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Measurement of Microwave Dielectric Properties of $Pb(Zr_{1-x}Tr_x)O_3$ Thin Films

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Abstract. Ferroelectric $Pb(Zr_{1-x}Ti_x)O_3$ (PZT) thin films were prepared by sol-gel deposition method. The structural and surface morphologies were investigated using X-ray diffractometer and atomic force microscope. Microwave dielectric properties were obtained using co-circle electrode patterns, which were made by photolithography and etching process. The dielectric constant of PZT films was about 450 from 0.05–1 GHz range.

1. Introduction

Recently, ferroelectric thin films have been intensively studied for applications such as non-volatile ferroelectric random access memory (NVFRAM), dynamic access memory (DRAM), piezoelectric devices, infrared sensor and microwave devices [1]. $Pb(Zr_{1-x}Ti_x)O_3$ (PZT) thin films are most promising candidates for NVFRAM because of the high remanent polarization and low deposition temperature.

In the material characteristics for microwave devices, generally the high dielectric constant tunability and the low dielectric loss are necessary. But in some device, the contribution of the dielectric loss to the device loss is smaller. In this point, in spite of the relatively high dielectric loss, it is valuable to investigate the electric properties of the PZT material for microwave tunable devices. It is important to know the dielectric constant of the material because 50 ohm matching design is needed to minimize the return loss caused by the impedance mismatches. But the measurement technique for dielectric constant is difficult in the devices for operating frequencies which range from hundreds of megahertz (MHz) to tens of gigahertz (GHz) [2–4], there have mainly been studied in the kHz–MHz frequency range.

In this paper, we report the measured microwave dielectric properties of $Pb(Zr_{1-x}Ti_x)O_3$ (PZT) thin films for metal-ferroelectric-metal (MFM) devices with frequency from 0.05 to 1 GHz, and compared the dielectric properties at low frequency with those at high frequency.

2. Experimental

Polycrystalline $Pb(Zr_{1-x}Ti_x)O_3$ (PZT) films were deposited onto $Pt(111)/Ti/SiO_2/Si$ substrates by sol-gel deposition method using metal alkoxide solution. All the films were post-annealed in the air-filled furnace above 550°C for 30 min. The detailed fabrication processes are described elsewhere [2]. The composition $x = 0.48$ corresponds morphotropic phase boundary between Zr-rich rhombohedral and Ti-rich tetrahedral phase region. The structural properties of PZT films were characterized by X-ray diffraction (XRD) measurement using a Rigaku X-ray diffractometer equipped with Cu K_α radiator source. The microstructure of the films was observed by atomic force microscope (AFM). Thickness of the deposited PZT film was confirmed by a cross sectional scanning electron microscope (SEM), and the composition analysis for PZT films was done by analyzing Rutherford Backscattering Spectrum.

To measure dielectric properties of PZT thin films, metal-ferroelectric-metal (MFM) trilayer structure on $Pt(111)/Ti/SiO_2/Si$ substrates which is consist of the center circular patch and surrounding ground was used.

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In MFM capacitor, all the test structures used here have a 150 μm outer diameter. All the measurements were done on two test structures with 50 and 80 μm inner diameters.

In all devices, the electrode of a 2 μm thick gold layer with a thin chromium adhesion layer was deposited by DC sputtering deposition method and the electrode pattern was fabricated on the PZT film using photolithography and dry etching process.

Low frequency dielectric properties of the PZT films were measured using RT6000S in home-made probe station.

The measurement in microwave frequency is one-port reflection measurement using the HP 8510C vector network analyzer at frequency range of 0.05–10 GHz. Coaxial cables and equipment were calibrated up to the microwave picoprobes to minimize measurement uncertainty using a commercial standard kit. The measured reflection coefficient of the device under test was converted into impedance parameters using the following equation

$$Z_{\mathrm{dut}} = Z_0 \frac{1+\Gamma}{1-\Gamma}$$

Where Z_0 is the impedance of the measurement port of the vector network analyzer which is usually 50 Ω, and Γ is the complex reflection coefficient.

In MFM capacitor, there is no direct contact to the bottom metal layer and the electrical path to the bottom electrode of the capacitor is made through the large capacitor formed between the bottom metal layer and the large top ground. Since the capacitance of the capacitor formed between the top circular metal patch and the bottom metal layer is much smaller than that between the large top ground and the bottom metal layer, the response of the whole circuit is dominated by the smaller capacitor. The ground-signal-ground (GSG) probe makes contact to the center circular patch of metal which functions as the top electrode of the capacitor, and the metal outside the ring which functions effectively as the ground in MFM structure device. The data are used in an equivalent circuit model which was designed with a parallel resistor/capacitor and a series resistor to extract the dielectric constant of PZT thin films [5].

