



Controlled thermal oxidation of nanostructured vanadium thin films



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ABSTRACT

Pure V thin films were dc sputtered with different pressures (0.4 and 0.6 Pa) and particle incident angles α of 0°, 20° and 85°, by using the Glancing Angle Deposition (GLAD) technique. The sputtered films were characterized regarding their electrical resistivity behaviour in atmospheric pressure and in-vacuum conditions as a function of temperature (40–550 °C), in order to control the oxidation process. Aiming at comprehending the oxidation behaviour of the samples, extensive morphological and structural studies were performed on the as-deposited and annealed samples. Main results show that, in opposition to annealing in air, the columnar nanostructures are preserved in vacuum conditions, keeping metallic-like electrical properties.

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1. Introduction

Vanadium oxides (VO_x) such as VO_2 and V_2O_5 in the form of thin films display interesting optical and electrical properties that make them suitable for a wide range of applications [1–3]. The reversible first-order semiconductor-to-metal transition exhibited by the VO_2 [2,4–6] and V_2O_5 phases [1,7] makes them especially interesting for gas sensing purposes [3,8,9]. Furthermore, the sensitivity of these VO_x phases are commonly attributed to the presence of oxygen vacancies [2,3,6,7,8]. Consequently, two main strategies are currently being explored to obtain the thin film materials either by (i) ab initio sputtering of the referred oxides [5,10] or by (ii) subsequent thermal oxidation of pure V films [4,11]. Also, the combination of both strategies is often used [2,6,12].

In this work, the authors sputtered pure V thin films using the GLAD technique. It is known that porous nanostructured films display interesting electrical and optical properties, thus making them ideal candidates for sensing applications [13,14]. Hence, the effects of controlled thermal oxidation on the resistivity behaviour of the samples sputtered with normal and inclined columnar structures were investigated in atmospheric pressure and in-vacuum conditions. The aim of the present letter is to assess how the combination of nanostructured features and a controlled formation of the VO_x phases contribute to the enhancement of the electrical behaviour of the films.

2. Experimental details

Pure V films were deposited by dc magnetron sputtering inside a 40 L stainless-steel custom-made vacuum chamber. The reactor was equipped with a circular planar and water-cooled magnetron sputtering source, which was evacuated with a turbomolecular pump, backed by a mechanical one, in order to obtain a base pressure of 10^{-6} Pa. A vanadium target (purity 99.6 at%, 75 mm diameter) was used. This target was dc sputtered in a pure argon atmosphere, using a constant current $I=200$ mA. The incident angle of the particle flux was changed by tilting the substrate holder with an angle α taken from the substrate normal: $\alpha=0^\circ$, 20° and 85° (GLAD technique). The argon flow rate was kept at 5.7 sccm ($\alpha=0^\circ$ sample) and 2.1 sccm ($\alpha=20^\circ$ and 85° samples), corresponding to a partial pressure of 6×10^{-1} Pa ($S=16 \text{ L s}^{-1}$) and 4×10^{-1} Pa ($S=8.9 \text{ L s}^{-1}$), respectively. The substrates, introduced through a 1 L airlock, were glass microscope slides (ISO norm 8037-1, with roughness better than 0.5 nm) and (100) silicon wafers (p-type, $\rho=1\text{--}30 \text{ } \Omega \text{ cm}$). Before each run, all substrates were cleaned with acetone and alcohol, and the target was pre-sputtered in a pure argon atmosphere for 5 min, in order to remove the target surface contamination layer. The target-to-substrate distance was kept at 65 mm in all runs. The substrates were grounded and all depositions were carried out at room temperature. The deposition time was adjusted in order to obtain a thickness close to 500 nm.

The thickness of the coatings was assessed using a Tencor Alpha Step IQ profilometer, while the morphological features of the samples were probed by scanning electron microscopy (SEM) at 5 keV in a Dual Beam SEM/FIB FEI Helios 600i microscope. The coatings were also characterized by X-ray diffraction (XRD). Measurements were carried out using a Bruker D8 focus

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diffractometer with a cobalt X-ray tube ($\text{Co } \lambda_{\text{K}\alpha} = 1.78897 \text{ \AA}$) in a $\theta/2\theta$ configuration. Scans were performed with a step of 0.02° per 0.2 s and a 2θ angle ranging from 20 to 80° . The resistivity measurements were performed using the four-probe van der Pauw method in the temperature range of 40 – 550°C (1st cycle of 40 – 100 – 40°C with a ramp of 2°C min^{-1} followed by 10 similar cycles with 50°C increments each until a maximum temperature of 550°C is reached), for both in-vacuum (10^{-5} Pa) and atmospheric pressure conditions. The atmospheric pressure measurements were done in a custom-made chamber, which is covered in order to have a dark environment; humidity and cleanness were considered as constant. The error associated to all resistivity

measurements was always below 1% and the attachment of the contacts was checked prior to every run (I/V correlation close to 1) to ensure that an ohmic contact was attained (use of gold coated tips). All films were characterized in as-deposited and annealed (after atm. pressure and in-vacuum resistivity tests) conditions.

