Verification of Grain Boundaries in Annealed Thin ZrO_2 Films by Electrical AFM Technique

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Motivation

Investigation of high-k materials on a nanoscale using new advanced methods for both, physical and electrical characterization.

High-k materials have been identified as promising candidates to replace SiO_2 based dielectrics for the continued down scaling of semiconductor devices. In high-k films, amorphous and crystalline phases coexist depending on the thermal budget.

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Compared to SiO_2, different and more complex current conduction mechanisms occur at the nanometer scale.

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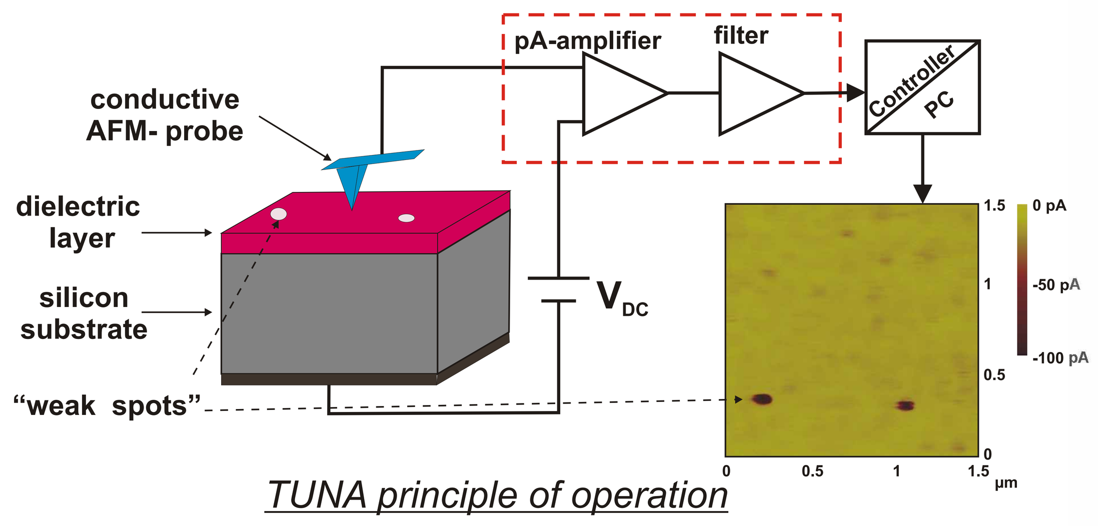
New advanced characterization methods with extremely high lateral resolution are needed.

Tunneling Atomic Force Microscopy (TUNA) is a very promising method which allows a simultaneous characterization of surface topography and electrical properties of thin dielectric films on a nanometer scale.

TUNA Principle



TUNA principle of operation

- Atomic Force Microscope (AFM) with a conductive probe operated in contact mode
- DC bias voltage (V) is applied between the tip and the sample
- Leakage (tunneling) current (I_{tuna}) through the dielectric layer is measured in dependence on V
- I_{tuna} range: 50 fA to 100 pA; noise level: 40 fA

Experimental

High-k sample preparation:

- Atomic Layer Deposition (ALD) of ZrO_2 films on native oxide (1 nm) on p-type silicon wafers
- Film thicknesses of about 5 nm
- Rapid Thermal Annealing (RTA) in Ar at different temperatures (450 °C and 600 °C) and for various times (30 s to 300 s)

Surface topography measurements:

- Veeco DI5000 SPM system operated in tapping mode to obtain optimum accuracy
- Standard Si probes (nominal tip radius of about 10 nm)

TUNA measurements:

