Comprehensive Measurement Protocol

Metal-HZO(10nm)-Metal Ferroelectric Memristor

Research Protocol Documentation

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Contents

1 Material System Overview

Target System: $Hf_xZr_{1-x}O_2$ (HZO) Ferroelectric Memristor Stack.

- **Top Electrode (TE):** TiN, Pt, W, or RuO_2 ($10 - 50$ nm).
- **Ferroelectric Layer:** HZO (10 nm, $x \approx 0.5$).
- **Bottom Electrode (BE):** TiN, Pt, W, or RuO_2 ($10 - 50$ nm).
- **Substrate:** Si, SiO_2/Si, or Sapphire.

Neuromorphic Advantages of HZO

- **Non-Volatile:** Retains state without power due to spontaneous polarization.
- **CMOS Compatible:** Unlike PZT or BTO, HZO integrates into standard silicon flows.
- **Scalable:** Robust ferroelectricity down to < 10 nm thickness.
- **Energy Efficient:** Low switching energy (~ 10 fJ/bit potential).

2 Instrument Requirements (Best Case Scenario)

2.1 Primary Electrical Characterization

Semiconductor Parameter Analyzer (SPA): Keithley 4200A-SCS

- 4 SMU channels for simultaneous measurements.
- Current resolution: 1 fA (critical for low leakage HZO).
- Built-in Pulse Measurement Unit (PMU) for pulses down to 200 ns.

Ferroelectric Tester: Radiant Precision Premier II

- **Critical Requirement:** Measures charge (Q) directly, not just current.
- Automated P-E loops, PUND protocols, and fatigue testing up to 10^{10} cycles.

Impedance Analyzer: Keysight E4990A (20 Hz - 120 MHz)

- Required for C-V butterfly curves and dielectric constant extraction.

2.2 Advanced Characterization

Pulse Generator & Oscilloscope: Keysight 33600A + DSOX6004A (6 GHz).

Cryogenic Probe Station: Lake Shore CRX-4K (4 K to 475 K) for Arrhenius plots.

Piezoresponse Force Microscopy (PFM): Asylum Research Cypher ES for domain imaging.

3 Phase 0: Initial Device Screening & Wake-Up

CRITICAL: Wake-Up Effect

As-deposited HZO is often in a mixed phase (monoclinic/tetragonal). It **must** be electrically cycled to transition into the ferroelectric orthorhombic phase. Without wake-up, polarization (P_r) will be negligible.

3.1 Test 0.1: Pre-Measurement Inspection

1. Visual inspection for shorts/damage.
2. Measure pristine capacitance at 1 kHz (check for open circuits).
3. Check leakage at 0.5 V (should be < 1 nA for a good device).

3.2 Test 0.2: Wake-Up Cycling

Parameters:

- **Frequency:** 10 kHz (Triangular/Bipolar).
- **Amplitude:** ± 3 V to ± 5 V.
- **Cycles:** 10^4 to 10^5 cycles.
- **Stop Condition:** When remnant polarization (P_r) stabilizes.

```
- WAKE-UP CYCLING FOR HZO
reset()
smu.measure.terminals = smu.TERMINALS_REAR
local wake_voltage = 4.0
local num_cycles = 10000
local frequency = 10000
local half_period = 1 / (2 * frequency)

smu.source.func = smu.FUNC_DC_VOLTAGE
smu.source.limiti = 100e-6
smu.source.output = smu.ON

for cycle = 1, num_cycles do
    - Positive half-cycle
    smu.source.levelv = wake_voltage
    delay(half_period)
    - Negative half-cycle
    smu.source.levelv = -wake_voltage
    delay(half_period)
end
smu.source.levelv = 0
smu.source.output = smu.OFF
print("Wake-Up Complete")
```

Listing 1: TSP Script for Wake-Up Cycling

4 Phase 1: Ferroelectric Characterization

4.1 Test 1.1: Polarization-Electric Field (P-E) Loop

Instrument: Radiant Tester (Preferred) or Keithley 4200A (via Integration).
Goal: Extract Remnant Polarization (P_r) and Coercive Field (E_c).

Procedure: Sweep voltage $0 \rightarrow +V_{max} \rightarrow -V_{max} \rightarrow 0$. If using Keithley (SMU), measure Current (I) vs Time (t) and integrate:

$$P = \frac{1}{A} \int I(t) dt \tag{1}$$

Where A is the device area.

4.2 Test 1.2: PUND Measurement

Purpose: To separate true ferroelectric switching from leakage current and dielectric capacitance.

Pulse Sequence:

1. **Preset:** Saturates device to $+P_r$.
2. **P (Positive):** Switches state (Includes Switching Current + Leakage).
3. **U (Up):** Non-switching (Includes Leakage only).
4. **N (Negative):** Switches state to negative.
5. **D (Down):** Non-switching negative.

Calculation:

$$Q_{sw} = \frac{(Q_P - Q_U) + (Q_D - Q_N)}{2} \tag{2}$$

4.3 Test 1.3: C-V Butterfly Curve

Instrument: LCR Meter / Impedance Analyzer.
Parameters:

- Frequency: 1 kHz - 1 MHz.
- AC Amplitude: 30 mV (Small signal).
- DC Bias: Sweep ± 5 V.

