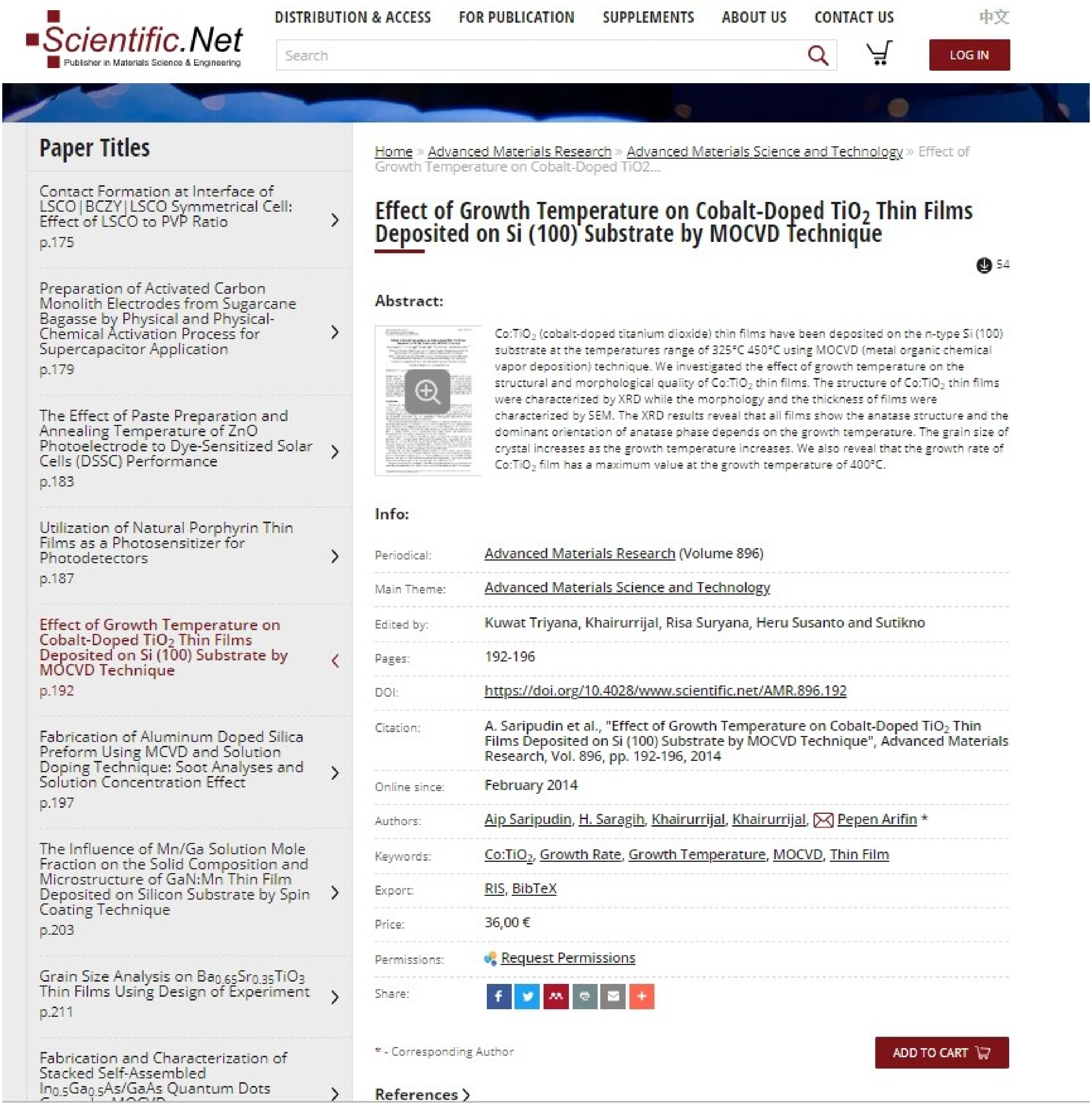

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Home » Advanced Materials Research » Advanced Materials Science and Technology » Effect of Growth Temperature on Cobalt-Doped TiO2...