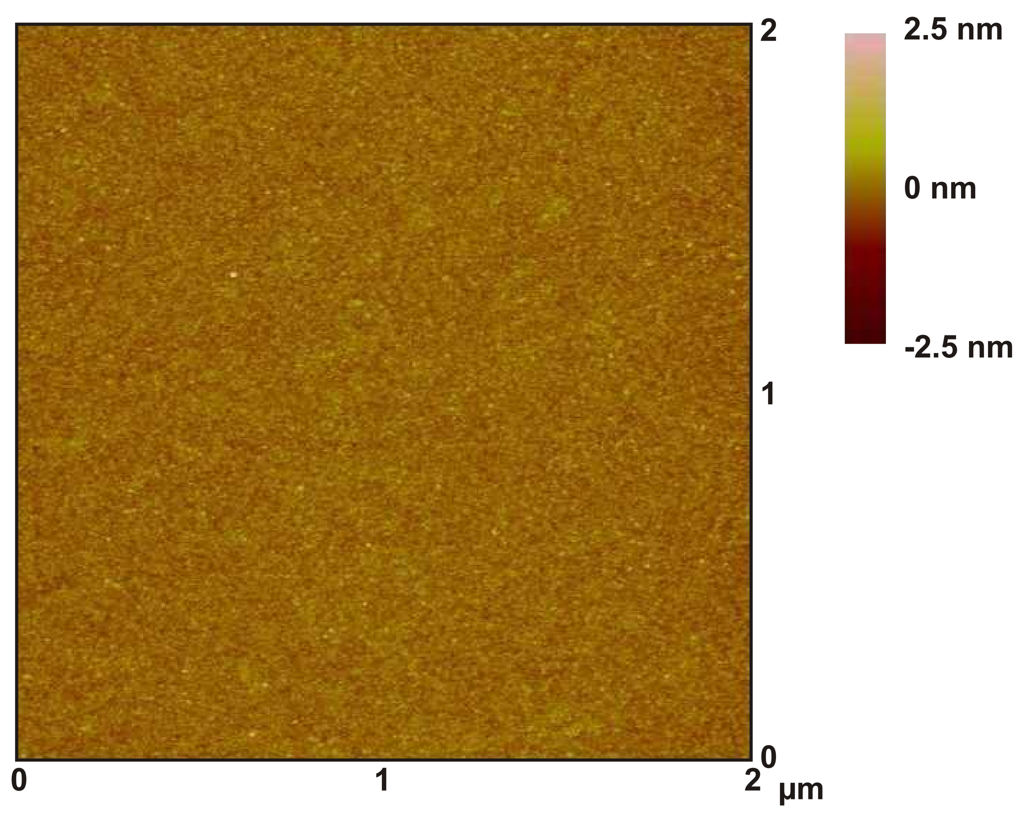
- Veeco DI5000 SPM system operated in contact mode
- Pt/Ir coated Si probes (nominal tip radius of about 20 nm)
- Negative DC bias between sample and tip (grounded) to avoid anodic oxidation of the surface

High resolution TEM (HR-TEM) analysis:

- Philips CM300 system
- Cross sectioned high-k samples

Results and Discussion

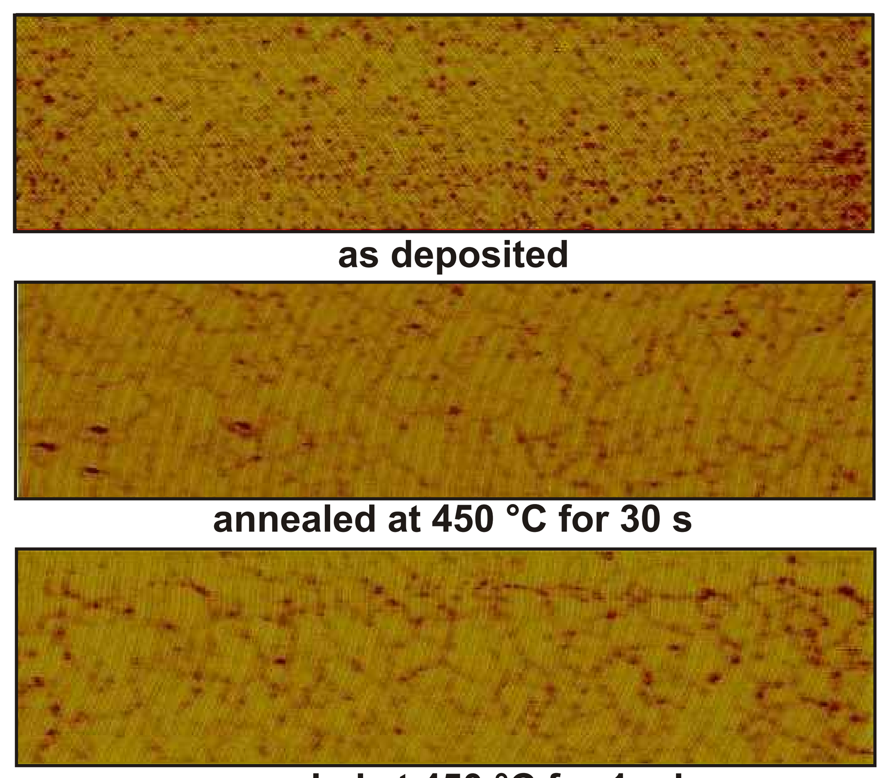
Surface topography:



