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Arnaud Devos, P. Emery, E. Defay, N. Ben Hassine, G. Parat. Ultrafast optical technique for measuring the electrical dependence of the elasticity of piezoelectric thin film : demonstration on AlN. Review of Scientific Instruments, 2013, 84, pp.015007-1-4. 10.1063/1.4788936 . hal-00796410

HAL Id: hal-00796410

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Submitted on 2 Jun 2022

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Cite as: Rev. Sci. Instrum. **84**, 015007 (2013); <https://doi.org/10.1063/1.4788936>

Submitted: 29 November 2012 • Accepted: 08 January 2013 • Published Online: 29 January 2013

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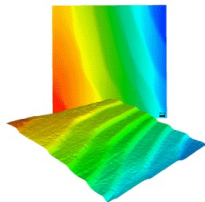
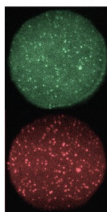
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Ultrafast optical technique for measuring the electrical dependence of the elasticity of piezoelectric thin film: Demonstration on AlN

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(Received 29 November 2012; accepted 8 January 2013; published online 29 January 2013)

We present a technique based on ultrafast acoustics which permits us to measure the electrical dependence of the elastic properties of a thin piezoelectric layer. Ultrafast acoustics offers a unique way of measuring elastic properties of thin-layer in a non-destructive way using ultrashort optical pulses. We apply this technique to a thin layer to which a dc voltage is simultaneously applied. Both the film thickness and the sound velocity are affected. The two effects can be separated by use of a semi-transparent top electrode. A demonstration is made on a thin aluminum nitride (AlN). From that the d_{33} piezoelectric coefficient and the stiffness variation induced by the bias in AlN are measured.

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I. INTRODUCTION

When a piezoelectric material is submitted to a force, it generates an electrical voltage proportional to the magnitude of the force. Conversely, when a voltage is applied to the same material, it is submitted to a deformation. Both effects have been used for a long time to convert mechanical energy into electrical energy in transducer.

Recently, thin film piezoelectric materials have attracted much attention due to their wide applications such as microactuators, microsensors, motors or pumps, medical transducers, and filters. Devices based on thin piezoelectric films can be integrated in microelectronics processes especially since the introduction of aluminium nitride (AlN) as piezoelectric material.¹

In designing such devices it is required to know the elastic and piezoelectric properties of the material which can differ from the macroscale material. Most of the piezoelectric thin films are polycrystalline materials. The piezoelectric effect is averaged over all the grains. Elasticity and piezoelectricity depend on the material growth process and dedicated characterization techniques have to be developed. An accurate method to determine the piezoelectric coefficient d_{33} is to use a double side laser interferometer, which measures the thickness change unambiguously and avoids errors due to sample deflections.² Elastic properties of thin AlN films can be measured using ultrafast acoustics an optical technique³

Being able to tune the AlN elastic properties using a bias would find interest in many other applications such as micro- and nano-resonators, tunable filters, phononic crystals electrically controlled by a dc field. Consider for example a bulk acoustic wave resonator which is very similar to the basic quartz crystal scaled down in size. It is commonly used in filtering components for wireless communications⁴ and frequency oscillators.⁵ An important limitation of such acoustic devices is that after elaboration the frequency of the device cannot be tuned.

Chen *et al.* have shown that the resonance frequency of an AlN resonator is shifted by superimposing a dc voltage to the radio-frequency signal.⁶ Lanz *et al.* attributed the effect to a linear change in the elastic constant of the piezoelectric material due to the electrical bias (known as elastoelectric effect).⁷ A similar effect was observed in a quadratic manner for electrostrictive resonators including perovskite materials as SrTiO₃ or (Ba,Sr)TiO₃.⁸ Up to now no direct measurement has confirmed this point.

In this paper we describe an optical technique for measuring the elasticity changes induced by a dc voltage on a thin piezoelectric film. The technique is derived from the so-called ultrafast acoustics (UA) which is an optical technique that permits to implement a sonar at nanoscale. Using a semi-transparent top electrode and some specific wavelength conditions, we can access to piezoelectric and elastoelectric effects. A demonstration is made on AlN for which the d_{33} piezoelectric coefficient and the elastoelectric coefficient are independently measured. The results we obtained are compared to those previously published and obtained on acoustic devices.

