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Citation: Journal of Applied Physics **86**, 6376 (1999); doi: 10.1063/1.371700
View online: http://dx.doi.org/10.1063/1.371700
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Improvement in the electrical properties in Pt/Pb($Zr_{0.52}Ti_{0.48}$)O_3/Pt ferroelectric capacitors using a wet cleaning method

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(Received 12 March 1999; accepted for publication 10 August 1999)

A wet cleaning solution was designed to specifically eliminate nonferroelectric phases, such as pyrochlore, PbO, and the etching damaged layer. Scanning electron microscopy pictures clearly showed that treatment with the cleaning solution completely removed these nonferroelectric phases. After removing the nonferroelectric phases, ferroelectric properties such as remnant polarization, coercive voltage, and leakage current, were remarkably improved. In addition, the wet cleaned ferroelectric capacitors yielded superior endurance against hydrogen-induced damage compared to those of the noncleaned capacitors. © *1999 American Institute of Physics.*
[S0021-8979(99)02622-5]

I. INTRODUCTION

Recently, there has been a tremendous interest in ferroelectric thin films especially with regards to their applications in nonvolatile memory devices.[1–3] Despite the large amount of effort in this field, several issues still remain to be solved for the integration of ferroelectric capacitor modules with standard silicon technology.[4–11] The major issues under current investigation are plasma damage, hydrogen-induced damage, and conductive diffusion barriers. Thus, considerable effort has been made to minimize plasma damage in connection with the undesirable effects of damaged layers. Recently, Lee *et al.* reported that the side region of ferroelectric film is susceptible to damage during the dry etching process. They further suggested that the damaged layer appears to be amorphous in nature and plays a parasitic role in the ferroelectric capacitor, which would lead to deteriorated performance.[10] Torii *et al.* attempted oxygen rf-plasma treatment in order to cure the etching damaged layer, but could not achieve complete recovery.[11] On the other hand, the existence of a secondary phase, namely, pyrochlore or PbO is also thought to be an additional parasitic source. This can be avoided by optimizing process parameters, such as excess lead content, annealing ambient, heating rate, and the use of seed layers.[12] However, the growth and confirmation of pure perovskite Pb($Zr_{0.52}Ti_{0.48}$)O_3 [PZT or PZT(52/48)] thin films without trace amounts of second phases appear to be very difficult, in particular, on a production scale. For these reasons, we have attempted to develop a wet cleaning technique to remove the second phases as well as the etching damaged layer.

Preliminary results concerning the properties of the etching damaged layer and secondary phases have already been reported.[10,13] A detailed understanding of the damaged layer permits the formulation of a wet cleaning solution so that the nonperovskite phases can be selectively removed. In this work, we report on the effectiveness of our wet cleaning solution, which was designed to eliminate secondary phases and the etching damaged layer. Additionally, endurance to hydrogen-induced damage is also examined, since it is thought that a relationship exists between hydrogen-induced damage and the existence of nonferroelectric phases. We also propose a method for the effective removal of the secondary phases which are formed on the surface of PZT thin films.

II. EXPERIMENTAL PROCEDURE

PZT thin films, 240 nm in thickness, were spun coated onto Pt/TiO_2/SiO_2/Si substrates by a sol–gel method. The precursor with a Zr/Ti ratio 52/48 and 10% excess Pb was purchased from the Mitsubish Materials Co. After depositing the top Pt electrode at a thickness of 200 nm, PZT and Pt films were dry etched by means of a Tegal® 6540 dry etcher using a gas mixture of Ar–Cl_2–CF_4. The size of the patterned capacitors was $100\times 100\,\mu m^2$. To understand the nature of the etching damaged layer, partially etched PZT specimens, without the protection of the photoresist, were produced. The physical and chemical properties of the films were characterized using x-ray diffraction (XRD), x-ray pho-

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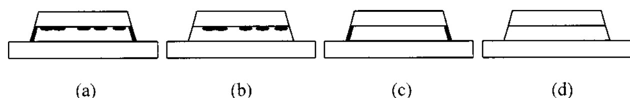

