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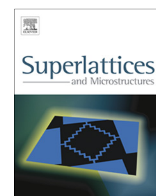

The influence of substrate temperature on the optical and micro structural properties of cerium oxide thin films deposited by RF sputtering

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ABSTRACT

In this investigation, CeO_2 thin films were deposited on glass substrates with different substrate temperatures by using RF Magnetron sputtering technique. The deposited films were characterized by X-ray diffraction (XRD), UV–visible spectroscopy, room temperature photoluminescence spectroscopy, micro-Raman and X-ray photoelectron spectroscopy. The thicknesses of the film were determined by using profilometer and it was varied from 0.70 μm to 0.87 μm. From the XRD results, it is evident that the film deposited at room temperature (RT) is amorphous in nature. The remarkable change in character from amorphous to crystalline is observed by increasing the substrate temperature from RT to 300 °C, and the crystallites were found to be in cubic phase with preferred orientation along (220). Calculated crystallite sizes were in the range of ~8.6 nm. Optical characteristics were studied as a function of change in substrate temperature and thickness in air. Overall transmittance percentages of the films (85–60%) were found to decrease with increase in substrate temperatures. The results show that the energy band gap was found to be decreased (3.95–3.75 eV) with the increase of substrate temperature as well as thickness of the film. Optical constants like refractive index (n), extinction coefficient (k), optical conductivity (σ), real and imaginary part of dielectric constant (ε_1 & ε_2), volume energy loss function (VELF) and surface energy loss function (SELF) were evaluated by using UV spectra. It is found from PL spectra that the decreased defects for the films prepared at a higher substrate temperature. A strong near band edge emission and weak green emission were observed at ~360 and ~519 nm respectively. The micro-Raman results show the characteristic peak of CeO_2 F_{2g} at ~465 cm^{-1}. It is found from the spectrum that the peak intensity of the films increased with increase in substrate temperature. XPS analysis confirms the highly non-stoichiometric nature of the films with dominant occurrence of Ce^{4+} (CeO_2) and subsidiary occurrence of Ce^{3+} (Ce_2O_3).

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1. Introduction

In recent years, cerium (IV) oxide (CeO_2, also known as ceria) thin films have received much attention due to their many interesting characteristics, such as unique UV absorption ability, high stability at high temperature, high hardness and reactivity. CeO_2 is an excellent and desired buffer layer material for the exponential growth in many applications such as high

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temperature superconducting and semiconductor thin films due to its small lattice mismatch, good stability and similar thermal coefficient [1,2]. It exhibits two structures mainly cubic fluorite cerium dioxide or ceria (due to tetravalent Ce) and a hexagonal sesquioxide Ce_2O_3 (due to trivalent Ce). Cerium oxide is a notable functional material with an extraordinary capacity to store and release oxygen without losing its fluorite cubic structure [3]. Cerium oxide based materials have been extensively used in wide variety of applications such as solid oxide fuel cells (SOFCs) [4], catalysis [5], luminescent materials [6], gas sensors [7,8], polishing materials [9], ferromagnetic oxides [10] and so on. CeO_2 is also suitable for various optical, electro-optical and optoelectronic devices because it is transparent oxide in visible and near-IR spectral regions [11,12].

Several methods have been proposed to prepare CeO_2 thin films such as molecular beam epitaxy (MBE) [13], chemical vapor deposition (CVD) [14], pulsed laser deposition [15], dip coating [16], spray pyrolysis [17], spin coating [18] and radio frequency (RF) magnetron sputtering [19]. Among these techniques RF sputtering method has many attractive advantages which include better film growth control, repeatability, low temperature deposition, large scale stability and uniform film properties. Thus, RF Magnetron sputtering was chosen for the deposition of CeO_2 thin films. The properties of sputtered CeO_2 thin films are known to depend not only on deposition parameters such as RF power, pressure, substrate temperature and ambient atmosphere [20], but also on post deposition processes, such as thermal annealing.

In this study, the effect of substrate temperature on micro structural, optical properties and chemical composition of the deposited CeO_2 thin films is reported. The results of crystalline structure, grain size, and the optical band gap of CeO_2 thin films with respect to the substrate temperature treatment have been investigated.

2. Experiment details

2.1. Film deposition

CeO_2 thin films were deposited by RF-sputtering (HINDHIVAC; Planar Magnetron RF/DC Sputtering Unit Model-12″ MSPT) onto glass substrates. The substrates were chemically cleaned by using acids and finally acetone. The cleaned substrates were then loaded into the deposition chamber for the deposition of CeO_2 thin films. 5 cm diameter and 5 mm thickness of target was prepared by using commercially available CeO_2 powder (Sigma Aldrich). This powder was compacted to pellet with the help of stainless steel pelletizer and hydraulic press and the target was sintered at 1000 °C for 12 h by using box furnace (Model: VB Ceramic Consultant, India.). The prepared target was loaded into the chamber and the chamber was evacuated at a base pressure of $\sim 5 \times 10^{-6}$ mbar. The coating parameters like target to substrate distance, working pressure, RF power and coating time for the deposition of CeO_2 were set as 6 cm, 2×10^{-3} mbar, 150 W and 30 min respectively. Substrate temperature was varied from room temperature to 300 °C at the time of film coating.

2.2. Characterization techniques

The thickness of the deposited film was measured by Stylus profilometer (Mitutoyo, SJ-301). The structural property of CeO_2 thin film was studied by X-ray diffraction (XRD) by using Cu-Kα ($k = 0.154$ nm) radiation source (X' pert Pro PANalytical) over a 2θ scan range of 10–70. The effect of substrate temperature on the optical properties of CeO_2 films were studied by using UV–Vis–NIR spectrophotometer (JASCO; V-670) in the wavelength range of 300–1100 nm. Room temperature photoluminescence (RTPL) study was performed by using Varian Cary Eclipse fluorescence spectrophotometer. Raman spectra of the films were recorded using a micro Raman spectrometer (Acton SpectraPro 2500i, Princeton Instruments, Acton Optics & Coatings). The XPS measurements were performed with XPS instrument (Carl Zeiss) equipped with Ultra 55 FESEM with EDS and all the spectra were recorded under ultra high vacuum with Al Kα excitation at 250 W.

3. Results and discussion

3.1. Structural studies

The thickness of the film was increased from 0.70 μm to 0.87 μm by varying the substrate temperature. XRD patterns of the sputtered CeO_2 thin films grown at different substrate temperatures are shown in Fig. 1. No diffraction peak is observed in the films grown at RT of the substrate which indicates the amorphous nature of the film. When the substrate temperature increases to 100 °C, two diffraction peaks corresponding to (1 1 1) and (2 2 0) crystallographic plane with cubic fluorite structure (space group Fm3m (2 2 5)) of the CeO_2 phase is observed with weak intensities, and their intensities increase as the substrate temperature further increases to 200 °C and 300 °C. However, the FWHM for (2 2 0) diffraction peak does not show any remarkable change in for the films.

The indexed peaks are well matched with the standard JCPDS data card No. 34-0394. By applying Scherrer's equation to the 2θ and FWHM of the (2 2 0) peaks [21], it is found that both crystallite size and thickness of the films got increased with increase in substrate temperature. The crystallite size (D), dislocation density (δ) and strain (ε) are calculated by using the relations:

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