3. Results and Discussion

The deposited PZT thin films on Pt(111)/Ti/SiO$_2$/Si substrates had perovskite phase and preferentially oriented along the [111] direction as shown in Fig. 1. Using the formula

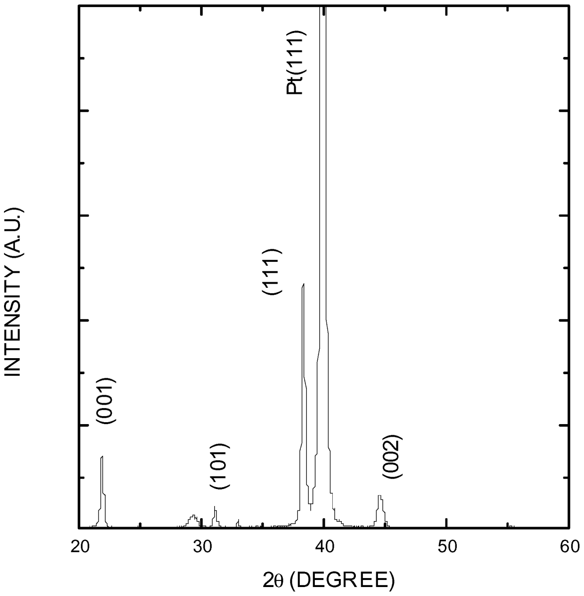

Fig. 1. X-ray diffraction pattern of the PZT films on Pt(111)/Ti/SiO$_2$/Si substrates.

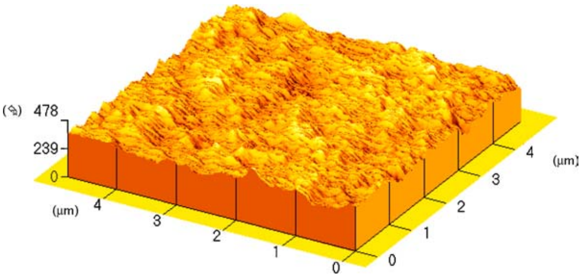

Fig. 2. AFM image of the PZT films on Pt(111)/Ti/SiO$_2$/Si substrates.

$$\alpha_{111} = \frac{I(111)}{I(001)+I(101)+I(111)}$$

where I is the XRD peak intensity, the degree of preferred orientation of the (111) plane was determined to be 65%. This result may be due to not only the lower surface energy of the (111) surface but also the small lattice parameter difference between perovskite PZT (111) plane and face center cubic Pt (111) plane, about 4.36%.

Figure 2 shows the AFM image of the PZT films on Pt(111)/Ti/SiO$_2$/Si substrates. The microstructure of the films observed by AFM was crack-free and uniform with a spherical grain of 0.1–0.2 μm having the

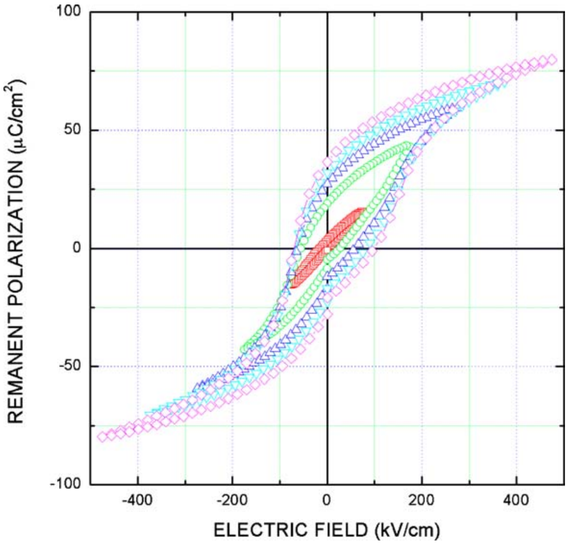

Fig. 3. The polarization versus electric field (P-E) curve of the planar type PZT capacitor with a MFM structure.

columnar structure. The average roughness was about 2 nm. Thickness of PZT films was 0.4 μm, which was confirmed by cross-sectional scanning electron microscope.

Figure 3 shows the polarization versus electric field (P-E) curve of the planar type PZT capacitor with a MFM structure. The polarization exhibits strong applied voltage dependence, the values of P_r, E_c, and the switching polarization P*-P^ were about 32 μC/cm^2, 80 kV/cm, and 55 μC/cm^2, respectively. The P-E curve shows typical hysteresis due to polarization reversal found in conventional ferroelectric thin films.

In MFM capacitor device, the circular-patch capacitor used for measuring the thin film dielectric properties consisted of a disk-shape capacitor and an outer capacitor surrounding it, as shown in Fig. 4(a). Accordingly, to remove the effect of the outer capacitor, the impedances of two test structures, which had the same outer diameter but different inner diameters, were subtracted. However, the subtracted impedance had an additional series resistance, which was caused by resistance in the bottom/top electrode and contact resistance (between the GSG probe and inner/outer top electrodes). Therefore, to quantify the additional resistance including the contact resistance, an equivalent circuit model was designed with a parallel resistor/capacitor and a series resistor in this study, as shown in Fig. 4(b). The series resistor in this equivalent circuit model represents the difference (in two test structures) in both the resistance from the electrodes and the contact resistance.

