PERFORMANCES OF HIGH-K DIELECTRIC MATERIALS (Al_2O_3, HfO_2, ZrO_2) FOR LIQUID DIELECTROPHORESIS (LDEP) MICROFLUIDIC DEVICES

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ABSTRACT

This paper reports performances of improved Liquid Dielectrophoresis (LDEP) devices made of Al_2O_3, HfO_2 or ZrO_2 as dielectric layers, rather than using classical dielectric layers (SiN, SU-8 resin, SiO_2…). These layers have been evaluated on several parameters, such as the threshold actuation voltage, the resistance to the electric field and the droplet generation process. The High-K materials implementation open-up new perspectives for a low-voltage, robust LDEP transduction mechanism and promotes it as a relatively relevant and promising liquid handling technique for sample preparation in µTAS devices.

KEYWORDS: Liquid Dielectrophoresis, High-K materials, threshold actuation voltage, dielectric breakdown.

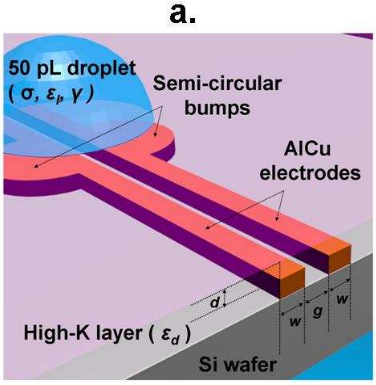
INTRODUCTION

LDEP microfluidic technique is known to displace liquids and create an array of micrometric size-controlled droplets (0.1 pL to 0.1µL) within a very short time (~100 ms) onto a surface. The principle of operation requires an AC sinusoidal signal applied between two coplanar electrodes separated by a gap [1]. Most studies in the literature still report quite high actuation voltage amplitudes (>200 V_{RMS}) [2-3] and suffer from dielectric breakdown of insulating stacks coated over the driving electrodes. Our previous theoretical study pointed out the importance of the dielectric stacks implemented atop of the LDEP electrodes on the overall actuation performances [4]. In particular, some dielectric stacks with thin thicknesses combined with high dielectric constants should reduce significantly the liquid actuation voltages, while keeping a robust technology with regard to high electric fields trough the various layers.

MATERIEL AND METHODS

Open single-plate architecture devices, (see Fig.1a), have been fabricated from a 200mm wafer Si technology. The driving electrodes are made of a 10 nm Ti (adhesion layer) / 200 nm AlCu layer atop of a SiO2 isolating layer and designed with specific geometries to create an array of 50 pL droplets (electrodes width w = 10µm separated with a gap g = 4 µm). Then, the electrodes are covered with three different High-K materials deposited through Atomic Layer Deposition (ALD) process: Al_2O_3, HfO_2 and ZrO_2 (see Table 1 and Fig 1b).

The LDEP electrodes are energized with a 100 kHz AC signal pulse (duration: 100 ms), for each DI water (σ = $5.5.10^{-6}$ $S.m^{-1}$) droplets creation event, in ambient conditions. For each dielectric stack, three parameters have been evaluated: the threshold actuation voltage V_{th} (minimum voltage corresponding to the initiation of the liquid finger), the total actuation voltage V_{tot} (minimum voltage corresponding to the complete liquid finger actuation along the 1.5 mm long coplanar electrodes) and the number of successful and successive actuations N_{a1} (number of actuations when the liquid finger reaches a position between the end of the coplanar electrodes and the middle of those ones) and $N_{a1/2}$ (number of successful liquid actuations which do not overreach the middle of the coplanar electrodes).



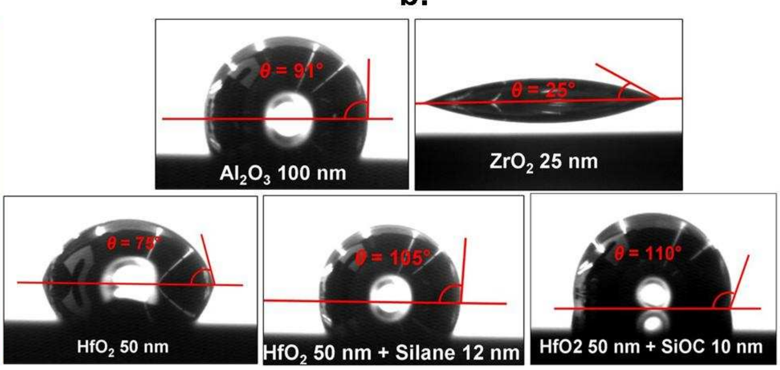


Figure 1: (a) Schematic view of an LDEP structure. (b) 2 µL droplet liquid contact angle with DI water on the High-K layer surfaces.

