Microwave Characterization of Ba-substituted PZT and ZnO Thin Films

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Abstract — The microwave dielectric properties of $(\text{Ba}_{0.1}\text{Pb}_{0.9})(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ (BPZT) and ZnO thin films with thicknesses below $2\ \mu\text{m}$ were investigated. No significant dielectric relaxation was observed for both BPZT and ZnO up to 30 GHz. The intrinsic dielectric constant of BPZT was as high as 980 at 30 GHz. The absence of strong dielectric dispersion and loss peaks in the studied frequency range can be linked to the small grain diameters in these ultrathin films.

Index Terms — Capacitors, Ferroelectric films, Permittivity, Piezoelectric materials.

I. INTRODUCTION

FERROELECTRIC materials are known for their excellent piezoelectric properties and are therefore widely used in electromechanical transducers [1-3]. $\text{Pb}(\text{Zr}_{1-x}\text{Ti}_x)\text{O}_3$ (PZT) has been of particular interest due to its large piezoelectric response, especially near the morphotropic phase boundary at $x = 0.48$ [4, 5]. Besides ionic displacement, piezoelectricity occurs in ferroelectrics via additional mechanisms due to the movement of non- 180° domain walls and the displacement of interphase boundaries, both strongly influencing the electromechanical behavior [6]. Thus, ferroelectric materials typically show a non-linear piezoelectric response and are very sensitive to crystal orientation and defectivity, as well as external boundary conditions, *e.g.* the electrode material [7]. However, ferroelectric piezoelectrics often show high losses and strong dielectric dispersion at microwave frequencies [8-11]. Several mechanisms for the observed dispersion in the frequency range between 10^7 and 10^{10} Hz have been proposed, including translational domain wall vibration [12], acoustic shear wave emission from 90° ferroelastic domain walls [9, 13], as well as piezoelectric resonances within grains [14]. Since the mechanical response in a piezoelectric material is a response to the induced polarization, such dielectric dispersion may impose severe limitations on actuators operating in the microwave regime.

Recently, thin films of ferroelectric piezoelectrics with thicknesses in the sub- μm range have received increasing attention for high frequency bulk acoustic resonators [15, 16] as well as

emerging magnetoelectric applications [17,18]. Only few studies have addressed the microwave dielectric properties of PZT films with film thicknesses in the sub- μm range [19, 20], with conflicting results. In this paper, we characterize the dielectric properties of both Ba-substituted PZT (BPZT) and ZnO thin films with thicknesses in the 100 nm range at microwave frequencies up to 30 GHz. BPZT has shown superior properties with respect to unsubstituted PZT in terms of fatigue [21], breakdown field, as well as piezoelectric response [22]. Among non-ferroelectric piezoelectrics, ZnO has also been widely used for various transducer applications [23] because of its good electromechanical coupling coefficient and deposition conditions compatible with CMOS technology. However, the microwave characteristics of very thin ZnO layers have not yet been reported in the literature.

II. EXPERIMENTAL PROCEDURE

At low frequencies ($< \text{MHz}$), metal-insulator-metal (MIM) capacitors have typically been used for the characterization of dielectric layers. However, due to large parasitics at microwave frequencies, it is difficult to extract accurate dielectric parameters from such test structures. Several solutions to this issue have been presented [19, 20, 24] but one of the most successful is the method originally proposed by Ma *et al.* [25,26]. A schematic of the test structure is shown in Fig. 1a. The dielectric layer under test is deposited onto a planar metallic bottom

Fig. 1. (a) Schematic and (b) equivalent circuit of the test structures.

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Fig. 2. 2θ - ω x-ray diffraction patterns of (a) BPZT and (b) ZnO layers with indicated thicknesses. Reflections due to the Pt BE are also visible.

electrode (BE), followed by the definition of a concentric top electrode (TE) structure. In this approach, unwanted parasitic effects induced by the outer grounded top electrode are compensated by using two test structures with the same outer electrode but with different sizes for the inner signal electrode.

Low frequency capacitance-voltage curves were measured using MIM capacitors. Here, two electrodes with different areas were patterned on top of the dielectric layer and thus two capacitors in series were characterized, similarly to the concentric circular capacitors used for microwave measurements.