The impedance of the equivalent circuit can be expressed in the following equations:

$$Z_{\text{dut}} = R_s + \frac{R_p}{1 + i\omega C_p R_p} \approx R_s + \frac{1}{i\omega C_p}$$

where C_p is capacitance, and R_p is intrinsic resistance term of thin film dielectrics. The second equation was obtained using the approximation that R_p is inversely proportional to the frequency [6].

The total impedance of the device under test (DUT) is then

$$Z_{\text{dut}} = (R_{\text{center}} + R_{\text{ring}} + R_{\text{outer}}) + \frac{1}{i\omega}\left(\frac{1}{C_{\text{center}}} + \frac{1}{C_{\text{outer}}}\right)$$

This corresponds to the same equivalent circuit as mentioned with a series resistor added. The complex C_{center} is what we are interested in obtaining. The way to extract the dielectric constant of the PZT films is to

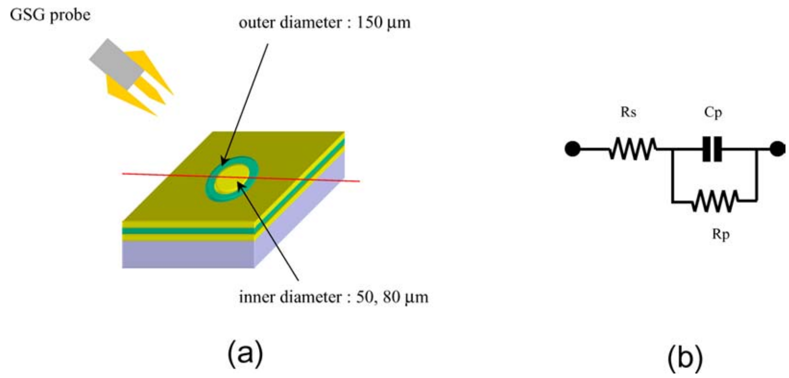

Fig. 4. (a) Schematic diagram of the co-circle electrodes (b) Equivalent circuit of MFM co-circle capacitor.

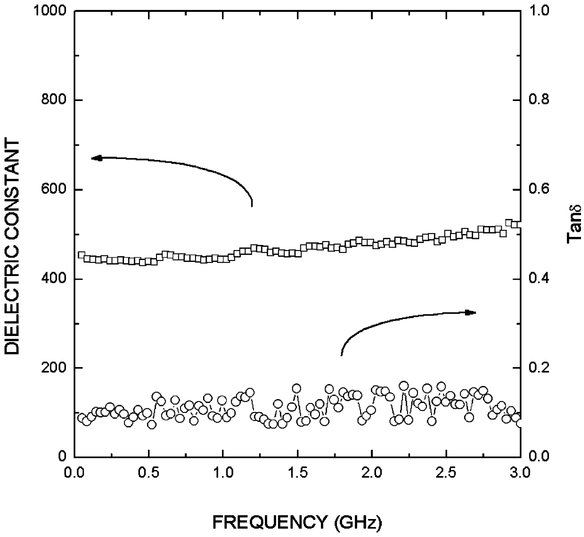

Fig. 5. Frequency dependent dielectric constant and dielectric loss of the PZT films on Pt(111)/Ti/SiO$_2$/Si substrates.

measure the input impedance of two test structures with the same outer diameter but different inner diameters, and then subtract them to remove the effect of the outer ground. Given the two inner diameter values of a and b, the impedance difference is

$$Z_1 - Z_2 = \frac{R_s}{2\pi} \ln\left(\frac{b}{a}\right) + \frac{t}{i\omega\pi}\frac{1}{\varepsilon_0\varepsilon_r}\left(\frac{1}{a^2} - \frac{1}{b^2}\right)$$

The complex dielectric constant is then easily obtained using the above equation.

In Fig. 5, the dielectric constant and the dielectric loss of the PZT thin film is about 450, 0.1 at 1 GHz, respectively, and showed an almost constant value up to 1 GHz range. Above 1 GHz, small increase in the dielectric constant of the PZT films is observed, which is mostly caused by the sensitivity of the instrument and possibly by some uncompensated parasitic effect.

4. Summary

Ferroelectric polycrystalline PZT films were prepared on Pt(111)/Ti/SiO$_2$/Si substrates by sol-gel deposition method. The dielectric constant of PZT films in MFM capacitor was calculated with frequency upto 1 GHz using both the reflection S-parameter data for the co-circle electrode patterns and the equivalent circuit. We observed that the PZT films have a dielectric constant of about 450 at 1 GHz for MFM capacitor.

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