II. EXPERIMENTAL SETUP

A. Ultrafast acoustics

UA is an optical technique which uses a femtosecond laser to excite and to detect acoustic waves at very high frequency (100 GHz to 1 THz). Such an optical setup offers a unique way of implementing a sonar at nanoscale. By measuring the time-delay between successive echoes it is basically possible to access to the thickness or to the longitudinal sound velocity in films whose thickness can be as small as 5 nm. The UA capabilities can be enhanced if one uses several laser wavelengths. More parameters are measurable, for example, thickness and sound velocity are simultaneously reachable and the measurement accuracy can be significantly enhanced.

Figure 1(a) gives a schematic diagram of the experimental setup which is a two-color pump-probe experiment

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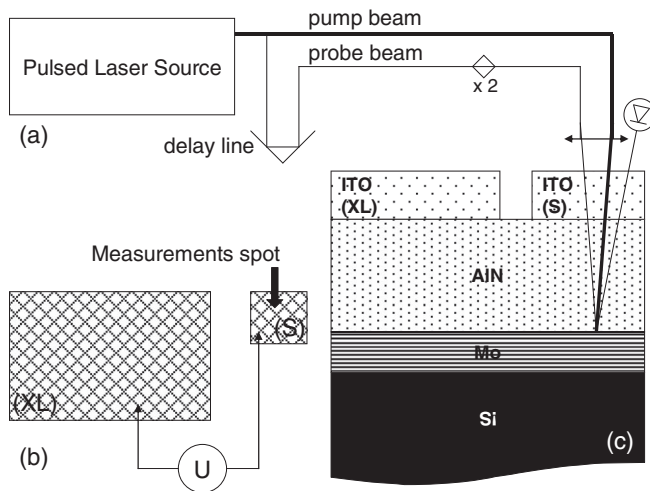


FIG. 1. (a) Scheme of the experimental setup, the probe beam is focused in a BBO crystal to generate the second harmonic. (b) Top view of the studied sample, showing the pattern of the ITO layer. The two surfaces (designated as XL and S, with a 30:1 surface ratio) realize in-series capacitors connected by the full-sheet Mo layer. The measurements are performed on the small capacitor. (c) Cross section sketch of the sample.

associated with a tunable femtosecond laser. Details can be found in Ref. 9. The absorption of a first optical pulse (the pump pulse) generates a thermal strain pulse whose extension can be less than ten nanometers. The resulting acoustic pulse propagates in the sample normally to the surface at the longitudinal sound velocity. The reflection at the film/substrate interface yields returning echoes which are detected at the surface by another delayed optical pulse (the probe pulse). From an accurate measurement of the echo delay, one can deduce the sound velocity or the thickness of the film.¹⁰

In transparent media, the reflectivity can also present an oscillating part so-called Brillouin oscillation, which is the result of interferences between the probe that is reflected from the interfaces of the sample and the light partially reflected from the strain pulse. Brillouin oscillation permits to measure the sound velocity and the thickness from the optical index.⁹

In case of a strain pulse generated in a buried layer, a third acoustic contribution appears in the reflectivity curve known as a reflectivity step. It is a step, experienced by the transient reflectivity when the strain pulse enters or is reflected within a transparent layer.¹¹ The probe beam is reflected at both interfaces of the transparent layer and these reflections interfere. If the thickness is slightly modified the phase relation between both reflections is modified and so is the resulting intensity of the reflected probe. Due to the interferometric nature of the effect, displacements as small as those carried by picosecond acoustic pulses can be detected. The sound velocity of the film and the thickness determine the delay at which the step is observed which is the acoustic time-of-flight. But it is important to point that the step magnitude does not depend on the sound velocity. It is determined by the optical path namely the film thickness, the film optical index and the probe wavelength.¹² As shown in the following, we can take advantage of the high sensitivity of such a contribution to measure the small thickness change induced by the electric field on the piezoelectric film.

B. Ultrafast acoustics under electric field

In this paper, we present a technique for measuring the effect of an electric field on the elastic property and on the thickness of a thin piezoelectric film. To apply the electric field, the piezoelectric layer must be sandwiched between electrodes. A first basic idea would be to take advantage of the high sensitivity of the ultrafast acoustic technique to generate acoustic wave in the top electrode and to detect the returning echoes issued from the interface between the piezoelectric layer and the bottom electrode. Measuring the time-shift induced by the dc voltage would lead to an estimation of the impact of the dc voltage on the piezoelectric layer elasticity.

