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Young modulus and Poisson ratio of PZT thin film by Picosecond Ultrasonics

F. Casset, A. Devos, S. Sadtler, A. Le Louarn, P. Emery, G. Le Rhun, P. Ancey, S. Fanget, E. Defay

Abstract— Thin-film sol-gel $\text{Pb}(\text{Zr}_{0.52}, \text{Ti}_{0.48})\text{O}_3$ (PZT) can be used in many actuator applications thanks to its high piezoelectric coefficients. The accurate knowledge of its mechanical properties such as Young modulus (E) and Poisson ratio (ν) or stiffness matrix is a key point for predictive design, whereas the state of the art reports E_{PZT} ranging from 70 to 139GPa. Those parameters can be accurately characterized by Picosecond Ultrasonics (PU) as detailed in this paper.

Index Terms— Picosecond Ultrasonics, Young modulus, Poisson ratio, PZT

I. INTRODUCTION

$\text{Pb}(\text{Zr}_{0.52}, \text{Ti}_{0.48})\text{O}_3$ (PZT) thin films are well suited to actuator applications such as micro mirror [1], RF MEMS [2], inkjet [3] or loudspeaker [4] due to their large piezoelectric coefficients [5]. The accurate knowledge of their mechanical properties such as Young modulus (E) and Poisson ratio (ν) is a key point for predictive MEMS design. The state of the art reports E_{PZT} ranging from 70 [6] to 139GPa [7], characterized with different techniques such as nano indentation, X-ray diffraction or vibrating method. Here we propose to use Picosecond Ultrasonics (PU) to measure both constants for sol gel PZT thin films. Although extracting E with PU is rather well established now [8], it is much more challenging to characterize ν with this technique, though feasible. Indeed, E only requires longitudinal out-of-plane waves into the film, which are easily generated with a laser [8]. ν requires in-plane transversal waves that can be obtained by adding onto the film a dedicated pattern of metallic bumps [9]. In this paper, we detail first the PU technique. Then we describe how the samples were prepared to generate transversal waves. The longitudinal and transversal

measurements are therefore successively described. Finally, we detail the model used and extract both $E=82.1\text{GPa}$ and $\nu=0.39$ for sol gel thin film PZT.

II. PICOSECOND ULTRASONICS TECHNIQUE

A. Set up

PU consists in the use of femtosecond optical pulses generated by a laser (cf Fig. 1). The so-called pump pulse generates a thermal strain at the metal/PZT interface and consequently induces the propagation of an out-of-plane longitudinal acoustic wave. When this wave reaches the free surface, a variation of reflectivity can be detected with a second optical probe for which a controllable delay is performed (cf Fig. 1). Several reflections on the different interfaces of the sample stack are detectable. Therefore, PU allows to measure accurately the trip duration of the acoustic wave through the PZT layer. Besides, knowing the PZT thickness allows us to calculate the longitudinal acoustic velocity in the PZT (c_L). To get ν , the use of nanostructured cubes above the PZT are required. They generate transversal in-plane waves as sketched in Fig. 1. Finally, by using a dedicated acoustic model, one can extract E and ν from those in-plane and out-of-plane measurements.

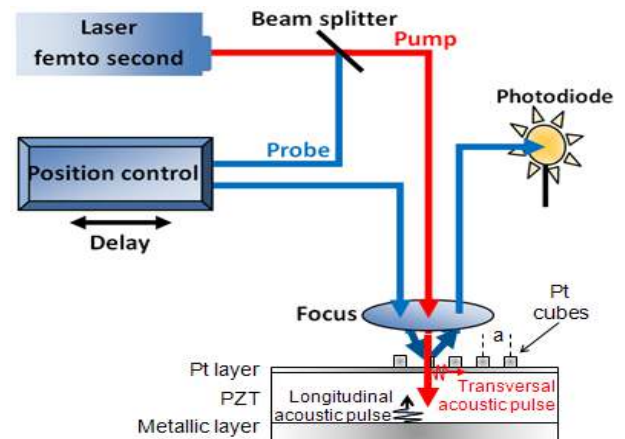


Fig. 1. Picosecond Ultrasonics schematic set up. The pump pulse delivery by a femtosecond laser generates an acoustic pulse propagating through the thin-film layer. The probe pulse detects the reflected signal.

