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Scientific paper
ISSN 0351-9465, E-ISSN 2466-2585
UDC: 621.793.1+546.24
doi:10.5937/zasmat1904379M

Zastita Materijala 60 (4)
379 – 384 (2019)

Effect of deposition rate and substrate microstructure on gas sensitivity of Te thin films

ABSTRACT

Tellurium thin films have been prepared using different rates (0.1 ÷ 30 nm/s) by physical deposition in vacuum on glassy, sintered alumina and electrochemically nanostructured Al_2O_3 substrates. The sensitivity to nitrogen dioxide of fabricated films was tested at room temperature. It is shown that the deposition rate strongly influences the microstructure of the films in question, as well as their gas sensing properties. The increasing of deposition rate results in transformation of microcrystalline structure of the film into an amorphous one. Simultaneously, both the gas - sensitivity and the response time decrease. The results are explained in terms of interaction between gas molecule and lone – pair electrons of tellurium atoms.

Keywords: *deposition rate, gas sensitivity, response time, substrate, thin film.*

1. INTRODUCTION

Tellurium thin films can be successfully used for the detection of toxic gases and environment monitoring [1-3].

Firstly, the microcrystalline Te films grown on the glass Pyrex substrates were shown to be sensitive to nitrogen dioxide in ambient air [4,5]. Soon, it was found out that some other gases as CO, H_2S, or propyllamine [6,7] can be successfully detected at room temperature, but the sensitive parameters can be affected by technological conditions of the sensor preparation [8]. In the last communication it was observed that the increasing of the substrate temperature, results in increase the tellurium grain dimensionality. The microstructures of the substrate influence the gas sensitivity, as well. So, the films deposited on the glass substrate show the maximum sensitivity to H_2S and NO_2, while those deposited on sapphire substrate show a minimal sensitivity [8].

In the present work the tellurium films have been prepared by different rates of growing on different substrates, not heated and not cooled, which allowed to establish the influence of both deposition rate and substrate microstructure on gas sensitivity.

2. MATERIALS AND METHODS

Tellurium (purity 99.999 %) based thin films of different thicknesses were deposited either onto Pyrex glass or nanostructured Al_2O_3 substrates with thermal vacuum evaporation. The evaporation was performed from a tantalum boat at the working pressure of $\approx 10^{-4}$ Pa, without heating or cooling the substrate. The growing rate of the film was varied from 1,0 to 30 nm/s, the area of deposition being around 70 mm^2. Rectangular samples of different thicknesses were prepared by variation of evaporation time, while the distance between the evaporation boat and the substrate has been kept the same - 20 cm. The thicknesses and the shape of the films have been measured after their preparation using an Atomic Force Microscope (AFM) (SIS SCAN Control/C). The surface morphology of the films was investigated, using a VEGA TESCAN TS 5130 MM scanning electron microscope (SEM). X– ray analyses using the DRON –YM1 diffractometer by Fe$K\alpha$ radiation was applied for the structural investigations of the grown films. Rotation velocity of the scintillation counter was 2 (or / and 4) angle degrees /min.

Two Indium pillows were pressed on top of the Te film surface in order to serve as electrical contacts for the gas-sensing element. The distance

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Paper received: 06. 08. 2019.
Paper accepted: 12. 09. 2019.
Paper is available on the website: www.idk.org.rs/journal

between electrodes was ~ 5mm so that the sensing aria consists of about 30 mm^2.

NO_2 vapor with a concentration of 0.15 to 1.0 ppm was obtained by using the experimental set up described in [9]. The thin film sensing devices were put into a test cell (of 10 ml volume) and the gases were injected with a flow rate of 100 ml /min, maintained by mass flow controllers (MFC, Wigha, Germany), parallel to the film surface.

Humidification of the carrier gas was accomplished using saturated solution of the salt $CaCl_2$ in water with relative humidity of 32%, and the platinum resistance temperature detector PT – 100 close to the sensor served as a temperature controller. The current transient characteristics have been carried out with different gas concentrations at room temperature with an applied voltage of 5V. In order to transform the resistance signal into voltage signal, the sample was connected in series to a load resistance using a direct current (d.c) voltage supplier. In all measurements, the load resistance was chosen to be approximately by an order of magnitude less than the sample resistance.

