Chapter 5

Direct Measurement of Electrical Hysteresis of Micron-Sized Pb(Zr,Ti)O_3 Capacitors using the Constant Current Method

5-1. Introduction

As the area of ferroelectric capacitors declines toward the submicron regime, measuring their electrical hysteresis is becoming difficult. Over the last few years, many approaches for determining the polarization loops of capacitors with small areas have been proposed [30-31, 42-46]. One approach uses an array of capacitors in parallel, but only average results can be obtained [30]. Another method is by determining the piezoelectric coefficient $\boldsymbol{d}_{33}$ of the sample, using a piezoresponse force microscope (PFM) [42-45]. The remanent polarization can be calculated using some connections between piezoelectric coefficients and electrical polarization [47]. Additionally, Tiedke *et al.* applied a virtual ground method (VGM) to obtain the electrical hysteresis loops of a single nanoscale ferroelectric capacitor using an atomic force microscope to make contact [31]. Open compensation and a linear correction yielded electrical hysteresis loops for capacitors with areas as low as 0.04 μm^2. A numerical method for compensating Sawyer-Tower (ST) hysteresis measurements was recently presented [32]. This approach corrects the parasitic capacitance of the probe station and the sense capacitor to obtain the ferroelectric property of micron-sized PZT capacitors (2.5 μm×2.5 μm).

In this chapter, we present a constant current method (CCM) [28-29], which uses a constant current and measures the corresponding voltage, to obtain the hysteresis plot for single micron-sized ferroelectric capacitors. The parasitic capacitance of the probe station can be calculated from the voltage buildup using $V(t) = (I_0 \times t)/C_{\mathrm{para}}$. The corrected hysteresis loops of small capacitors can also be easily obtained. The proposed approach represents a low-cost approach for measuring directly the electrical hysteresis of a micron-sized capacitor.

5-2. Experiments

In this experiment, 250-nm-thick Pb($Zr_{0.3}Ti_{0.7}$)O_3 (PZT(30/70)) films were deposited onto Pt(100nm)/Ta(20nm)/SiO2(100nm)/Si substrates using a sol-gel-derived method (See chapter 2). The deposited films were then crystallized by annealing in a furnace at 600°C for

30 min in oxygen ambient. Then, 50-nm-thick platinum top electrodes were deposited onto the PZT films at room temperature by DC sputtering. A 20-nm-thick TiN was then deposited as a hard mask. Next, lithography and dry etching were used to define capacitors with areas of 100 μm×100 μm, 50 μm×50 μm, 20 μm×20 μm, 10 μm×10 μm, 5 μm×5 μm, 3 μm×3 μm, and 2 μm×2 μm. A postannealing process was carried out at 600°C for 1 min, involving rapid thermal annealing (RTA) in an O_2 atmosphere, which was used to remove etching damage.

After the samples were prepared, the micron-sized capacitors were placed in contact with a probe station via a needle with a tip having a radius of 0.2 μm. A constant current array with an optimized range was used and the corresponding voltage was measured using a Keithley 4200 semiconductor characterization system, to determine the hysteresis loops. The hysteresis of the 100 μm×100 μm capacitor was also measured using a standardized ferroelectric test system RT66A, in virtual ground mode, to verify the results obtained using the CCM method.

5-3. Results and Discussions

The experimental procedures were mentioned in the previous section. In the following we shall verify the measured results of CC method. The results will also be compared with that of virtual ground (VG) method. We will first compare hysteresis curves corresponding to both methods. Additionally, the performance on the measuring hysteresis loops of micro-sized ferroelectric capacitor and the effect of parasitic capacitance will be characterized.

5-3-1. Verification of CC method

Figure 5-1 plots the voltage that corresponds to the constant currents used in the constant current method, at a constant current density of 0.2 mA/cm^2. The voltage profile is almost noiseless. The single cycle time is 0.68 s and the maximum voltage was approximately ± 6.5 V. Under these test conditions, the testing time was shorter than the reported cycle time of 360 s, because the testing current density was higher than the reported value of 0.156 μA/cm^2 [28].

