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Nanostructured alumina films by E-beam evaporation

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Abstract

The E-beam evaporation technique is utilised at room temperature to deposit 90, 120 and 150 nm thin alumina films on 75 μm thin titanium foils. As-grown films are annealed at 500, 700 and 800 °C in air. The phase analysis, morphology and electronic structure of the as-grown and annealed thin films are respectively investigated by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and X-ray photoelectron spectroscopy (XPS) techniques. The XRD results show that the as-grown thin films are amorphous. The annealed thin films show crystalline peaks corresponding to a mixture of different phases of alumina. The FESEM studies reveal tripod-like nanostructure and dense nanorods in the alumina thin films annealed at 700 and 800 °C, respectively. These results are explained on the basis of experimental evidences provided by the corresponding XPS studies.

Keywords: Alumina; Film; E-beam evaporation; Nanorod; Tripod

1. Introduction

The nanostructures particularly 1-dimensional (1D) nanostructures e.g., nanorods, nanotubes, nanowires, etc. assume very special significance today than ever before particularly due to the emerging application prospects in micro/nanodevices that can be useful in a wide variety of technologically important fields e.g., electronic, optoelectronic, magnetic, chemical, mechanical, biomedical, etc. [1,2]. In this context, alumina nanostructures have potential applications in both structural and high temperature resistant composites, catalysts, adsorbents, transparent structures, electrochemical processes and future nanodevices [1–5]. The alumina nanostructures in the form of rods [3,5–12], wires [1,2,4,5,11,13–17], tubes [1,15–18], platelets [19], leaves [20], flowers [20], belt [13], fibres [1,21,22], walls [5], pillars [16] and polygon [12] have been widely studied. These are summarised in Table 1. Mainly alumina nanopowder synthesised by various methods are reported [3,4,6–10,13,14,18–22]. Typically these methods include hydrothermal [4,6,7,10,18–20,22], solvothermal [21], carbothermal [14], sol–gel [3,8] and in-situ chemical vapour deposition (CVD) [13] techniques with or without the use of surfactant or template or base. Development of integrated alumina coating nanostructure on aluminium substrate by anodic oxidation [1,11,15] or subsequent chemical etching [5,16,17] is also reported. However, studies on nanostructured alumina films/coatings are scarce in the literature [2,12].

In the present work, through variations in post-deposition annealing temperature different nanostructured alumina films are deposited by the electron beam (E-beam) evaporation technique on titanium (Ti) substrate. The phase analysis, surface morphology and electronic structure of the deposited and post-annealed films are studied respectively by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and X-ray photoelectron spectroscopy (XPS) techniques.

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Table 1
Literature status of alumina nanostructures.

Form of alumina i.e. as film/powder	Phase	Processing details	Calcination/annealing temperature	Type of nanostructures	Dimensions (nm)	Remarks	References
Integrated coating on Al substrate	A	Anodic oxidation	–	Fibres, wires, tubes	Nanowire- *D*: 30 Nanotube- D_o: 220	–	[1]
Film on Si substrate	–	Thermal evaporation method: Al and alumina powder as precursors	Processing temperature- 1000 °C for 2 h	Wires	*L*: 2000–3000 *D*: 40	Catalyst free growth	[2]
Powder	γ	Sol–gel	500 °C for 4 h	Rods	*L*: 26–9 *D*:1–7	Soft template	[3]
Powder	B, γ	Hydrothermal route: $AlCl_3$ and NaOH as precursors	500 °C for 2 h	Wires	*L*: 100–200 *D*: 10–30	–	[4]
Integrated coating on Al substrate	A, γ, α+ γ	Anodic oxidation and subsequent chemical etching by NaOH	600–1000 °C	Wires (A), rods (γ), walls (α+γ)	Nanowire- *D*: 60 Nanorod- *D*:180 Nanowalls- *T*:50	–	[5]
Powder	γ	Hydrothermal route: Al $(NO_3)_3 \cdot 9H_2O$ as precursor	800 °C for 2 h	Rods	AR:2–4	Without templates	[6]
Powder	γ	Hydrothermal route: AIP as precursor	350–600 °C for 6 h	Rods	*L*: 500 *D*:100–300	Phenol formaldehyde as a template	[7]
Powder	B, γ+ δ+α	Sol–gel route: AIP as precursor	500–700 °C for 4 h	Rods	*L*: 17–18 *D*: 6–8	–	[8]
Powder	γ	Solvothermal route: $AlCl_3$ and NaOH as precursors	500 °C for 3 h	Rods	*L*: 170–320 *D*:15–25	Surfactant assisted	[9]
Powder	B, γ	Hydrothermal route: ATB as precursor	500 °C for 4 h	Rod	*L*: 200	Without added organic solvents	[10]
Integrated coating on Al substrate	–	Anodic oxidation	–	Wires, rods	Nanowires- *L*: 1000–2500 *D*: 35–80 Nanorods- *L*: 100	–	[11]
Film on SS304 substrate	A, α+δ+γ+θ	E-beam evaporation method	700 °C for 2 h	Rods, polygon (preferential growth)	*L*: 3000–7000 *D*: 28–570	Room temperature deposition	[12]
Powder	α	Integrated furnace assembly with CVD facility: alumina and Al as precursor	Processing temperature- 1350 °C for 1 hr (Belts), 1250 °C for 1 h (Wires)	Belts, wires	–	–	[13]
Powder	α	Carbothermal route: Al and graphite as precursors	Processing temperature- 1300 °C for 6 h	Wires, tubes	Nanotubes- D_i=20–25 D_o=40–50	–	[14]
Integrated coating on Al substrate	–	Anodic oxidation	–	Wires, tubes	Nanotubes: *L*: <1000 *D*: 50–60	–	[15]
Integrated coating on Al substrate	–	Anodic oxidation and subsequent chemical etching by NaOH	450 °C	Tubes (half), wires, pillars	Nanowire- *D*: 23 Nanopillars- *D*: 28	–	[16]
Integrated coating on Al substrate	–	Anodic oxidation and subsequent chemical etching by NaOH	420 °C	Wires, tubes	Nanowire- *L*: >1000 *D*: 50 Nanotube- *D*: 100	–	[17]
Powder	A (upto 700 °C), γ (800 °C)	Hydrothermal route: Al $(NO_3)_3 \cdot 9H_2O$ as precursor	500–800 °C for 5 h	Tubes	*L*: <200 *D*: 3–10	Anionic surfactant assisted	[18]
Powder	B, γ	Hydrothermal route: AIP as precursor	600 °C for 15 min	Platelets	D_m: 30–80 (B) D_m: 60–70 (γ)	–	[19]
Powder	B, γ	Hydrothermal route: $AlCl_3$ and NaOH as precursors	600 °C for 4 h	Leaves (B), flowers (γ)	Leaves-lateral size: 4500 × 9000 nm^2 *T*: 60–90	Surfactant assisted	[20]
Powder	B, γ	Solvothermal route: $AlCl_3 \cdot 6H_2O$ as precursor	500 °C for 3 h	Fibres	*L*: 2000 *D*: 330	Without any surfactant or template or base	[21]
Powder	γ	Hydrothermal: ATB as precursors	500 °C for 4 h	Fibres	–	Cationic surfactant assisted	[22]

A: amorphous, B: boehmite, AIP: aluminium isopropoxide, ATB: aluminum tri-sec-butoxide, *D*: diameter, D_m: mean diameter, D_o: outer diameter, D_i: inner diameter *L*: length, AR: aspect ratio (*L*/*D*), *T*: thickness, CVD: chemical vapor deposition.