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Procedia Materials Science 11 (2015) 464 – 468

Procedia Materials Science

www.elsevier.com/locate/procedia

5th International Biennial Conference on Ultrafine Grained and Nanostructured Materials, UFGNSM15

Fabrication of ZnS Zigzag Sculptured Nanostructured Thin Films

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Abstract

In This work, the design, manufacture and implementation of new attached equipment to the physical vapor deposition system was reported. Using this system ZnS films were prepared under glancing angle deposition (GLAD) technique with the help of varying polar and azimuthal angles of substrate simultaneously without vacuum drop during deposition process. In addition, the designed system will help to control substrate temperature which makes the growth of sculpture thin films possible. The whole system is controlled by a programmable microcontroller. The formation of ZnS zigzag sculptured nanostructured thin films was confirmed by Field Emission Scanning Electron Microscopy (FESEM) and Atomic Force Microscopy (AFM) analyses.

Peer-review under responsibility of the organizing committee of UFGNSM15

Keywords: Zinc Sulfide; Thin film; zigzag nanostructure; Glancing Angle Deposition.

1. Introduction

Previously people believed that material properties could be changed only by varying the chemical composition. But later it was found that the material properties could be tuned by varying the size of the material without changing the chemical compositions. Fabrication of new materials in nanoscale has an enormous impact on a wide variety of technological areas. As nanotechnology is increasingly used, a number of attempts have been made to improve and upgrade productivity of nanostructures features. In doing so, sculptured nanostructure thin films (SNTFs) have been produced, Taschuk et al. (2010). SNTFs deposited by oblique angle deposition (OAD) or glancing angle deposition (GLAD) Robbie (1998), are new generation of thin films. Such films consist of nano-columns with 1 to 100 nm height, Robbie et al. (1998). The main difference between conventional thin films and SNTFs is in production of SNTFs with desired geometries (e.g. inclined columns, Messier et al. (2000), straight

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Peer-review under responsibility of the organizing committee of UFGNSM15
doi:10.1016/j.mspro.2015.11.055

pillars, Dick et al. (2003), helix Robbie et al. (1995), zigzag Lintymer et al. (2004) shapes). Thereby, many novel physicochemical properties can also be easily engineered, Lakhtakia and Messier (1999). Such a geometrical feature affects physical properties of thin films remarkably, so that they are very good candidates for applications such as optics, sensors, energy and micro fluidics. The changes in basic physical properties arise because the ratio between volume and diameter is much different from that of macroscopic materials. The small sizes can result in quantum effects when the dimensions are smaller than the wavelength of electrons. Materials consisting of a closely packed array of nanorods are examples of very porous structures with interesting properties. Nanorods are believed to be one of the important building blocks for the assembly of integrated nano-electrical, nano-optical and nano-mechanical systems, Ye et al. (2004). They are also of great interest as individual nanodevices including nanoelectronics, field emitters, nano-optics, and photonic crystals, Ye et al. (2004).

This article, reports on the design and implementation of an indigenously made glancing angle deposition with a water circulating system that will maintain the substrate temperature at room temperature through thermal contact. The FESEM and AFM images of ZnS zigzag thin films were employed to confirm the efficiency of this system.

2. Experimental setup

2.1. Apparatus

One of the most important parameters for obtaining nano-structure thin films is the ability to accurate control the substrate position with respect to incident vapor flow (polar and azimuthal angles) as well as control of substrate temperature during the deposition process. This is achieved by means of glancing angle deposition (GLAD) technique. Therefore, for equipping physical vapor deposition system to produce thin films by GLAD technique; a proprietary apparatus was designed, manufactured and installed.

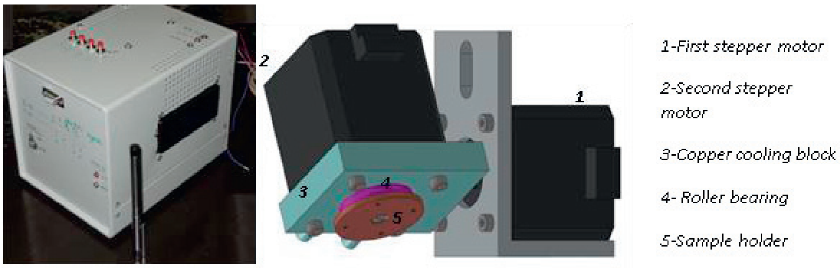

Fig. 1. The schematic of designed apparatus (right) and control box (left), Rahchamani et al. (2015).

