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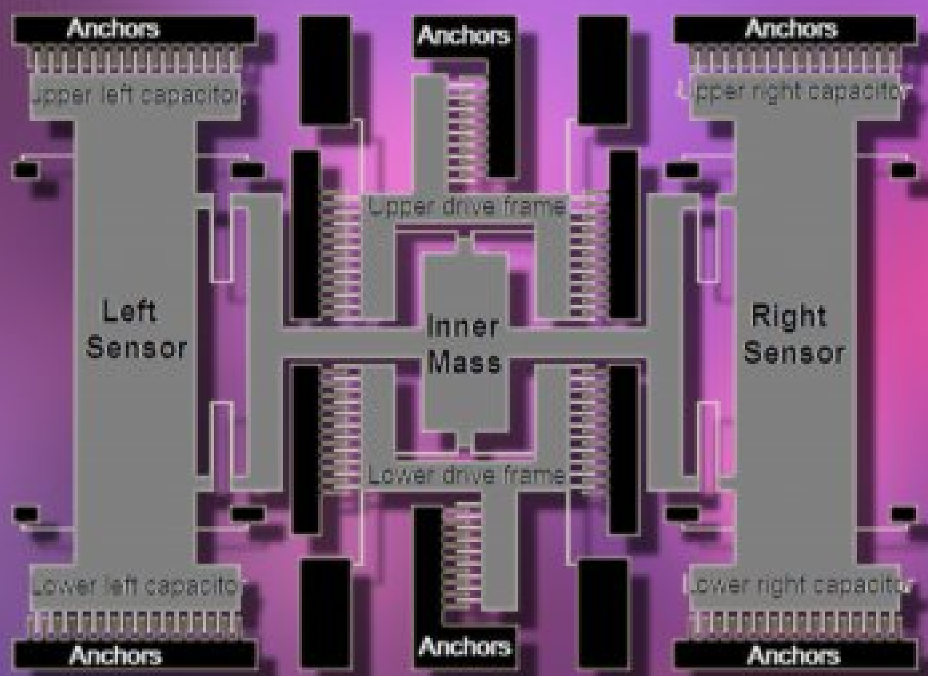

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Contents

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Research Articles

Piezoresistive Sensors Development Using Monolithic CMOS MEMS Technology
A. Chaehoi, M. Begbie, D. Weiland, D. O'Connell and S. Ray 1

A Fully Symmetric and Completely Decoupled MEMS-SOI Gyroscope
Abdelhameed Sharaf, Sherif Sedky, Mohamed Serry, Amro Elshurafa, Mahmoud Ashour and S. E.-D. Habib.......... 10

Surface Acoustic Wave Strain Sensor Model
William Wilson, Gary Atkinson.......... 23

$Pb(Zr,Ti)O_3$ (PZT) Thin Film Sensors for Fully-Integrated, Passive Telemetric Transponders
Richard X. Fu, Ryan C. Toonen, Eric H. Ngo, Melanie W. Cole Samuel G. Hirsch, Mathew P. Ivill, Clifford W. Hubbard 34

Low Cost Three-Dimensional Anemometer for High Spatial Resolution Wind Profiling
John Hirano and David Garmire 43

Mechanical Robustness of FPA in a-Si Microbolometer with Fine Pitch
Hee Yeoun Kim, Kyoung Min Kim, Byeong Il Kim, Won Soo Jang, Tae Hyun Kim and Tai Young Kang.......... 56

Point-Mass Model for Nano-Patterning Using Dip-Pen Nanolithography (DPN)
Seok-Won Kang and Debjyoti Banerjee.......... 64

Biomedical Applications of Modified Carbon Glassy Electrode Sensor with Nanoparticles and Dendrimers
Solomon W. Leung, Yuanhong Wang and James C. K. Lai 74

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How to reach 80,000+ addressable mobile audiences?

