Chapter 2

Experimental Details

2-1. DC sputtering Deposition

The insulator materials (HfO_2) and metal electrode (Pt/Ta) were deposited by an Ion Tech Microvac 450CB sputtering system.

The sputtering system is composed of the following: (1) Sputtering chamber (2) vacuum pumps, consisting of one Cryo pump and mechanical pump (3) DC power (4) 4-inch magnetron gun (5) gas flow meter (6) pressure gauges (7) film thickness monitor. The 4-inch or 6-inch Si substrates were placed in the spin holder driven by a motor. The targets (Pt, Ta and Hf) are 4-inch. The DC source can provide up to 250 W power. Normally the base pressure is around 1.0×10^{-6} torr and the working pressure is around 7.6 mtorr.

For insulating film HfO_2, the flow rate of Ar and O_2 were around 24 sccm and 3 sccm, respectively. The DC power was kept at 100 W. The deposition rate of this insulating film was around 0.02 nm/s.

For the bottom electrode, the flow rate of Ar was around 24 sccm. The DC power was kept at 40 W for Pt or at 150 W for buffer metal Ta. The deposition rate of Pt and Ta were around 0.2 nm and 0.35nm, respectively.

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2-2. Dual E-gun Deposition

The metal electrode (Pt/Ti) was deposited by an ULVAC EBX-10C Dual E-Gun Evaporation System. The base pressure is around 2.0×10^{-6} torr. The deposition rate of Pt and Ti were kept at around 1 nm/s by adjusting the power supply current.

2-3. Thermal Evaporation Deposition

The top metal electrode or backside electrode Al was deposited by an ULVAC EBX-6D Thermal Evaporation Coater. The base pressure is around 2.0×10^{-6} torr. The deposition rate of Al was kept at around 10 nm/s by adjusting the power supply. Normally, the thickness of the electrode was 500 nm.

2-4. Preparation of $Pb(Zr,Ti)O_3$ Precursor Solution

The precursor solution of PZT ferroelectric materials was prepared using the sol-gel method. The detail procedures of precursor preparation were shown in Fig. 2-1.

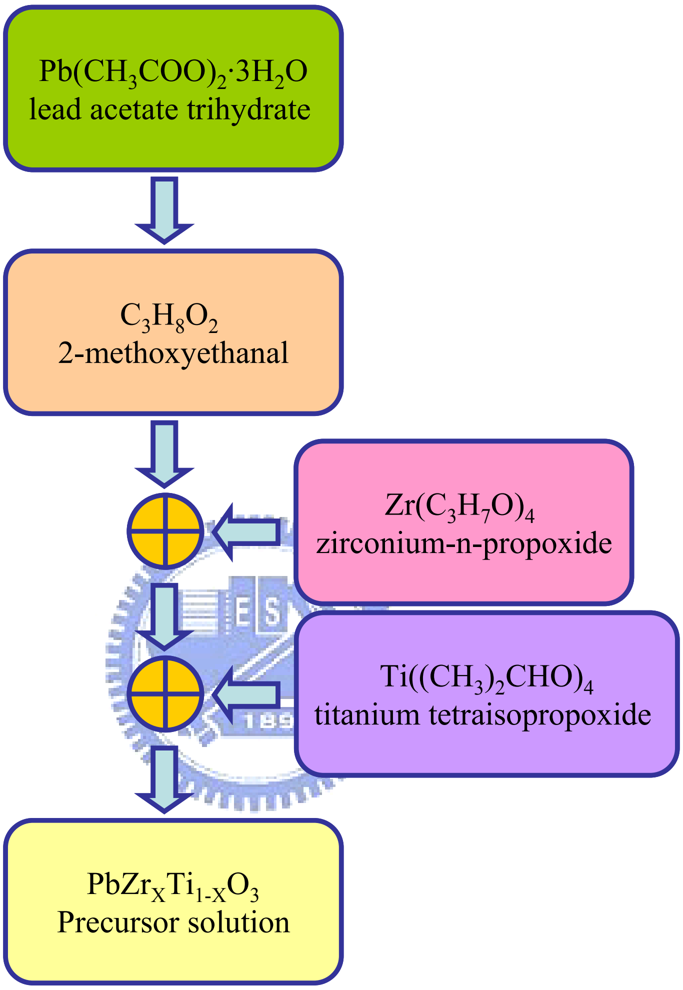

Fig. 2-1 Procedures of precursor preparation of PZT ferroelectric materials using the sol-gel method.