FIG. 1. Four types of Pt/PZT/Pt capacitors were fabricated. (a) Annealed at 450 °C in ambient O_2 after dry etching, (b) sidewall cleaning processed to eliminate the etching damaged layer, (c) surface cleaning processed to eliminate secondary phases, and (d) surface and sidewall cleaning processed.

toelectron spectroscopy (XPS), scanning electron microscopy (SEM), and atomic force microscopy (AFM) techniques. The thickness of the PZT film was monitored using Nanospec®. Electrical properties, including ferroelectric characteristics, were evaluated using Radiant Technologies RT6000 system and the Keithley $I-V$ measurement system.

In wet cleaning experiments, a variety of solution mixtures were examined in order to determine the optimum solution composition. In the beginning, we determined the proper etchants for PZT components such as titanium oxide, zirconium oxide, and lead oxide. The etching rate of PZT can be adjusted by altering the concentration of the etching solution by a suitable solvent. Among the investigated solutions, a mixture of ethanol (EtOH), acetic acid (CH_3COOH, AcOH), and BOE (buffer oxide etchant, 6.6% HF) with a 14.0/1.5/0.5 volumetric ratio showed the best property as a cleaning solution.

In an effort to determine whether any particular parasitic effects exist, four types of metal–ferroelectric–metal (MFM) capacitors were fabricated, as shown in Fig. 1. First, capacitor (a) has both the etching damaged layer in the side region and secondary phases on the surface of the PZT film. This was used as a reference capacitor to correlate variations in properties with the cleaning process. In the case of capacitor (b), the etching damaged layer was eliminated by the sidewall cleaning process, but secondary phases on the PZT surface still remain. In capacitor (c), the secondary phases were removed by the surface cleaning process, but the etching damaged layer exists in the side region of the capacitor. Finally, both sidewall cleaning and surface cleaning processes were employed for the type-(d) capacitor, and thus leaving behind only a pure perovskite phase.

III. RESULTS AND DISCUSSION

A. Characterization of the etching damaged layer

As a first step in the development of the cleaning solution, a detailed analysis of the etching damaged layer was made using the intentionally etch damaged PZT thin films, which were obtained by etching the PZT thin film in the absence of photoresist protection. Based on the previous study, the etching damaged layer was shown to be noncrystalline but amorphous, and the thickness of the damaged layer was about 10 nm. It was also shown that this damaged layer could not be crystallized completely even after thermal annealing at 650 °C. The XRD spectra in Fig. 2 clearly indicates that amorphous phases remain on the surface of the damaged PZT film even though it was annealed at the PZT formation temperature. However, when the damaged layer was eliminated by the treatment with the cleaning solution,

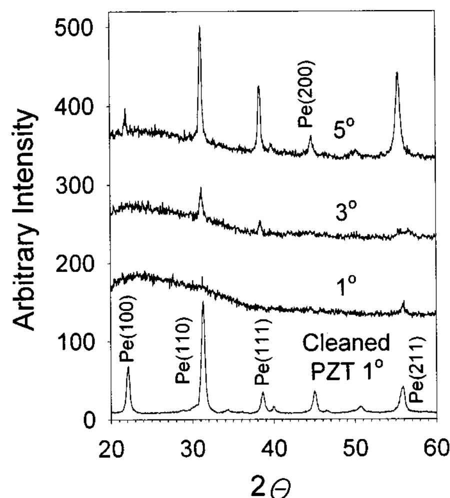

FIG. 2. XRD spectra of the surface cleaned PZT film with a glance angle of 1°, and the 650 °C-annealed etching damaged PZT film with glance angles of 1°, 3°, and 5°.

clear PZT peaks were observed. In addition, we analyzed the chemical composition of the etching damaged layer by using XPS. As shown in Fig. 3, fluorine (F) and chlorine (Cl) atoms were detected in the survey spectrum. Furthermore, the insets of Fig. 3 clearly show shifts in binding energies for lead (Pb $4f_{7/2}$), zirconium (Zr $3d_{5/2}$), and titanium (Ti $2p_{3/2}$) peaks. In the etching damaged layer Pb, Zr, and Ti peaks were shifted 2–4 eV to higher-binding energy compared to those obtained from the pure perovskite phase, indicating that parts of the PZT film might have chemical reactions with the Cl_2 and CF_4 of the etching gas. This also provides an explanation for why the effort to convert the damaged layer by the thermal treatment was not successful. Details of the etching damaged layer can be found in Ref. 10.