But one cannot be satisfied with such a basic ultrafast acoustic measurement in the top electrode for the following reason. The electric field affects simultaneously the sound velocity and the film thickness. Indeed, the electric field changes the C_{33} elastic constant through the elastolectric effect and thus the longitudinal sound velocity. It also modifies the film thickness through the inverse piezoelectric effect. Both effects affect the acoustic propagation and measuring a shift of the echo delay does not permit to separate the two contributions.

We propose to use the acoustic detection through the reflectivity step to isolate the thickness change induced by piezoelectric effect. The time-delay at which such a step is detected is as for an echo only dependent on the ratio between thickness and sound velocity. But, the magnitude of a reflectivity step is strongly dependent on the film thickness and a tiny change in thickness can induce a sign change of a reflectivity step. So, by combining a measurement of the time-shift and a measurement of a magnitude change both induced by the dc voltage, one can separate both effects.

A condition concerning the top electrode is needed. As reflectivity steps are detected only in transparent layers, we cannot use an absorbing layer as a top electrode. That is the reason why we propose to use a thin indium tin oxide (ITO) layer as top electrode which combines transparency and electrical conductivity.

As a resume, we propose to deposit a thin ITO layer on top of the studied thin piezoelectric film to be characterized. Then we perform ultrafast acoustic experiments under various dc voltages. An acoustic pulse is emitted from the buried electrode due to the transparency of the piezoelectric layer and ITO electrode. Its arrival at the free surface is detected as a reflectivity step whose time-delay and magnitude are studied as a function of the dc voltage. In the following we illustrate the technique by applying it on a thin AlN film.

III. DEMONSTRATION ON ALN

A. Sample description

In the following we present the results obtained on a thin AlN film to which an external electric field is applied. From that we deduce the d_{33} piezoelectric coefficient and the stiffness variation in AlN induced by the electric field. The studied sample is composed of the following stack, from the top layer to the substrate: ITO(200 nm)/AlN(1 μ m)/Mo(150 nm)/Si substrate. All the layers were sputtered on a 200 mm (100) Si substrate. The AlN is strongly c-axis oriented,

exhibiting a rocking curve as low as 1.2° . A cross section of the sample is sketched in Fig. 1(b). The ITO layer is a semi-transparent and conducting layer that has been patterned by wet etching, providing the top electrode of the capacitors. As the AlN and Mo layers are not patterned, there is no direct access to the bottom Mo layer. The dc voltage is applied to two ITO top electrodes exhibiting very different surfaces which defines two in-series capacitors. As the surface ratio is huge, the voltage is mainly applied to the small capacitor, which is the device under study. The size of the capacitor where the laser probe is focused is 0.25 mm^2 . The dc voltage range is $\pm 150 \text{ V}$.

B. Results with no dc voltage

Figure 2(a) presents two transient reflectivity results obtained on the sample probed at 440 nm without any dc voltage. The first signal has been measured on a part of the sample where there is no ITO, the second on the whole stack. In the first case the pump light is absorbed only in the Mo layer and two acoustic pulses are emitted, one towards the free surface and the second towards the substrate. The propagation of a strain pulse within the AlN layer is evidenced by the detection of a Brillouin oscillation immediately after the generation. When it reaches the free surface, the pulse is reflected and the strain is sign changed and a strong reflectivity step is detected close to 90 ps . The smaller step detected near 75 ps results from the entrance of the other strain pulse in AlN after one round trip in the Mo bottom electrode.

The signal measured on the whole sample is slightly more complicated due to some slight absorption of the pump