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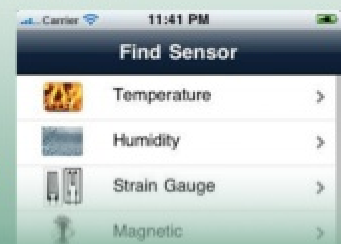

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Pb(Zr,Ti)O_3 (PZT) Thin Film Sensors for Fully-Integrated, Passive Telemetric Transponders

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Abstract: The great potential of taking advantages of PZT in a single chip to achieve inexpensive, fully-integrated, passive telemetric transponders has been shown in this paper. The processes for the sputter deposition of Pb(Zr,Ti)O_3 (PZT) thin films from two different composite targets on both Si and c-plane sapphire substrates have been demonstrated. PZT thin films have been deposited by sputter technique. PZT films were deposited onto substrates (Si [(100) Cz wafer] and c-plane sapphire (0001)//Ti//Pt) followed by sputter-deposited Pt top electrodes. X-ray diffraction results showed that both sputtered PZT films were textured along the [110] direction. The degree of preference for the [110] direction was greater on sapphire substrate where the intensity of that peak is seen to be larger compared to the intensity one Si substrate. TEM data revealed that both sputtered PZT films were polycrystalline in nature. Selected area diffraction (SAD) pattern showed that the degree of disorientation between the crystallites was smaller on sapphire substrate compared to on Si substrate, which confirmed the results from the XRD. The remnant polarization P_r on sapphire substrate was larger than on Si's. The leakage current for the 11 % Pb target sputtered film was much less than 22 % Pb target sputtered film. The breakdown voltage on sapphire substrate was the best. However, for the 11 % Pb target sputtered film's breakdown voltage was much higher than 22% Pb target sputtered film. *Copyright © 2011 IFSA.*

Keywords: PZT, Sputter deposition, Ferroelectric properties, Transponder

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1. Introduction

There is a critical need to develop the technology of fully-integrated, passive telemetric readout systems. Ultrasonic sensors accessed via microwave transponders could allow for a new and improved means of remotely testing structural integrity in a nondestructive manner. One of the many applications of this potential innovation is the remote detection of defects in aging aircraft. The objective of this research is to explore, develop and demonstrate the feasibility of providing customers with inexpensive, scalable, passive and ultra-low power consuming fully-integrated circuits that enable analog information from a piezoelectric sensor to be telemetrically monitored via a RFID-like backscatter transponder.

Current structural health monitoring devices employ wire-dependent sensors that must be inconveniently routed and secured without influencing the performance of the system being monitored. We envision a compact, wireless, cost-effective, disruptive technology that can be field-implemented by simply adhering this single-chip device to a monitored structure. This passive component would require no maintenance; its non-disruptive size would easily allow for redundant placement in order to ensure long-term reliability. As shown in Fig. 1(a), microstructural defects could be detected with a biostatic-sonar-like approach. A signal generator would drive an ultrasonic transducer which would send acoustic waves throughout a monitored structure such as an aircraft panel. The signals received from an array of N detection nodes would be wirelessly transmitted to a centralized digital signal processing (DSP) station where a multilateration technique would interpret the data and determine the location and severity of microfaults resulting from structural fatigue. By using high quality factor (Q) resonator based frequency-select filters, a specific frequency channel (centered on f_n for n = 1, 2, ..., N-1, N) could be assigned to each of the N nodes. Each detection node would be composed of a lead zirconate titanate (PZT) based sensor that would modulate the tunable barium strontium titanate (BST) based reflection phase-shifter of a backscatter transponder. PZT is a piezoelectric material that responds to an acoustic waveform by generating a proportional voltage signal [1-5]. BST is a ferroelectric material whose relative permittivity is a function of the magnitude of an externally applied electric field.

