Application Note

Keywords: Ferroelectric devices, Non-volatile memory, Defect engineering

Accurate Oxygen Vacancy Profiling in Ferroelectric Hafnia Using HAXPES

Differentiating True Defect Concentrations from Sputtering Artifacts

Introduction

Ferroelectric hafnium oxide (HfO_2), particularly in its doped form as hafnium zirconium oxide (HZO), is a cornerstone material for next-generation non-volatile memory and logic devices. Its CMOS compatibility and scalability to ultra-thin dimensions make it ideal for advanced semiconductor architectures. However, device performance and reliability are highly sensitive to oxygen vacancy (V_O) concentration and distribution, which influence phase stability, leakage currents, and endurance.

Conventional V_O quantification using X-ray Photoelectron Spectroscopy (XPS) with Ar^+ sputtering introduces significant artifacts, leading to overestimation of the V_O concentration by up to an order of magnitude. This study demonstrates how Hard X-ray Photoemission Spectroscopy (HAXPES) provides a non-destructive, accurate alternative for depth-resolved V_O analysis.

Objective

To accurately quantify oxygen vacancy concentrations in ultra-thin ferroelectric HZO capacitors and assess the impact of sputtering-induced artifacts on conventional XPS measurements.

Methods

- **XPS**: Scienta Omicron XPS Lab with Argus CU and Al Kα source, Ar^+ sputtering
- **HAXPES**: Scienta Omicron R4000 analyzer at the 7-ID-2 beamline, NSLS-II, Brookhaven National Laboratory
- Comparative analysis supported by first-principles calculations

Key Benefits of HAXPES

1. Non-Destructive Depth Profiling

Photon energy tuning in HAXPES achieves comparable probing depths to sputtered XPS without altering film chemistry. This eliminates preferential oxygen sputtering and preserves the integrity of ultra-thin ferroelectric layers.

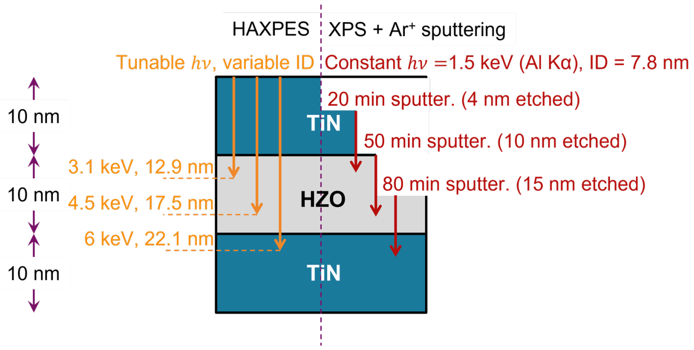

Fig 1: Illustration of depth sensitivity of XPS (with sputtering) vs. HAXPES (via photon energy tuning).

2. Accurate V_O Quantification

HAXPES measurements reveal realistic VO concentrations (~1%), compared to inflated values (up to 13%) from sputtering-based XPS.

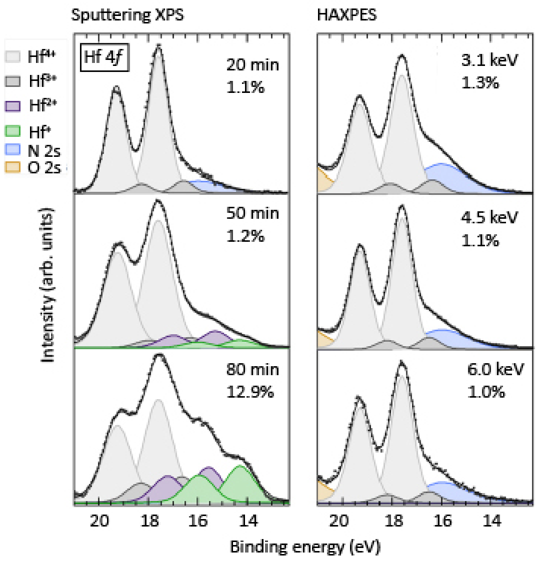

Fig 2: Side-by-side comparison of Hf 4f spectra from XPS (with sputtering) and HAXPES.

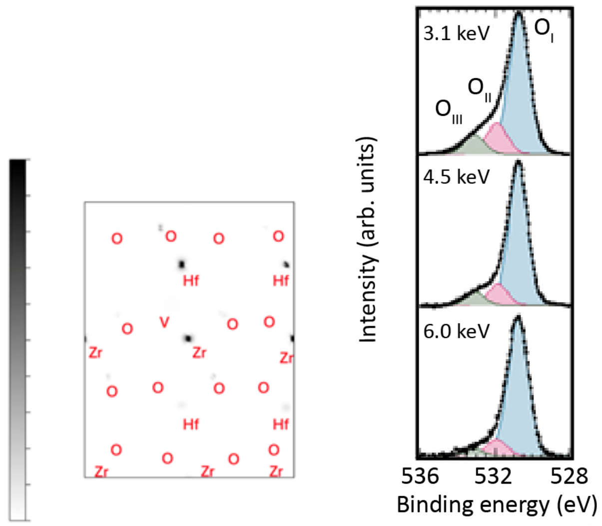

Fig 3: Charge density difference induced by a single VO, confirming no distinctive O 1s signature.

3. Correction of Misinterpretations

The study shows that high-binding-energy O 1s components often attributed to V_O are actually due to surface contamination. This finding corrects widespread errors in the literature.

Results & Insights

- HAXPES reveals a V_O gradient, highest near the TiN/HZO interface, consistent with oxygen scavenging effects.
- XPS falsely indicates higher V_O near the bottom electrode due to sputtering artifacts.
- Reliable interpretation of Hf 4f and O 1s spectra is critical for accurate defect characterization.

Conclusion

HAXPES provides a robust, non-destructive solution for V_O profiling in ferroelectric hafnia. This approach enables precise control of material properties for next-generation semiconductor technologies and avoids misinterpretations.

Recommended For

- Materials scientists developing hafnia-based ferroelectric devices
- Researchers optimizing non-volatile memory and logic architectures
- Scientists seeing accurate oxygen vacancy concentrations in ultra-thin films

DeepCore-X

Whereas this study relied on laboratory XPS and synchrotron HAXPES, the DeepCore-X system provides both capabilities in a single laboratory-based platform.

Reference:

Pérez Ramírez, L., Barrett, N., 2025. Journal of Applied Physics 138, 134106. https://doi.org/10.1063/5.0288354

Learn more at **www.scientaomicron.com/DeepCore-X**