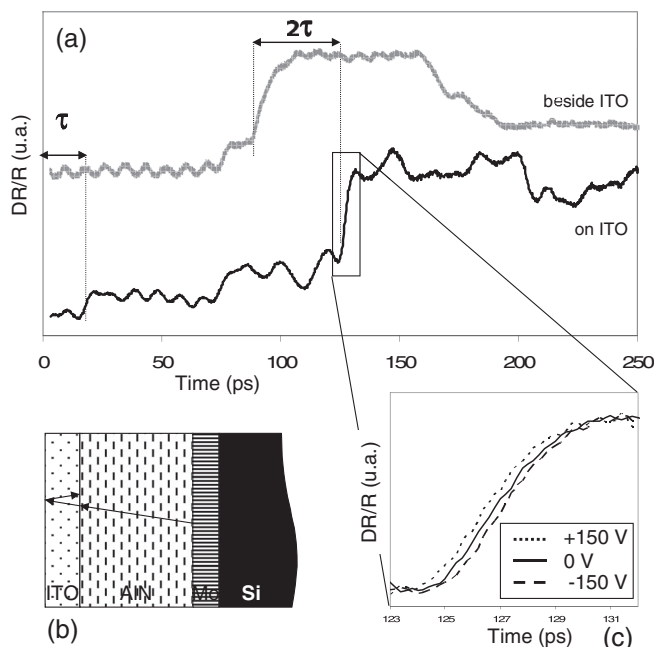


FIG. 2. (a) Transient reflectivities obtained on the sample, beside and on the ITO layer, pumped at 880 nm and probed at 440 nm (the thermal background has been subtracted). (b) Cross section of the sample and acoustic path of the pulse that is responsible for the studied reflectivity step on the ITO/AlN/Mo stack. (c) Zoom of the studied reflectivity step detected at three different dc voltages ($+150 \text{ V}$, 0 V , -150 V).

pulse in the ITO. This is supported by the detection of a small reflectivity step near 20 ps [referred as τ in Fig. 2(a)]. Both signals have also many points in common. First, one retrieves a Brillouin oscillation in AlN. Second, the step due to the entrance of the echo issued from the Mo electrode is still visible at the same time-delay. One also detects a strong reflectivity step but it is time-delayed compared to first experiment (from 90 to 125 ps). The delay (2τ) is due to propagation of one round trip in the ITO layer [illustrated in Fig. 2(b)]. In the following we focus on this step and we measure the effect of an electric field on its time-delay and on its magnitude.

C. Acoustic measurements under electric field

We performed three experiments at -150 V , 0 V , and $+150 \text{ V}$ and we focus on the temporal drift of the strong reflectivity step induced by the dc voltage as shown in Fig. 2(c). One can note that the time of flight t decreases for a positive voltage. The time of flight variation is $dt = -0.85 \text{ ps}$ when the dc polarization varies from -150 to $+150 \text{ V}$. The relative change in time-of-flight per volt is -23 ppm/V . It is important to note that this variation results from a change of the thickness (induced by the piezoelectric effect) and a change of the sound velocity (induced by the elastolectric effect).

To separate the thickness and the stiffness effects, we take advantage of the sensitivity of the magnitude of a reflectivity step to the optical path in the transparent layer. Furthermore, for some given probe wavelengths, it is particularly sensitive around some specific thicknesses of the AlN layer. Thus we select a probe wavelength around which a tiny change in the AlN layer thickness can change the reflectivity step sign. In the present case we retain 435 nm as a probe wavelength. Figure 3(a) shows the step's magnitude detected at 435 nm versus the AlN thickness. We perform experiments at $+150$, 0 , and -150 V . The application of a dc voltage significantly changes the step's magnitude, as shown in Fig. 3(b). According to the sign of the applied bias, the step is positive or negative. That means that the bias induces a change in the film thickness. In the present case, a positive bias gives a negative step and following the curve reproduced in Fig. 3(a) it means

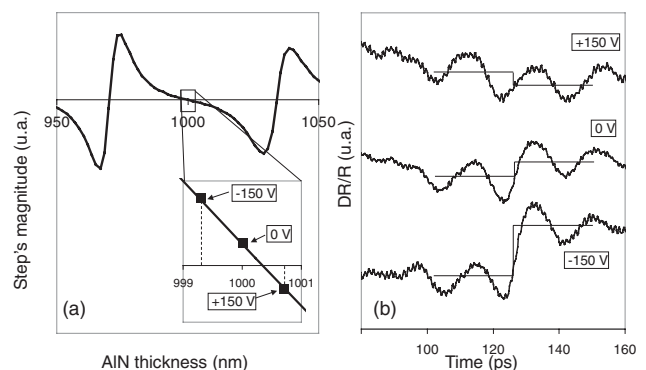


FIG. 3. (a) The plain line is the model for the reflectivity steps' magnitude, probed at 435 nm , as a function of the AlN thickness. The inset is a zoom around $1 \mu\text{m}$, where the steps' magnitude at $+150$, 0 , and -150 V has been reported. (b) Evolution of the reflectivity step's magnitude, pumped at 870 nm and probed at 435 nm , for three different dc voltage ($+150 \text{ V}$, 0 V , -150 V).

that the thickness increases. Assuming no change in the AlN refractive index, it can be converted in a thickness change of 1.4 nm for a voltage varying from -150 V to 150 V. This value can also be given in meter per volt as 4.7×10^{-3} nm/V.