The different parts of the designed apparatus have been introduced in Fig.1. The stepper motors (No. 1 and 2) used to tilt and rotate the substrate. The first stepper motor controls the tilting angle of the substrate with respect to incident flow angle. The second one controls the substrate azimuthal angle with respect to normal of the substrate surface. Their step angle is 0.9˚. For triggering the stepper motors, the necessary signals are produced by two suitable drivers which are sent to the vacuum chamber through two separate electrical feedthroughs. These signals have been produced through provided pulses by an AVR microcontroller (ATM128-IC). These pulses used to supply the drivers. The microcontroller is programmable in different models. The desired substrate position is given to the microcontroller as initial information. In fact, the command section controls the variations of polar and azimuthal angles and substrate rotation angles.

Generally, substrate temperature is not controlled in conventional GLAD systems. Therefore, to create high quality films, high melting point materials are used due to their low surface mobility on substrates. So for achieving the columnar structure of materials with low melting temperature, it is necessary to dissipate the heat generated at the substrate surface.

To maintain the substrate temperature during the deposition process a circulating cold water system including a copper block acting as a heat exchanger attached to the sample holder was used. The heat exchanger is connected to the revolving sample holder (RSH) using a roller bearing. Heat exchanger is made in a grooved form. Because of the large thermal energy transfer and appropriate conductivity, copper was used. The cold water flows through a U-shaped channel within the block. The design demands a minimum of 10 mm wall thickness. Because of the way in which the fluid channel was bored out, some of the bore holes needed to be plugged; plugs were made from copper, machined to fit the holes, and a fixed by silver soldering for a vacuum-compatible seal. The center of the heat exchanger is bored out to accommodate the rotating shaft with the interchangeable head piece. The heat exchanger was designed to mate to stepper motor body by four screws, then the RSH screws to shaft of rotating stepper motor. When installed, the gap between the heat exchanger and RSH is 4 mm. To make contact between block and RSH, to creative possibility rotation of RSH, a fitted roller bearing stainless steel with suitable diameter was used.

The main problem when attempting heat removal from a RSH is the need to preserve substrate tilt and rotation. The tilt of the substrate in GLAD system creates a dilemma in maintaining link between the two sections with enough area and large heat transfer, and source of fluid in the out of chamber. To flow cold water within the vacuum chamber, a liquid feed-through was installed. A feed-through tube with 1 inch diameter was used. A flexible Silicon tube connected the feed-through to the heat exchanger. This tube is suitable for ultrahigh vacuum (UHV) environments and is capable of operating up to 500 ˚C. A tube of 120 cm length (60 cm for entering the water and 60 cm for flowing water out of the vacuum) was chosen to anticipate pitfalls and lapses in design. The connection to the cooling block was made by ordering a stainless steel clamp. To create a parallel vapor flux, the distance between the evaporation sources to the substrate is possibility adjusted by moving the apparatus.

2.2. The deposition process

ZnS zigzag nanostructured thin films have been deposited onto glass substrates in Hind-HIVAC coating unit (Model 15F6) equipped with already described controlling system. The substrates were cleaned in acetone using ultrasonic bath and then dried by purified nitrogen gas. Base pressure of the chamber was about 10-6 mbar. ZnS tablet of 99.99% purity, supplied by Aldrich Company, was evaporated from a molybdenum boat. Deposition rate and thickness of film were measured and controlled in-situ for non-angled non-rotating substrate using Hind-HIVAC thickness monitor (Model DTM-101). The average deposition rate was 5 Å/s. In order to achieve a uniform layer on the substrate, the distance between the evaporation source and the substrate was 30cm. The prepared samples were systemically examined by the following techniques; Field Emission Scanning Electron Microscopy (FESEM) (HITACHI S-4160) with operating voltage equal to 15 kV, and Atomic Force Microscopicy (AFM) (NT-MDT, Scanning probe, model BL022) 2D and 3D.

3. Result and discussion

FESEM and AFM studies of the deposition ZnS thin films proved the capability of the GLAD system equipped with a substrate controlling facility to produce zigzag sculptured nanostructures. The top and side views obtained by FESEM are shown in the Fig. 2a and 2b. Fig. 2a shows the porosity of the prepared film. From Fig. 2b, one can easily observed the formation of columnar angled film. The formation of zigzag structure observed in these figures, is due to rotating the substrate by 180˚ around its axis.