B. Properties of the cleaning solution

Based on the fact that the thermal treatment is not sufficient to recover the damage which originates from the dry etching process, the wet cleaning approach was attempted.

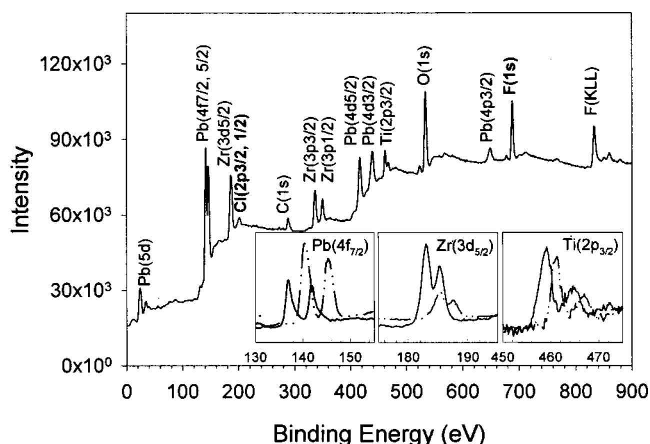

FIG. 3. XPS spectrum of the etching damaged layer. Fluorine and chlorine peaks were detected and metal (Pb, Zr, and Ti) peaks were shifted to higher-binding energy. In the insets: solid lines, perovskite PZT and dotted line, etching damaged layer.

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TABLE I. Etching rates of various PZT thin films.

PZT film property		EtOH/AcOH/BOE (volumetric ratio)	
		85/10/5 (Å/min)	85/10/10 (Å/mim)
PZT (52/48)	(111) highly oriented	27–28	60–63
	(110) highly oriented	18–19	40
	Etch damaged layer	22	⋯
PZT (30/70)	(111) highly oriented	6–8	15
	Etch damaged layer	21	⋯

The cleaning solution was designed to dissolve PZT components such as lead oxide, zirconium oxide, and titanium oxide. A variety of mixed solutions were examined. A critical problem was the removal of the lead component, namely, lead oxide. Zirconium and titanium components can be easily eliminated by the treatment with a dilute HF solution, but a residual lead component always remains on the surface after the solution treatment. Given the fact that nitric acid (HNO_3) and acetic acid are known to be typical etchants for PbO, we attempted to mix these solutions with the BOE. However, when the HF and HNO_3 solutions were mixed together, precipitation was observed on the PZT surface. The precipitation is thought to arise from salt formation between the anion (F^-, NO_3^-) and PZT metal components (Pb, Zr, and Ti). Thus, a combination of BOE and acetic acid was more efficient as a PZT echant than that of BOE and nitric acid. Along with the appropriate selection of the etchants, another feature to point out is the control of the etching rate, since the etching rate of the aforementioned solution was too high to use for the purpose of cleaning. As an approach to reduce the etching rate, various types of organic solvents were added to decrease etchant activity. Among these, ethanol showed the best properties in view of solution homogeneity and proper etching rate. Therefore, we chose a combination of EtOH–AcOH–BOE as a PZT cleaning solution. The etching rates of various PZT film properties are listed in Table I. Etching rates varied according to the PZT film property, such as the Zr/Ti ratio, film orientation, and etching damaged layer. In the case of Ti-rich PZT(30/70), the etching rate decreased drastically to one third of that for PZT(52/48) but, surprisingly, the etching rates of the damaged layers showed a similar rate in spite of the difference in the Zr/Ti ratio. As shown in Fig. 4, the etching rate is monotonically proportional to the amount of BOE, whereas it is independent of the amount of AcOH. This suggests that the removal of ZrO_2 and TiO_2 by HF is the key step in PZT cleaning.

