Thin Solid Films 547 (2013) 198–201

Contents lists available at ScienceDirect

Thin Solid Films

journal homepage: www.elsevier.com/locate/tsf

# Optical and electrical properties of $V_2O_5$ nanorod films grown using an electron beam

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ARTICLE INFO

Available online 3 April 2013

*Keywords:*
$V_2O_5$
Nanorod
Electron beam
Photoluminescence
Metal-to-insulator transition

ABSTRACT

An electron beam irradiation method is suggested for growing $V_2O_5$ nanorods. $V_2O_5$ nanorods with an orthorhombic structure (α-$V_2O_5$) were well grown by electron beam irradiation and the nanorod growth was greatly enhanced by inserting a buffer layer. The X-ray diffraction pattern of $V_2O_5$ nanorods grown at a dose rate of 800 kGy considerably changed due to an increase in structural inhomogeneity formed by vanadyl–oxygen vacancies. The transition temperature increased due to the increasing surface resistance and was steeply enhanced by the increase in crystallinity with growing nanorods. Two photoluminescence peaks were observed at 530 nm and 710 nm for the $V_2O_5$ nanorods grown at a dose rate of 800 kGy due to oxygen vacancies and a band edge transition.



## 1. Introduction

Vanadium pentoxide ($V_2O_5$) is the most stable of vanadium oxides and exhibits highly anisotropic electric and optical properties due to its orthorhombic structure [1,2]. $V_2O_5$ film has a transition temperature ($T_c$) of 280 °C [3] and its transition accompanies great variation in electrical and optical properties near $T_c$ [3–5]. Owing to its outstanding chemical, electronic, and thermal properties, nanostructured $V_2O_5$, such as nanowires, nanorods, and nanocrystals, are promising for electronic and optical devices such as gas sensors, optical–electrical switches, and electrochromic devices [6–9].

There are several methods, including chemical and physical processes, for growing $V_2O_5$ nanostructures [10–12]. For example, Wang et al. reported a thermal oxidation method to form $V_2O_5$ nanorods [10]. $V_2O_5$ nanorods were grown by heating a $V_2O_3$ thin film deposited on silicon substrates in air at 400 °C. Ajayan et al. reported the synthesis of $V_2O_5$ nanofibers by heating a mixture of carbon nanotubes and $V_2O_5$ powder in air at 750 °C [11], and Takahashi et al. reported single crystal $V_2O_5$ nanorod arrays synthesized inside polycarbonate templates by sol electrophoretic deposition [12]. In this study, an electron beam irradiation method is proposed for growing $V_2O_5$ nanostructures. The method is shorter than other methods and does not require additional processes such as post-annealing or a chemical treatment.

In the present study, an electron beam irradiation method is proposed to grow $V_2O_5$ nanorods. $V_2O_5$ films prepared by a radio frequency (RF) sputtering method at room temperature were irradiated by an electron beam in air. The nanorod growth was enhanced by inserting a buffer layer between the $V_2O_5$ film and substrate. The dependences of the structural, electrical, and photoluminescence (PL) properties of $V_2O_5$ nanorods were also investigated.

## 2. Experiments

Fig. 1(a) and (b) shows a schematic diagram for the cross-section of the amorphous $V_2O_5$ film that was grown on an $Al_2O_3$(0001) substrate and the amorphous $V_2O_5$ film with a crystalline $V_2O_5$ as buffer layer was grown on the substrate, respectively. The film thickness was measured by a spectroscopic ellipsometer (SE; Jobin-Yvon, Uvisel UV/NIR) with a photon energy range of 0.75–4.0 eV at an incident angle of 70° and the thickness of the amorphous and crystalline buffer layers were 200 and 140 nm, respectively. SE data for the $V_2O_5$ film were analyzed via an optical model based on the Bruggeman effective medium approximation. The double new amorphous formula combined with two oscillators.

