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Characterisation of thin layers of parylene at high frequency using PZT thick film resonators

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Abstract

High frequency electrical impedance measurements on PZT thick film structures were used to characterise thin layers of parylene for acoustic matching applications. The parylene properties (i.e. longitudinal wave velocity and acoustic impedance) were obtained in a realistic configuration for transducer applications. The measured parylene properties were compatible with medical imaging requirements, in particular for high frequency, since the deposited thickness can be controlled with high accuracy (around 1 μm). The mean longitudinal wave velocity was measured at 2135 ms^{-1} and corresponding acoustic impedance was 2.75 MRa. Three high frequency single element transducers were simulated to show that using parylene as a matching layer is a good trade-off between transducer performance and the technical process.

Keywords: Tape casting; PZT; Impedance; Mechanical properties; Biomedical applications

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1. Introduction

It is necessary to use matching layer(s) for medical imaging applications to improve the electro-acoustic performance of a transducer. The layer is placed between the piezoelectric element (acoustic impedance around 33 MRa for PZT) and the propagation medium (water 1.5 MRa). For high frequency ultrasound transducer applications (here around 30 MHz) the thickness of the matching layer, which is around a quarter wave-length, is only a few tens of microns and the corresponding acoustic properties (longitudinal wave velocity and attenuation) are thus difficult to measure accurately. Some authors have obtained parylene properties by acoustic or electrical methods.[1–3]

Parylene is a good candidate for matching layers in terms of moisture resistance, acoustic properties, deposition technology and precise control of the deposited thickness. Measurements are performed using high frequency piezoelectric thick film resonators (around 65 μm) fabricated by tape-casting (Ferroperm Piezoceramics Pz29). The fabrication process for thick films is described in next section. The method for parylene characterisation (thickness between 10 and 30 μm) is based on measurement of the electrical impedance of a multilayer structure (epoxy resin substrate, piezoelectric disc with electrodes and parylene layer) for which the only unknown properties are those of the parylene layer. The parylene properties are deduced (Section 3) from a fitting process with a KLM model (1D equivalent electrical circuit) of the electrical impedance of the whole structure and presented in Section 4. Finally, high frequency transducer simulations and corresponding performance of devices integrating tape-cast PZT thick films and parylene matching layer are shown.

2. Sample fabrication

2.1. Thick film processing

Pz29 was fabricated by a conventional mixed oxide route. The discs were manufactured by a direct method, where

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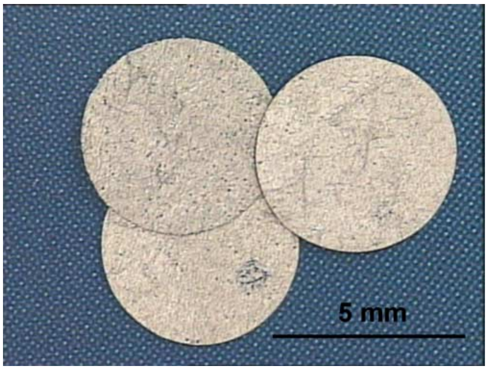


Fig. 1. Photographs of tape-cast Pz29 discs used for parylene characterisation.

time-consuming and rejection-generating steps such as lapping and polishing were eliminated. The materials were tape-cast in layers of 20–40 μm by a conventional organic solvent route. Two individual layers were then stacked in order to obtain the desired thickness (and thereby resonance frequency). Platinum electrodes of approximately 2 μm thickness were applied by a screen-printing method. The stacks were then laminated at high temperature and pressed to form a dense and homogeneous green body. Individual discs could then be punched out using a sharp circular stainless steel tool where dimensions were adjusted to correct for sintering shrinkage, so as to obtain a sintered disc of desired diameter. Subsequently, binder burn-out and sintering were performed by conventional production techniques. After sintering, poling was performed in oil for 2 min at 130 °C with an electrical dc field of 2 kV mm^{-1} (Fig. 1).