IV. DISCUSSION AND SUMMARY

First, the thickness change induced by the dc voltage is a direct measurement of the d_{33} piezoelectric coefficient. Indeed, due to the inverse piezoelectric effect, when the layer is submitted to a voltage U , the thickness e is expected to change as $de = d_{33}U$. From the de reported in Sec. III we obtain $d_{33} = 4.7 \times 10^{-3}$ nm/V. This value is in very good agreement with literature 5×10^{-3} nm/V.¹³ It is important to know the sign of d_{33} which depends on the direction of AlN polarization. In a previous study it was observed that the AlN under study has a downward polarization which means that the thickness increases when a positive bias is applied.¹⁴

From the measured d_{33} we can now separate the two contributions of the time of flight variation. Indeed, the stiffness $C_{33} = \rho v^2$, where ρ is the mass density and v is the longitudinal sound velocity. The sound velocity is given by $v = e/t$, e being the AlN film thickness and t the time-of-flight of the longitudinal acoustic wave in the AlN. A change in the film thickness also induces a change in the mass density. Combining both effects we can write

$$\frac{dC_{33}}{C_{33}} = \frac{de}{e} - 2\frac{dt}{t}. \quad (1)$$

From that expression and from the measured values, it is possible to get the relative change of C_{33} which is 51 ppm/V. This value has been obtained on a $1 \mu\text{m}$ thick layer with a dc voltage of 300 V. In order to get an intrinsic value of the material, we divide this relative stiffness change by the electric field. We thus obtain $51 \text{ ppm} \cdot \mu\text{m/V} = 51 \times 10^{-3}$ nm/V for the intrinsic change in stiffness induced by the bias.

We can compare our results to those obtained by Lanz *et al.* from electrical measurements.⁷ They studied the frequency shift induced by a dc voltage on the antiresonance of a AlN-based resonator. The antiresonance frequency f_a of the first thickness mode can be written as

$$f_a = \frac{1}{2e} \sqrt{\frac{C_{33}^D}{\rho}}, \quad (2)$$

where e is the thickness of the AlN layer, C_{33}^D is the stiffness at fixed D , and ρ is the mass density. The stiffness variation can be extracted from the antiresonance frequency f_a shift experienced by a resonator induced by a bias is applied. A differential calculation on Eq. (2) gives

$$\frac{dC_{33}^D}{C_{33}^D} = d_{33} \frac{U}{t} + 2 \frac{df_a}{f_a}. \quad (3)$$

Lanz reported a f_a variation reaching 72 ppm/V for a 380 nm thick AlN film.⁷ By reporting this result in Eq. (3) and assuming that d_{33} is identical to ours, the relative stiffness vari-

ation is 59 ppm. $\mu\text{m/V}$. This result is also in good agreement with the value obtained by our technique.

We presented an ultrafast optical technique for measuring the effects induced by an electric field on a thin piezoelectric layer. First, the inverse piezoelectric effect induces a tiny change in thickness which has been measured directly using a transparent top electrode and ultrafast acoustics at a specific wavelength. From that, we measured the d_{33} piezoelectric parameter in AlN which has been found to be in very good agreement with literature. This is a first interest for the presented method, being able to measure d_{33} on thin film which is known to be different from bulk. In this work, the technique has been applied to AlN a material which has attracted considerable attention recently. It is important to note that d_{33} in AlN is about twenty times smaller than in PZT that demonstrates the high sensitivity of the method.

Secondly, the electric field also affects the sound velocity and the corresponding elastolectric coefficient has been measured from d_{33} and the change in the time-delay propagation. The linear elastolectric effect is found to dominate significantly the piezoelectric effect in AlN which is consistent to what has been proposed by Lanz from electrical measurements. This work also demonstrates that it is possible to electrically tune the elastic properties of a thin piezoelectric material. That suggests some promising applications such as tunable radio-frequency filters or switchable phononic crystals filters.

ACKNOWLEDGMENTS

The authors want to thank Grégory Caruyer, Alexandre Volatier, Christophe Zinck, and Denis Mercier for fruitful discussions. This work was partly funded by the French Public Authorities through the NANO 2012 program and was partially realized in the framework of the French ANR program “Mélusine.”

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