

# Preparation of Sword-type Leaf-like Zirconium Oxide Films by Electrolysis

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## Abstract

Zirconium oxide ( $\text{ZrO}_x$ ) films were successfully prepared on a transparent conducting substrate (fluorine-doped tin oxide) by galvanostatic cathodic electrolysis of a solution containing ammonium hexafluorozirconate (AFZ) and ammonium nitrate at 323 K. The morphology of the film evidently changed based on the initial concentration of AFZ in the solutions. The film prepared at comparably higher AFZ concentration exhibited aggregates of sword-type leaf-like units tens of micrometers in size in the longitudinal direction, radially growing and dividing into secondary structure. On the other hand, normal planar films of a micrometer order were deposited at a lower concentration of the AFZ solution. The films were classified as zirconium oxyfluorides ( $\text{Zr-O-F}$ ) from the atomic concentration ratios of Zr, O, and F determined by X-ray fluorescent spectrometry analysis. Regardless of the initial AFZ concentrations, the crystallographic structures of the films were converted to monoclinic crystalline  $\text{ZrO}_2$  (baddeleyite) by the calcination at 773 K. The leaf shape of the

film obtained in the solution of higher AFZ concentration was maintained following the calcination process, indicating the development of a 3D ZrO<sub>2</sub> film. The present work evaluated the chemical character of the films and gave deeper insight into the growth mechanism of these remarkable materials.