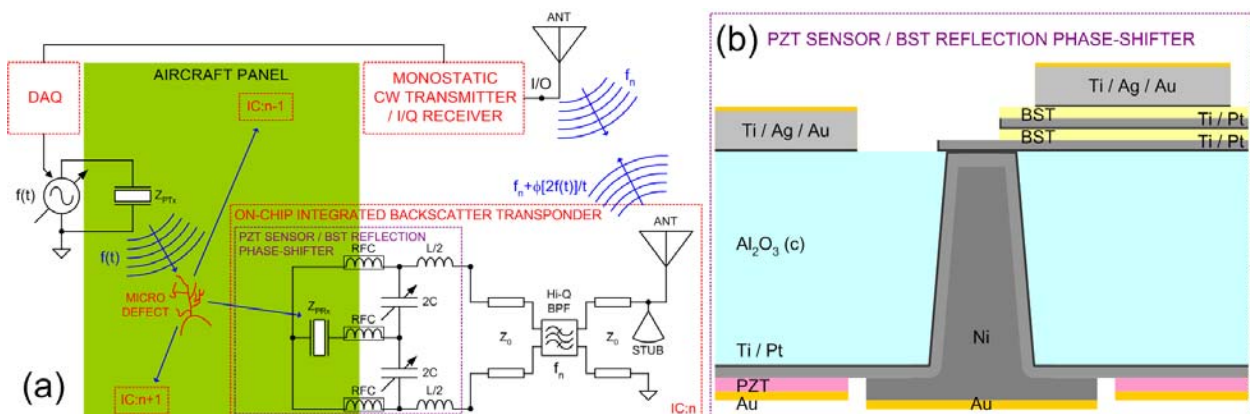

Fig. 1 (a) Diagram depicting the role of integrated PZT/BST in a wireless, telemetric, microfault detection system. The voltage generated by a PZT-based, ultrasonic sensor modulates the reflection coefficient of a tunable, BST-based, backscatter transponder. **(b)** The PZT sensor is fabricated on the bottom-side of a sapphire (Al_2O_3 (c)) wafer while the BST reflection phase-shifter is fabricated on the top-side.

One of the advantages of transmitting the analog data of the PZT sensor using phase modulation (PM) is that this type of data encoding is robust against atmospheric signal attenuation. There is a strong

interest in the radio frequency identification (RFID) community to develop S-band (2 to 4 GHz) transponders. However, the short-range data link needed for our application could operate in the K-band (18 to 26 GHz). Operating within this frequency band could reduce the long-range electronic signature of these sensor nodes as this particular band is known to experience heavy signal absorption due to atmospheric humidity. The goals of this research include: enhancing the piezoelectricity of PZT-based thin films and optimizing the ferroelectric properties of BST-based thin films; developing PZT-based interdigital sensors (IDSs) and stacked BST-based metal-insulator-metal (MIM) capacitor arrays; characterizing PZT-based, ultrasonic sensors and BST-based, microwave, reflection phase-shifters; and developing a process that successfully enables the integration of PZT and BST-based thin films onto a single chip—as depicted in Fig. 1(b). This paper is focused on PZT MIM capacitor fabrication and test on both Si and sapphire substrates.

2. Experimental

The PZT studied in this work was deposited by physical vapor deposition (sputtering) using two different PZT targets with compositions of 22% Pb $Pb_{1.22}Zr_{0.52}Ti_{0.48}O_{3.22}$ and 11% Pb $Pb_{1.11}Zr_{0.52}Ti_{0.48}O_{3.11}$. The sputtering was performed in an Oerlikon Clusterline® 200. The PZT itself was grown on top of a film stack (consisting of Si (or Sapphire)/Ti/Pt/PZT/Pt) in order to facilitate the manufacturing of a capacitive test structure used for ferroelectric and breakdown characterizations. All layers were sputter deposited in an Oerlikon Clusterline®. The sputtered PZT films were produced from a 99.995 % pure target with a Zr to Ti ratio of 52:48 and an excess of Pb.

Ferroelectric data was acquired from the simple capacitor devices where the underlying Pt in the above stated underlying film stacks served as the bottom electrode. The top Pt electrode was incorporated through lift-off of a blanket Pt film. The mask used produced an array of 2.5 X 10^{-3} cm^2 squares (top electrodes). To expose the bottom electrode, a portion of the PZT was etched down to the underlying Pt film by use of an HCl:HF:H_2O solution.