Success Criteria: Characteristic "butterfly" shape where capacitance peaks at the coercive voltages ($\pm V_c$).

5 Phase 2: Multi-Level States (Neuromorphic Weights)

5.1 Test 2.1: Partial Polarization Switching

To emulate synaptic weights, we need analog conductance states, not just binary 0/1.

Protocol (Incremental Pulse Programming):

1. Reset device to Negative State (-5V).
2. Apply positive pulse stream with increasing amplitude (e.g., 1.0V, 1.1V... 3.0V).
3. Read conductance ($G = I/0.5V$) after each pulse.

```
- MULTI-LEVEL PULSE PROGRAMMING
local v_start = 1.0
local v_stop = 3.0
local v_step = 0.1
local read_voltage = 0.5

for v_pulse = v_start, v_stop, v_step do
    - Program
    smu.source.levelv = v_pulse
    delay(0.001) - 1ms pulse
    smu.source.levelv = 0
    delay(0.01)

    - Read
    smu.source.levelv = read_voltage
    local i_read = smu.measure.i()
    smu.source.levelv = 0

    print(string.format("Pulse: %.2f V, Conductance: %.2e S", v_pulse, i_read/read_voltage))
end
```

Listing 2: TSP Script for Multi-Level Programming

5.2 Test 2.2: State-Wise Retention

Unlike binary memory, each intermediate state must be stable.

- Program to State N (e.g., 50% polarization).
- Read Conductance continuously for 10^4 seconds.
- **Pass criteria:** Drift $< 5\%$ over the measurement period.

6 Phase 3: Synaptic Behavior (Neuromorphic)

6.1 Test 3.1: LTP and LTD

LTP (Long-Term Potentiation): Gradual conductance increase.
LTD (Long-Term Depression): Gradual conductance decrease.

Protocol:

- Apply 50 identical pulses (+2V, 100ms) for LTP.
- Apply 50 identical pulses (-2V, 100ms) for LTD.
- **Metric:** Linearity and Dynamic Range (G_{max}/G_{min}).

6.2 Test 3.2: Spike-Timing-Dependent Plasticity (STDP)

Emulates biological learning rules (Hebbian Learning).

$$\Delta G = f(\Delta t) \quad \text{where} \quad \Delta t = t_{post} - t_{pre} \tag{3}$$

- If $\Delta t > 0$ (Causal): Potentiation (Synapse strengthens).
- If $\Delta t < 0$ (Anti-causal): Depression (Synapse weakens).

7 Phase 4: Reliability & Endurance

7.1 Test 4.1: Fatigue (Endurance) Cycling

Requirement: HZO must survive switching cycles for computing applications.

- **Target:** $> 10^9$ cycles.
- **Frequency:** 1 MHz (using Pulse Generator).
- **Monitor:** P_r and On/Off ratio every logarithmic decade ($10^3, 10^4, \ldots$).

7.2 Test 4.2: Temperature Retention (Arrhenius Plot)

Accelerated aging to predict 10-year retention.

1. Measure retention decay τ at 85°C, 100°C, 125°C.
2. Fit to Arrhenius Equation:
$$\tau(T) = \tau_0 \exp\left(\frac{E_a}{k_B T}\right) \tag{4}$$
3. Extrapolate to room temperature (25°C).

8 Novel Neuromorphic Demonstrations (Future Work)

8.1 Proposal 1: Reservoir Computing

Utilize the inherent non-linearity and short-term memory of the HZO film to process temporal data (e.g., speech recognition) without training the HZO layer itself, only the readout layer.

8.2 Proposal 2: Asymmetric STDP

Demonstrate biologically realistic STDP where Potentiation is stronger than Depression using asymmetric pulse engineering (e.g., +3V Set / -2V Reset).

8.3 Proposal 3: 5-Bit Weight Storage

Attempt to stabilize 32 distinct conductance levels (2^5) with high yield, surpassing standard 3-bit or 4-bit memristive demonstrations.

9 Appendix: Full Fatigue Script (Keithley TSP)

```
- ENDURANCE/FATIGUE TESTING
reset()
smu.measure.terminals = smu.TERMINALS_REAR

local set_voltage = 5.0
local reset_voltage = -5.0
local pulse_width = 1e-3
local read_voltage = 0.5
local num_cycles = 1000000       - 1 Million Cycles
local read_interval = 1000       - Check every 1k cycles

smu.source.func = smu.FUNC_DC_VOLTAGE
smu.source.output = smu.ON

```

```
print("Endurance Cycling Started")

for cycle = 1, num_cycles do
    - SET Pulse
    smu.source.levelv = set_voltage
    delay(pulse_width)
    smu.source.levelv = 0
    delay(0.01)

    - RESET Pulse
    smu.source.levelv = reset_voltage
    delay(pulse_width)
    smu.source.levelv = 0
    delay(0.01)

    - Periodic Readout
    if cycle % read_interval == 0 then
        smu.source.levelv = read_voltage
        local i_read = smu.measure.i()
        smu.source.levelv = 0
        print(string.format("Cycle %d: I_read = %.2e A", cycle, i_read))
    end
end

smu.source.output = smu.OFF
print("Endurance Test Complete")
```

Listing 3: Endurance Test Script