C. Effects of secondary phases and the etching damaged layer

The cleaning process can be utilized in different ways. The first method is to remove the etching damaged layer formed in the side region of the capacitor. (This process is referred to as sidewall cleaning.) The second is to remove the secondary phases that were grown on the surface of the PZT film. (This process is referred to as surface cleaning.) In fact, the approach adopted to eliminate the etching damaged layer led us to develop the surface cleaning technique. In accordance with the previous results, first the blanket etching on the PZT surface was carried out to intentionally produce the damaged amorphous area, which was then treated with cleaning solution to dissolve the etching damaged layer. Because the etching rates between perovskite PZT and secondary phases were quite different, we should produce primarily the amorphous layer on the surface. Otherwise, the result would be a rough PZT surface. When a 250 Å PZT(52/48) film was etched by the cleaning solution without the blank etching, the surface roughness increased from 20 to about 80 Å.

The effectiveness of the sidewall cleaning process in view of ferroelectric properties is clearly shown in Fig. 5. Remnant polarization (P_r) values were obtained from 15 capacitors at different positions in 6 in. wafers. Prior to the sidewall cleaning process, the average value of P_r was around 14.3 $\mu C/cm^2$. However, after the etching damaged layer was eliminated by the sidewall cleaning process, the P_r values were increased about 3 $\mu C/cm^2$. The total thickness of the etched layer is controllable by altering the etching time. When 60 Å of the etching damaged layer was eliminated, the average value of P_r was about 16.9 $\mu C/cm^2$. As the thickness of the etched layer is increased, higher values of P_r were obtained. Finally, when about 90 Å of the damaged layer was eliminated, the average P_r increased from 14.3 to 17.5 $\mu C/cm^2$. However, additional increases in the value of P_r were not observed when the thickness of the etched layer was increased above 90 Å. This can be attributed to the fact that the thickness of the etching damaged layer is about 100 Å. The surface morphology of the side region of Pt/PZT/Pt

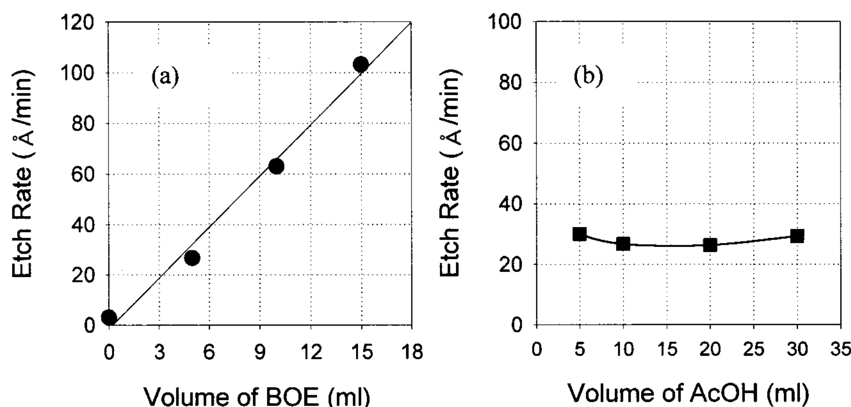

FIG. 4. Effect of cleaning solution components. (a) Etch rate of PZT film with increasing amounts of BOE, EtOH/AcOH/BOE=85/10/x, x=0, 5, 10, and 15 ml. (b) Etch rate of PZT film with increasing amounts of acetic acid, EtOH/AcOH/BOE=85/y/5, y=5, 10, 20, and 30 ml.