Remnant polarization P_r was determined by use of a two point probe station. The probes were attached to a HP Agilent 4275A LCR Meter, which was controlled using a Radiant Technologies Charge 2.1 software package. X-ray diffraction (XRD) analysis was performed on Bruker GADDS system. Transmission electron microscopy (TEM) analysis was performed using a JEOL 2010F transmission electron microscope with an EDAX energy dispersive spectroscopy (EDS) attachment.

3. Results and Discussion

3.1. X-ray Diffraction

From Fig. 2 XRD results showed that both sputtered PZT films were on different substrates textured along the [110] direction. The degree of preference for the [110] direction was greater in the sapphire substrate film where the intensity of that peak is seen to be larger compared to the intensity for the sputtered PZT on Si substrate. Additionally, in the sputtered film, all reflections were comparatively low in intensity and were shifted with respect to the bulk PZT values due to strain the films. Overall the PZT films on sapphire substrates were more oriented with all peaks present having higher intensities.

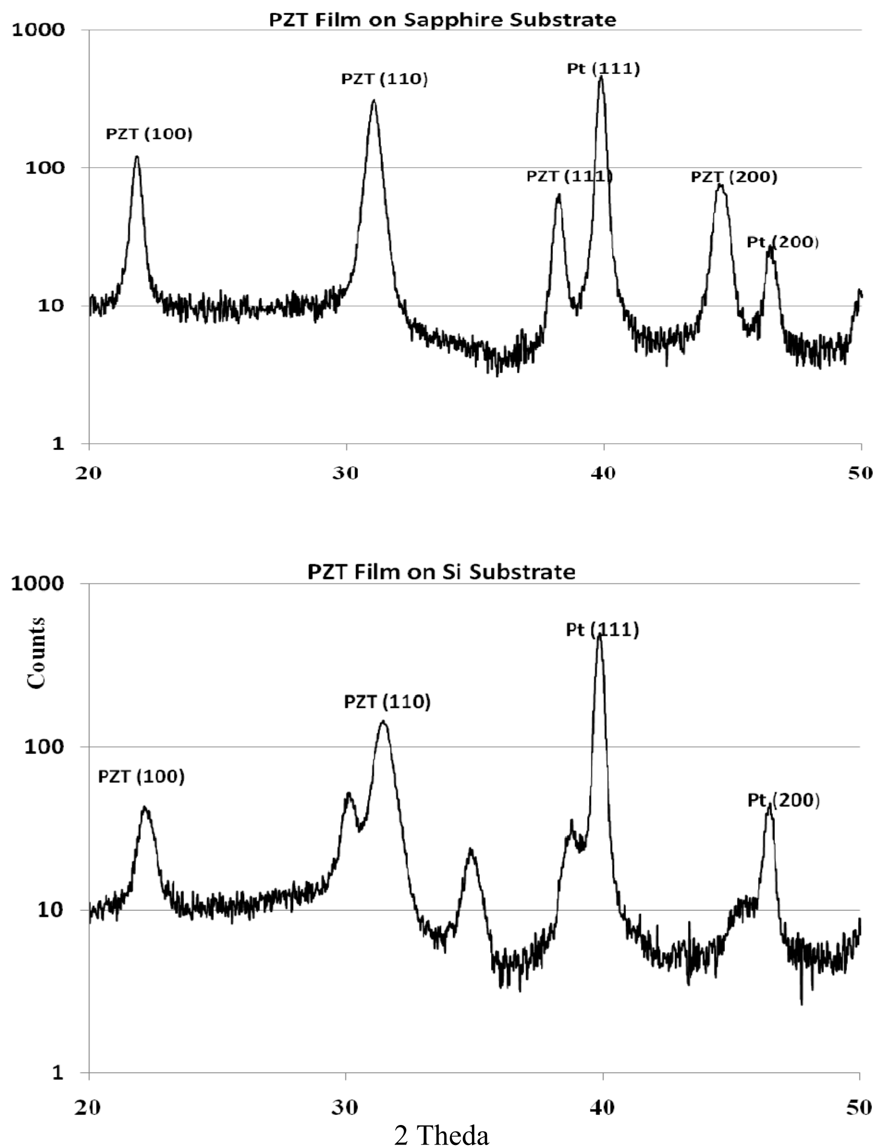

Fig. 2. Powder diffraction PZT film patterns sputtered on sapphire substrate (Top) and Si substrate (Bottom).