2.2. PZT thick films on substrate

Epoxy resin was used for substrate fabrication. These small cylinders have a diameter of 12.5 mm and a thickness of 19 mm. The properties of the substrate have been previously measured. The longitudinal wave velocity was determined from the measurement of the time of flight by inter-correlation between the reference signal (in water) and the signal transmitted through the epoxy substrate. Attenuation measurements were also performed by a transmission method in water with 15 MHz wideband transducers. The attenuation curve between 3 and 23 MHz is quasi-linear and by extrapolation to 35 MHz the same behaviour as a function of frequency is assumed. Characteristics are given in Table 1. An acoustically thin gold layer (around 200 nm) was sputtered on the upper face and PZT discs were bonded using the same epoxy resin, relatively high pressure being applied to the disc to minimise adhesive thickness.

Table 1
Characteristics of epoxy resin substrate

	V_l[a] (m s^{-1})	α[b] ($\text{dB mm}^{-1}\,\text{MHz}^{-1}$)	ρ[c] (kg m^{-3})
Epoxy resin substrate	2650	0.56	1170

[a] V_l: Longitudinal wave velocity.
[b] α: Attenuation coefficient.
[c] ρ: Density.

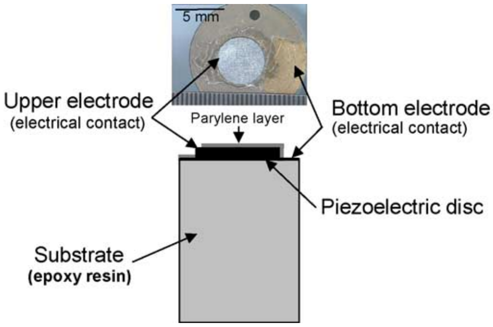


Fig. 2. Cross section representation of samples for parylene characterisation and photograph of the front face of a sample.

2.3. Parylene layer deposition

The upper faces of the piezoelectric disc were first carefully cleaned and pre-treated with Silane. The parylene C film was formed by vaporising the powdered dimer over 100 °C, creating molecular changes in the gaseous dimer by thermal energy at approximately 690 °C, and polymerisation on the chosen substrate at room temperature.[4] A small area on the upper electrode was protected before coating to maintain the electrical contact. Fig. 2 shows a cross section diagram of samples where the three elements (substrate, PZT disc, parylene layer) are represented. The sputtered gold electrode on the substrate and the glue layer are clearly seen in Fig. 3 (SEM photograph). The mean thickness of the adhesive layer was around 7 μm, but irregularities in this thickness still maintained the electrical contact.

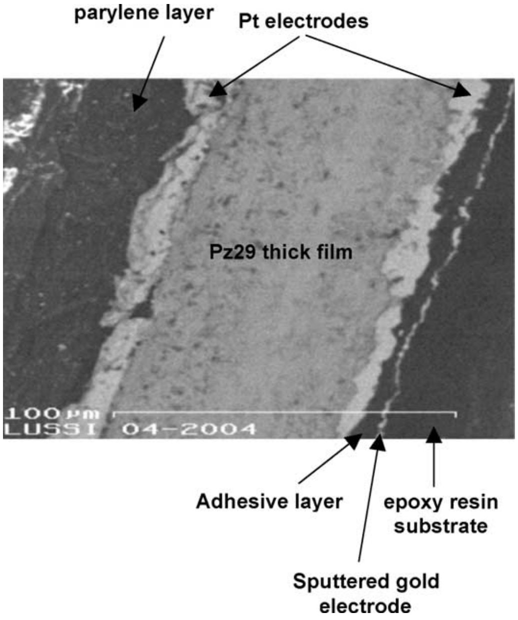


Fig. 3. Cross section (SEM photograph) of a sample.

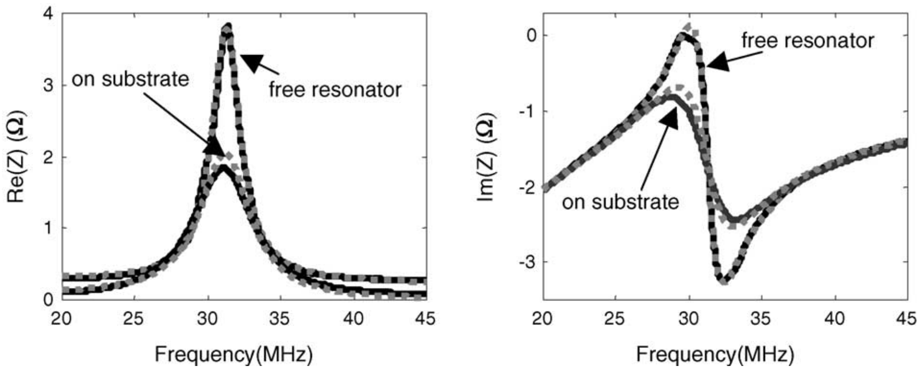


Fig. 4. Experimental (grey dashed lines) and theoretical (black solid lines) complex impedance of Pz29 tape-cast disc in free resonator conditions and on the substrate.

3. Functional characterisation

First, the electromechanical properties of the PZT disc were measured in free resonator conditions. The film dimensions make it possible to isolate the thickness mode so that a 1D model is valid. An equivalent electrical circuit model (K.L.M.)[5] was chosen since it is very suitable for multilayer structures such as ours. The effects of platinum electrodes are not negligible. Electrical impedances were measured with a set-up composed of an HP4395 impedance analyser, its impedance test kit and specific spring clip fixture. The theoretical impedance was obtained by a fitting process to deduce the thickness mode parameters. Fig. 4 represents complex experimental and theoretical electrical impedances of a thick film disc in free resonator conditions. The results for five PZT discs are given in Table 2. Their electromechanical constants were found to be similar to those of bulk samples with the same composition, in particular the thickness coupling factor. Variations of few MHz were observed between the antiresonant frequencies of the disc with and without electrodes (Table 2), showing clearly the influence of the electrodes at such high frequencies.

Measurements of electrical impedance were also performed before parylene deposition on PZT discs bonded on the substrate. These measurements were compared to those simulated with the K.L.M. scheme (with values from Tables 1 and 2). The results were in close agreement and confirmed that the characteristics of the substrate, sputtered gold electrode and adhesive layer were correctly taken into account or negligible (Fig. 4).

Table 2
Properties of five tape-cast discs (Pz29)

Samples	1	2	3	4	5
e_{piezo}[a] (μm)	67	68	70	66	65
ρ[b] (kg m^{-3})	6810	6810	6810	6810	6810
V_{l}[c] (m s^{-1})	4960	5005	5270	5015	5025
f_{a}[d] (MHz)	37	36.8	37.6	38.0	38.7
k_{t}[e] (%)	40.7	45.2	42.8	44.6	43.2
$\varepsilon_{33\mathrm{r}}{}^{S}$[f]	1075	1245	1240	1250	1055
δ_{m}[g] (%)	8.4	9.7	16.4	8.9	10.2
δ_{e}[h] (%)	3.8	3.7	3.7	5.2	2.2

[a] e_{piezo}: Thickness of the piezoelectric disc.
[b] ρ: Density of the piezoelectric material.
[c] V_{l}: Longitudinal wave velocity.
[d] f_{a}: Anti-resonant frequency.
[e] k_{t}: Thickness coupling factor.
[f] $\varepsilon_{33\mathrm{r}}{}^{S}$: Relative dielectric constant at constant strain.
[g] δ_{m}: Mechanical losses.
[h] δ_{e}: Electrical losses.

Finally, after parylene deposition (different thicknesses) on five samples, electrical impedances were measured

Table 3
Parylene thicknesses measured after deposition (on the five samples) and corresponding longitudinal wave velocities deduced in parylene

Samples	1	2	3	4	5
e_{min}[a] (μm)	12	20	21	29	29
e_{max}[b] (μm)	13	22	23	31	31
V_{lmin}[c] (m s^{-1})	2110	2046	1945	1974	2080
V_{lmax}[d] (m s^{-1})	2280	2252	2129	2216	2222

$V_{\mathrm{l}} = 2135 \pm 85$ m s^{-1}[e]; $\rho = 1289$ kg m^{-3}[f]; $Z = 2.75 \pm 0.1$ MRa[g]

Mean value (velocity) and acoustic impedance are specified.
[a] e_{min}: Minimum parylene thickness measured.
[b] e_{max}: Maximum parylene thickness measured.
[c] V_{lmin}: Minimum velocity calculated.
[d] V_{lmax}: Maximum velocity calculated.
[e] V_{l}: Mean velocity obtained.
[f] ρ: Density.[4]
[g] Z: Acoustic impedance deduced from e and f.

between the two electrodes specified in Fig. 2 to deduce parylene longitudinal wave velocity.

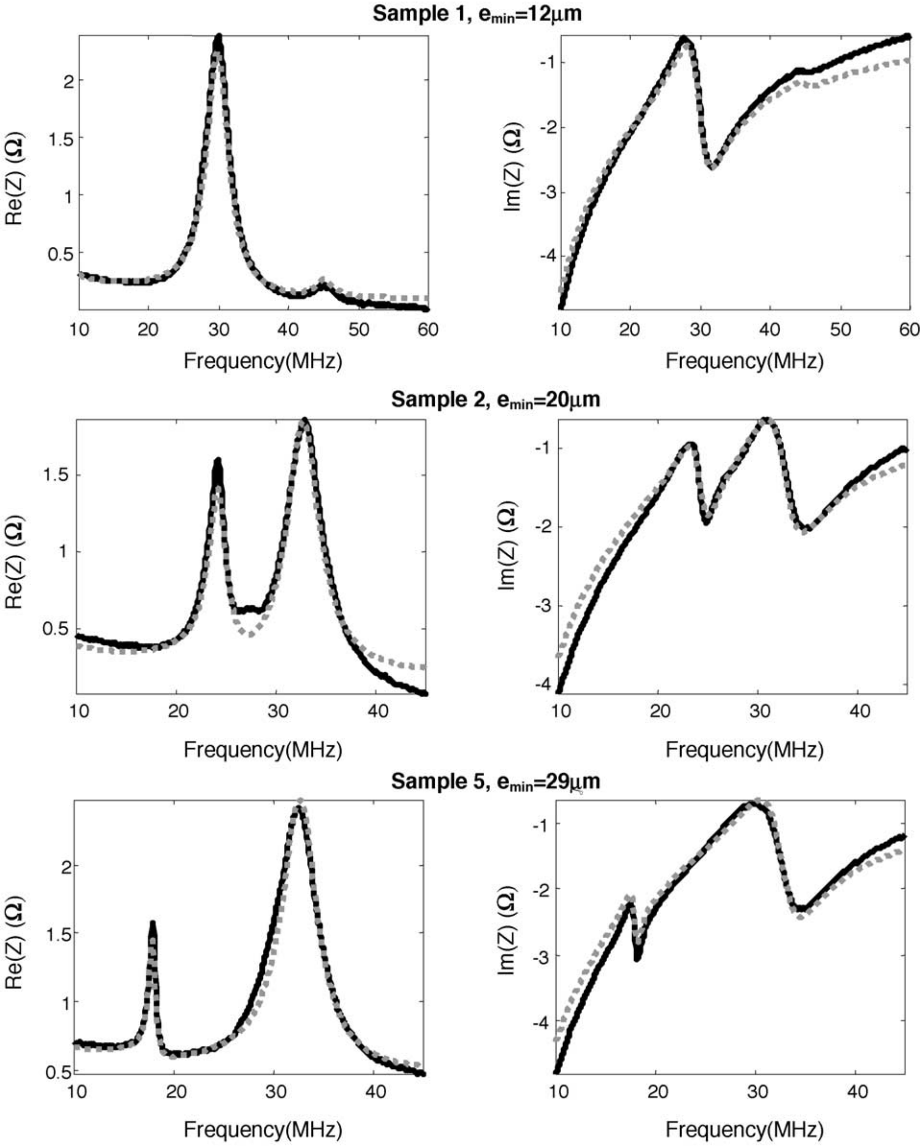
4. Results

Five samples of different parylene thickness (10, 20 and 30 μm) were studied. After measurement of the electrical impedance of each sample, parylene layers were removed and thicknesses measured with a micrometer. Minimum and maximum values are given in Table 3 and fits using both values were performed to obtain longitudinal wave velocity and attenuation of parylene layers. Fig. 5 presents fits corresponding to the three different parylene thicknesses and the results are given in Table 3. Two resonance peaks are observed corresponding to the coupled resonator structure composed of the piezoelectric and parylene layers. The mean value of the longitudinal wave velocity (from the ten fits), measured at around 30 MHz was 2135 $\mathrm{m\,s^{-1}}$ with an error of ± 85 $\mathrm{m\,s^{-1}}$. The precision obtained for attenuation was very low, the mean value being 0.56 dB $\mathrm{mm^{-1}\,MHz^{-1}}$ with a possible error around ±0.25dB $\mathrm{mm^{-1}\,MHz^{-1}}$.

The values obtained for velocity were in close agreement with those of other publications[1,2] and manufacturers datasheets[4,6].

5. High frequency transducers

The characteristics of sample 2 (Table 2) were used to simulate three high frequency single element transducer configurations. A heavy backing was chosen with an acoustic


Sample 1, e_{min}=12μm

Sample 2, e_{min}=20μm

Sample 5, e_{min}=29μm

Fig. 5. Experimental (grey dashed lines) and theoretical (black solid lines) complex impedances of multilayer samples (substrate, PZT disc and parylene layer).

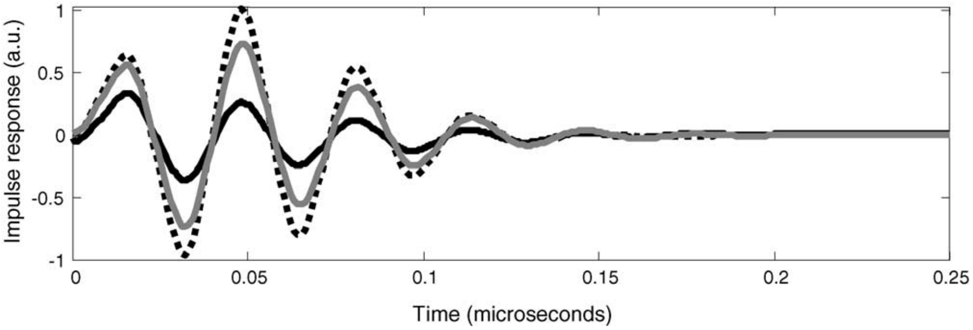


Fig. 6. Simulated impulse electro-acoustic responses of three transducers (solid black line = no matching layer, solid grey line = parylene matching layer, dashed black line = optimal matching layer).

impedance of 13 MRa, which corresponds to a porous PZT substrate which can be integrated in the fabrication process of screen-printed films.[7]

The KLM model was used to calculate the impulse electro-acoustic responses in water of a transducer with no acoustic matching layer (configuration 1), with an optimised thickness parylene matching layer (configuration 2) and a matching layer whose thickness and acoustic impedance had been optimised (configuration 3). For the last two configurations, the optimisation process was based on a performance index specifically developed for medical imaging.[8] Minimisation of this index by a recursive algorithm provided the optimum acoustic properties of each layer. The impulse electro-acoustic responses are superimposed in Fig. 6: the 2nd configuration delivered intermediate performance between the two others. Due to an heavy backing, the bandwidth was not greatly influenced by the addition of a matching layer. Sensitivity of the 2nd configuration was 6 dB lower than that of 3rd configuration but 12 dB higher than that of 1st configuration.

6. Conclusions

The acoustic properties of thin parylene (between 10 and 30 μm) were measured using piezoelectric thick films made by tape-casting (thickness around 65 μm and resonance around 30 MHz) and bonding on an epoxy substrate before parylene deposition on the upper face. With variations in electrical impedance due to parylene deposition, a longitudinal wave velocity of $2135 \pm 85\ \mathrm{m\,s^{-1}}$ was measured on five samples. Simulated high frequency transducers (around 25 MHz) with parylene matching layer, were shown to deliver satisfactory performance.

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