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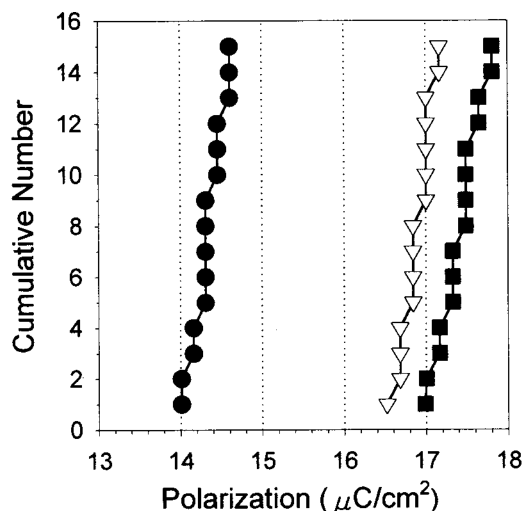

FIG. 5. Remnant polarization values of Pt/PZT/Pt capacitors. 15 capacitors in a 6 in. wafer were measured for accurate comparison. ●, no sidewall cleaning; ▽, 60 Å of the etching damaged layer was eliminated by the sidewall cleaning process; and ■, 90 Å of the etching damaged layer was eliminated.

capacitors before and after the sidewall cleaning process is compared in Fig. 6. Small particulates in Fig. 6(a) were completely eliminated by the sidewall cleaning process, and the surface morphology appeared to be smooth, as shown in Fig. 6(b). XPS analysis indicated that the Pb/Zr/Ti ratios in an as-deposited PZT film and the etching damaged layer are 52.6/35.9/11.5 and 42.1/37.1/20.8, respectively. Therefore, small particulates in Fig. 6(a) were thought to be lead-deficient and titanium-rich PZT. It is unclear at present how they can be formed at the sidewall of the capacitor after the etching process. It is assumed that these particulates are formed by the redeposition of vaporized by-products during the dry etching process.

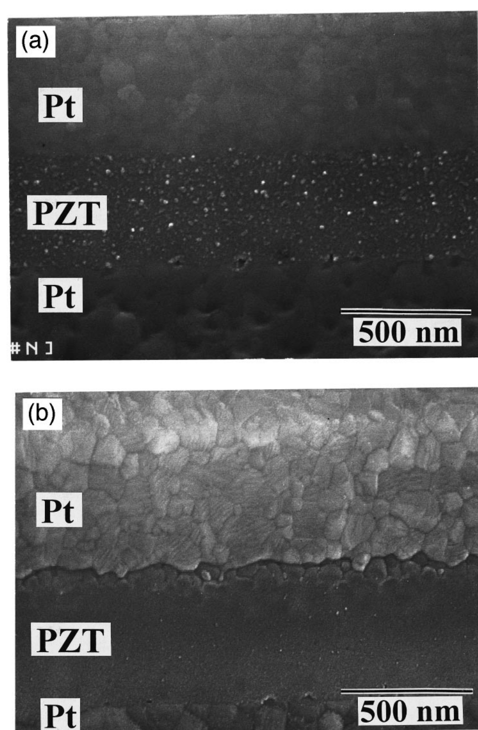

FIG. 6. SEM pictures of the side region in Pt/PZT/Pt capacitors. (a) Before sidewall cleaning and (b) after sidewall cleaning.

To investigate the effectiveness of the cleaning process, four types of capacitors were fabricated, as shown in Fig. 1. The sidewall cleaning process was employed in the case of the (b) and (d) capacitors, and the surface cleaning process was employed in the case of the (c) and (d) capacitors. Therefore, capacitor (a) contains secondary phases and an etching damaged layer, and capacitor (b) contains only the secondary phases at the interface between PZT and the top Pt. Capacitor (c) contains only the etching damaged layer, whereas capacitor (d) contains only the perovskite phase and no nonferroelectric phases. We initially evaluated leakage current variations by measuring $I-V$ and $I-t$ (at 5 V) before and after hydrogen annealing. As shown in Figs. 7(a) and 7(d), the leakage current of the capacitors was closely related to the existence of the damaged layer at the sidewall of the capacitors. The surface cleaning process itself does not lead to a noticeable improvement in the leakage current behavior without incorporating the sidewall cleaning process. This suggests that the improvement in the leakage current can be attributed to the elimination of the parasitic paths, rather than the elimination of the secondary phases on the surface. This clearly implies that the etching damaged layer plays a key role in leakage behavior. In the next step, an attempt was made to relate hydrogen-induced damage with the existence of nonferroelectric phases in the ferroelectric capacitor. When four capacitors were exposed to hydrogen damage conditions for 2 min, namely, 200 °C and 5 mTorr H_2, the leakage current level of capacitors (a) and (c) increased about two orders of magnitude, as shown in Figs. 7(b) and 7(e). This demonstrates that the etching damaged layer in the side region is the primary cause of PZT capacitor degradation. By continuous hydrogen annealing for 4 min, capacitor (b), which contains the secondary phase on the PZT surface, also showed a drastically increased leakage current by four orders of magnitude, as shown in Figs. 7(c) and 7(f). This observation indicates that the secondary phase on the PZT surface is the second cause of PZT capacitor degradation. Capacitor (d), which consists of pure perovskite PZT, still has around 10^{-7} A/cm^2 of leakage current, even though it has been exposed to hydrogen annealing for 4 min.