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B. Technology

We prepared a 506nm thick, (100) oriented sol-gel PZT layer, as detailed in [2]. Fig. 2 shows a SEM cross section of this PZT layer exhibiting a very dense microstructure.

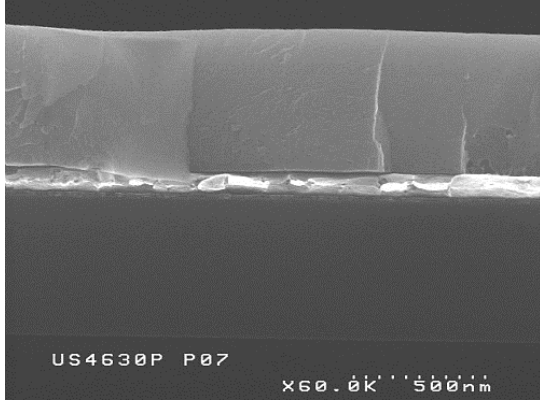


Fig. 2. Sol-gel 506nm thick, (100) oriented PZT layer SEM cross section.

As reported in [9], one can use an array of nanostructured metallic cubes (Fig. 3) in order to generate transversal waves once the laser pump shines on those cubes. We used a thin metallic film between the cubes and the thin film material to characterize. It allows obtaining the in-plane Rayleigh velocity (c_R) of the studied material from the Rayleigh velocity of the considered material and metallic layer system. Thus we deposited and patterned by e-beam lithography square Pt cubes ($h_c=100\text{nm}$ thick, $d=200\text{nm}$ long) exhibiting various pitches a from 400 to 800nm. We realized two samples with Pt layer thickness, between PZT and Pt cubes, of $h=15$ and 50nm .

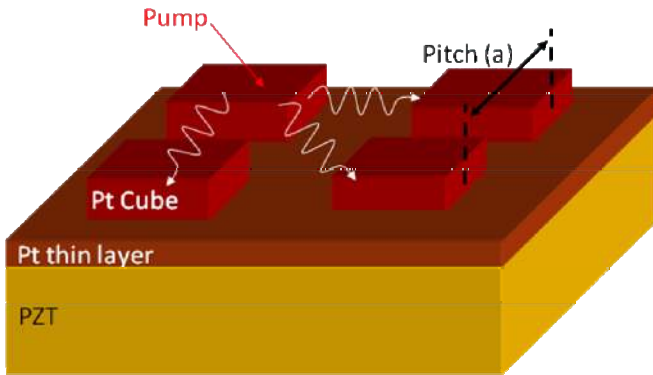


Fig. 3. Pt nanostructured cubes arrays on thin film PZT layer schematic view.

III. LONGITUDINAL MEASUREMENTS

Without nanostructured cubes, we can work on longitudinal waves. On transparent material such as PZT, the acoustic pulse partially reflected by the interface under PZT can interfere with the probe. This induces the so-called Brillouin oscillations exhibiting a period T given by

$$T = \frac{\lambda}{2nc_L \cos \theta} \quad (1)$$

λ is the probe wavelength, n is the optical index at λ , c_L is the longitudinal sound velocity and θ is the pump angle of incidence (typically 5°).

Fig. 4 shows the relative variation of reflectivity ($\Delta R/R$, or PU signal) versus time obtained on our PZT sample at $\lambda=425\text{nm}$ ($n \approx 2.71$). The inset focuses on the Brillouin oscillations. This experiment and equation 1 yields $c_L=4.73 \pm 0.07\text{nm/ps}$.

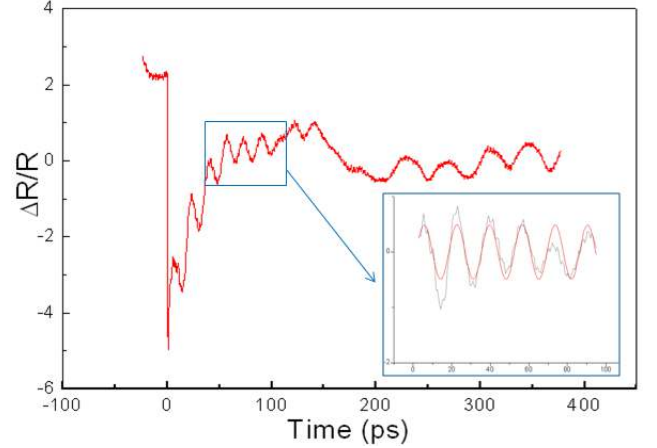


Fig. 4. Relative variation of reflectivity ($\Delta R/R$) versus time at $\lambda=425\text{nm}$ on sol-gel 506nm thick, (100) oriented PZT layer. Inset: focus on the Brillouin oscillation. Blue line: experiment, red line: model.