3. Results and discussion

The resistivity (ρ) behaviour of the normal incidence and GLAD sputtered films with increasing annealing temperature is shown in Fig. 1. Major differences can be seen between atmospheric

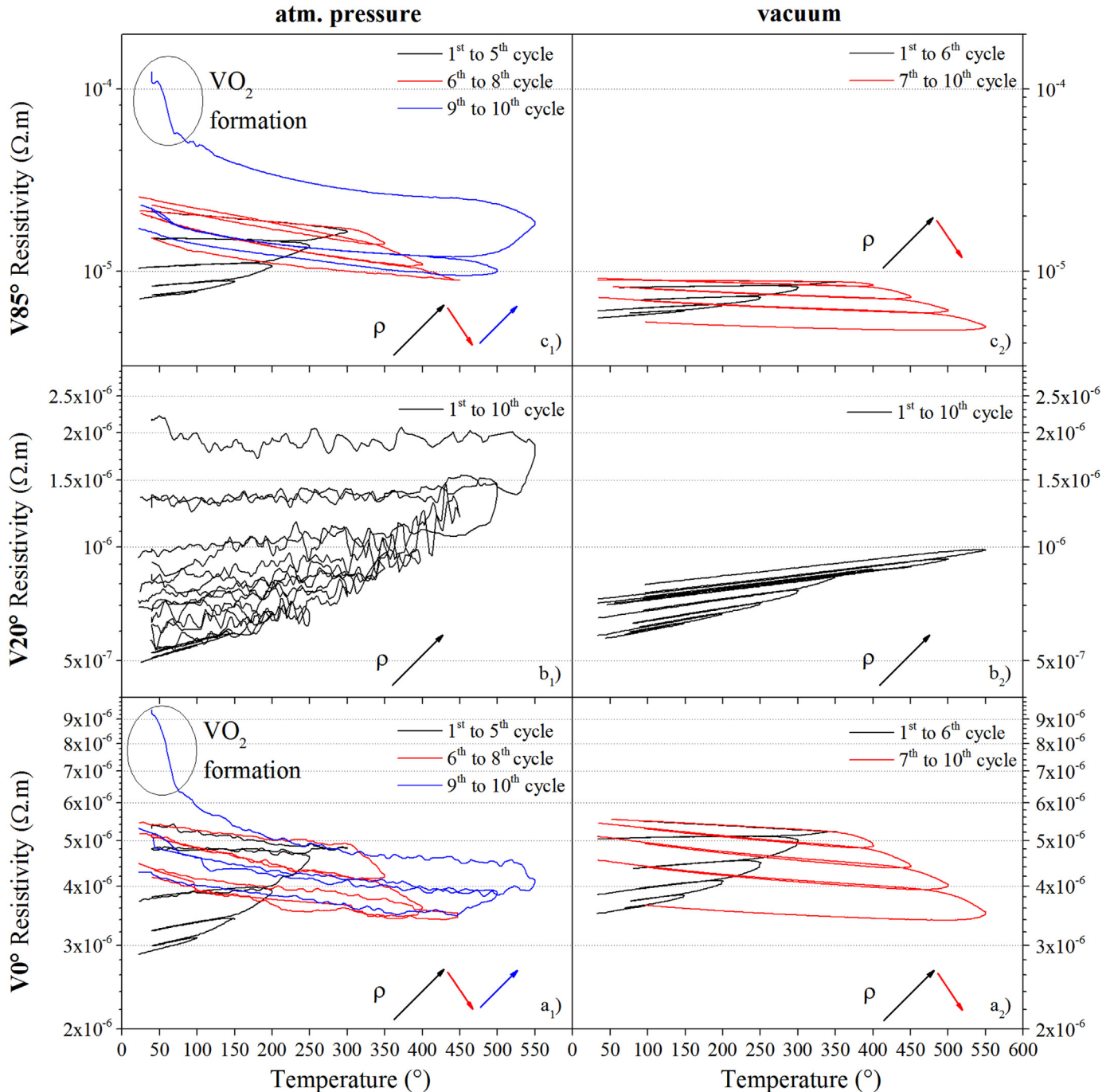


Fig. 1. Atmospheric pressure (a_1 – c_1) and in-vacuum (a_2 – c_2) resistivity measurements for the V thin films deposited with particle incidence angles of 0° , 20° and 85° . Coloured arrows represent the resistivity evolution with increasing cycle number. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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