2-5. Deposition Procedures of Ferroelectric Capacitor

The ferroelectric films were deposited using the sol-gel method or MOD technique. The detail procedures were shown in the Fig. 2-2.

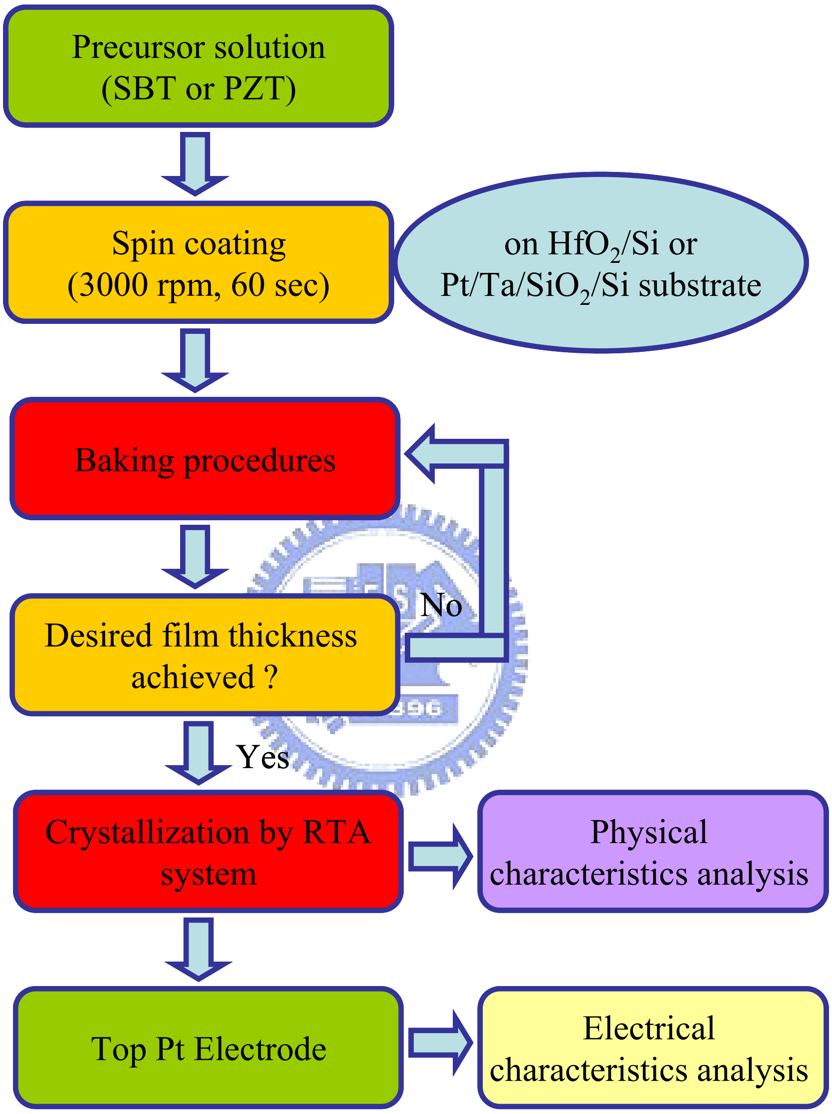

Fig. 2-2 Deposition procedures of ferroelectric film using the sol-gel method or MOD technique.

2-6. X-Ray Diffraction Analysis (XRD)

The film microstructure was characterized by a Mac Diffractometer (at 200 kV and 50 mA) with Cu Ka 1 radiation.

2-7. n&k Optical Analysis

The film thicknesses were obtained by the spectrophotometer (n&k analyzer 1200, Nikon). The n&k analyzer incorporates the formulation for optical constants along with a parameterized model for multi-layer configuration. The thickness and the refraction index of dielectric layers can be obtained by curve fitting according to this method.

2-8. Atomic Force Microscope (AFM)

The film roughness and surface microstructure was characterized by a Digital Instruments DI 5000.