IV. TRANSVERSAL MEASUREMENTS

Now the pump is guided toward the Pt nanostructured cubes. Two kinds of vibration are generated: individual resonance of each cube and collective modes of resonance. We studied those last modes which resonance frequency depends on the cubes pitch, namely [9]

$$f_{i,j}(a) = c \sqrt{\frac{a^2 h}{a^2 h + V} \left(\frac{\sqrt{i^2 + j^2}}{a} \right)} \quad (2)$$

V is the volume of each cube ($V=d^2 h_c$), c is the velocity of the considered system (here Pt/PZT), i and j are the integer coordinates of the wave vector in the reciprocal lattice.

The PU signal of a 400nm-pitch array ($h=50\text{nm}$) and its Fourier transform amplitude are reported in Fig. 5. We can identify for each pitch and for this 50nm-thick Pt thin layer (cf Fig.3) the three first collective mode frequencies as reported in Figure 5. There is a good agreement between the model (Equation 2) and the measurements (Table 1). Indeed, successive branches are separated by the right factors ($\sqrt{2}, 2, \sqrt{5} \dots$) that correspond to the excitation of the same mode in different directions [9].

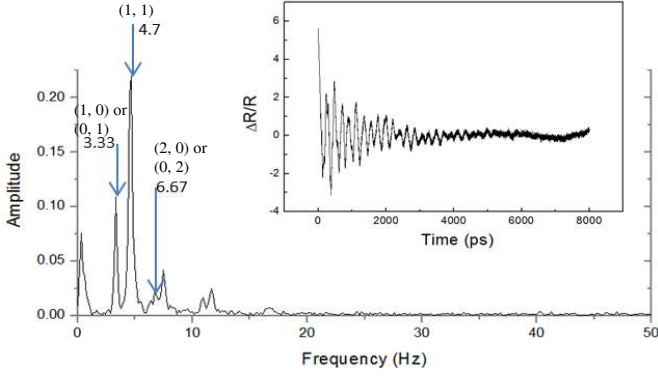


Fig. 5. Fourier transform (main graph) and PU signal (inset) on Pt nanostructured cubes ($a=500\text{nm}$)/ 50nm-thick Pt thin layer /PZT.

TABLE I
3 FIRST COLLECTIVE MODE FREQUENCIES – PT CUBES (PITCH=400NM)/PT LAYER 15 NM THICK/PZT

Measured frequencies (GHz)	Theoretical factor $\sqrt{i^2 + j^2}$	Measured factor $\sqrt{i^2 + j^2}$
3.33	1	1
4.7	1.41	1.41
6.67	2	2

We performed the same kind of measurements for several pitch ranging from 400 to 800nm, both for $h=15\text{nm}$ and $h=50\text{nm}$. We reported in Figure 6 the collective mode frequencies versus $1/a$ for $h=15\text{nm}$. Measurements can be fitted by Equation 2 by adjusting c , which is the velocity of the considered system (Pt/PZT) for a given Pt thin layer. We extracted $c=1.55\text{nm/ps}$ for $h=15\text{nm}$. With a 50nm-thick Pt layer, we extracted $c=1.36\text{nm/ps}$ (Figure 7).

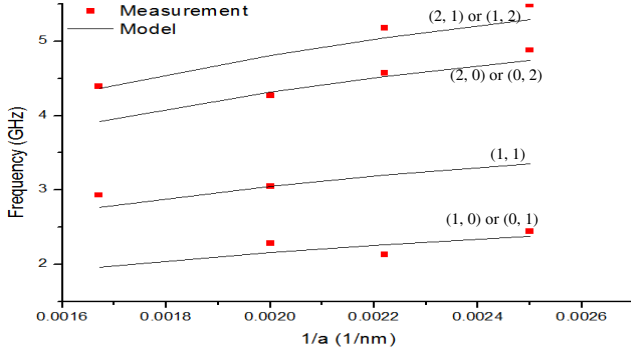


Fig. 6. Collective mode frequencies versus $1/a$ - Pt layer 15nm thick/PZT.

