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Materials Today: Proceedings 4 (2017) 6015–6021

materialstoday: PROCEEDINGS

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STEMa2016

The influence of post annealing on the structure and optical properties of vanadium oxide thin films

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Abstract

Vanadium and vanadium oxide films were deposited onto glass and silicon substrates at room temperature by DC magnetron sputtering and reactive DC magnetron sputtering, respectively. The effect of post annealing in air atmosphere were explored. The as-deposited vanadium and vanadium oxide films were annealed under air atmosphere at different temperatures (100-500°C) and annealing times (6, 12 hours). The structural properties of the films were studied by X-ray diffraction and scanning electron microscopy. The optical properties of the films were studied by UV-Vis spectrophotometer. The results showed that the annealed vanadium films have a formation of polycrystalline V_2O_5 structure while the optical transmittance is low due to the imperfect thermal oxidation. For the annealed vanadium oxide films, the vanadium oxide with the phase of V_2O_5 is also presented while the transmittance decreased when the annealing temperature is increased. This is due to the light loss by the scattering at the rough surface. A comparison of the results with the annealed vanadium and annealed vanadium oxide films will be discussed. Moreover, the effect of the post annealing temperature and the annealing time on the structural and optical properties will also be discussed.

Selection and Peer-review under responsibility of International Conference on Science and Technology of the Emerging Materials.

Keywords: Vanadium oxide; Sputtering; XRD; Annealing

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1. Introduction

Over the years, vanadium oxide thin films has been investigated and published by various researchers. Due to their excellent properties and there are many phases in vanadium oxide, at present vanadium oxide is used in wide range of industrial applications. For example, V_2O_5 is used as catalyst in chemical reactions and electrochromism [1] VO_2 is used as smart window [2] and optical switching device [3]. V_2O_3 may be used to fabricate a low-noise microbolometer [4] etc. Thin films of vanadium oxide can be deposited using various techniques including magnetron sputtering [5-9], which generally uses the vanadium metal or ceramic of vanadium oxide as a sputtering target. For vanadium metal target, during the deposition, an oxygen gas was fed and mixed with a working gas (e.g. Argon). Oxygen gas in the discharge will react with the sputtered vanadium atoms and form the desired vanadium oxide compound at the substrate. This technique is referred as reactive magnetron sputtering [10].

The deposition conditions such as oxygen flow rate, substrate temperature or post annealing for depositing vanadium oxide films have been extensively investigated [5-7]. The influence of the deposition conditions on the structural and optical properties of vanadium oxide films have also been reported [5-7]. Although many research works have focused on the effect of substrate temperature or post annealing temperature on the structure and optical properties. Nothing has been reported about a comparison of the results between the annealed vanadium and vanadium oxide thin films.

The aim of this present study is to compare the deposition processes, annealed vanadium films process and annealed vanadium oxide films process, for synthesis of crystalline vanadium oxide films with high optical transmittance. The effect of post annealing temperature on the structural and optical properties of the vanadium and vanadium oxide films were investigated. Moreover, the effect of the annealing time on the films properties is also presented.

2. Experimental procedure

This work was carried out in home-made DC magnetron sputtering system. The vanadium (V) (a purity of 99.7%) sputtering target with diameter of 75 mm and thickness of 6 mm is used. The substrates are silicon wafers (100) and microscopy glass slides. They were ultrasonically cleaned in acetone for 10 min., cleaned in methanol for 10 min. and dry with nitrogen gas. The grounded substrates were placed facing the target at the distance is approximately 85 mm. The vacuum chamber was evacuated down to a base pressure below 5×10^{-3} Pa using a diffusion pump assisted by a rotary pump. Argon gas was introduced into the chamber with the flow rate corresponding to a constant pressure 0.4 Pa which set as a sputtering pressure. A MDX 1K direct current (DC) power supply (Advanced Energy) operated in a constant power mode was used to deposit the vanadium metal thin films at sputtering power of 200 W for 15 min. In order to deposit the vanadium oxide thin films, the oxygen reactive gas was fed into the chamber corresponding to the change of the discharge voltage reach to the compound mode as described in the previous work [7]. For reactive sputtering case, the sputtering power was 200 W and the deposition time was 60 min. Before the deposition, the metal vanadium target was pre-sputtered by argon gas at the power of 200 W for 10 min to remove the compound layer and contamination coated on the target surface. It should be note that all coating films in this study were deposited without intentional heating. The vanadium metal films and the vanadium oxide films were annealed at the temperature during 100 to 500°C under air atmosphere for 6 hrs and 12 hrs. For the previous work [7], the annealed vanadium oxide films showed the higher crystallinity as compared to the annealed vanadium films at the same annealing temperature and time. This suggests that the case of compound films used lower thermal energy to improve the films crystallinity. However, the thermal energy depends on both the temperature and time. Therefore, for the case of the annealing time of 12 hrs, the temperature is desired to vary in smaller step as compared to the 6 hrs. The crystal structure of the films was characterized using RIGAKU MiniFlex II x-ray diffractometry (XRD) with Cu Kα (λ = 0.15406 nm) operated at 30 kV and 15 mA to measure in 2θ was 35-65° including the glazing incidence x-ray diffractometry (GIXRD) using a TTRAX III-RIGAKU with Cu Kα (λ = 0.15406 nm) operated at 40 kV and 40 mA. The GIXRD measurements were performed with incident beam angle ω = 1°. The scanning range in 2θ was 10-70°. The surface morphology and the films cross sectional analysis were obtained using JSM-7001F Field Emission Scanning Electron Microscope (FE-SEM). The optical transmittance was measured by UV-Vis spectrophotometer with the range of 300-800 nm.