Effect of Growth Temperature on Cobalt-Doped TiO_2 Thin Films Deposited on Si (100) Substrate by MOCVD Technique

54

Abstract:

Co:TiO_2 (cobalt-doped titanium dioxide) thin films have been deposited on the n-type Si (100) substrate at the temperatures range of 325°C 450°C using MOCVD (metal organic chemical vapor deposition) technique. We investigated the effect of growth temperature on the structural and morphological quality of Co:TiO_2 thin films. The structure of Co:TiO_2 thin films were characterized by XRD while the morphology and the thickness of films were characterized by SEM. The XRD results reveal that all films show the anatase structure and the dominant orientation of anatase phase depends on the growth temperature. The grain size of crystal increases as the growth temperature increases. We also reveal that the growth rate of Co:TiO_2 film has a maximum value at the growth temperature of 400°C.

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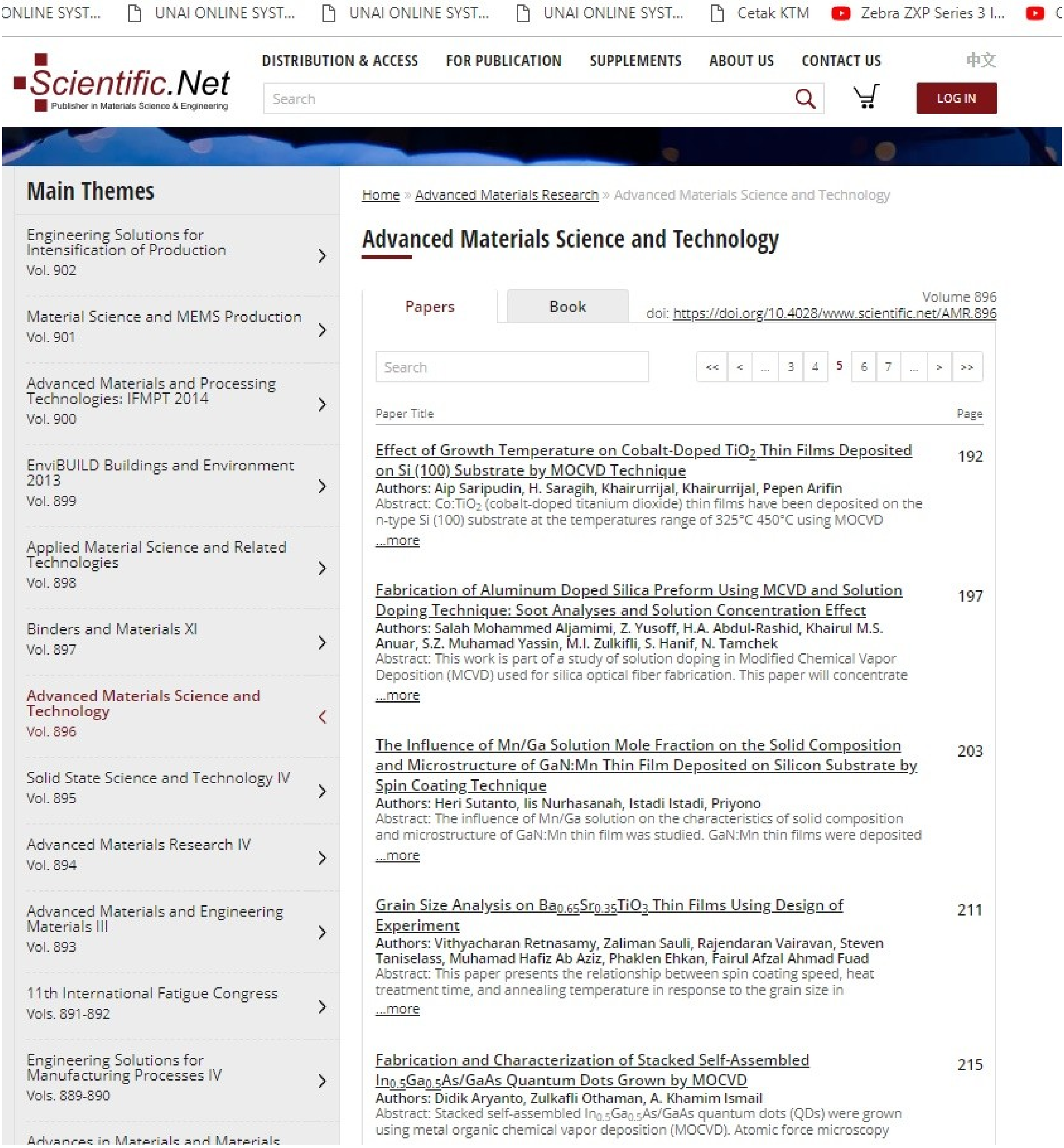

