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Study of free-standing electroded PZT thick-films: from materials to microsystems

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Abstract. Free-standing electroded piezoelectric PZT thick-films are straightforward fabricated thanks to the association of the low-cost screen-printing technology with the sacrificial layer method. Au/ PZT/ Au bridges are directly attached onto the alumina substrate on top of it they are processed. In addition, completely released disks are also processed. A study of the behaviour of these components shows the influence of both the releasing process and the densification on the piezoelectric properties of the PZT layer. From the electromechanical measurements, electroded PZT cantilevers and disks are promising for actuator, sensor or SHM applications.

Introduction

Microsystems (MEMS) are mostly issued from the transposition of silicon micromachining processes used in IC manufacturing to the fabrication of movable elements [1]. MEMS can also be made by using alternative technologies such as LIGA, LTCC or direct processes of prototyping (inkjet, micropen, microstereolithography, etc.) [2]. In the same way, the cost-effective screen- printing technology may also be considered for the fabrication of thick-films MEMS [3]. However, the fabrication of free-standing thick-film actuators suffered from the lack of technological solutions for releasing the structural layers which need to be actuated independently of the substrate.

Recently, free-standing piezoelectric devices made of screen-printed thick-films have been developed [4],[5]. In this paper, we report straightforward electroded piezoelectric Au/PZT/Au thick-films for the realization of bridges and disks based components. Bridges are directly attached onto the alumina substrate on top of it they are processed whereas the disks are completely released. After processing, physicochemical and electromechanical characterizations of each type of component are performed. The piezoelectric properties of PZT thick-films are compared to those of ceramics prepared with the same materials. In other respects, properties of PZT disks are evaluated through actuating and sensing applications as well as the ElectroMechanical Impedance (EMI) of the disks bonded on a metallic structure, for Structural Health Monitoring (SHM) applications.

Study of free-standing and totally released piezoelectric microsystems

Bridge-type free-standing piezoelectric devices

The combination of screen-printing technology and sacrificial layer method allows the fabrication of bridge-type piezoelectric microsystems Au/PZT/Au, partially released from an alumina substrate after removal of sacrificial layer. The sacrificial layer, made of mineral filler in an epoxy-type ink, must fulfill requirements such as thermal and mechanical stability for the structural layer deposition, chemical harmlessness, etc. Strontium carbonate (SrCO3) is chosen as the mineral filler because of its chemical stability up to the usual firing temperatures $(850-900°C)$. SrCO3 layer sustains the structural layers during the thermal treatment at high temperature $(T > 500°C)$ to prevent mechanical deformation of the sample. SrCO3 is easily dissolved in a weak acidic solution at the end of the firing process. Au ink is commercially available (ESL8836 from ElectroScience Laboratory) whereas PZT and sacrificial layer inks are prepared in our laboratory. Commercial SrCO3 powder is first sieved and mixed with an epoxy-based paste. Concerning the piezoelectric paste, the PZT (PbZr0.52Ti0.48O3) powder is mixed with a standard organic binder ESL400 in a mortar and then homogenized in a planetary ball mill during 12 hours in order to fulfil the required rheological properties. A borosilicate glass-frit (5%wt) is added to the PZT ink for densification improvement while decreasing the firing temperature.

Then, the sacrificial layer ($\approx 50\mu m$) is screen-printed on the substrate and polymerized at $120^{\circ}C$, prior to successive depositions of bottom electrode ($\approx 10\mu m$), PZT layer ($\approx 120\mu m$) and top electrode ($\approx 10\mu m$) consecutively dried at $120^{\circ}C$ during 20min. After firing the multilayer samples 15 min at $850^{\circ}C$ in air atmosphere, dissolution of sacrificial layer is performed in the 0.9mole.1-1 H_3PO_4 aqueous solution. As shown in Fig. 1a, Au electrodes are symmetrically anchored on each side of the cofired multilayers to maintain the suspended bridge-type piezoelectric device over the alumina.

Estimation of a compacity of 70% ($\rho PZT \sim 5.5g.cm^{-3}$) for the porous microstructure of the PZT layer is obtained from SEM analysis of free-standing PZT devices ($3.3x3.3x0.08mm^3$). Moreover, microprobe analyses (CAMECA SX 100), performed at different locations of the free-standing PZT thick-film, disclose no traces of Sr in Au and PZT layers.

Before the electrical characterizations performed with an HP4194A impedance analyzer, PZT devices are placed under dry helium atmosphere in a closed cell and poled 10 min under an electric field of $5kV.mm^{-1}$ at $T = 270^{\circ}C$ lower than the Curie temperature of PZT ($TC\ 280^{\circ}C$).

Conversely to clamped PZT devices, impedance measurements of released PZT bridge-type samples show clearly in-plane vibrations at 463kHz. However, both type of PZT devices exhibit mechanical out-of-plane vibrations parallel to the poling axis in the MHz range. From the frequency resonances measured for free-standing sample, it is possible to calculate the piezoelectric parameters like the dielectric constant (K3T), the quality factor (Q), the dielectric loss (tan δ) and the piezoelectric coefficient d_{31} (Table 1). Comparison of these parameters is made with those of the bulk ceramic fabricated with the same PZT composition and fired 10min at $850^{\circ}C$. The lower electromechanical properties of the free-standing samples compared to those of PZT ceramics may be attributed to the high porosity rate of screen-printed PZT layers.