Considering the growth behavior of columns shown in figure, one can easily observe that the column diameter is bigger at its top than its bottom. This kind of tapering may be due to the shadowing effect. That is any column acts as a barrier to its adjacent column to receive flow of material as the time elapses, Karabacak et al. (2003), Main et al. (2004). Buzea et al. (2005) observed the same behavior in the case of silicon nanocolumn growth by GLAD technique.

The relation between the column angle (β) and the flux angle (α) (Fig. 2b) is given by Driks et al. (1977)

$$tan\beta = \frac{1}{2} tan\alpha \quad (1)$$

Another relation suggested by Tait et al. (1993) is represented as following:

$$\beta = \alpha - sin^{-1}(\frac{1-cos\alpha}{2}) \quad (2)$$

Both the relations have been used for GLAD deposited films by Xiao et.al, however they found the later one was better to give best results for larger values of α (>50°) Xiao et al. (2010). Also, using the ImageJ software on FESEM side view image, the growth angle (β) was measured about 51˚ which is close to the calculated value 55.6˚ obtained from Eq. 2. The column diameter has been estimated to be between 60 to 80 nm. Fig. 2c demonstrated the AFM image of the specimen. In Fig.2c, the dark spots indicate the absence of columns and creation of porosity.

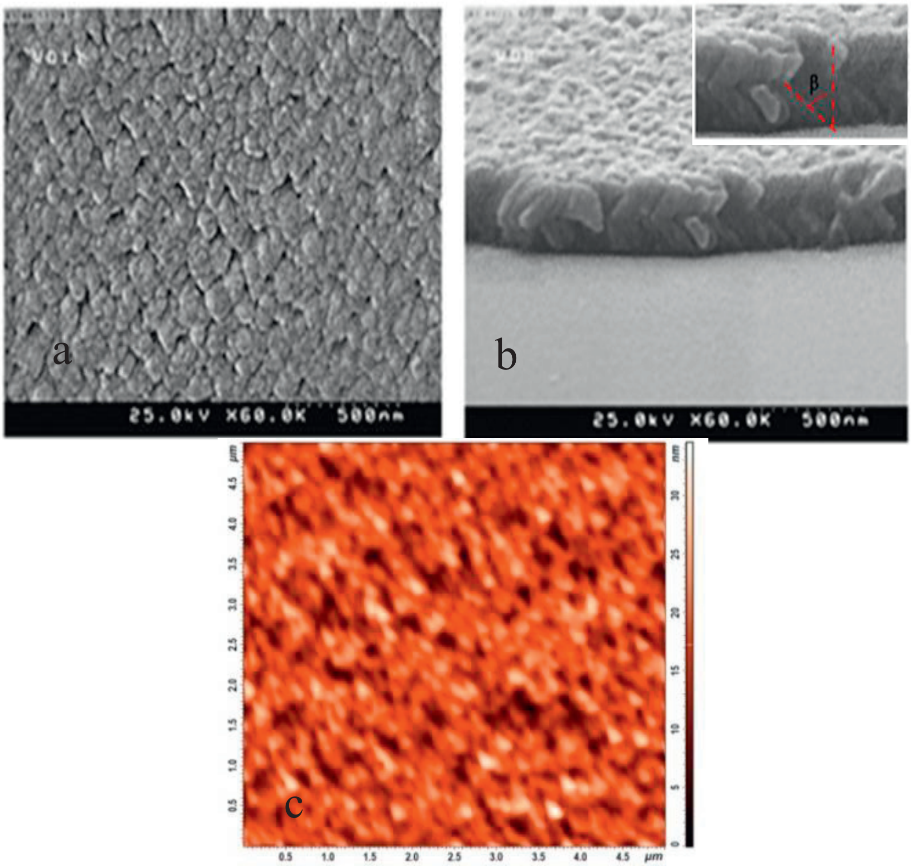

Fig. 2. The FESEM image of (a) top; (b) side views of the specimen; and (c) the AFM image of the specimen.

4. Conclusion

This work focuses utilizing GLAD technique to grow nanostructures. For this purpose, a homemade system consisting sample folder, two step motors, heat exchanger unit and AVR microcontroller was designed .This setup can control the polar and azimuthal angles of substrate during deposition process. This setup was used to produce nanostructures of ZnS. The FESEM analysis of specimen confirmed the formation of tilt columnar structure.

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