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Home » Advanced Materials Research » Advanced Materials Science and Technology

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Paper Title	Page
Effect of Growth Temperature on Cobalt-Doped TiO$_2$ Thin Films Deposited on Si (100) Substrate by MOCVD Technique Authors: Aip Saripudin, H. Saragih, Khairurrijal, Khairurrijal, Pepen Arifin Abstract: Co:TiO$_2$ (cobalt-doped titanium dioxide) thin films have been deposited on the n-type Si (100) substrate at the temperatures range of 325°C 450°C using MOCVD ...more	192
Fabrication of Aluminum Doped Silica Preform Using MCVD and Solution Doping Technique: Soot Analyses and Solution Concentration Effect Authors: Salah Mohammed Aljamimi, Z. Yusoff, H.A. Abdul-Rashid, Khairul M.S. Anuar, S.Z. Muhamad Yassin, M.I. Zulkifli, S. Hanif, N. Tamchek Abstract: This work is part of a study of solution doping in Modified Chemical Vapor Deposition (MCVD) used for silica optical fiber fabrication. This paper will concentrate ...more	197
The Influence of Mn/Ga Solution Mole Fraction on the Solid Composition and Microstructure of GaN:Mn Thin Film Deposited on Silicon Substrate by Spin Coating Technique Authors: Heri Sutanto, Iis Nurhasanah, Istadi Istadi, Priyono Abstract: The influence of Mn/Ga solution on the characteristics of solid composition and microstructure of GaN:Mn thin film was studied. GaN:Mn thin films were deposited ...more	203
Grain Size Analysis on Ba$_{0.65}$Sr$_{0.35}$TiO$_3$ Thin Films Using Design of Experiment Authors: Vithyacharan Retnasamy, Zaliman Sauli, Rajendaran Vairavan, Steven Taniselass, Muhamad Hafiz Ab Aziz, Phaklen Ehkan, Fairul Afzal Ahmad Fuad Abstract: This paper presents the relationship between spin coating speed, heat treatment time, and annealing temperature in response to the grain size in ...more	211
Fabrication and Characterization of Stacked Self-Assembled In$_{0.5}$Ga$_{0.5}$As/GaAs Quantum Dots Grown by MOCVD Authors: Didik Aryanto, Zulkafli Othaman, A. Khamim Ismail Abstract: Stacked self-assembled In$_{0.5}$Ga$_{0.5}$As/GaAs quantum dots (QDs) were grown using metal organic chemical vapor deposition (MOCVD). Atomic force microscopy	215

Advanced Materials Research Vol. 896 (2014) pp 192-196
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Effect of Growth Temperature on Cobalt-doped TiO_2 Thin Films Deposited on Si(100) Substrate by MOCVD Technique

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Keywords: Co:TiO_2, MOCVD, growth temperature, growth rate, thin films.