$V_2O_5$ films were prepared using an RF sputtering system with a $V_2O_5$ (99.99%) disk target with a 10 cm diameter and sputtering was performed at an RF power of 200 W. The amorphous layer was grown at room temperature for 200 min and the buffer layer was deposited at a substrate temperature of 500 °C for 150 min. The distance between the target and substrate was 10 cm. The sputtering gas and reactive gas were Ar and $O_2$, respectively, each with 99.999% purity. The gases were injected into the chamber with an $O_2$ partial pressure of 10% and a total flow rate of 30 sccm. The base pressure of the chamber was less than $6.67 \times 10^{-3}$ Pa and the working pressure during sputtering was approximately $1.33 \times 10^{-1}$ Pa. The sputtering rates for the amorphous and crystalline films were approximately 1.0 and 0.93 nm per minute, respectively. Electron beam irradiation was used to grow the $V_2O_5$ nanorods. Two $V_2O_5$ films were irradiated by an electron beam with an energy of 0.7 MeV using an electron beam

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0040-6090/$ – see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.tsf.2013.03.060

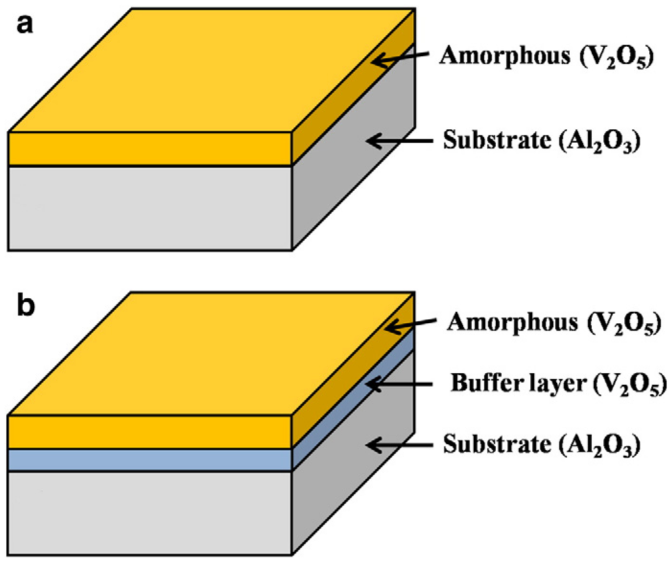


**Fig. 1.** Schematic diagram for the cross-section of (a) amorphous $V_2O_5$ film grown on an $Al_2O_3$ (0001) substrate and (b) amorphous $V_2O_5$ film with a crystalline $V_2O_5$ as buffer layer.

accelerator (BINP, ELV-0.5). The dose rates of the electron beam were 0, 300, 600, 800, and 1000 kGy.

The microstructures of the $V_2O_5$ films were investigated using a scanning electron microscope (SEM; JEOL, JSM6335F) at an operating voltage of 10 kV and an X-ray diffractometer (XRD; Rigaku, D/MAX-Rc) with Cu Kα radiation. The XRD measurements were performed using a 2$\theta$ method at an interval of 0.02° and a scanning speed of 4° per minute.

The temperature dependence of the resistance of the $V_2O_5$ films with dose rates were measured by a 2 probe method using an electrometer (Keithley, 2400) from room temperature to 600 °C. The temperature dependence of the PL spectra was measured using a Raman/PL spectrometer (Horiba Jobin-Yvon, LabRAM HR) under the excitation of a He–Cd laser with a 325 nm wavelength at temperature range from 10 to 300 K.

## 3. Results and discussion

Fig. 2 shows surface morphologies of the $V_2O_5$ films without the buffer layer with a dose rate of electron beam irradiation. As the dose rate increases, the films showed considerable variation in their surface morphology. In particular, the film with a dose rate of 800 kGy showed growth of nanorods. The length and diameter of the nanorods were 350 nm and 67.6 nm, respectively. The inset shows the cross-sectional image of nanorods grown at a dose rate of 800 kGy.

At a dose rate of 1000 kGy, the film showed planar growth of nanorods due to over irradiation by the electron beam via a thermal effect. The electrons accelerated by an electron beam accelerator strike the film and continuously transfer energy to the film. Consequently, the collision energy generates heat in the film. The crystallization temperature of $V_2O_5$ film is approximately 500 °C [3,13]. Thus, the film temperature with a dose rate of 800 kGy is greater than 500 °C.