A. Materials and test vehicle

All BPZT and ZnO layers were deposited on Pt/TiO₂/SiO₂/Si substrates. The TiO₂ layer (10 nm) acted as an adhesion layer for the Pt (70 nm) BE, which was deposited by e-beam evaporation and annealed at 750 °C for 3 min. This resulted in smooth polycrystalline Pt layers with strong (111) texture. BPZT, *i.e.* (Ba_{0.1}Pb_{0.9})(Zr_{0.52}Ti_{0.48})O₃ was deposited by pulsed laser deposition (PLD) in a Solmates SIP-700 system using a 10 nm thick LaNiO₃ (LNO) buffer layer, also deposited by PLD. The usage of the LNO buffer layer resulted in a predominant (110) texture (Fig. 2a) and improved dielectric [27] and piezoelectric [28] properties. ZnO was deposited by RF sputtering, followed by annealing at 500 °C for 3 h in air. Resulting x-ray diffraction (XRD) patterns are shown in Fig. 2b, indicating (predominant) (002) texture. The Scherrer grain sizes in the BPZT films deduced from the XRD patterns in Fig. 2a were between 20 and 25 nm with little dependence on the film thickness. For ZnO,

Fig. 3. Cross-sectional scanning electron micrographs and atomic force microscopy surface analysis (insets) of (a) 400 nm, (b) 200 nm, (c) 100 nm BPZT and (d) 250 nm ZnO films.

values between 25 and 28 nm were obtained for the 30 nm and 250 nm thick films, respectively. The cross-sectional scanning electron micrographs in Fig. 3a-c indicate a columnar structure of the BPZT films whereas ZnO was more granular (Fig. 3d). ZnO grain sizes were in reasonable agreement with the Scherrer grain sizes determined by XRD.

Finally, the Ti/Au (10 nm/1.5 μm) TE was defined by a lift-off process using standard optical lithography. The TE outer radius b (Fig. 1a) was 80 μm whereas the TE inner radius a , was either $a_1 = 15$ μm or $a_2 = 10$ μm. No poling process was performed on any of the samples unless specified.

An equivalent circuit of the test structure is shown in Fig. 1b. The low sheet resistances measured for the BE ($2.0 \pm 0.1 \Omega/\square$) and the TE ($< 30 \text{ m}\Omega/\square$) led to low series resistances R_G , R_B , and R_S . The small inner capacitor area minimized C_C .

B. Measurement setup

Microwave measurements were performed using an Agilent HP E8361C vector network analyzer (VNA) with 2.4 mm ports between 1 and 30 GHz. The VNA is equipped with a bias-tee to

control the dc bias applied during the measurements by means of an Agilent E5260 voltage source. Adaptors were used to connect the VNA to a 40A Picoprobe manufactured by GGB Industries Inc., using a high-performance SMK (2.92 mm) connector. The 40A Picoprobe had a ground-signal-ground (G-S-G) configuration with a 150 μm pitch and a bandwidth of 40 GHz.

The ground needles were in contact with the outer top electrode of the test vehicle (Fig. 1a) whereas the signal needle was in contact with the central circular electrode. Before starting the measurements, a one-port short-open-load-thru (SOLT) calibration procedure was performed on the whole setup using a CS-5 calibration substrate provided by the probe manufacturer. All measurements were performed at -17 dBm; this is equivalent to an applied field over the inner capacitors of about 1 to 2 kV/cm for BPZT and up to 10 kV/cm for ZnO. For such low applied fields, no significant nonlinear effects are expected.

Fig. 4. S_{11} Smith charts (normalized to an impedance of 50 Ω) for test structures including a 400 nm BPZT film and inner capacitor radii of 10 and 15 μm , as indicated. Lines in (a) represent experimental data whereas open symbols in (b) represent the result of HFSS simulations (see text).

III. RESULTS AND DISCUSSION

A. Microwave permittivity of unpoled dielectrics

The measured one-port S -parameter (see Fig. 4a for S_{11} of capacitors with 400 nm thick BPZT) was converted to a complex impedance Z [25]. The real part of the permittivity ϵ_r and the loss tangent $\tan\delta$ were then extracted from measurements of the impedances $Z_{1,2} = R_{1,2} + jX_{1,2}$ of two devices with inner TE radii a_1 and a_2 , respectively, using the following equations derived from Eq. (21) in [25]:

$$\epsilon_r = \frac{h \left(\frac{1}{a_2^2} - \frac{1}{a_1^2} \right) (X_1 - X_2)}{\omega \pi \epsilon_0 \left\{ (X_1 - X_2)^2 + \left[R_1 - R_2 - \frac{R_B}{2\pi} \ln \left(\frac{a_2}{a_1} \right) \right]^2 \right\}} \quad (1)$$

$$\tan\delta = \frac{\epsilon_2}{\epsilon_1} = \frac{\frac{R_B}{2\pi} \ln \left(\frac{a_2}{a_1} \right) - R_1 + R_2}{X_1 - X_2} \quad (2)$$

