

## TEM Study of The Structure of Thin Zirconia Films Obtained by Self-Assembled Monolayer Mediated Deposition

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The structure of as-grown and annealed sulphated zirconia films was studied by means of TEM and EELS. The zirconia films were prepared through wet-chemical deposition with subsequent annealing. Si(100) wafers were first functionalized with a self-assembled monolayer (SAM). The aqueous deposition medium was prepared by dissolving  $\text{Zr}(\text{SO}_4)_2 \cdot 4 \text{H}_2\text{O}$  in 0.4 N HCl to give a Zr concentration of 4 mmol/L; this solution was heated to 50°C. The SAM-functionalized Si-wafers were immersed into the deposition solution. The films were annealed at temperatures ranging from 150°C to 600°C for 2 hours.

Electron diffraction and high-resolution images show that the as-grown films are amorphous or short-range ordered. EDX confirms the presence of sulphur in the films. Annealing at temperatures below 525°C does not change the structure of the film, but its thickness decreases. Voids or cracks as a result of shrinking were not observed. Annealing at temperatures above 550°C causes the crystallization of films in tetragonal  $\text{ZrO}_2$ . In films annealed at 600°C, the tetragonal phase and a small amount of monoclinic phase of  $\text{ZrO}_2$  were found to coexist. Annealed films consist of grains from 10 to 50 nm in size. The sulphur signal was still visible in EDX spectra obtained from films annealed at 600°C.