Figure 5-2 shows the results concerning leakage properties with a delay of 1 s. The leakage current (I) is negligible relative to the constant current (I_0) used in testing. In the case of a negligible conduction current (I_C), the total current $I_0(t) = A\,dD(t)/dt + I_C$ obtained using the CCM technique is [28-29]

$$I_0(t) = A\frac{dD(t)}{dt} = A\frac{\varepsilon\varepsilon_0}{L}\frac{dV(t)}{dt} + A\frac{dP(t)}{dt}, \qquad (3\text{-}1)$$

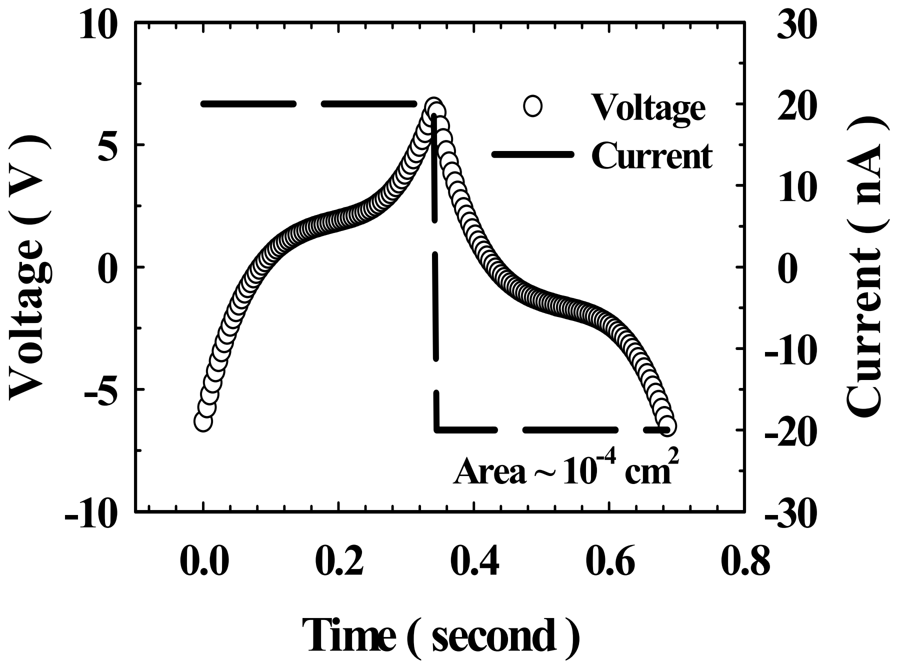

Fig. 5-1 Electric constant current array (dashed line) and resulting voltage (open circle) vs time plot, obtained using the constant current method, for a 250-nm-thick PZT(30/70) film.

