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Formation of anodic zirconia nanotubes in fluorinated ethylene glycol electrolyte with K_2CO_3 addition

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Abstract

The influence of K_2CO_3 on the morphology of anodic zirconia (ZrO_2) nanotubes array were investigated by anodizing zirconium (Zr) foil at 60 V in fluorinated ethylene glycol (EG) electrolyte added to it varying amount of K_2CO_3: 0.5 vol.%, 1 vol.%, 2 vol.% and 3 vol.%. The adhesion of ZrO_2 on Zr is affected by the volume of K_2CO_3 added whereby at lower volume, i.e. 0.5 vol.% and 1 vol.%, poor adhesion of anodic film was observed leading to the formation of loose ZrO_2 flakes. At higher 2 vol.% and 3 vol.% addition the adhesion was improved. All anodic films are comprised of nanotubes with length increases when more K_2CO_3 was added in EG. Nanotubes grown in 3 vol.% K_2CO_3 are 9.4 µm long with 48.8 nm outer diameter and 9.1 nm wall thickness. Reducing the applied potential to 20 V resulted in compact oxide and at 40 V, nanotubes with smaller diameter of < 50 nm were produced. Crystallization of the ZrO_2 nanotubes was achieved by annealing at 400 °C. The crystalline ZrO_2 nanotubes (mostly in monoclinic and tetragonal phasess) grown in 3 vol.% K_2CO_3 exhibits the highest photocurrent density (0.12 mA/cm^{-2}) and rapid methyl orange (MO) degradation under ultra-violet (UV) radiation. This is attributed to the good adhesion of ZrO_2 on Zr, longer length of the tubes and perhaps from the effect of adsorbed carbonate ions on the surface of the oxide.