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## ACCEPTED MANUSCRIPT

# Formation of anodic zirconia nanotubes in fluorinated ethylene glycol electrolyte with K<sub>2</sub>CO<sub>3</sub> 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<sub>2</sub>CO<sub>3</sub>: 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<sub>2</sub> 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<sub>2</sub>CO<sub>3</sub> was added in EG. Nanotubes grown in 3 vol.% K<sub>2</sub>CO<sub>3</sub> 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<sub>2</sub> nanotubes was achieved by annealing at 400 °C. The crystalline ZrO<sub>2</sub> nanotubes (mostly in monoclinic and tetragonal phasess) grown in 3 vol.% K<sub>2</sub>CO<sub>3</sub> exhibits the highest photocurrent density (0.12 mA/cm<sup>-2</sup>) and rapid methyl orange (MO) degradation under ultra-violet (UV) radiation. This is attributed to the good adhesion of ZrO<sub>2</sub> on Zr, longer length of the tubes and perhaps from the effect of adsorbed carbonate ions on the surface of the oxide.

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