Finally, we aimed to extract the Rayleigh velocity of the PZT c_R^{PZT} . This can be obtained by extrapolating the data obtained on the Pt/PZT stack when Pt thickness is set to naught. Indeed, the Pt/PZT system acoustic velocity according to each individual layer velocity and to the Pt layer thickness h (Equation 3) [10, 11].

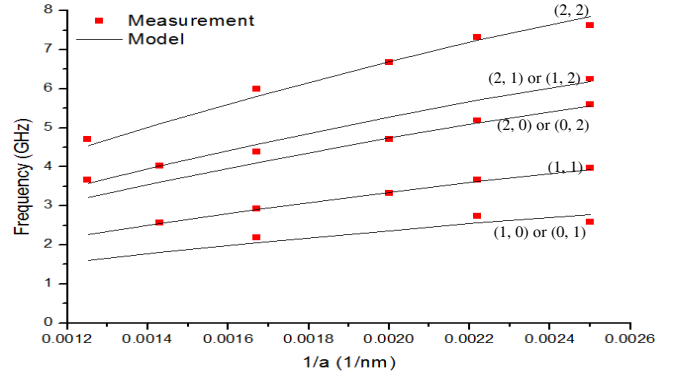


Fig. 7. Collective mode frequencies versus $1/a$ - Pt layer 50nm thick/PZT.

$$c(a) = \left(c_R^{PZT} - c_R^{Pt} \right) e^{\frac{h}{\delta}} + c_R^{Pt} \quad (3)$$

δ is a corrective factor and c_R^{Pt} is known to be 1.31nm/ps .

We plot c extracted from measurements on Fig.8 and we adjust c_R^{PZT} to fit the model with experimental data. Thus we subtract the Pt layer contribution and we are able to extrapolate the PZT in-plane Rayleigh velocity as being 1.84nm/ps .

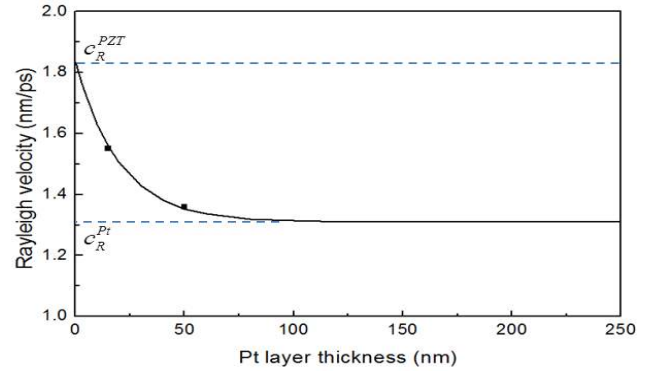


Fig. 8. Rayleigh velocity of the Pt/PZT system versus Pt layer thickness.

The Viktorov's approximation is given in Equation 4. It allows calculating the transverse velocity c_t from Rayleigh and longitudinal velocities for isotropic materials. We find $c_t=1.95\text{nm/ps}$.

$$\frac{c_R}{c_t} = \frac{0.718 - \frac{c_t^2}{c_L^2}}{0.75 - \frac{c_t^2}{c_L^2}} \quad (4)$$

V. YOUNG'S MODULUS AND POISSON'S RATIO CALCULATION

On isotropic materials, we can extract E and ν from longitudinal and transverse velocities using the theory of elasticity [11]. Equations (5) give the relations between c_L , c_t and E, ν .

$$\begin{cases} c_L = \sqrt{\frac{E(1-\nu)}{\rho(1+\nu)(1-2\nu)}} \\ c_t = \sqrt{\frac{E}{2\rho(1+\nu)}} \end{cases} \Rightarrow \begin{cases} \nu = \frac{c_L^2 - 2c_t^2}{2(c_L^2 - c_t^2)} \\ E = 2c_t^2 \rho \left(1 + \frac{c_L^2 - 2c_t^2}{2(c_L^2 - c_t^2)} \right) \end{cases} \quad (5)$$

Finally we can extract $E=82.1\text{GPa}$ and $\nu=0.39$ for the sol gel thin film PZT.