The surface morphology of $V_2O_5$ films with an inserted buffer layer with respect to the dose rate of the electron beam irradiation are shown in Fig. 3. Remarkable rod-like crystallites were observed at a dose rate of 300 kGy and started to transform into nanorods with increasing dose rate of the electron beam irradiation. At a dose rate of 800 kGy, the film showed greatly enhanced growth of nanorods with a maximum length of 3794 nm and a diameter of 198 nm, indicating that the nanorod growth is affected by the crystalline structure of the substrate. Generally, the initial growth of a film depends on the crystalline structure of the substrate, and the crystallinity of a film is greatly affected by the substrate. In our study, crystalline $V_2O_5$ used as buffer layer had an orthorhombic ($\alpha$-$V_2O_5$) structure and the crystalline structure of the $Al_2O_3$ substrate is hexagonal. Thus, the buffer layer is responsible for the enhanced growth of nanorods at a dose rate of 800 kGy.

Fig. 4 shows the XRD pattern of the $V_2O_5$ nanorods grown at a dose rate of 800 kGy with a buffer layer. The pattern of the nanorods without the buffer layer shows typical peaks of $\alpha$-$V_2O_5$ [2,3,14–16]. The peaks at $2\theta = 20.36°$ and $2\theta = 20.66°$ correspond to reflections in the (001)

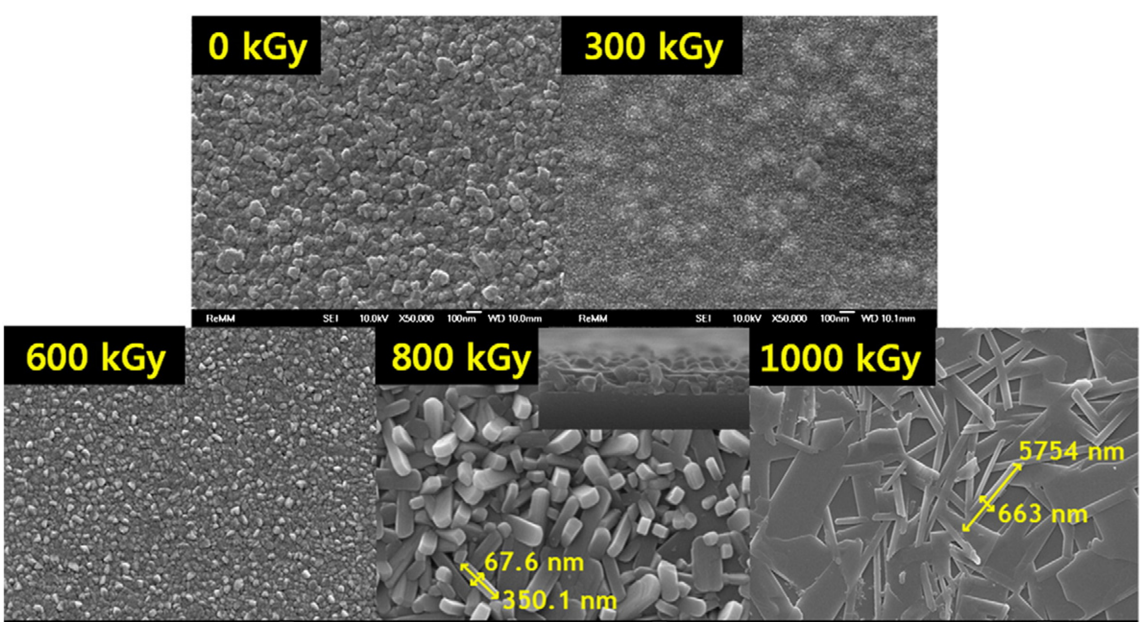


**Fig. 2.** Surface morphologies of the $V_2O_5$ films without the buffer layer with electron beam dose rate. The inset shows the cross-sectional image of nanorods grown at a dose rate of 800 kGy.