Fig. 5. (a) Effective permittivity ϵ_r and (b) loss tangent $\tan\delta$ of BPZT films with thicknesses as indicated.

Here, h is the dielectric layer thickness, $\omega = 2\pi f$ the angular frequency of the measurement, ϵ_0 the vacuum permittivity, and R_B the sheet resistance of the BE.

Figs. 5 and 6 show resulting values of ϵ_r and $\tan\delta$ as a function of frequency for BPZT and ZnO, respectively. Films of different thicknesses (100 nm to 2 μm for BPZT, 30 to 250 nm for ZnO) were studied. The data show only very weak dielectric dispersion with ϵ_r decreasing at most by 10% for all BPZT (Fig. 5a) and ZnO (Fig. 6) films up to 30 GHz. The slightly lower effective permittivity measured for the 30nm-thick ZnO can be

ascribed to the lower density of the very thin film. The loss tangent of BPZT increased from about 0.05 at 1 GHz to values between 0.25 and 0.35 at 30 GHz according to the film thickness (Fig. 5b), similar to the results of Defy et al. [19]. The films differ a little from each other starting from 20 GHz but a clear trend was observed: thinner layers show higher $\tan\delta$ values while thicker layers are closer to a lossless dielectric (see next sections and Fig. 9). By contrast, the loss tangent of ZnO was low (Fig. 6), about 0.05 and nearly independent of the frequency up to 20 GHz with only a very small increase at higher frequencies up to 0.08 for the 30 nm thick film.

Fig. 6. Effective Permittivity (closed symbols) and loss tangent $\tan\delta$ (open symbols) of ZnO layers with thicknesses as indicated.

B. Thickness dependence of permittivity and loss tangent

Dielectric losses in thin films are typically found to be an order of magnitude larger than in bulk materials. This is mainly an extrinsic effect, often ascribed to charged defects, and thus related to the film quality. Losses have been reported to be proportional to the permittivity value and inversely proportional to the applied field [1]. We have not observed a clear thickness dependence of $\tan\delta$ of BPZT (Fig. 5b), suggesting that the dielectric properties of BPZT were not strongly degraded as the thickness was decreased from 2 μm down to 100 nm. Very similar results were obtained for ZnO (Fig. 6). The origins of the measured loss parameters are discussed in more detail below.

By contrast, the BPZT permittivity showed a strong dependence on the film thickness over the entire frequency range. The behavior is summarized for a frequency of 1 GHz in Fig. 7a. The measured effective permittivity of the dielectric stack increased with thickness with signs of saturation at larger thicknesses. Such a response has commonly been reported for thin ferroelectric films [29-33] and has been ascribed to the presence of a thin “dead layer” with a low permittivity at the electrode-ferroelectric interface resulting in a series capacitor with an increasing influence as the thickness of the dielectric is reduced. This additional interfacial capacitor can be linked to a finite potential across the charge at the interface, either due to screening effects by the metallic electrode [29] or, more likely, to internal screening by a space charge layer of mobile oxygen vacancies [33].

To correct for the influence of the dead layer and to extract the intrinsic permittivity of BPZT, ϵ_{BPZT} , the dielectric stack with thickness h and (effective) permittivity ϵ_r can be described

by two series capacitors. ϵ_{BPZT} as well as the ratio of the thickness h_{DL} and the permittivity ϵ_{DL} of the dead layer can then be obtained by

$$\frac{h}{\epsilon_r} = \frac{(h - h_{\text{DL}})}{\epsilon_{\text{BPZT}}} + \frac{h_{\text{DL}}}{\epsilon_{\text{DL}}}. \quad (3)$$

Fig. 7. (a) Effective permittivity ϵ_r at 1 GHz of BPZT as a function of film thickness. The dashed line indicates the extracted intrinsic value ϵ_{BPZT} at 1 GHz. (b) Linear fit of the reciprocal normalized effective permittivity values (h/ϵ_r) at 1 GHz using Eq. (3). (c) Frequency dispersion of the intrinsic permittivity of BPZT, ϵ_{BPZT} , obtained from the data in Fig. 5(a) using the dead layer model described in the text.