VI. CONCLUSION

We have shown in this paper the PU technique, which can be used to characterize the in-plane and out-of-plane acoustic velocities of thin films such as PZT. We extracted a longitudinal out-of-plane acoustic velocity of 4.73nm/ps . We also used nanostructured cubes to study in-plane waves, and we extracted an in-plane Rayleigh velocity of 1.84nm/ps . We made the approximation of isotropy of PZT to use the Viktorov's approximation which allows us to calculate the transverse velocity $c_t=1.95\text{nm/ps}$. Finally, using the theory of elasticity one can extract both $E=82.1\text{GPa}$ and $\nu=0.39$ for the sol gel thin film PZT.

REFERENCES

- [1] T. Bakke, A. Vogl, O. Zero, F. Tyholdt, IR. Johansen, D. Wang, "A novel ultra-planar, long stroke and low-voltage piezoelectric micromirror", *Journal of Micromechanics and Microengineering* 2010; 20, 064010.
- [2] M. Cuffe, E. Defay, P. Rey, G. Le Rhun, F. Perruchot, C. Ferrandon *et al.* "A fully packaged piezoelectric switch with low-voltage actuation and electrostatic hold", *IEEE Int Conf on Micro Electro Mechanical Systems (MEMS)*, 2010, pp. 212-215.
- [3] E. Fujii, R. Takayam, K. Nomura, A. Murata, T. Hirasawa, A. Tomozawa *et al.* "Preparation of (001)-oriented $\text{Pb}(\text{Zr},\text{Ti})\text{O}_3$ thin films and their piezoelectric applications", *IEEE transactions on Ultrasonics, Ferroelectrics and Frequency Control*, Vol. 54, n°12, December 2007, pp. 2431-2438.
- [4] R. Dejaeger, F. Casset, B. Desloges, G. Le Rhun, P. Robert, S. Fanget *et al.* "Development and characterization of a piezoelectrically actuated MEMS digital loudspeaker", *26th European conference on Solid-State Transducers (Euroensors)*, 2012, To be published.
- [5] M. Cuffe, M. Allain, J. Abergel, G. Le Rhun, M. Aid, E. Defay, D. Faralli, « Influence of the crystallographic orientation of $\text{Pb}(\text{Zr}, \text{Ti})\text{O}_3$ films on the transverse piezoelectric coefficient d_{31} », *IEEE Int. Ultrasonics Symposium*, 2011.
- [6] MA. Trindade and A. Benjeddou, "Effective electromechanical coupling coefficients of piezoelectric adaptive structures: critical evaluation and optimization", *Mechanics of advanced Materials and Structures*, Vol. 16, n°3, pp. 210-223, 2009.
- [7] R. Zachariasz, A. Zarycka and J. Ilczuk, "Determination of the lead titanate zirconate phase diagram by the measurements of the internal

friction and Young's modulus", *Materials Science-Poland*, Vol. 25, n° 3, pp. 781-789, 2007.

- [8] F. Casset, A. Devos, A. Le Louarn, P. Emery, G. Le Rhun, M. Cuffe, E. Defay, P. Ancey, S. Fanget, "PZT thin film elastic characterization using picosecond acoustics", " *Int. Conf. on Photoacoustic and Photothermal Phenomena (ICPPP)*, 2011.
- [9] PA. Mante, A.Devos, JF. Robillard, "Towards thin film complete characterization using picosecond ultrasonics", *IEEE Int. Ultrasonics Symposium*, 2008, pp. 1203-1206.
- [10] JF. Robillard, "Etude par acoustique picoseconde des vibrations individuelles et collectives de nanostructures organisées", Ph.D. dissertation, Université des Sciences et Technologies de Lille – Lille I, 22/07/2008.
- [11] PA. Mante, JF. Robillard, A. Devos, "Complete thin film mechanical characterization using picosecond ultrasonics and nanostructured transducers: experimental demonstration on SiO_2 ", *Applied Physics Letters* 93, 071909, 2008.