14	-	-	25	235 ^{3}	[29]
PVDF, Biaxially Oriented	no sub.	-	-	-	11.6	11–22 ^{5}	-	[30]

*GPa, ^{1} copolymer molar ratio 70/30, ^{2} copolymer molar ratio 55/45, ^{3} calculated with the data from the reference, ^{4} only ceramic phase poled, ^{5} stress dependent.

Piezoelectric ceramic-polymer composite cantilevers were fabricated by stencil printing on two metal substrates: brass and steel. Due to the thinness of the substrates (50 µm), the structure remained flexible and the fabricated unimorph cantilevers showed clear piezoelectric properties in converse piezoelectric measurements. The effective transverse piezoelectric coefficient was found to increase with the stiffness of the metal substrate (steel vs. brass). The measured d_{31eff} values for steel and brass substrates were -22 pm/V and -17 pm/V, respectively and higher by factors of 4.3 and 3.3 than those reported earlier with similar composites on polymer substrates. The highest achieved d_{31eff} corresponded to 9 % of the piezoelectric filler ceramic. The results showed that by careful design (selecting a thin enough substrate and its Young's modulus accordingly), the effective piezoelectric performance of a printed ceramic-polymer unimorph cantilever can be enhanced. In addition to the high piezoelectric response, the low permittivity makes these cantilevers suitable, for example, for sensing or energy harvesting applications.

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SUPPORTING INFORMATION

See the supplementary material for measurement results for Young's modulus of the substrate materials and the composite and the calculation of mechanical parameters of the unimorph cantilevers.

AUTHOR INFORMATION

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. The authors declare no conflict of interest.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding authors upon reasonable request.