A surprising aspect of hydrogen annealing is that leakage currents were lowered in capacitors (b) and (d), which were subjected to the sidewall cleaning process. Similar phenomena were also observed by another research group.[14] At low voltage, below 5 V, the leakage level was similar but at high voltage, above 5 V, the leakage current decreased by about one order of magnitude. It is known that Pt/PZT/Pt capacitors follow the Schottky conduction mechanism at high voltage.[15] It is thought that dangling bonds between the top Pt and PZT interface are cured by hydrogen atoms and, therefore, increase the Schottky barrier height. As a result, hydrogen-annealed capacitors showed lower leakage current values. This mechanism is commonly accepted and has been applied to improve gate properties in silicon semiconductor devices, so-called hydrogen passivation, or forming gas anneals.[16] In terms of hysteretic properties, the hysteresis

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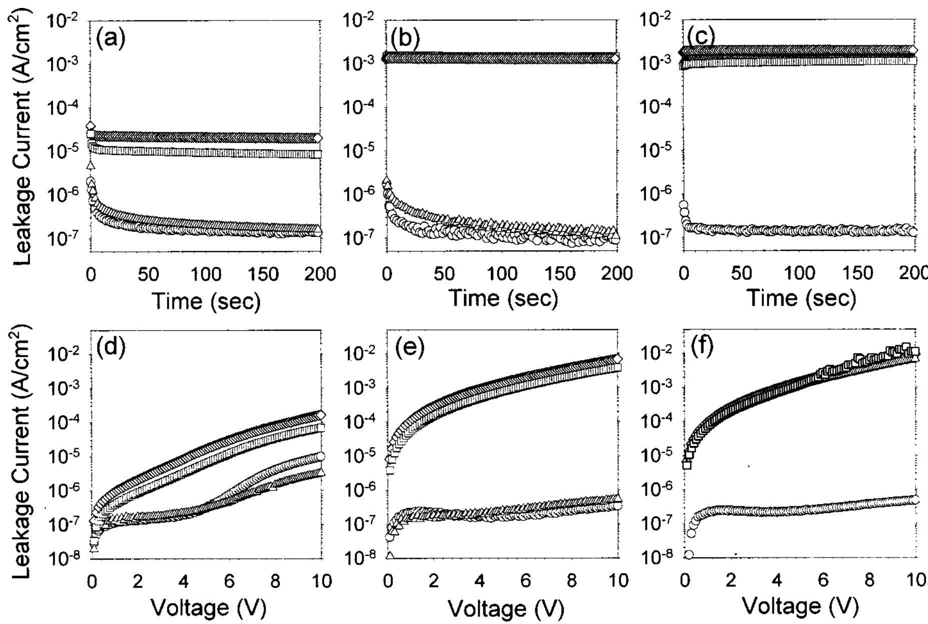

FIG. 7. Leakage current behavior of Pt/PZT/Pt capacitors before and after hydrogen anneals. $I-t$ properties were measured at 5 V [(a), (b), and (c)]. $I-V$ properties were measured with a voltage step 0.1 V and delay time 0.3 s. ○, surface and sidewall cleaning processed; △, sidewall cleaning processed; ◇, surface cleaning processed; and □, no wet cleaning.

loops of four types of capacitors are shown in Fig. 8. Prior to hydrogen annealing, all loops showed a similar shape except for minor variations, particularly in the polarization and coercive voltage values. After 2 min of hydrogen annealing, one can see loops with a distorted shape for Figs. 8(a) and 8(c), while Figs. 8(b) and 8(d) show loops with a proper shape, indicating that the sidewall cleaning process is effective in minimizing the hydrogen damage. By a further annealing for 4 min, the loop of only the sidewall cleaning processed capacitor also adopted a distorted shape, but the capacitor which consisted of only perovskite PZT retained its shape.