Abstract. Co:TiO_2 (cobalt-doped titanium dioxide) thin films have been deposited on the n-type Si(100) substrate at the temperatures range of 325°C – 450°C using MOCVD (metal organic chemical vapor deposition) technique. We investigated the effect of growth temperature on the structural and morphological quality of Co:TiO_2 thin films. The structure of Co:TiO_2 thin films were characterized by XRD while the morphology and the thickness of films were characterized by SEM. The XRD results reveal that all films show the anatase structure and the dominant orientation of anatase phase depends on the growth temperature. The grain size of crystal increases as the growth temperature increases. We also revealed that the growth rate of Co:TiO_2 film has a maximum value at the growth temperature of 400°C.

Introduction

TiO_2 has many interesting properties, such as high refractive index, high dielectric constant, and chemically stable. TiO_2 is commonly used as a pigment, absorbent, catalyst supports, filters, coatings, photoconductor, and the dielectric material [1]. Today, TiO_2 is also known as a semiconductor with photocatalytic activity and has a high potential for applications such as environmental purification, decomposition of carbonic acid gas, and the generation of hydrogen gas [2]. In addition, cobalt-doped TiO_2 has demonstrated a ferromagnetic semiconductor at room temperature to allow for use as spintronics devices [3].

TiO_2 has three different types of crystal structure, namely rutile, anatase, and brookite. Anatase is anisotropic, tetragonal (a = b = 3,785 Å, c = 9,514 Å) with a bandgap of 3.2 eV. Anatase structure can be produced at low temperatures around 350°C. Rutile is also tetragonal (a = b = 4.593 Å, c = 2.959 Å) with a bandgap of 3.0 eV [4, 9]. Rutile structure can be produced at the temperature of 400°C or higher. Brookite structure can be produced at high pressure and temperature but thermodynamically very unstable compared to the two other structures.

TiO_2 thin films can be obtained by different growth techniques, for example, atomic layer deposition, MBE (molecular beam epitaxy) [3], PLD (pulsed laser deposition), DC and RF Sputtering [5 – 7], and MOCVD [1, 8, 10]. Beyond these technicques, MOCVD technique has many advantages such as epitaxial growth possibility, selective deposition, and growth parameter controlling simplicity. Therefore, MOCVD technique is known as a powerfull technique and suitable for stoichiometric and microstructural thin film deposition [10].

Undoped TiO_2 and cobalt-doped TiO_2 can be deposited on $LaAlO_3$, $SrTiO_2$, and Si substrates. Cobalt-doped TiO_2 deposited on $LaAlO_2$ and $SrTiO_2$ substrates have been extensively investigated due to a small lattice mismatch [11]. But, silicon substrates are low cost, available in large diameters, and have well characterized electrical and thermal properties [12] so many reseachers used silicon substrate to deposite TiO_2 or cobalt-doped TiO_2 thin film [1,2,4-11].

This paper focuses on the investigation of the effect of growth temperature on the structural and morphological quality of Co:TiO_2 thin films deposited on Si(100) substrate by MOCVD technique.

Experimental Details

Co:TiO$_2$ thin films were grown by MOCVD technique. The films were deposited on n-type Si(100) substrate. We used TTIP (titanium (IV) isopropoxide) as TiO$_2$ precursor, Co(TMHD)$_3$ (tris (2,2,6,6-tetramethyl-3,5-heptanedionate cobalt (III) as Co precursor, and THF (tetrahydrofuran). The type of MOCVD reactor is cold-wall cylindrical vertical MOCVD reactor.

In this experiment, we mixed 20 mL TTIP, 900 mg Co(TMHD)$_3$, and 20 mL THF in a bubbler tube. The bubbler tube was then heated at a constant temperature of 100^{o}C to evaporize the precursor. The precursor vapor with pressure of 0,3 kg/cm^2 were transported by the argon gas to the reactor chamber. The flowrate of argon gas was 70 sccm. In the reactor chamber, Si(100) substrate was placed on the heater (molybdenum disc) using silver paste. The growth temperatures were set at 325^{o}C, 350^{o}C, 375^{o}C, 400^{o}C, and 450^{o}C, respectively. The reactor chamber pressure was 2×10^{-3} torr and the growth time was 120 minutes.