2-9. Secondary Ion Mass Spectroscopy (SIMS)

The element depth profile of MFIS stack was characterized by a CAMECA ims 4f Secondary Ion Mass Spectrometer.

2-10. Auger electron spectrometry (AES)

The element depth profile of the ferroelectric films was studied by Auger electron spectrometry (VG Microlab 310F).

2-11. Thermal Desorption Spectra (TDS)

The residual gases and contamination left in the bulk of the films were characterized by a Hitachi Tokyo Electronics.

2-12. Capacitance-Voltage Measurements (CV)

Computer-controlled HP 4284A was used for high-frequency CV measurements. The measurement frequency was kept at 100 kHz or 1 MHz for these systems. The signal level (small signal amplitude) was fixed at 50 mV and the maximum applied bias may be ranged from ±20 V. The option of integration time was chosen to be short. The measurements were controlled by a personal computer using the Labview program.

2-13. Capacitance-Frequency Measurements (CF)

Computer-controlled HP 4284A was used for CF measurements. The measurement frequency was ranged form 20 Hz to 1 MHz. The signal level was fixed at 50 mV. The option of integration time was chosen to be short. For MIM capacitor, the DC bias was fixed at 0 V. For the MIS structure, the DC bias was fixed at the voltage, operating at accumulation region of CV curves. The measurements were controlled by a personal computer using the Labview program.

2-14. Current-Voltage Measurements (IV)

A Keithley 4200 semiconductor characterization system was used to measure the leakage currents of the thin films. Figure 2-3 shows the capacitor measurement of the metal-ferroelectric-metal (MFM) and metal-ferroelectric-insulator-semiconductor (MFIS) structures.

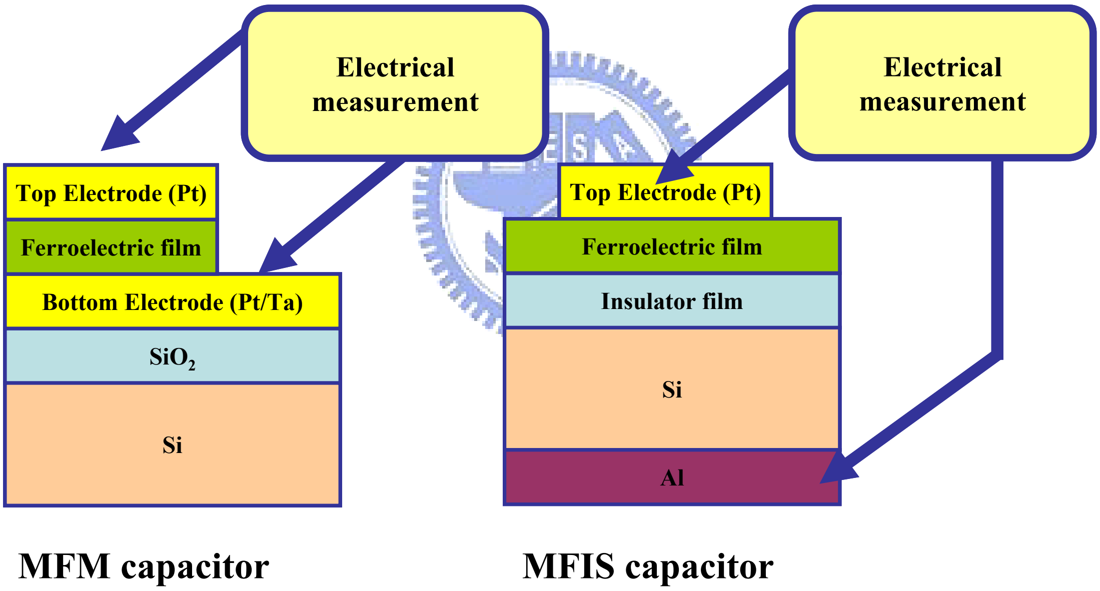

Fig. 2-3 MFM and MFIS capacitor structures for the measurements.

2-15. Polarization-Voltage Measurements (P-V)

A standardized ferroelectric test system RT66A was used to perform the hysteresis measurements. This system was operating in virtual ground mode. The measurement point was fixed at 200. For the fast mode, the frequency of one hysteresis measurement was around 56 Hz. For slow mode, the frequency was around 5.6 Hz. The maximum bias may be ranged from ±20 V.