3.2. TEM

High resolution transmission electron microscopy, HRTEM, analysis was performed on cross- sections of sputtered PZT films on both Si and sapphire substrates. As both PZT samples displayed near identical TEM results, only one figure is presented. TEM sample area diffraction (SAD) data revealed the sputtered PZT films on both substrates were polycrystalline and had the Perovskite structure, as shown in Fig. 3 and Fig. 4. Furthermore, the energy dispersive spectroscopy (EDS) data consistently showed that both films were Zr-rich with respect to Ti. A particularly noteworthy aspect of these PZT samples was the fact that they had a Pb concentration gradient that was most pronounced near the films' surfaces. It was at the surface where this gradient reached its maximum in terms of excess Pb. It is suspected that during the annealing step of the PZT film fabrication, the crystallization process initiated at the Pt/PZT interface and propagated toward the surface. As the requisite amount of Pb was consumed, an excess may have been pushed upward which might have resulted in this residual Pb-rich region. An additional observation that supports this directional crystallization hypothesis is the fact that the degree of crystallinity was greater toward the bulk of the film as data was collected in a direction away from its surface. A previous study on the same material system [6] has also identified the presence of an interfacial layer between the top electrodes of sputter Pt/PZT/Ti/Pt type structures.

Further investigation is required to better understand the formation of this layer. Nevertheless, the structural quality of both films is comparable.

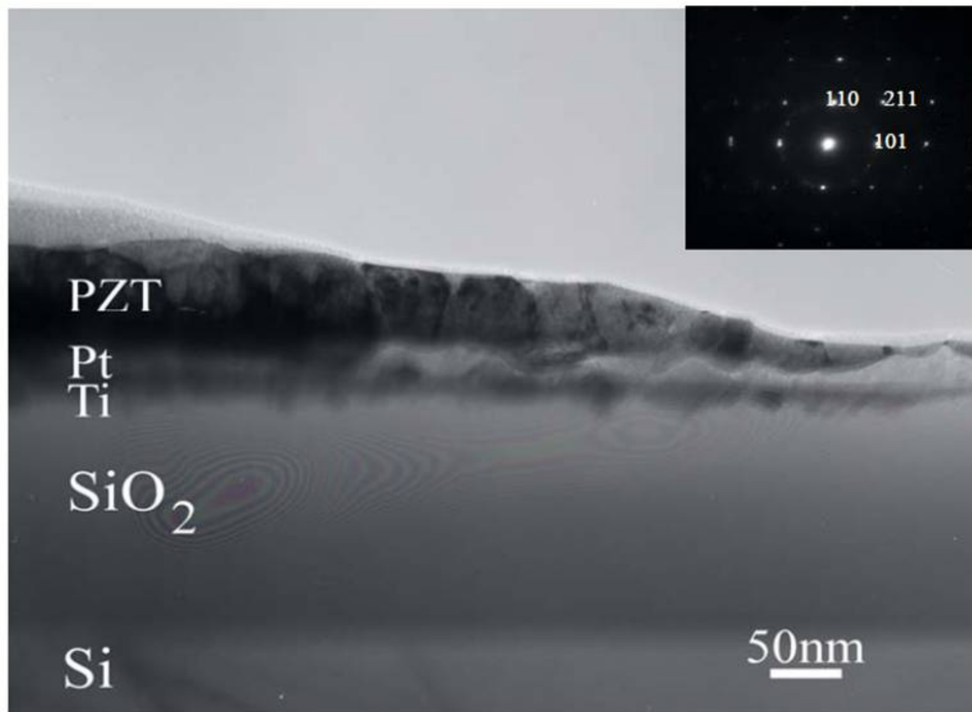

Fig. 3. TEM image of PZT displaying film cross section along with Si substrate and SAD pattern.