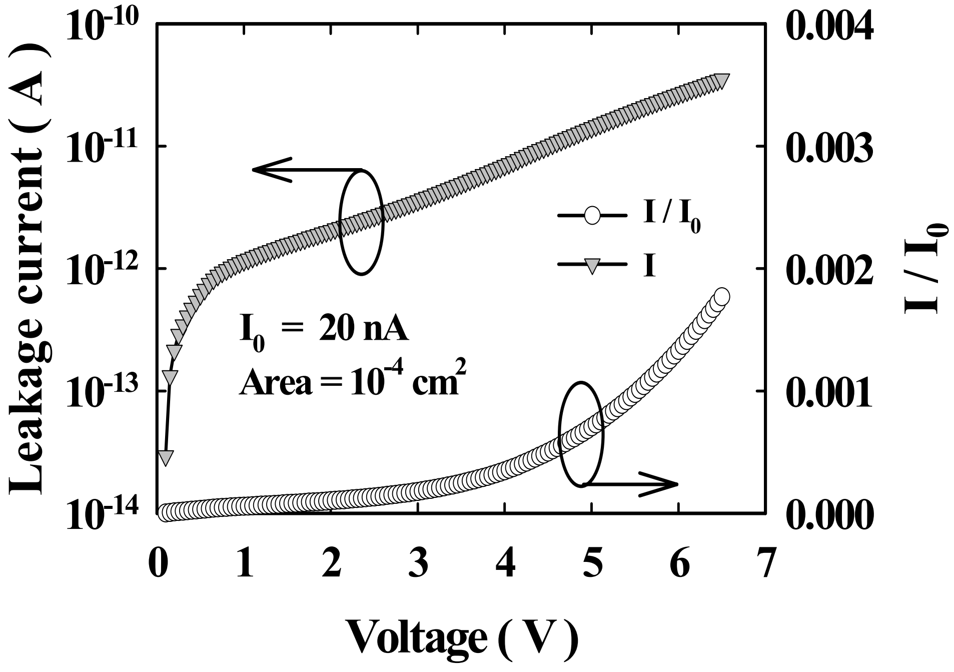

Fig. 5-2 Current-voltage characteristics and the ratio I/I_0 for a 250-nm-thick PZT(30/70) film, where $I_0 = 20nA$ is the testing constant current.

where $D(t) = (1/A)\int_0^t I_0(t)dt$ is the dielectric displacement; ε is the instantaneous dielectric constant; ε_0 is the dielectric permittivity of free space; $P(t)$ is the electric polarization; $V(t)$ is the voltage across the specimen; A is the area of the specimen, and L is the thickness of the specimen.

Hence, this assumption, the dielectric displacement $D(t) = (1/A)\int_0^t I_0(t)dt$, can be determined. Figure 5-3 shows the hysteresis plots obtained using the CCM and the VGM techniques. The two plots are almost identical, even though the testing methods are different. These results imply that the CCM method is also suited for measuring the hysteresis of a relatively thin ferroelectric film.

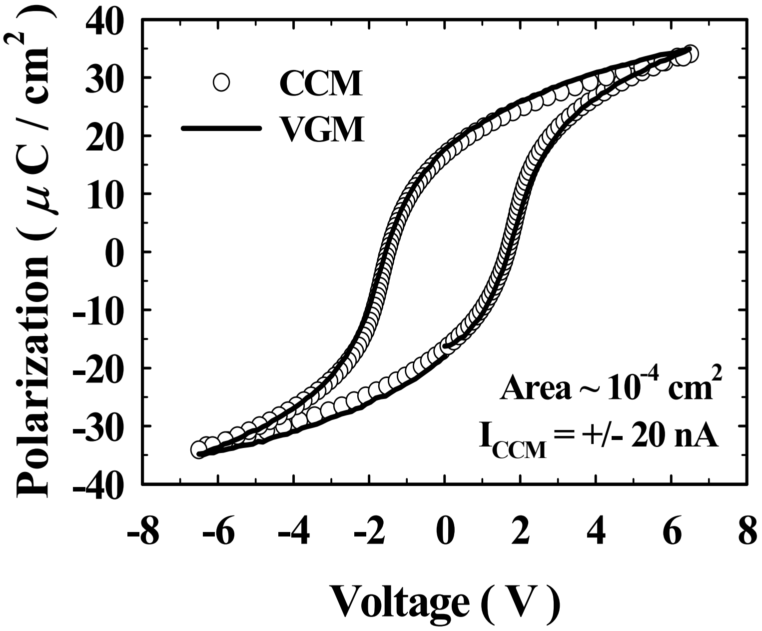

Fig. 5-3 Hysteresis curves of electric displacement ***D*** vs voltage *V*. The unbroken line plots the curve obtained by the virtual ground method (VGM); the open circles represent results obtained using the constant current method (CCM). The area of the sample was 10^{-4} cm^2.

5-3-2. Hysteresis measurement of micron-sized ferroelectric capacitor

Figure 5-4 shows the hysteresis plots measured for 100 μm×100 μm, 50 μm×50 μm, 20 μm×20 μm, 10 μm×10 μm, 5 μm×5 μm, 3 μm×3 μm, and 2 μm×2 μm capacitors. As in other studies [31-32], the maximum polarization increases markedly as the area of the capacitor declines, perhaps because of the presence of the parasitic capacitance of the probe station.