Table 1: properties of the implemented High-K materials

Layers ID	Dielectric Materials	Thickness d	Dielectric constant ε_d	Equivalent dielectric thickness α ($\alpha = d/\varepsilon_d$)	Contact angle θ (with DI water)	Deposition process
L1	Al_2O_3	100 nm	~ 8	12.5 nm	90°	ALD
L2	Al_2O_3	50 nm	~ 8	6.2 nm	90°	ALD
L3	HfO_2	50 nm	~ 15	3.3 nm	75°	ALD
L4	HfO_2	25 nm	~ 15	1.7 nm	75°	ALD
L5	ZrO_2	50 nm	~ 25	2 nm	25°	ALD
L6	ZrO_2	25 nm	~ 25	1 nm	25°	ALD

THEORETICAL BACKGROUND

According to the theory detailed in [4-5], the threshold liquid actuation voltage V_{th} depends on several parameters: mainly the liquid properties, the dielectric layers properties and the electrodes geometry.

$$V_{th} = \sqrt{\frac{2F_{\gamma}\left(\sum_{i}^{N}\frac{2}{C_i^{*}}+\frac{1}{C_{liq}^{*}}\right)\left(\sum_{i}^{N}\frac{2}{C_i^{*}}+\frac{1}{C_{air}^{*}}\right)}{\left(\frac{1}{C_{air}^{*}}-\frac{1}{C_{liq}^{*}}\right)}} \quad (1) \qquad C_i^{*} = \frac{\varepsilon_0\varepsilon_i w}{d_i} \quad (2) \qquad \alpha = \sum_{i=1}^{N}\frac{d_i}{\varepsilon_i} \quad (3)$$

In the expressions (1) and (2), w, d_i, ε_0, ε_i refer respectively to the electrodes width, the dielectric layer i thickness, the vacuum permittivity and the dielectric constant of the dielectric layer i. C_i^{*}, C_{liq}^{*}, C_{air}^{*} represent the electrical capacitance of the dielectric layer i, the liquid and the air derived with respect to the direction of liquid motion. F_{γ} is the force generated by the liquid surface tension in the air. In the expression (3), α is defined as the sum of dielectric thicknesses of a layers stack and strongly affects the V_{th} value.

RESULTS AND DISCUSSION

The electrochemical model described in the previous section predicts actuation voltage should range below 100 V_{RMS} by implementing the layers listed in Table 1. As expected, the implementation of High-K materials improves the LDEP technique efficiency. As shown in the Fig.2a, the DI water liquid finger motion can be initiated from 50 (L6 layer) to 70 V_{RMS} (L1 layer). A total liquid actuation through 1.5 mm with 50 pL droplets generation occurs from 70 (L6 layer) to 110 V_{RMS} (L1 layer). The general tendency of experimental data is in agreement with the theoretical model, since the actuation voltages decrease as the dielectric thickness decreases. As a conclusion, these values get closer to signal amplitudes typically used for EWOD actuations and there is no need to use sophisticated high-voltage amplifier anymore.

Concerning the number of successive actuations on a same design, High-K dielectric materials are characterized by a high resistance to the electric field since some dielectric layers can support more than 200 successive actuation cycles without dielectric breakdown (Fig.2b). Note that the dielectric breakdown of the layer L6 early occurs when the potential difference between coplanar electrodes reaches a value between 50 and 70 V_{RMS}.

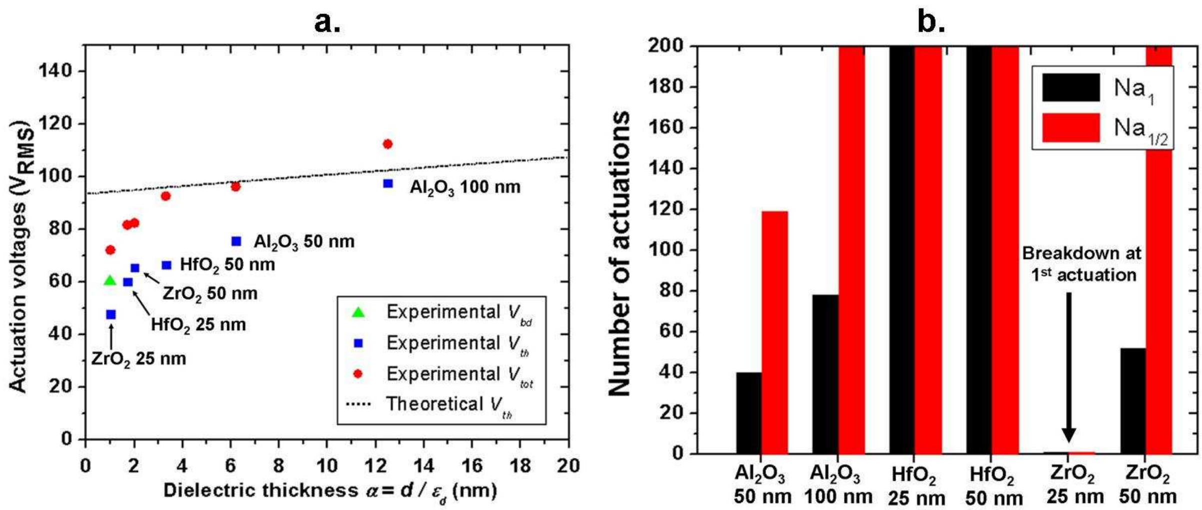


Figure 2: (a) Threshold actuation voltages V_{th} (in blue) and total actuation voltage V_{tot} (in red) for each layer. (b) Number of successive and successful actuation on a same design for each layer. (Na_1 / $Na_{1/2}$: the liquid finger stops after ***(in black)*** */ before (in red) the middle of electrodes).*

On the other hand, one issue is related to the droplets creation quality, once the liquid finger breaks up. Indeed, these materials, more or less hydrophilic (Fig.1b), may prevent the controlled and reproducible droplets generation at each bump position. Some of these materials would have adapted natural surface properties for LDEP actuations [5], since DI water contact angles on Al_2O_3 and HfO_2 layers are respectively 90 and 70°, almost hydrophobic. In that case, the droplets generation process is not as sluggish as experiments on very hydrophilic surfaces such as SiN or SiO_2 layers. Nevertheless, an additional coating made of either 10 nm thick SiOC (Silicone Oxycarbide) or 12 nm thick FDTS (Perfluorodecyltrichlorosilane) carried out onto the High-K layers. These top surfaces increase the contact angles with DI water samples and the droplets generation process is significantly improved while keeping quite low actuation voltages (see Fig. 3).

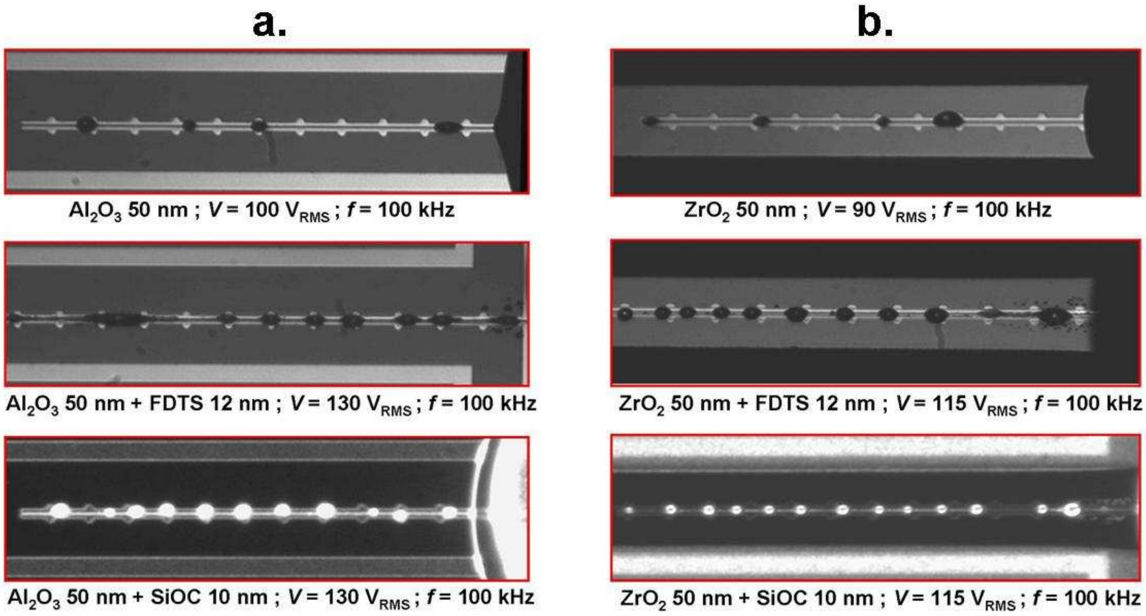


Figure 3: (a) Droplet generation process comparison between a single layer Al_2O_3 and Al_2O_3 layers coated with FDTS and SiOC. (b) Same photos sequence with ZrO_2 material.

CONCLUSION

Such results demonstrate the LDEP can be performed at low voltage with a robust silicon-based technology. Moreover the droplets generation process can be improved by adding thin hydrophobic coatings such as SiOC. These performances provide a strong interest to carry out sample preparations for massively parallel and complex biochemical protocols.

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