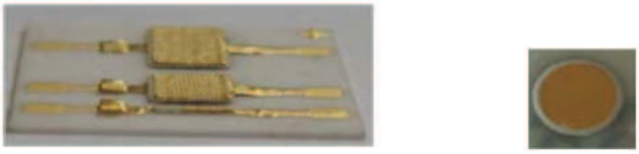
Table 1: Piezoelectric parameters of free-standing and ceramic of PZT

	Free-standing sample	Ceramic
dim. (mm)	3.3x3.3x0.08	Ø 6.8 x 0.8
density ($g.cm^{-3}$)	5.5	7
f_r (KHz)	463	308
K_{3T}	160	240
tan δ	0.05	0.01
Q	116	278
d_{31} ($pC.N^{-1}$)	14	10

Totally released microceramic for MEMS and SHM applications

The sacrificial layer process has also been applied for the fabrication of discrete Au/PZT/Au microceramics which must be totally release from the substrate. The two pastes (Au, SrCO3-epoxy) and process selected for disk fabrication are identical as those used for the cantilever processing [6]. For a better densification of PZT layers, the eutectic phase LBCu (25wt% Li2CO3, 40wt% Bi2O3, 35%wt CuO) replaces the borosilicate sintering aid previously used in the PZT paste for bridge-type samples.

The screen-printed sacrificial layer covers entirely the 2.5x2.5cm2 alumina substrate. After printing, the dried thicknesses of gold and PZT layers are respectively 5 and 200μm. In order to limit the presence of cracks or holes in the very thick PZT layer the drying step is performed with an heating rate of $1°C$/min for $30 < T(°C) < 400$ and $20°C$/min for $(400 < T(°C) < 900)$. The disk samples are then fired 2 hours at $900°C$ and cooled down to room temperature at $20°C$/min (Fig.1b).



(a) (b)

Figure 1: Photograph of a) piezoelectric bridges $(5x3.3x0.08mm^3$, $3.3x3.3x0.08mm^3$, $3.3x1x0.08mm^3)$ and b) straightforward electroded microceramic $(\varnothing 9.5mm$, thick. $190\mu m)$

The main piezoelectric and dielectric properties related to screen-printed piezoelectric disks are reported in Table 2. They are compared to those of a pellet of same composition and fired the same way and those of a PZT commercial ceramic (PI ceramic). The dielectric and piezoelectric performances of PZT thick-films are still lower than those of pellets or of those of commercial ceramics because of their poorer densification. Nevertheless, the screen-printed Au/PZT/Au microceramics are tested in three configurations -sensor, actuator and ElectroMechanical Impedance- in order to evaluate the potentiality for MEMS or SHM applications. For all the tests, the microceramics Au/PZT/Au are bonded with a $30\mu m$ rigid glue from EPOTECK (EPO-TK- E4110) on a steel beam clamped at one end $(200x20x0.48mm^3)$.

Table 2: Electromechanical parameters of Au/PZT/Au disk and pellet and commercial PZT sample.

Sample	K_T^{33}	$\tan\delta$	k_p (%)	$-d_{31}$ (pC/N)	f_{RES} (kHz)
Au/PZT/Au printed Ø9.5mm, Thick. 190µm	634	0.015	14	40	166
Au/PZT/Au pellet, Ø11.5mm, thick. 950µm	930	0.015	46	82	140
Commercial PI Ø10mm, thick. 200µm	2900	0.015	66	200	241

For the sensor test, the first resonance mode of the beam is excited by pulling it away from its equilibrium. The induced vibrations and the resonance frequency are thus measured with the screen-printed piezoelectric Au/PZT/Au microceramic through a charge amplifier. The discrepancies between the measured resonance frequency of the beam $(9.50Hz)$ and the calculated value $(10.17Hz)$ can be attributed to its sizes approximation. Concerning the actuator test, application of a voltage $V = 130V$ to the PZT disk at the resonance frequency of $9.5Hz$ induces beam oscillations which are measured with a laser vibrometer. The maximum speed of these oscillations $(\sim 130mm/s)$ for displacement amplitude of $2.18mm$ is measured. Finally, measurement of the ElectroMechanical Impedance (EMI) of the beam with the screen-printed piezoelectric transducer shows many resonance peaks, which is favourable for SHM applications (Fig.2).

Conclusion

The efficiency of this simple and low-cost process has been demonstrated with the fabrication of MEMS based on different materials (metals, ceramics, glass, glass-ceramics, etc.). Piezoelectric materials have been chosen for the processing of actuated MEMS, more precisely anchored bridges and

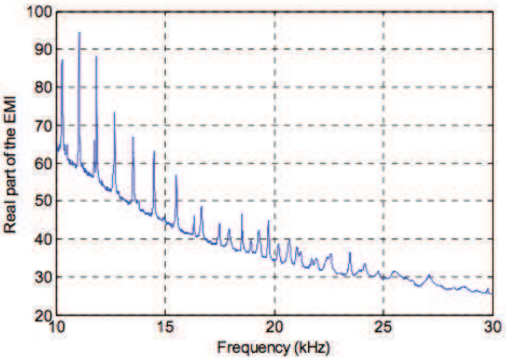


Fig. 2: Real part of the ElectroMechanical Impedance (EMI) measurements with the Au/PZT/Au microceramic bonded on the metallic structure.

totally free-standing disks. Studies of microstructure of the Au/PZT/Au bridges show the harmlessness of the process. Impedance analysis confirms the presence of out-of- plane and in-plane vibrations, the later being only observed for free-standing resonators. The electromechanical properties of PZT thick-films, still lower than those of PZT ceramics, are correlated to the high porosity rate ($\sim 30\%$) of the printed PZT films. However these free-standing PZT microcomponents present a good electromechanical behaviour and can be used in MEMS applications. Indeed, the Au/PZT/Au disks can be used as actuator or vibrations sensors. Their good electromechanical signature when bonded on a metallic structure also suggests SHM applications. EMI tests with damaged structures are under current development. The thick-film sacrificial layer process described in this paper may well open new routes of investigation for MEMS, complementary to silicon, LTCC or PCB ones.

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Materials and Applications for Sensors and Transducers III
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