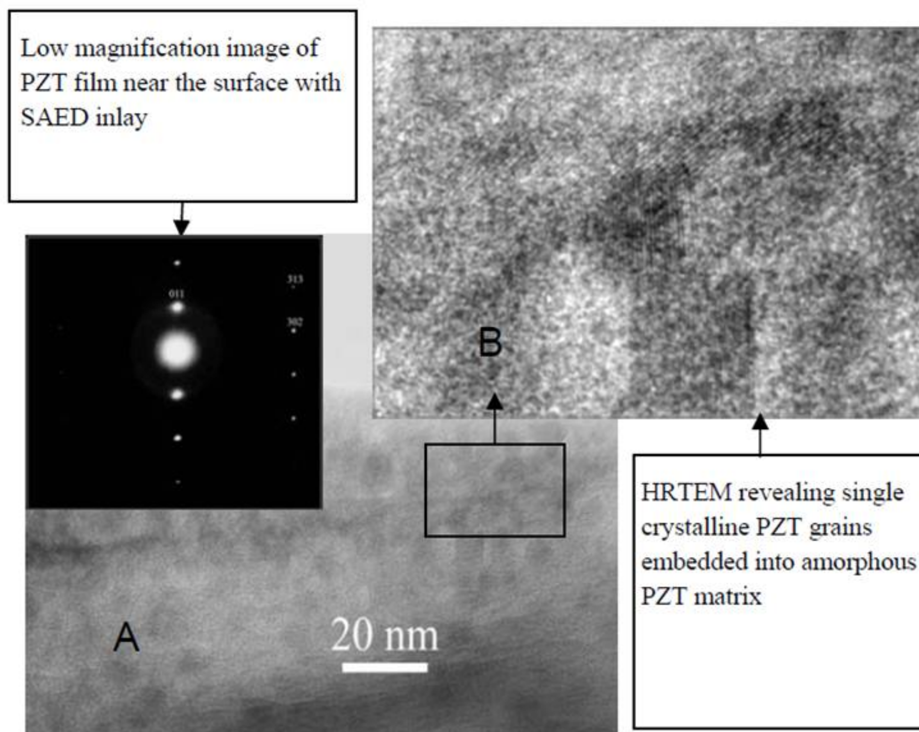

Fig. 4. TEM images of PVD deposited PZT displaying both low (A) and high (B) magnification of same sample on sapphire substrate.

3.3. Ferroelectric and Electric Data

PZT thin films with compositions of $Pb_{1.11}Zr_{0.52}Ti_{0.48}O_{3.11}$ and $Pb_{1.22}Zr_{0.52}Ti_{0.48}O_{3.22}$ were deposited on Si and c-plane sapphire wafers with metalized coatings consisting of 5 nm of Ti and 82 nm of Pt. Films with thicknesses of (300 and 600) nm were deposited using an Oerlikon CLC 200 Clusterline Sputter System. Following deposition, the films received a Rapid Thermal Annealing (RTA) treatment at a temperature of 700 °C for duration of 60 sec. The device electrodes (as shown in Fig. 5(a) on sapphire substrate) were patterned using a combination of photolithography and ion milling techniques. Low-frequency characterizations were performed using a Radiant Technologies LCR meter and software package. Capacitors with zero bias values of approximately (14 and 7) nF were made from the (300 and 600) nm thick films, respectively—yielding an extracted permittivity in the neighborhood of 950. The DC leakage currents were found to be on the order of 20 nA. The dielectric polarization density versus DC bias voltage trace, shown as Fig. 5(b), revealed a remnant polarization of $P_r = 39.7\ \mu C \cdot cm^{-2}$. Fig. 5(c) shows a trace of capacitance density versus DC voltage bias for the cases of sweeping the bias from low-to-high and high-to-low values.

