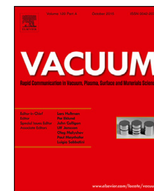
Vacuum 121 (2015) 105–112

Contents lists available at ScienceDirect

Vacuum

journal homepage: www.elsevier.com/locate/vacuum



The role of gas direction in a modified Grimm-type glow discharge for controlling the degree of crystallinity in brass alloy thin films

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A R T I C L E I N F O

Article history:
Received 9 March 2015
Received in revised form
22 July 2015
Accepted 6 August 2015
Available online 10 August 2015

Keywords:
Grimm type glow discharge
Brass thin films
Nanocrystalline structure

A B S T R A C T

Sputter-deposition rates in different gas directions were used for growing brass thin films in the modified Grimm-type direct current glow discharge. The sputtering and deposition rates were significantly improved by changing the direction of the argon gas flow. The degree of crystallinity and the nanoparticle size of the deposited thin films could also be controlled. Optimal operating conditions such as the gas pressure, applied power, deposition time, and distance from the substrate to the target surface were employed. The degree of crystallinity of the films was determined via the X-ray diffraction technique. Sequence changes in the brass thin film structure to nanocrystalline phases were obtained based on the direction of the Ar gas. However, in all cases, the obtained nanocrystalline films had polycrystalline or single crystalline structure with the normal and the new proposed gas flow direction respectively. Wavelength dispersive X-ray fluorescence analysis confirmed that the films had the same elemental compositions as that of the cathode material. Semi-amorphous and polycrystalline brass thin films were also easily obtained using the normal gas direction. Moreover, the new proposed gas direction in the modified Grimm-type glow discharge resulted in the successful deposition of single crystal thin films with controlled nanoparticle sizes.

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1. Introduction

The sputtering-deposition processes associated with direct current (DC) glow discharge have been used increasingly in various areas. These processes have been used in various semiconductor industries as well as for producing optical antireflection coatings on glass, and the etching of thin films including the deposit contact metals on the films. The resulting films are used in the electronic industries, as well as for solar cells, display panels, photovoltaic devices, and xerographic material formation of protective hard coatings [1–3]. Sputter-deposition via glow discharge offers many advantages, namely: the ability to deposit high-melting-point materials, excellent adhesion to the substrates, and compositional similarity between the deposited film and the source material. The experimental and theoretical studies of this deposition method are thoroughly described elsewhere [4–10]. The Grimm-type glow discharge is used extensively in analytical atomic emission spectroscopy. This technique may also be used as a thin film deposition technique and at optimal conditions, and significant improvements in the sputter-deposition yields can be achieved. As such, a modified Grimm-type glow discharge was investigated in this work. Two zones between the anode and the cathode, namely the cathode dark space (CDS) and the negative glow (NG) zones, play an important role in the deposition. The CDS and the NG are the relatively thin dark layer adjacent to the cathode surface and the most luminous region stemming from many excitation and ionization collisions, respectively. In the present work, the thin films were deposited via the sputter-deposition method; i.e., the sputtered target atoms reached the discharge plasma, where they diffused, and were then deposited at low gas pressures of 0.0075–0.075 Torr on the substrate [4,5]. Inert Ar gas is typically used as the discharge gas. Under optimal operating conditions, the Ar^{+} ions are accelerated to the cathode and subsequently sputter away the cathode atoms. The discharge plasma was revealed by the cathode atoms owing to the low operating pressure. Moreover,

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http://dx.doi.org/10.1016/j.vacuum.2015.08.003
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depending on the thermal-ionization atom concentration profile, the cathode atoms can also diffuse back to the cathode and/or migrate to the substrate positioned opposite to the cathode, where they are subsequently deposited [4,5]. The configuration of the present modified Grimm-type glow discharge is somewhat similar to the cylinder-jet configuration proposed by Kim et al. [11]. However, Kim et al. compared only the emission characteristics and analytical performance of the Cu emission lines resulting from three different types of jet configuration namely, cone-jet, six-jet, and cylinder-jet configurations; the sputtering-deposition phenomena occurring inside the discharge plasma of the proposed configuration were not considered in that work. Although the cylinder-jet configuration exhibits the worst analytical performance of the three types of jet configurations, this may be indicative of the significant number of sputtered atoms and ions present in the discharge plasma. In this work, the deposition of brass thin films using modified Grimm-type glow discharge is briefly demonstrated using gas flow directions 01 and 02. