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Highly transparent PZT capacitors on glass obtained by layer transfer process

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ABSTRACT

Transparent ITO/PZT/ITO capacitors were fabricated on 200 mm glass substrate. The PZT films of 1 μm and 2 μm thickness were first grown on platinized Si wafer by sol-gel method, and then transferred onto glass substrate together with ITO electrodes following an innovative process. The obtained PZT based stacks on glass show an average transmission of about 70 % in the visible range. PZT films keep their preferred (100) orientation after transfer process. The capacitors exhibit ferroelectric, dielectric and piezoelectric properties comparable to standard non-transparent PZT films with metal electrodes. Transverse piezoelectric coefficient $e_{31,f}$ as high as 16 C/m² was measured for both PZT film thicknesses. This proof of concept opens the way to the fabrication of transparent piezoelectric actuators on glass for high performances haptic devices, as well as for other emerging applications like self-cleaning or functionalization of smart windows.

Keywords: Piezoelectric, Transparent, Tin film, PZT, Glass

1. Introduction

Lead zirconate titanate, $\text{Pb}(\text{Zr},\text{Ti})\text{O}_3$ (PZT), is today the most powerful piezoelectric material for micro-actuator applications thanks to its large piezoelectric response [1-2]. The PZT technology is present in several MEMS foundries for realizing PZT film actuator devices that are integrated in commercialized devices like inkjet printheads, micro-speakers and autofocus camera. Other applications under current development like micro-mirror for LIDAR, haptics for human-machine interface, micro-pumps, as well as PMUT for fingerprint, medical probe or gesture recognition might also integrate thin film PZT actuators in the near future [3]. In most cases, the piezoelectric material is integrated on silicon substrate, but we see those recent years emerging applications aiming at functionalizing glass and flexible substrates. Additionally, transparency is sometimes highly desired for transducers and actuator functions in application domains like photoacoustic imaging [4-6], haptics [7-8] and acoustic [9].

PZT, which is transparent due to its large bandgap (3.35 eV) [10], is usually deposited on platinized Si substrate. It is then integrated into metal-insulator-metal (MIM) capacitor, which is at the basis of most piezoelectric actuator devices. In order to insure optimal piezoelectric performance, crystallization temperature above 500°C, and typically 650-700°C, is requested. This makes it difficult to produce a transparent MIM capacitor-type stack on glass or flex using the standard PZT film deposition processes used in the MEMS industry. The integrity of the substrate (glass or flex), the diffusion of Pb towards the substrate, the growth of oriented PZT film, as well as the degradation of both electrical conductivity and transparency of the electrode are among the main integration locks to be considered. In that general context, a very interesting paper published in 2021 reviews the numerous efforts made the past years for developing low-temperature processing of PZT in order to make it compatible with semiconductors (450°C), smart glasses (400°C), flexible electronics (350°C) as

well as layer stacking transparency required in some cases [11]. Regarding transparent PZT stack, few works have been reported in the literature. A first example of fully transparent PZT MIM capacitor deposited on glass was reported in 2007 [12]. PZT was deposited on 1 in.² substrate using hybrid LaNiO₃ (LNO)/In₂O₃ 90 % SnO₂ 10 % (ITO) electrodes. The 90nm thick PZT showed rather poor crystal structure and the optical transmittance of the stack did not exceed 60 %. More recently, fluorine doped tin oxide (FTO) that shows better resistance to temperature than indium tin oxide (ITO), which is reported to degrade at temperature above 300°C [13-14], was preferred for growing PZT films [15-18]. In those published works, PZT films are either deposited by sol-gel or sputtering methods, and crystallized at temperatures between 260°C and 550°C. In all cases, PZT films are crystallized without preferred orientation and the transmittance of the layer stack can be as high as 80%. The electrical functionality of the fabricated capacitors is also evidenced, but with very moderate properties compared to standard PZT deposited on silicon substrate. It is important however to note that Hua et al very recently reported encouraging results about sol-gel PZT films deposited on ITO/glass substrate [8]. The PZT film crystallized at 650°C is poly-oriented and the piezoelectric stack shows transparency of around 75 %. While the resistance sheet of the 500 nm thick ITO bottom electrode is increased by a factor 10, excellent piezoelectric performances are obtained. However, according to the authors, an optimization of the process is still required for controlling the PZT film orientation, and maybe also for decreasing the ITO bottom electrode thickness. Finally yet importantly, the type of glass used for this work (strain point of 752°C) was chosen so that the glass to withstand the thermal budget for crystallizing PZT film. However, most of typical commercial glass substrates have much lower strain-point. As an alternative to MIM capacitor structure, coplanar type capacitor with interdigitated electrodes (IDE) on top of PZT allows getting rid of bottom electrode use. This simplifies the integration of transparent PZT on glass. Defay's group successfully employed this solution for realizing fully transparent functional friction-modulation haptic device based on PZT and ITO films deposited on fused silica [7, 19]. The main drawback of their technology is the high driving voltage required using IDE electrodes that can be detrimental for some applications.

In this work, we use the film transfer processing as an alternative way for getting optimal PZT on transparent electrode. The generic principle of layer transfer is the growth of PZT film on suitable Pt/Si substrate followed by its transfer onto a host substrate following various possible techniques like the grinding of the donor substrate, the etching of sacrificial layer between PZT and the grown substrate or the laser lift-off. More details can be found in the review paper of Song [11]. In the present article, we report the fabrication of fully transparent PZT based capacitors on glass obtained by a recently developed wafer-to-wafer layer transfer process. The dielectric, ferroelectric and piezoelectric properties of the capacitor devices were characterized so that to evaluate their potential of use in piezoelectric actuator MEMS applications.

2. Experimental

We describe hereafter in details the wafer-to-wafer layer transfer process used for getting transparent PZT based layer stacks on glass (Fig. 1). At first, PZT thin film is deposited on a donor platinumized Si wafer following standard sol-gel deposition process. We used a commercial PZT (52/48) sol-gel solution provided by Mitsubishi Materials Corporation. Details about the deposition process can be found elsewhere [20-21]. The PZT layer is then topped by 100 nm thick sputter deposited ITO electrode layer, followed by 300 nm of SiO₂ as bonding layer. Contrary to standard PZT stack, we did not insert classical TiO₂ or ZrO₂ adhesion layer in between SiO₂ and Pt, leading to a low energy interface (~ 1 J/m²) between those two layers. The latter will eventually be key for allowing easy detaching the PZT stack from the donor wafer by mechanical separation. The host wafer made of glass is also coated with 300 nm thick SiO₂ as bonding layer. Both Si and glass wafers are then bonded together (Fig. 1a). Note that prior to bonding step, a chemical-mechanical polishing operation is necessary to insure proper molecular bonding. As an alternative to direct bonding SiO₂/SiO₂, polymer bonding can be used [22] as well as Au-Au thermo-compression in case of non-transparent device [23]. Once wafers are bonded together, an annealing step is performed at 300°C for 2h (Fig. 1b). Then, by inserting a thin

blade between the bonded wafers, a sharp separation occurs between Pt and SiO₂ layers (Fig. 1c). The PZT stack is thus transferred onto the receiving glass wafer, while the donor Si wafer can possibly be recycled (Fig. 1d). To finish, the Pt layer on top of PZT film is removed by dry etching and replaced by a 100 nm thick sputter deposited ITO layer (Fig. 1e). A photograph of the 200 mm glass wafer with ITO/PZT/ITO full sheet layer stack is shown on Fig. 1f. The PZT stack can then be structured into capacitor devices using standard UV lithography and dry etching processes.

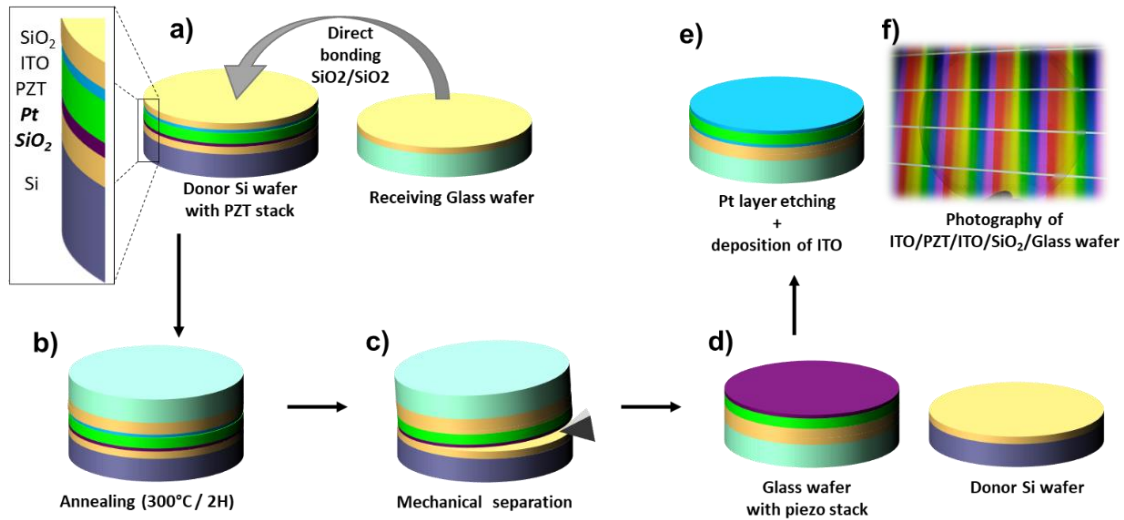


Fig. 1. PZT layer transfer process from Si to glass

The crystalline structure of the ITO/PZT/ITO stack was characterized by XRD. A scanning electron microscope (SEM) from HITACHI (S-5000) was employed to observe the cross cuts of PZT stacks done with focus ion beam (FIB) milling. The optical transmittance and reflectance were measured using an Agilent Technologies Spectrophotometer (Cary_7000). Dielectric measurements were performed with a TF Analyzer 2000 Measurement System, while ferroelectric polarization together with displacement curves were obtained using double beam laser interferometer (DBLI), both from aixACCT. Transverse piezoelectric coefficient $e_{31,f}$ was measured using the 4-Point Bending (aix4PB) system also from aixACCT company.

3. Results and discussion

XRD analysis was performed on the donor platinized Si substrate after depositing PZT film as well as on the receiving glass wafer on which the PZT film was transferred. Fig. 2 shows that PZT exhibits a pure perovskite phase with (100) texturation both before and after film transfer. The only difference between both theta-2 theta diffractograms is the presence of a peak from Pt before PZT transfer and peaks from ITO after PZT transfer followed by Pt etch. The thin film transfer process allows thus

obtaining PZT with desire (100) orientation on ITO electrode coated glass wafer. Indeed, PZT at MPB with (100) orientation is known to show the highest transverse piezoelectric coefficient d_{31} [1,24].

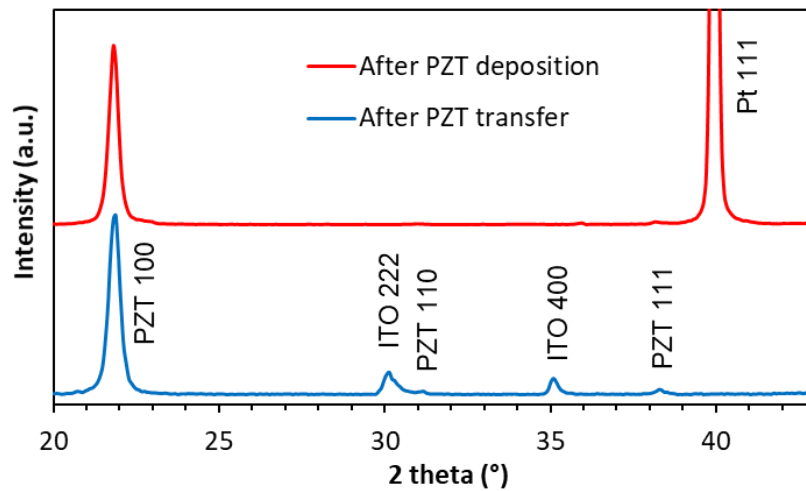


Fig. 2. X-ray Diffraction pattern of the PZT after deposition on Pt/Si (top red plot) and after transfer on glass (bottom blue plot).

SEM observations were performed to check the integrity of the PZT stacks after transfer process. Figs. 3a and 3b show SEM images of FIB cuts from PZT 2 μm and PZT 1 μm based stacks, respectively. The 2 μm thick PZT is topped with Pt layer, while the PZT 1 μm is observed after replacing top Pt layer by Pt/ITO electrode. The latter bilayer is used for piezoelectric measurements that require reflective coating. In both cases, PZT films appear dense without apparent voids and cracks. The horizontal lines visible inside the PZT films result from the RTA crystallization steps that are done every three-coated layers. Moreover, the interfaces are smooth and clean.

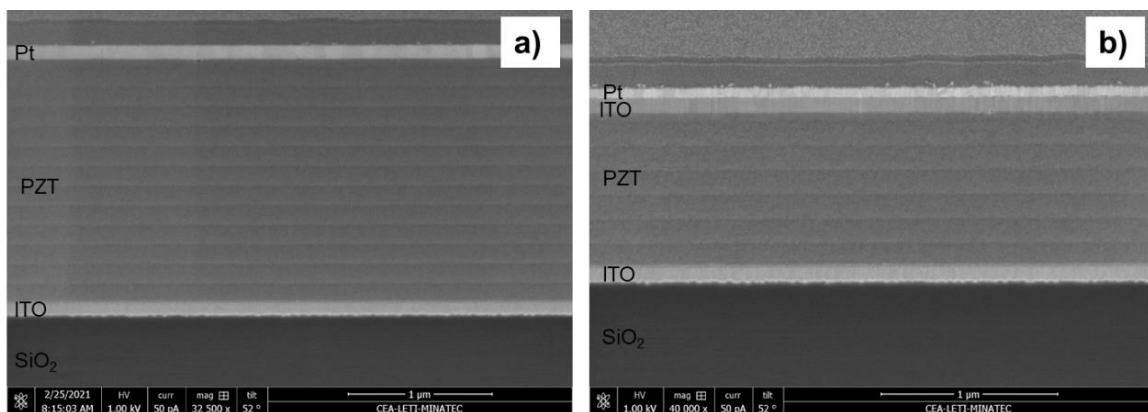


Fig. 3. FIB-SEM images of a) Pt/PZT 2 μm /ITO after transfer and b) Pt/ITO/PZT 1 μm /ITO stack.

The optical properties were measured in the visible spectrum (400 nm - 800 nm) using an Agilent Technologies Spectrophotometer (Cary_7000). Fig. 4a shows the transmittance of both 1 μm and 2 μm thick PZT films without top electrode ITO. The average transmittance is around 70 % whatever the film thickness. The oscillations observed on the curves are explained by the presence of several interfaces in the PZT capacitor stack where reflections can occur, creating interferences between the different transmitted beams. The only major difference between both curves is the period of the oscillations that is related to PZT thickness. In Fig. 4b we show the transmittance and reflectance spectra in case of PZT 2 μm with top electrode ITO. Both spectra were fitted using a multilayers model confirming that the oscillations arise from interference due to the internal reflections at the interfaces of the optically

dissimilar materials constituting the stack. Moreover, we observe that the transmittance minima occur at the reflectance maxima. An optimisation of the transmission intensity could thus be performed by minimising the reflectance, especially at low wavelengths, using for instance anti-reflective layers. In addition, we can say that the absorbance is as low as 10% in our stack, except below 450 nm where it increases up to around 25%. Nevertheless, the transparency of the PZT stack is high enough to easily read a text or an image through the capacitor devices (Fig. 4c).

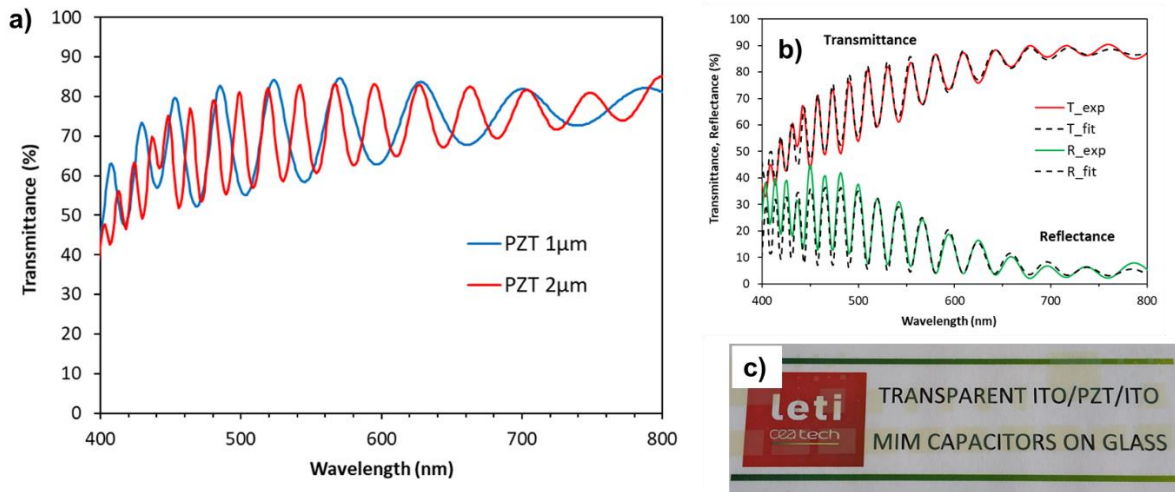


Fig. 4. a) Transmittance spectra of PZT 1 μm and 2 μm , b) Transmittance and reflectance spectra of PZT 2 μm with top ITO layer (experimental and simulation data), c) Photograph of logo and text through transparent PZT capacitors.

Capacitance and dielectric losses were measured on 1 mm^2 ITO/PZT/ITO capacitors by sweeping DC voltage between -25V and +25V while applying an AC excitation signal (1 kHz, 150 mV). The permittivity and losses curves registered for PZT 1 μm and PZT 2 μm are reported in Fig. 5a and 5b, respectively. The butterfly shape curves characteristic of ferroelectric materials are observed. The maximum permittivity is around 1100 for PZT 1 μm and slightly above 1300 for PZT 2 μm . The dielectric losses are below 7 % in both cases.

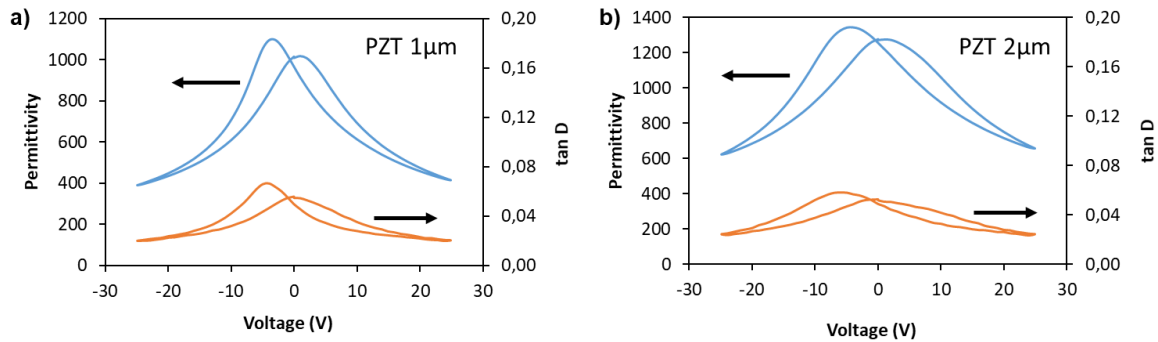


Fig. 5. Dielectric permittivity ϵ_r and losses $\tan \delta$ as a function of voltage for a) PZT 1 μm and b) PZT 2 μm

The large-signal polarization and displacement curves were measured on 1 mm^2 Pt/ITO/PZT(1 μm and 2 μm)/ITO capacitors (Fig. 6a and 6b). The ITO top electrode was coated with 50 nm thick Pt layer, while backside glass substrate was opacified with SiN/Ti bilayer, so that to get reflective surfaces, as required for DBLI measurements. The P-E loops measured at 300 Hz show typical hysteresis shape characteristic of a ferroelectric material. Ferroelectric properties are very similar for both PZT thicknesses. The maximum polarization reaches 38 $\mu\text{C}/\text{cm}^2$ for an applied electric field of 300 kV/cm , while the remnant polarization $2P_r$ is 28 $\mu\text{C}/\text{cm}^2$. The only significant difference is that the hysteresis loop is slightly larger in case of PZT 1 μm as the coercive field $2E_c$ amounts 85 kV/cm , while it is 67 kV/cm in the case of PZT 2 μm . The mechanical displacement of the 2 μm thick PZT film is

approximately twice the one of the PZT 1 μm . The large signal piezoelectric coefficients $d_{33,f}$ are 120 pm/V and 125 pm/V, for PZT 1 μm and PZT 2 μm , respectively. The measured ferroelectric and piezoelectric properties are thus comparable to standard PZT films of equivalent thicknesses grown on platinized Si substrate.

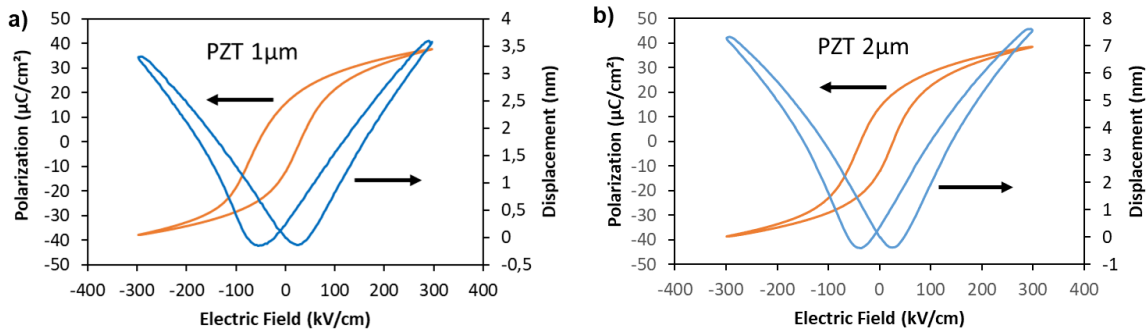


Fig. 6. Polarization and displacement as a function of electric field for a) PZT 1 μm and b) PZT 2 μm

The effective transverse piezoelectric coefficient $e_{31,f}$ was measured on $25 \times 3 \text{ mm}^2$ cut samples using the 4-point bending set up from aixACCT [25]. PZT films were polarized in both directions with maximum voltage of $\pm 20 \text{ V}$ for PZT 1 μm and $\pm 26 \text{ V}$ for PZT 2 μm . A static DC bias voltage was applied at each measurement step from maximum value down to 0V, with 2 V per step. The measurements results are plotted in Fig. 7a and 7b for PZT 1 μm and PZT 2 μm , respectively. Both PZT films show very similar results. Under optimal polarized state, either up or down, the $e_{31,f}$ coefficient reaches a maximum value around $16 \text{ C}/\text{m}^2$ for both PZT films. Those results are comparable to best values reported in literature for standard PZT films deposited on Pt/Si substrate [26]. It confirms the interest and the efficiency of our layer transfer process for getting transparent PZT capacitors on glass with properties at the level of state of the art.

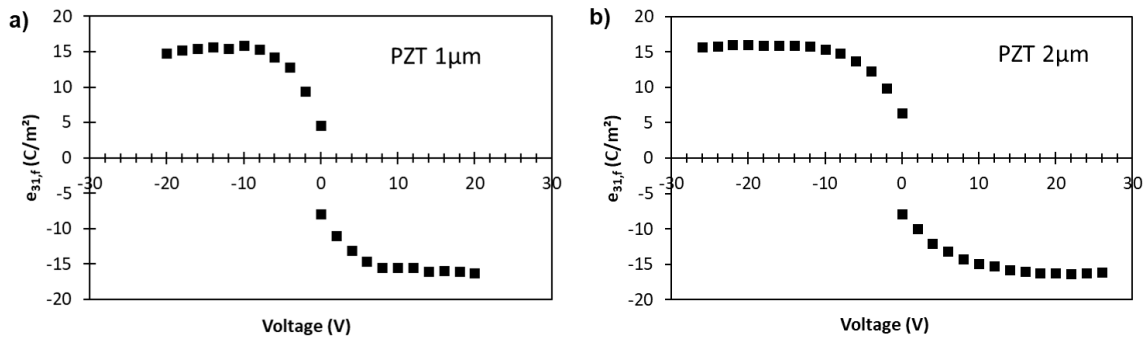


Fig. 7. Piezoelectric coefficient $e_{31,f}$ as a function of applied voltage for a) PZT 1 μm and b) PZT 2 μm

4. Conclusion

In this work, we fabricated transparent ITO/PZT/ITO piezoelectric capacitors on 200 mm glass wafer following a wafer-to-wafer layer transfer process. The PZT stack shows an average transmittance of around 70 % whatever the film thickness (1 μm and 2 μm) in the visible light range, making the capacitor devices transparent enough for human eyes. The PZT based capacitors show ferroelectric and piezoelectric characteristics similar to classically PZT films deposited on Si substrate with metal electrodes. In particular we measured piezoelectric coefficients $e_{31,f}$ at the level of best values reported in literature. These results pave the way to the fabrication of transparent piezoelectric actuators for piezomems applications, such as haptic for human-machine interfaces. In a future work, we thus aim at realizing a haptic device based on transparent PZT actuators.

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