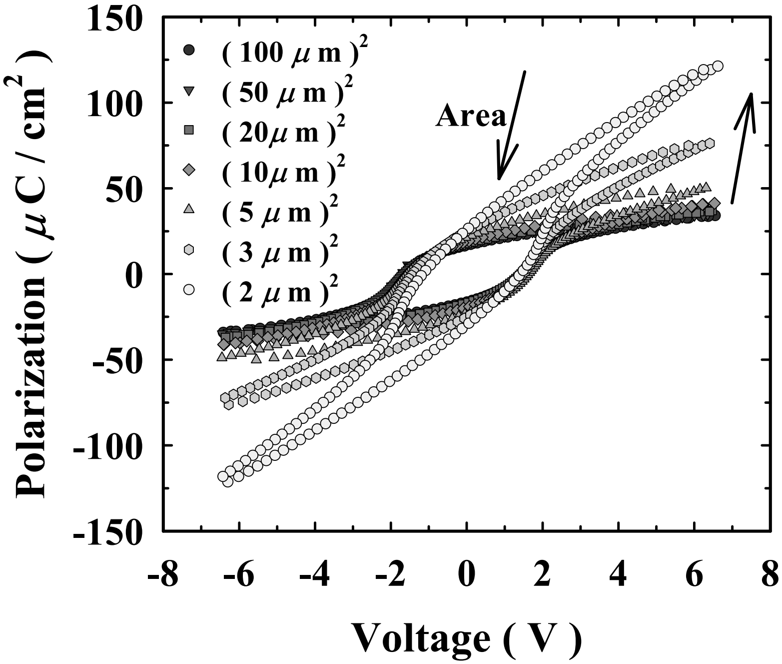

Fig. 5-4 Hysteresis plots obtained from 100μm×100μm, 50μm×50μm, 20μm×20μm, 10μm× 10μm, 5μm×5μm, 3μm×3μm, and 2μm×2μm capacitors using the CCM method.

The parasitic capacitance of the probe station was assumed to act in parallel with the capacitance of the ferroelectric capacitors. Figure 5-5 shows the equivalent circuit used in the CCM technique, including the parasitic capacitance (C_{para}) of the probe station. The voltage responses of the parasitic capacitor are obtained using the constant current method. The parasitic capacitance can be determined from the slope of the voltage buildup plots, $V(t)=(I_0\times t)/C_{\text{para}}$ [29], shown in Fig. 5-6, given that the charge of the parasitic capacitor is $Q_{\text{para}}=I_0\times t=C_{\text{para}}\times V(t)$. The voltage buildup plots are almost straight lines under a range of test conditions. The mean parasitic capacitance was determined to be around 0.4357 pF. Figure 5-7 shows the relevant results. The parasitic charge on the probe station can be calculated from the mean parasitic capacitance, using $Q_{\text{para}}=C_{\text{para}}\times V(t)$. Removing the contribution of parasitic effects yields the corrected hysteresis plots in Fig. 5-8. The hysteresis plots of these small capacitors show a slight increase in maximum polarization compared to the result of a 100 μm×100 μm capacitor. These increases may be related to increases in the linear dielectric constant, which can be calculated from the high-field slope of the hysteresis loops [32]. The variation of dielectric constant with the area of the capacitor was also investigated using a HP 4284A LCR meter with an AC signal of 50 mV at a DC voltage of 0 V, to elucidate these increases. Figure 5-9 shows the results. They show that the dielectric constant increases slightly as the area of capacitor decrease. This finding is consistent with the result concerning the linear dielectric constant obtained from the high-field slope of hysteresis loops corrected for parasitic effects. This figure also indicates the linear dielectric constants obtained from the high-field slope of uncorrected hysteresis loops. The parasitic capacitance is thus observed to influence strongly the hysteresis plot of a small capacitor.