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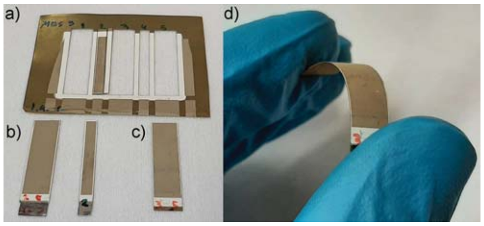
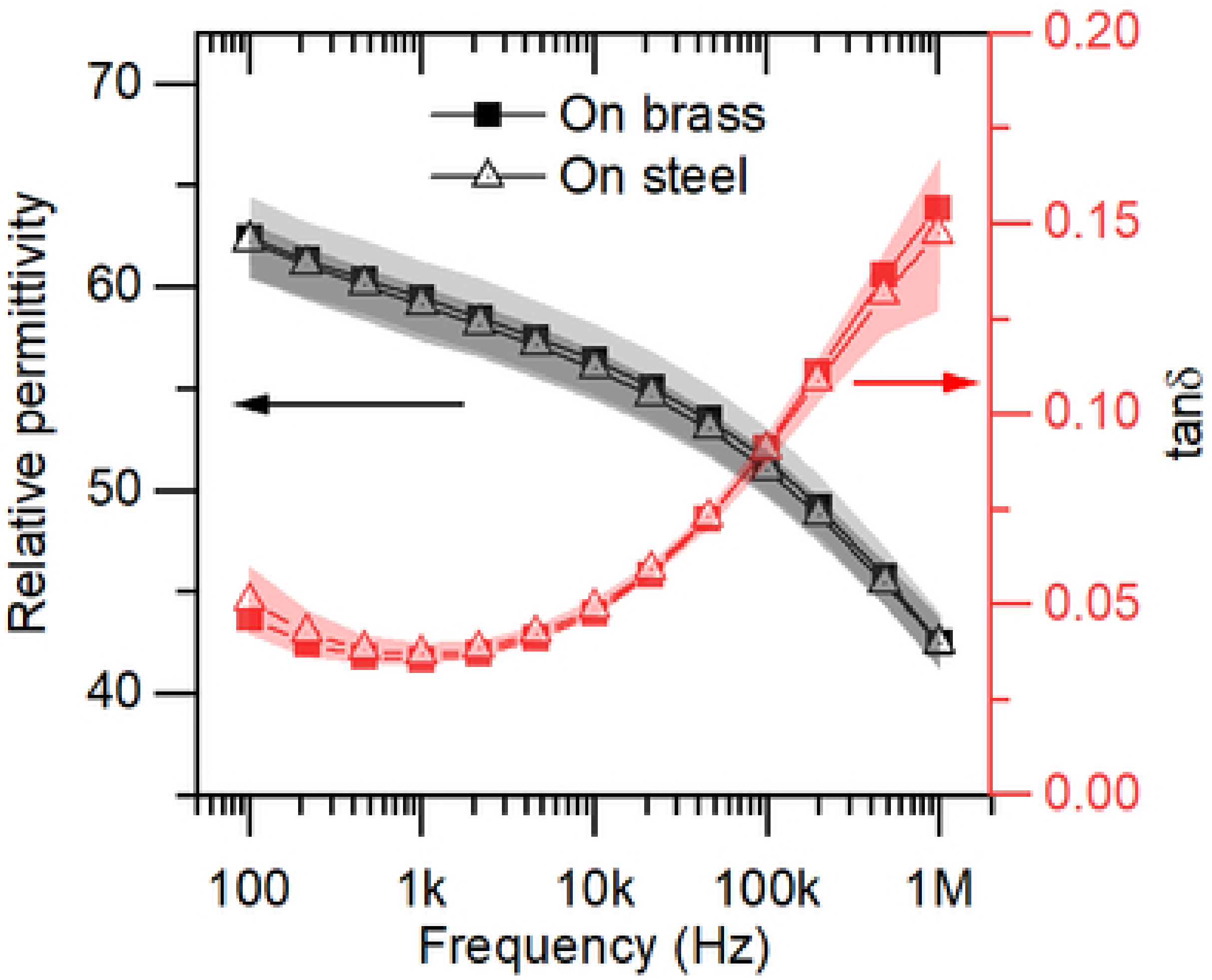
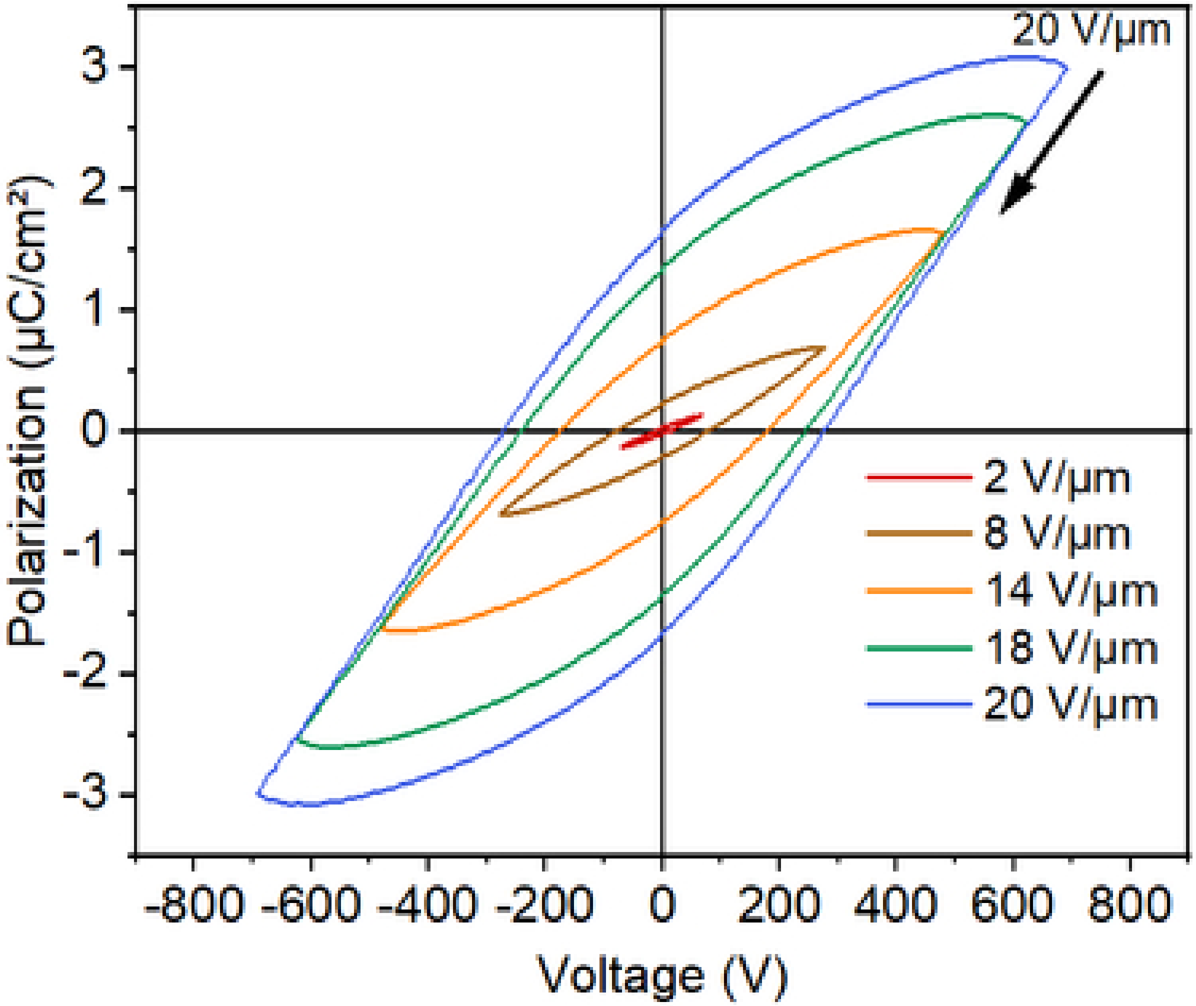
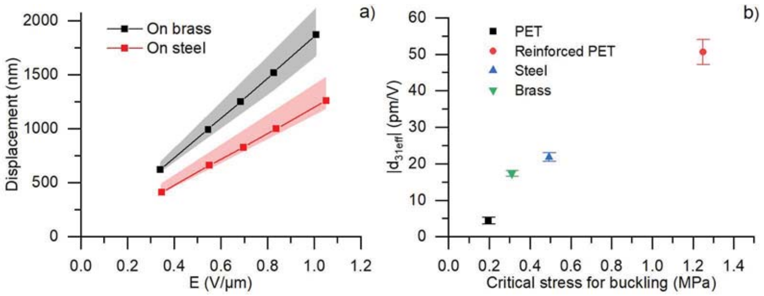



70
60
50
40
Relative permittivity
On brass
On steel
0.20
0.15
0.10
0.05
0.00
tanδ
100
1k
10k
100k
1M
Frequency (Hz)


20 V/μm
Polarization (μC/cm²)
3
2
1
0
-1
-2
-3
-800
-600
-400
-200
0
200
400
600
800
Voltage (V)
2 V/μm
8 V/μm
14 V/μm
18 V/μm
20 V/μm


a)
Displacement (nm)
2000
1500
1000
500
0
On brass
On steel
0.0
0.2
0.4
0.6
0.8
1.0
1.2
E (V/μm)
b)
|d31eff| (pm/V)
60
50
40
30
20
10
0
PET
Reinforced PET
Steel
Brass
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
Critical stress for buckling (MPa)