2-16. System of Capacitance Retention and Endurance Measurements

The measurement systems are composed of the following: (1) HP 4284A LCR meter (2) HP E5250 switching matrix (3) HP 8110A pulse generator (4) Labview programs (5) personal computer (6) GPIB interface and cables. Figure 2-4 shows the structure of the system for performing the capacitance retention measurement and endurance measurement of the metal-ferroelectric-insulator-semiconductor (MFIS) structures. Using this automatic system, the retention and endurance property can be monitored and recorded real-time.

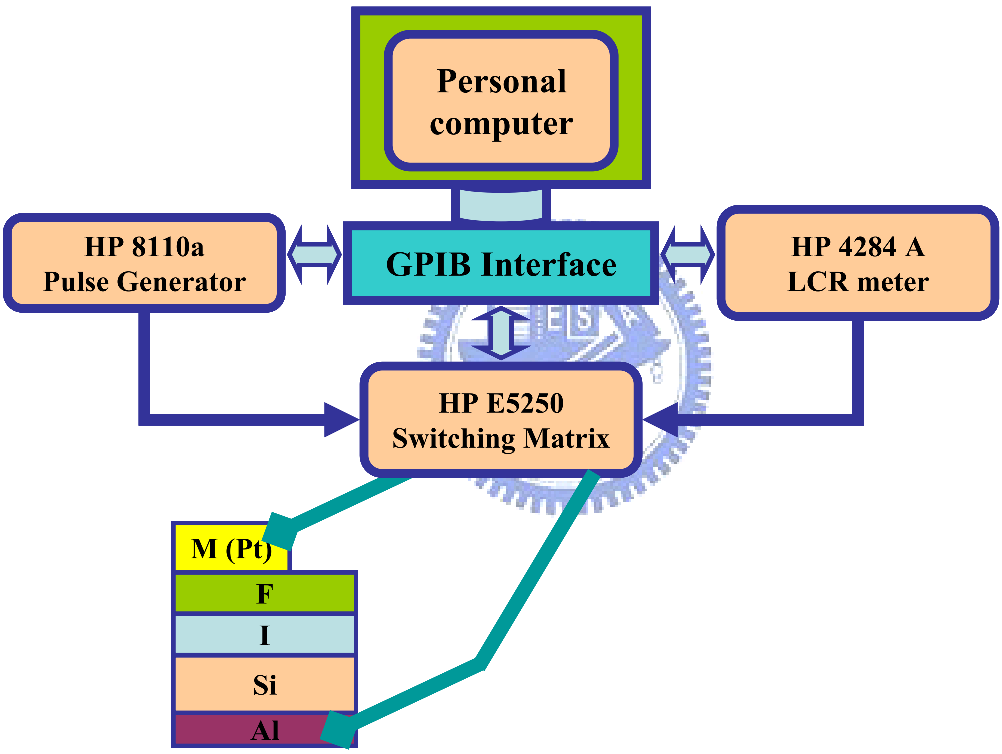

Fig. 2-4 Schematic system construction for performing the capacitance retention measurement and endurance measurement of the MFIS structure.

2-17. Capacitance Retention Measurements for MFIS Structure

Figure 2-5 shows the measurement procedures of the capacitance retention property of the metal-ferroelectric-insulator-semiconductor (MFIS) structures. Before the capacitance retention measurement, the central voltage of the hysteresis CV curve must be determined; thereby the CV measurement must be performed.