The parasitic charge and maximum polarization charge on a 100 μm×100 μm capacitor were considered to anticipate the parasitic effect on a submicron-sized capacitor, as indicated in Fig. 5-10. This figure presents results taken from ref. [31] and experimental results obtained herein. The parasitic charge dominates the measurements when the length of the side of the square capacitor is less than 3 μm. These results imply that the parasitic charge represents a challenge in the measurement of electrical hysteresis of submicron-sized ferroelectric capacitors.

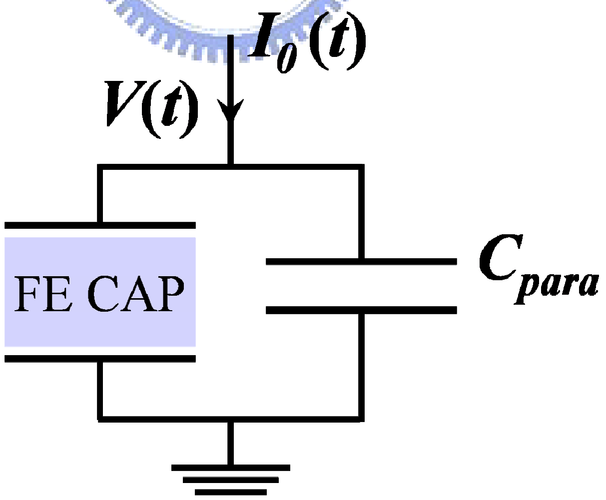

Fig. 5-5 Equivalent circuit associated with the CCM technique, including the parasitic capacitance (C_{para}) of the probe station.

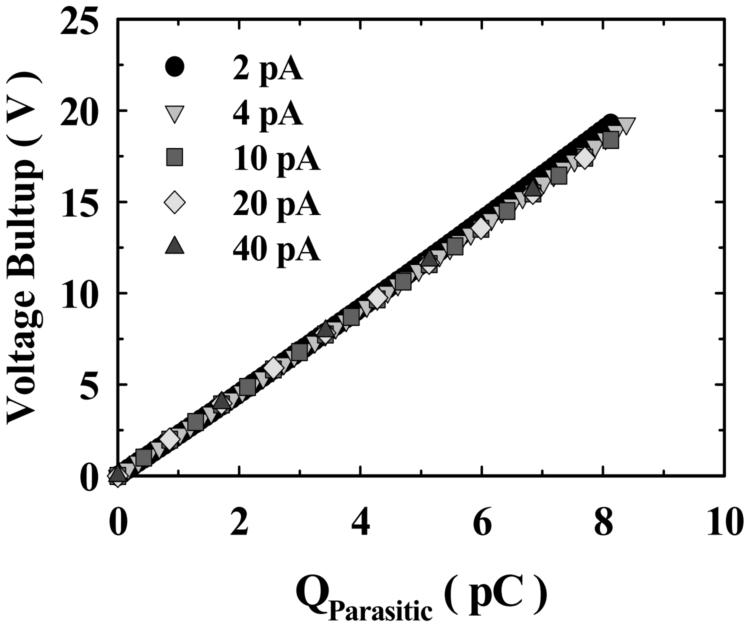

Fig. 5-6 Voltage buildup vs parasitic charge $Q_{\mathrm{para}}(t) = I_0 \times t$, for various charging currents.