The crystalline quality of Co:TiO$_2$ thin films was characterized by XRD (X-Ray Diffraction) using Cu Kα radiation (λ = 1,54060 Å). The grain size of the films was calculated using the Scherrer equation [7]:

$$D = \frac{0.89\lambda}{B\cos\theta} \tag{1}$$

where D is the grain size, λ is the wavelength, B is FWHM (full-width at half-maximum) of diffraction peak, and θ is diffraction angle.

The surface and cross-section morphology of film was characterized by SEM (scanning electron microscope).

Results and Discussion

The XRD patterns of Co:TiO$_2$ thin films are shown in Fig. 1. According to these results, the films have an anatase phase with different dominant orientation for each growth temperature. At the growth temperature of 325^{o}C and 350^{o}C, the dominant of crystal orientation is A(211), where "A" stands for anatase, and its relative intensity decreases as the growth temperature increases. At the growth temperature of 375^{o}C and 400^{o}C, the dominant of crystal orientation is A(112) and its relative intensity decreases as the growth temperature increases. At the growth temperature of 450^{o}C, the dominant of crystal orentations are A(112) and A(211). Based on these results, it is revealed that the growth temperature play an important role to the crystal orientation.

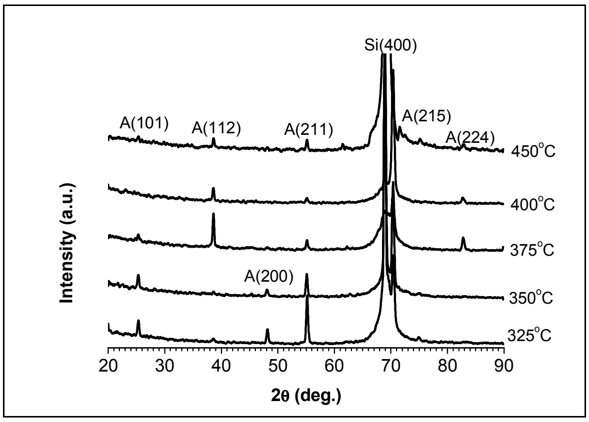

Figure 1. XRD patterns of Co:TiO$_2$ thin films grown at different growth temperatures.

The grain sizes of crystals were calculated using (1). The grain size dependence to the growth temperature is shown in Fig. 2. The grain size of crystal is about 70 nm at 325^{o}C and 75 nm at 350^{o}C. The grain size of crystal decreases to 74 nm at 375^{o}C, but increases as the growth temperature increases. Therefore, the grain size of crystal tends to increases as the growth temperature increases.

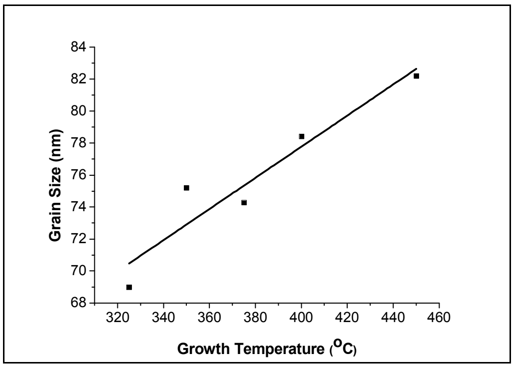

Figure 2. Grain size of crystal of the films grown at different growth temperatures.