As an example, the dependence of h/ϵ_r on the BPZT thickness h at 1 GHz is depicted in Fig. 7b. As expected, a linear dependence was observed, with the inverse slope equal to the intrinsic

permittivity of BPZT, ϵ_{BPZT} . Very similar behavior was found for higher frequencies also. By calculating such linear fits for each frequency in the entire studied range, the frequency dependence of ϵ_{BPZT} could be determined (Fig. 7c). A value of $\epsilon_{\text{BPZT}} = 1250$ was obtained at low frequencies with little frequency dispersion. This result falls within the range of 900–2000 previously reported for BPZT at low frequencies up to 1 MHz [22, 34]. The frequency behavior of ϵ_{BPZT} was essentially identical to that of ϵ_r in Fig. 5a. This indicates that the dead layer did not contribute any dielectric dispersion. The dead layer was characterized by $h_{\text{DL}}/\epsilon_{\text{DL}} = 0.4$ nm. The effective permittivity for the 2 μm -thick layer was very close to the extracted intrinsic permittivity value, consistent with the dead-layer model, as shown in Fig. 7a. It should be noted that the permittivity of this passive dielectric layer can be expected to depend on the test structures, the measurement technique, and the electrode material, which renders comparisons between different studies difficult.

Fig. 8. (a) Effective permittivity ϵ_r of BPZT as a function of applied dc electric field for different measurement frequencies between 1 and 30 GHz. Ferroelectric hysteresis was observed for all frequencies and the hysteresis area showed with little frequency dependence up to 20 GHz. (b) Low-frequency ϵ_r -V curves of a 400 nm thick BPZT layer.

C. HFSS simulations of the test structures and origin of measured losses

Due to the high dielectric constant of BPZT, the measurements may be very sensitive to RC limitations of the bandwidth [20].

To distinguish between intrinsic and parasitic losses, we have performed three-dimensional full-wave electromagnetic field simulations of the test structures using Ansys HFSS 17 including a 400 nm thick dielectric with a permittivity of 600 and a loss tangent of 0.08 (see below) in combination with experimental sheet resistances of the BE and TE. The simulations showed excellent agreement with the measurements (see the Smith charts in Fig. 4a and Fig. 4b). Analyzing S_{11} parameters calculated by HFSS with the same equivalent circuit model as the experimental data resulted in a permittivity values that were very close (typically within 3%) to the values assumed in the simulations. This confirms the excellent accuracy of the equivalent circuit model in the studied parameter (and frequency) range.

The black solid line in Fig. 9 shows the loss tangent extracted from simulations for a lossless dielectric with a thickness of 400 nm and the effective dielectric constant of BPZT, $\epsilon_r \sim 600$. The curve showed a similar frequency behavior but fell consistently below the experimental data. Good agreement between simulations and measured loss tangents was obtained when intrinsic BPZT losses of $\tan\delta = 0.06$ (red dotted-dashed curve) and $\tan\delta = 0.10$ (purple dashed curve) at low and high frequencies, respectively, were assumed in the simulations (Fig. 9). This indicates that a large part of the experimentally observed losses can be attributed to RC bandwidth limitations rather than to intrinsic dielectric dispersion in BPZT. For other thicknesses, essentially identical results were obtained since the absolute capacitances (and losses) were very similar (simulation data not shown) due to the reduction of permittivity for thinner films. Thus, intrinsic losses of BPZT can be estimated to be of the order of $\tan\delta \sim 0.1$ in the studied frequency range. Due to the much lower dielectric constant, the effect was much less pronounced for ZnO and contributed less than 0.01 to $\tan\delta$ for the 30 nm film at the highest frequencies.

Fig. 9. The solid black line indicates the parasitic loss tangent obtained from simulations for a lossless dielectric. The dotted-dashed red and the dashed purple lines indicate results of simulations with intrinsic BPZT losses of $\tan\delta = 0.06$ and 0.10, respectively.