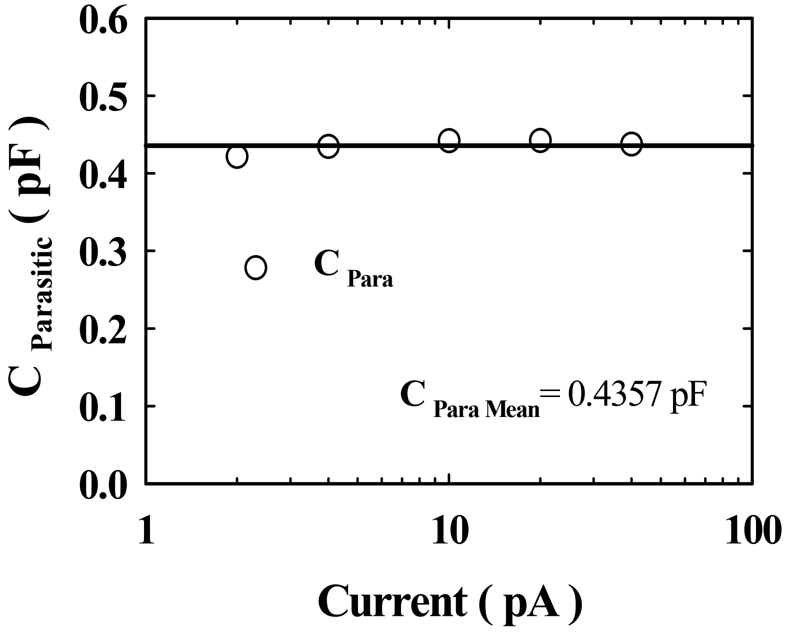

Fig. 5-7 Parasitic capacitance determined from the voltage buildup using $V(t) = (I_0 \times t)/C_{\mathrm{para}}$, for various charging currents.

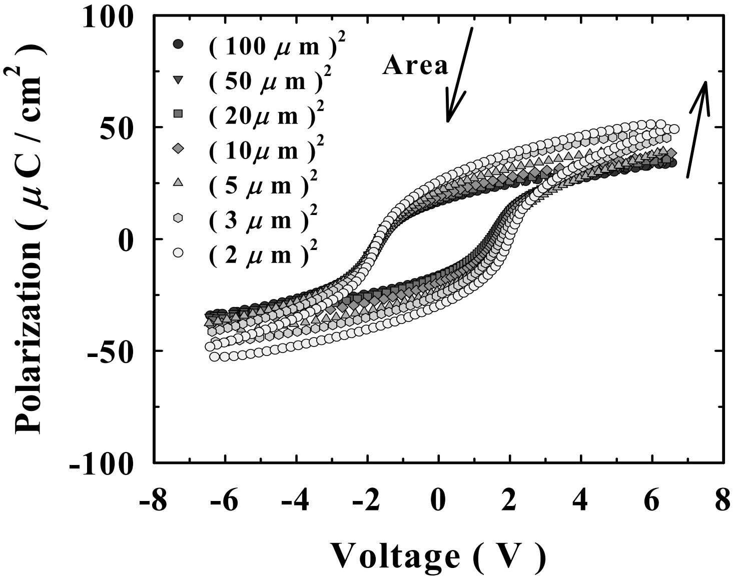

Fig. 5-8 Hysteresis plots of 100 μm×100 μm, 50 μm×50 μm, 20 μm×20 μm, 10 μm×10 μm, 5 μm×5 μm, 3 μm×3 μm, and 2 μm×2 μm capacitors obtained using the CCM technique with parasitic correction.