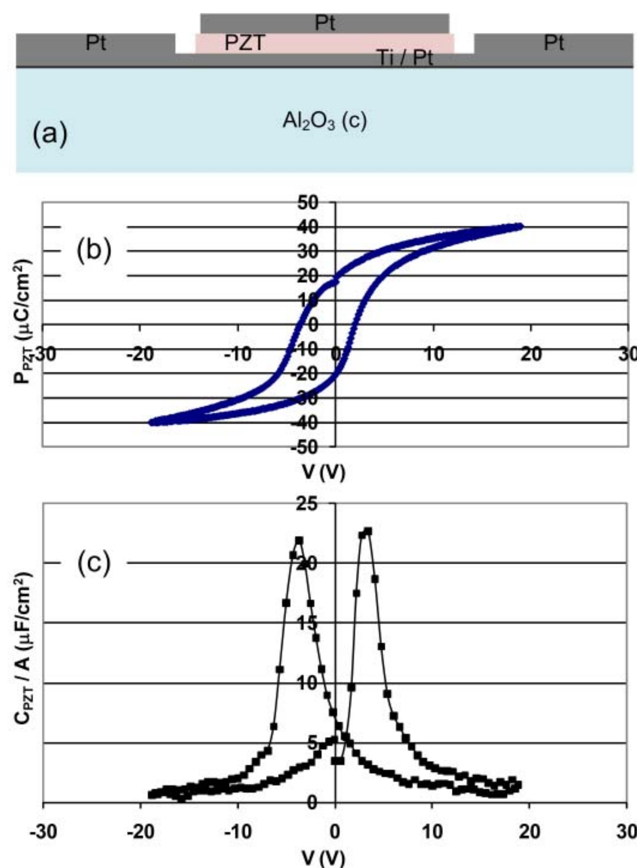

Fig. 5. (a) Lateral-view of MIM PZT test structure. (b) Dielectric polarization density versus DC bias voltage. (c) Capacitance density versus DC voltage bias (sweep up and down).

The electric data are summarized in Table 1 where the breakdown strength, leakage current, and resistivity are all displayed as a function of one another as well as the deposition conditions investigated. The breakdown voltages were determined from a collection of measurements taken from the same wafer to ensure accuracy of the value reported. As can be intuitively expected, a higher the breakdown strength is correlated with a lower the leakage current (and a correspondingly higher resistivity). The main point of interest, however, is the trend of increasing values of breakdown

strength. A reduction of excess Pb in the sputter target by a factor of one-half (22 % to 11 %) resulted in an increase in the breakdown strength by more than a factor of two on Si substrates. The PZT film withstood a voltage ~20 % higher than that required to break down the most optimal of the PVD conditions, 11 % excess Pb on sapphire substrates.

Table 1. Ferroelectric Properties on Si and Sapphire Substrates.

Sample	Prmax (μC/ cm^2)	Leakage Current (A)	Resistivity (Ohm·cm)	Breakdown Voltage (V)
Target 22 % Pb on Si	33.8	8.08 e-5	2.5 e+7	10~20
Target 11 % Pb on Si	31.8	7.69 e-9	2.6 e+11	30~50
Target 22 % Pb on Sapphire	39.7	3.23 e-9	5.4 e+11	50~70
Target 11 % Pb on Sapphire	37.3	1.62 e-9	7.5 e+11	60~80