The sensor sensitivity was defined as a relative variation of the resistance expressed in percent / ppm:

$$S = \frac{R_a - R_g}{cR_a}\mathbf{x100} \qquad (1)$$

were R_a and R_g are the electrical resistance of the sensor in air and in the presence of gas (NO_2) respectively, and c is concentration of the target gas [ppm].

3. RESULTS AND DISCUSSIONS

3.1. SEM and X-Ray analyses

To clarity the effect of deposition rate on microstructure, tellurium thin films were grown on Pyrex substrates and both X-Ray and SEM analyses have been used.

Figure 1 shows the SEM images of as-prepared Te thin films grown with deposition rate v = 1nm/s. It is seen that the film deposited with so low rate, exhibits a dense layer of polycrystalline Te with a crystallites size of about 0.5 to 1.0 µm, oriented along the substrate. The X-Ray diffraction pattern of such films (Figure 2) indicates the hexagonal phase of Tellurium crystallites.

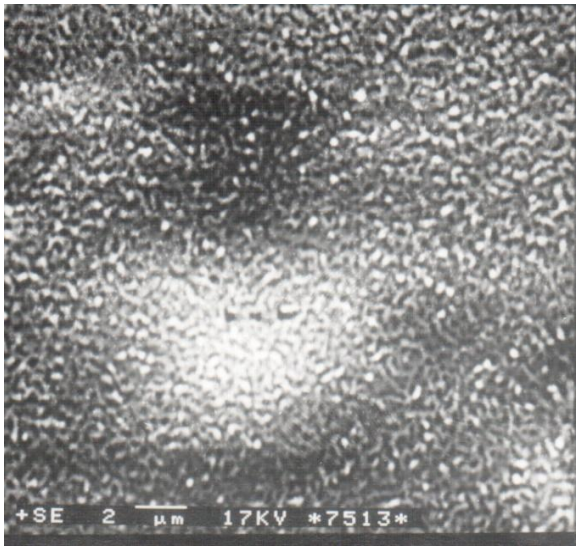

Figure 1. SEM micrographs of Te grown on Pyrex with on deposition rate v = 1nm/s

Slika 1. SEM mikrografije Te na Pyrex staklu sa brzinom taloženja v = 1nm/s

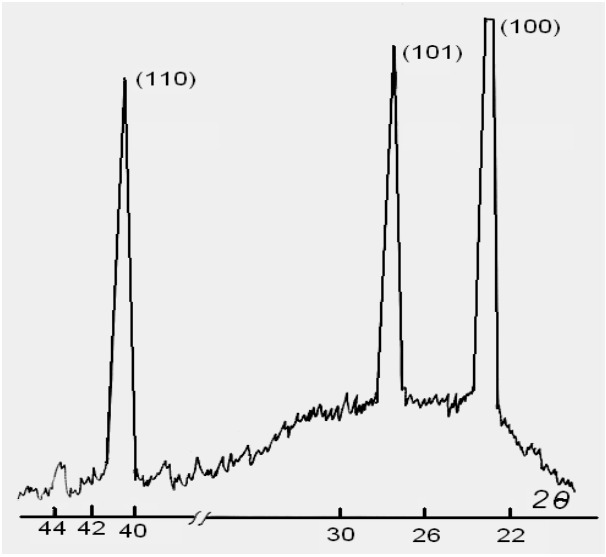

Figure 2. XRD diffraction micrographs of Te grown on Pyrex with deposition rate v = 1 nm/s

Slika 2. XRD difrakcione mikrografije Te na Pyrex staklu sa brzinom taloženja v = 1 nm/s

Increasing of the film deposition rate up *to v = 10 nm/s* results in transition from microcrystalline structure of the films to a nanocrystalline one (Figure 3)". Figure 4 shows a typical XRD pattern of a nanocrystalline tellurium film grown with a deposition rate v=10nm/s. This figure also confirms both the nanometric dimensionality of crystallites and the absence of preferential grow orientation.