Based on measurements of leakage current and the hysteresis loops, it is believed that the existence of nonferroelectric phases, such as the etching damaged layer and the secondary phases, would accelerate the hydrogen-induced degradation of PZT capacitors. The capacitor composed of pure perovskite PZT film showed very good endurance

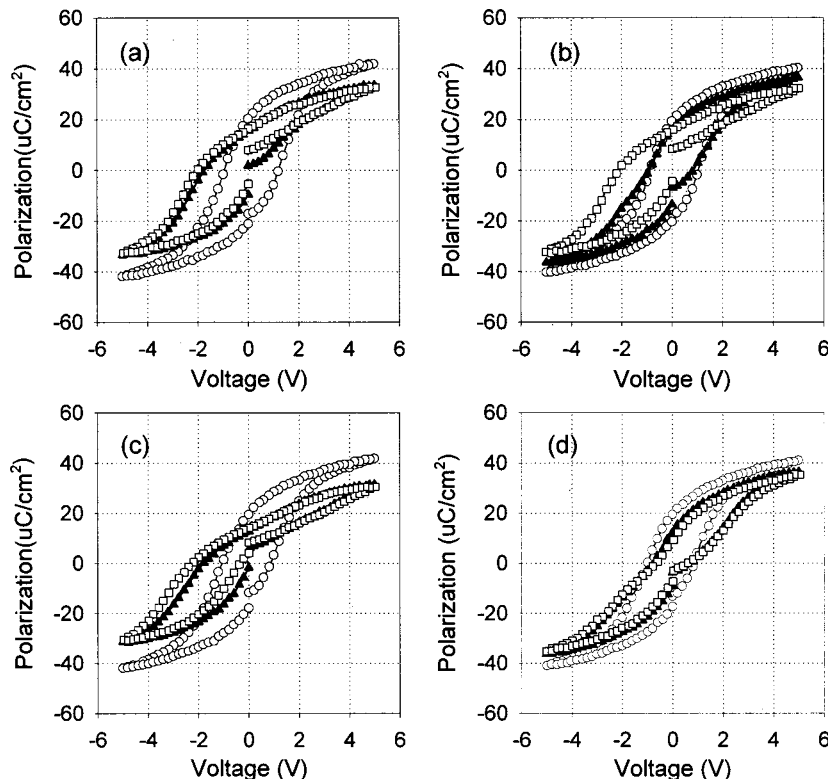

FIG. 8. Hysteresis loop changes before and after hydrogen anneals. (a) No wet cleaning; (b) sidewall cleaning processed; (c) surface cleaning processed; and (d) surface and sidewall cleaning processed. Hydrogen anneal time: ○, 0 min; ▲, 2 min; and □, 4 min.

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against hydrogen annealing, and retained its initial properties, even though it was exposed to a hydrogen annealing for 4 min.

IV. CONCLUSION

The existence of nonferroelectric phases in the Pt/PZT/Pt ferroelectric capacitors was known to play a parasitic role, which leads to degradation of the ferroelectric properties of Pt/PZT/Pt capacitors. We have developed a wet cleaning solution to selectively eliminate these nonferroelectic phases. The cleaning solution is composed of EtOH–AcOH–BOE in a volumetric ratio of 14.0/1.5/0.5. The etching rate of the solution was varied from 6 to 60 Å/min based on PZT film properties. Application of the aforementioned solution resulted in the successful elimination of both the etching damaged layer and the secondary phases, thereby remarkably improving electrical properties particularly in terms of hysteretic characteristics and leakage current. In addition, the elimination of nonferroelectric phases also improved the endurance of PZT against hydrogen-induced degradation.

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