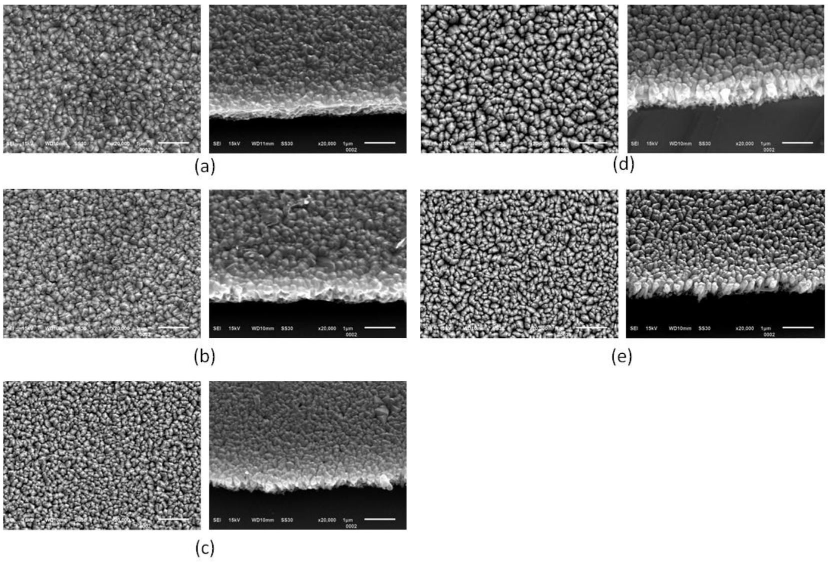

Figure 3. SEM images of Co:TiO_2 thin films grown at temperature of (a) 325°C, (b) 350°C, (c) 375°C, (d) 400°C, and (e) 450°C.

The increasing of the grain size of crystal could be roughly seen from the SEM images as shown in Fig. 3. On the left-hand size of Fig. 3, the surface morphologies of films are shown. Based on the SEM images, it is shown that the grain size of crystal increases between the growth temperature of 325°C and 350°C, then decreases at the growth temperatures between 350°C and 375°C, and increases again for the growth temperature between 375°C and 450°C. We can also see that the surfaces of films are generally smooth and homogeneous.

The cross-sectional of SEM image of the film is shown on the right-hand side of Fig. 3. Based on the SEM images, we found that films structure mainly composed of columnar-shaped grains. The columnar-shaped of crystal is perpendicular to the direction of film deposition. As the growth temperature increases, the grain size of crystal increases and the width of columnar-shaped of crystal also increases. The increasing of the crystal grain size as the increasing of growth temperature is supposed by the increasing of decomposition rate of the precursor at the substrate surface [9].

The dependence of the growth rate with the growth temperature is shown in Fig. 4. The growth rate of film has a maximum value at the growth temperature of 400°C. For the growth temperature less than 400°C, the growth rate increases as the temperature increases. This result indicates that the film deposition is controlled by diffusion. Furthermore, for the growth temperature higher than 400°C, the growth rate decreases as the temperature increases. It could be associated with some desorption, precursor decomposition at gas phase, or pre-reaction with residual gases. In the other word, the films deposited at high temperatures are denser than the films deposited at low temperatures. The same tendency of the growth rate has been reported for other MOCVD systems [9].

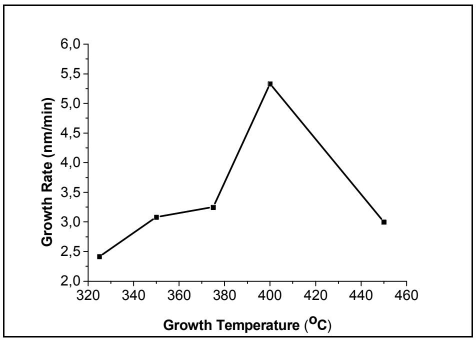

Figure 4. Dependence of the growth rate with the growth temperature.

Conclussions

We have deposited Co:TiO_2 thin films on Si(100) substrate by MOCVD technique at growth temperatures in the range of 325°C - 450°C. The structural and morphological qualities of films have been investigated. It is revealed that the films have anatase phase with the dominant of crystal orientation depends on the growth temperature. The grain size of crystal increases as the growth temperature increases. The growth rate of film increases as the growth temperature increases for the growth temperature in the range of 325°C and 400°C. On the other hand, the growth rate of film decreases as the growth temperature increases for the growth temperature in the range of 400°C - 450°C. The growth rate of Co:TiO_2 film has the maximum value at the growth temperature of 400°C.

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