When considering this material data in the context of integrated sensors, one must also consider the mechanisms of charge transport in PZT. In general terms, microstructure and the stoichiometry of both the crystalline and amorphous regions are the main contributors to said charge transport and therefore by extension breakdown strength of PZT. Since these films are approximately 0.5 μm thick and break down occurs at relatively high fields, surface related (ceramic metal contact band misalignment) phenomena will not be considered as a primary effect [7]. As such, we will focus more on the bulk portion of the PZT itself. The band gap of PZT is relatively low for a dielectric with reported values of approximately 3.0 eV [8]. It has been stated that PZT is intrinsically p-type due the seemingly inherent presence of vacant Pb sites [9]. Further, it is commonly understood that oxygen vacancies in oxide based ceramics created donor sites, which are n-type. From the crystallographic data it is seen that the degree of crystallinity and PZT crystal quality improves from the 22 % excess Pb to the 11 % excess Pb condition. This change can be directly correlated to higher breakdown strengths; so, it is seen that improved microstructure is a tenant of improved breakdown strength. A point of interest is the possibility that the excess Pb may be assumed to limit the formation of Pb vacancies and therefore reduce the conductivity of the films. However, this mechanism was not directly observed. It is believed that the Pb played a crucial roll in the PZT perovskite phase formation where too much of its excess decreased the quality of the crystal formation thereby possibly paving the way for excessive oxygen vacancy and defect formation. The lower resistivity of the 22 % excess Pb sample provided evidence for this suggestion. Furthermore, though not quantified in this work, defect density in poorer quality crystals can become an issue at higher field strengths where trapped charges become elevated to the conduction band. These additional charges contribute to the total current through the device and ultimately the initiating mechanisms of material breakdown. Additional composition analysis, defect density, and trap level are required to fully illustrate the mechanisms at work with these data.

Variations in the ferroelectric properties of the conditions studied are displayed in Table 1. There is a clear trend in increasing remnant polarization (37-40 μC/cm^2) and coercive field (50-80 kV/cm) on sapphire substrate, and 32-34 μC/cm^2 and 10-50 kV/cm on Si substrate, respectively, as excess Pb is reduced in the PVD samples. This also clearly correlates to the crystallographic data as did the breakdown strength. What is not so evident is the way by which the relative permittivity responded to each of the deposition conditions. Our results indicate that both of the PVD conditions performed better than the sol gel based devices. Within the PVD set though, the 11% excess Pb again produced the better result. The departure of the trend of the relative permittivity from the general trend observed thus far may exist within that which primarily contributes to permittivity. The permittivity of a material results from polarization events. In ferroelectrics, this typically results from ionic displacement. However, other mechanisms can contribute as well such as internal barrier layers

(IBL's) and charge collection at the contacts. The exact mechanism(s) in the case of this study were determined to be beyond the scope of the work and may be investigated in subsequent efforts. What can be asserted is that the PZT film on sapphire substrate yielded the highest remnant polarization and coercive field values, 39.7 $\mu C/cm^2$ and 80 kV/cm, respectively, and the PVD condition with 11% excess Pb produced the highest relative permittivity, ε_r = 965.

From Table 2, by comparison of lattice constant (a) of different materials, the lattice constant of c-plane sapphire is closer to PZT's and less lattice mismatch during crystal growth than Si's, which resulted in the better ferroelectric properties on sapphire substrate than Si's [10].

Table 2. Lattice Constant (a) of Different Materials.

Materials	**Si**	**C-Plane Sapphire**	**PZT**	**Pt**
Lattice Constant a (Å)	5.431	4.758	4.084	3.920

4. Conclusion

It has been demonstrated that sputtered PZT film on c-plane sapphire substrate has the great potential of taking advantages of PZT in a single chip to achieve inexpensive, fully-integrated, passive telemetric transponders. The data showed similar results in many aspects of the films investigated on both Si and sapphire substrates. In particular, for the 11% excess Pb target sputtered film, the breakdown voltage and ferroelectric data were significantly better than that of the 22 % excess Pb target, which revealed a broad potential of the sputtered PZT film applications, especially on sapphire substrate. PZT film depositions on both Si and sapphire substrates produced polycrystalline PZT with the Perovskite structure and a [110] texture. PZT films on sapphire substrate possessed a greater degree of crystallinity compared to that on Si substrate. However, the higher Pb content proved beneficial for the remnant polarization. It is believed that further refinement of the production friendly sputtering process and subsequent device treatment will allow for achievement of better material performance.

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