Typical topography map obtained for the sample annealed at 450 °C for 30 s.

- All topography maps show smooth and homogenous surfaces (RMS of about 0.2 nm).
- No grains due to crystallization are visible. (even in films annealed at relatively high temperatures)

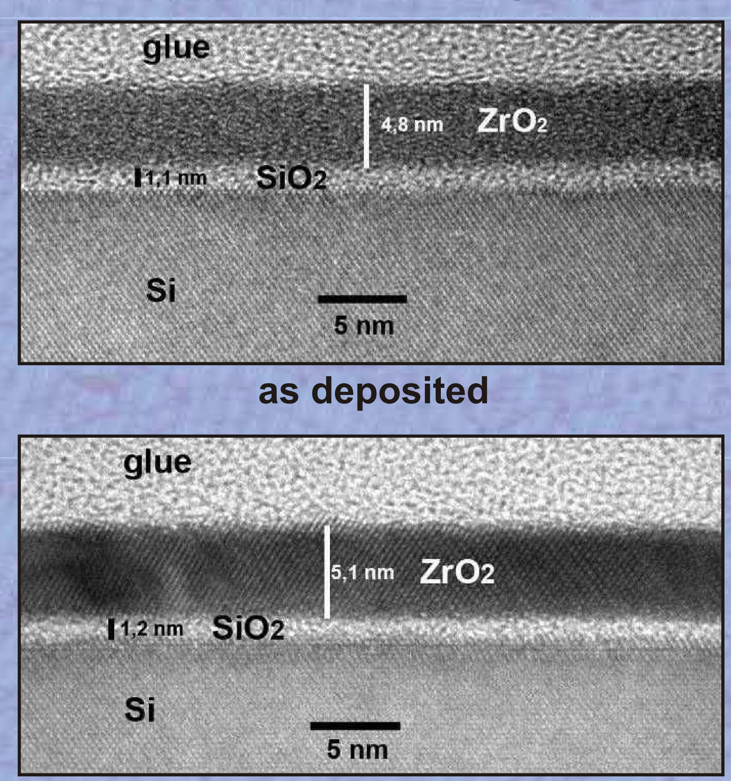
TUNA mapping:



TUNA current maps; scan size: 2 µm x 0.5 µm, applied bias voltage: -2.7 V, current scale: 0 pA (bright) to -5 pA (dark)

- Randomly distributed leakage current spots in the map of as deposited sample
- Ring shaped current structures in the maps of all annealed samples - crystallization after RTA

HR-TEM analysis:



HR-TEM pictures of ZrO_2 films

- ZrO_2 surface of all samples (before and after RTA) is very smooth.
- As deposited films have amorphous structure! All annealed films are nanocrystalline!

While the AFM topography measurements do not register the changes in film morphology (smooth surfaces), TUNA mapping visualizes the film crystallization as ring shaped current structures, which are due to grain boundaries.

Summary and Conclusions

- Electrical and surface properties of thin high-k ZrO_2 films as a result of post-deposition anneals were analyzed by AFM, TUNA and TEM.
- Surface topography measurements by AFM operated in tapping mode did not register changes in film morphology after RTA.
- TUNA mapping verifies the film crystallization after RTA (proved by TEM) as ring shaped current structures, which are due to grain boundaries.
- TUNA with its extreme current sensitivity is very well suited for the verification of grain boundaries in high-k layers at a nanometer scale.

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