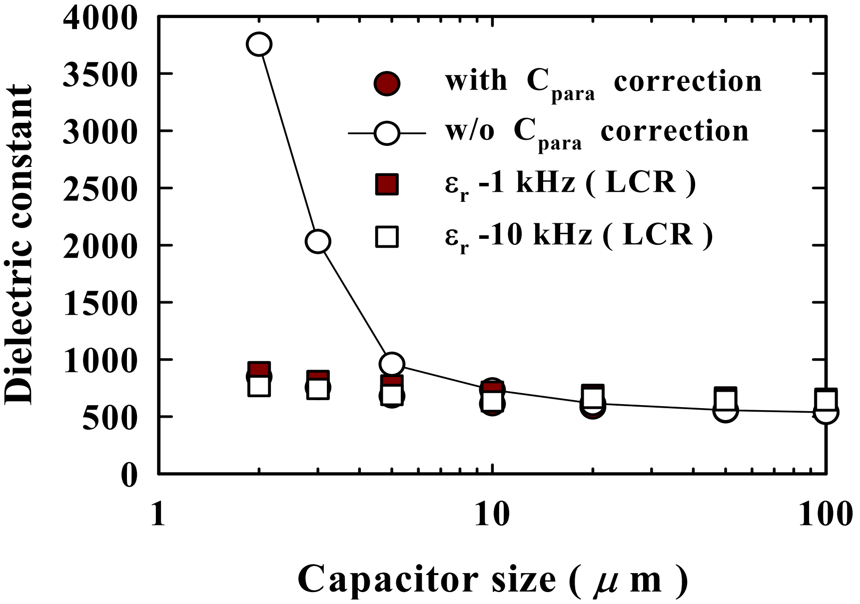

Fig. 5-9 Linear dielectric constant vs the size of the capacitor. Solid and open circles represent the dielectric constants obtained from the high-field slope of the hysteresis loops with and without parasitic correction, respectively. Full and open squares represent the results measured using an LCR meter at 1 kHz and 10 kHz, respectively.

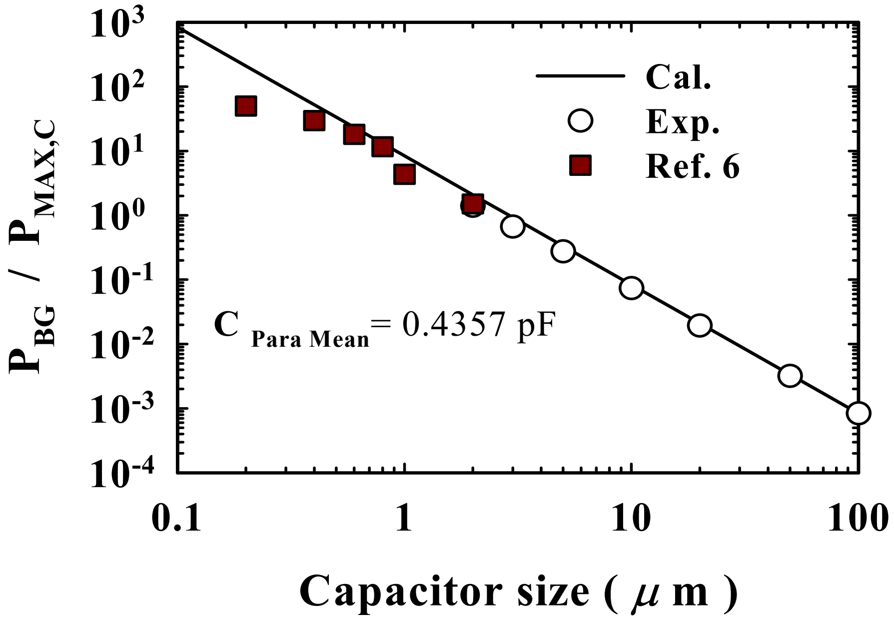

Fig. 5-10 Parasitic effect ($P_{BG} / P_{MAX,C}$) on small-sized capacitors, where P_{BG} is the parasitic charge of the probing setup, and $P_{MAX,C}$ is the maximum polarization charge with parasitic correction. The unbroken line is the expected result. The open circles and full squares represent the empirical results and the results obtained from ref. [31], respectively.

5-4. Summary

In this chapter, a constant current method (CCM) was implemented to yield electrical hysteresis loops of micron-sized ferroelectric capacitors. The results for a small capacitor show that the parasitic capacitance of a probe station may markedly increase the maximum polarization as the area of the capacitor declines. The CCM technique can be exploited to calculate the parasitic capacitance of the probe station and thus construct the corrected hysteresis loops. Additionally, the dielectric constants of small capacitors were measured using an LCR meter. Satisfactory agreement was obtained between the measured dielectric constants and those obtained from the high-field slopes of hysteresis loops that had been corrected for parasitic effects. These results suggest that the CCM technique represents a method for investigating the ferroelectric characteristics of small ferroelectric capacitors.