D. Ferroelectric behavior of BPZT

To gain further insight into the ferroelectric properties at microwave frequencies, we have studied ferroelectric hysteresis loops of BPZT as a function of an applied dc electric field at

frequencies between 1 and 30 GHz. Fig. 8a shows the hysteresis for a 400 nm thick BPZT film; very similar results were obtained for 200 nm and 100 nm thick BPZT films (not shown). Hysteresis was clearly observed and was very similar to low frequency ϵ_r -V curves shown in Fig. 8b that were recorded using an LCR meter between 100 Hz and 1 MHz. A decrease of the peak capacitance was observed as frequency increases. The asymmetry between peaks at positive and negative fields can be ascribed to the large area differences of outer and inner electrodes ($>10^3\times$) and the resulting difference in the amount of surface charges.

At microwave frequencies, the area of the hysteresis loop was rather independent of frequency; only at 30 GHz some area reduction was observed. However, Fig. 8a clearly proves that BPZT was still ferroelectric even at 30 GHz. Fig. 10 shows both the permittivity ϵ_r as well as the loss tangent $\tan\delta$ of the 400 nm thick BPZT film under a dc bias field near saturation of the BPZT polarization. As expected, the dielectric constant decreased significantly with respect to the value at zero dc bias.

Fig. 10. Frequency dependence of effective permittivity ϵ_r (closed symbols) and loss tangent $\tan\delta$ (open symbols) of a 400 nm thick BPZT film for different applied dc bias fields. Fields of 375 kV/cm and 500 kV/cm led to near saturation of the polarization.

However, neither the dielectric dispersion nor the loss tangent were significantly affected by the saturation of the polarization of BPZT. The reduction of the loss can be ascribed to reduced RC parasitics due to a lower capacitance with respect to the zero-field case.

E. Loss Mechanisms

As discussed above, the dielectric dispersion and the loss peaks of ferroelectrics have been linked to several mechanisms, both related to ferroelectricity, such as translational domain wall vibration [12] or shear wave emission from 90° ferroelastic domain walls [9, 13], as well as to piezoelectricity, in particular to acoustic resonances within grains [14]. The expected frequency $f_{\max}$ of the loss maximum due to acoustic resonances is determined by the acoustic wavelength λ_A in the material and related to the grain size g by $g = \lambda_A/2$ [10, 14]. With sound velocities of ~ 4300 m/s and ~ 6200 m/s for BPZT and ZnO [35, 36], respectively, we obtain $f_{\max} > 100$ GHz for both materials. Loss peaks due to ferroelectricity in BPZT can also be expected to occur at similarly high resonant frequencies if the domain

size is equal to the grain size, as it has commonly been assumed in the literature [8, 9, 11-13]. This is fully consistent with the absence of a clear loss peak and dielectric dispersion in the BPZT and ZnO data.

IV. CONCLUSION

In conclusion, we have studied the microwave dielectric properties (1–30 GHz) of polycrystalline BPZT (100–400 nm) and ZnO (30–250 nm) thin films. Both materials show a weak dispersion of ϵ_r over the characterized frequency range. The thickness dependence of the BPZT permittivity indicated the presence of a dead layer with $h_{DL}/\epsilon_{DL} \sim 0.4$ nm whereas losses were rather independent of thickness, as were the permittivity and losses of ZnO. The bulk ϵ_r and $\tan\delta$ values for BPZT were extrapolated by fitting the experimental data and by HFSS simulations, respectively. The calculated intrinsic permittivity of BPZT, ϵ_{BPZT} , was approximatively ~ 1200 and the effective permittivity of thicker (1–2 μm) BPZT layers approached this value. The extracted low $\tan\delta \sim 0.1$ for BPZT can be linked to the small grain sizes in the thin films. Estimations of the frequency of the loss maximum using measured Scherrer grain sizes were of the order of 100 GHz, well outside the measurement range. The results indicate that the dielectric loss peak of ferroelectrics can be shifted towards very high frequencies by reducing the grain size in the films without a decrease of the permittivity and a strong increase of the losses. The Ba substitution in PZT may play an important role since it has been reported to inhibit PZT grain growth [22]. Moreover, grain sizes are typically found to decrease with decreasing film thickness, which renders ultrathin BPZT films highly promising for microwave applications when the influence of the dead layer capacitance can be limited. Since their permittivity was strongly dependent on an applied dc electric field, BPZT films are also interesting for field-tunable capacitor (varactor) applications. Future work will focus on the piezoelectric properties of such ultrathin films.

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