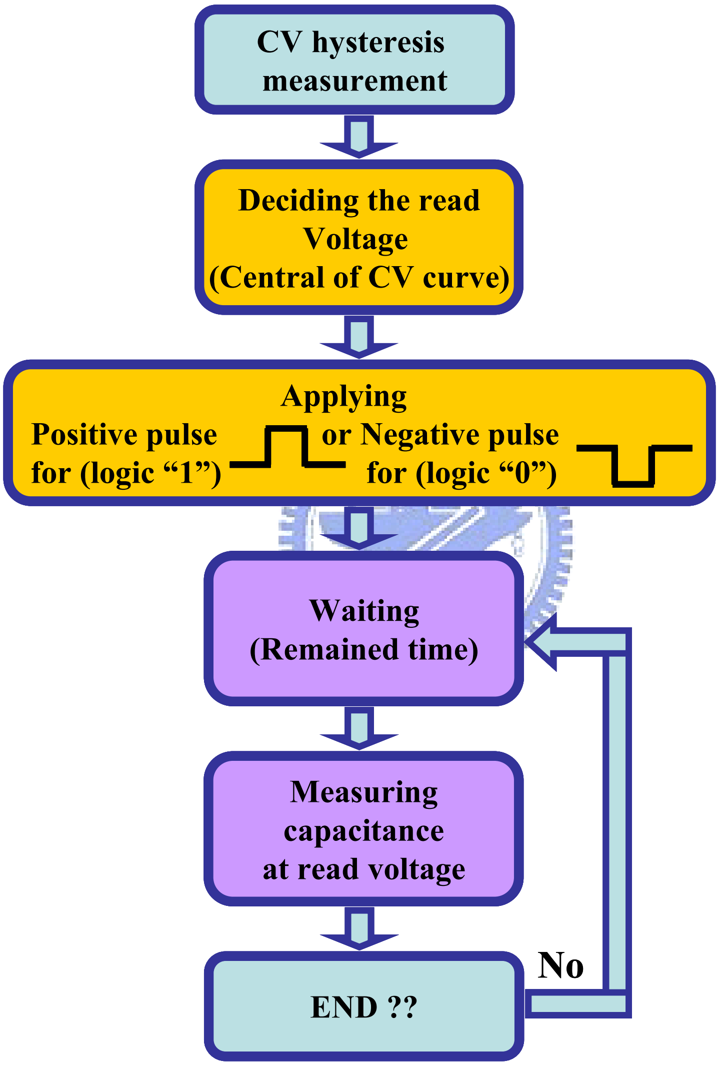

Fig. 2-5 Measurement procedure of the capacitance retention property for the MFIS structure.

2-18. Endurance Measurements for MFIS Structure

Figure 2-6 shows the measurement procedures of the endurance property of the metal-ferroelectric-insulator-semiconductor (MFIS) structures. Before the endurance measurement, the initial CV curve must be measured to determine the maximum amplitude of pulse. During the endurance testing, a bipolar pulse train was employed for testing the switching characteristics.

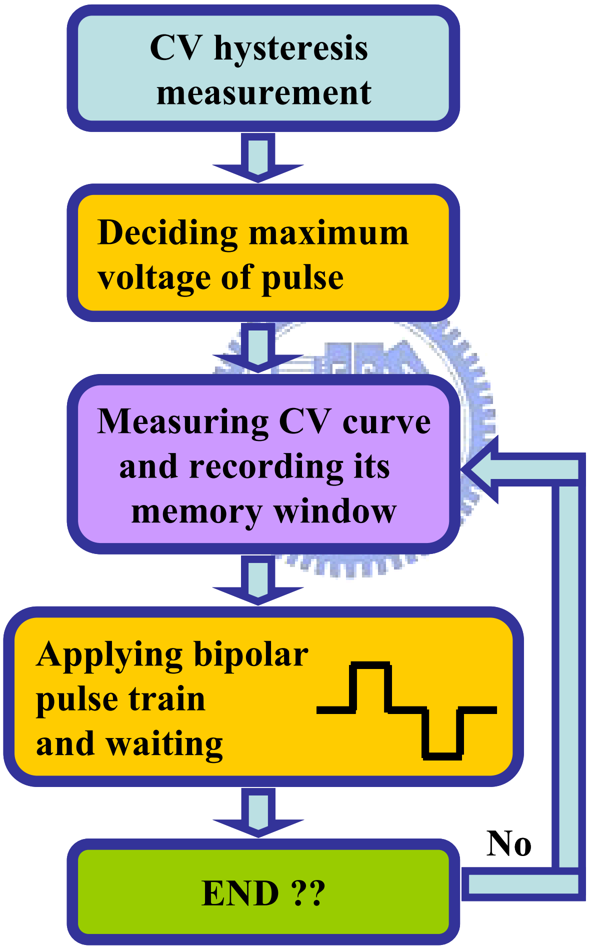

Fig. 2-6 Measurement procedure of the capacitance retention property for the MFIS structure.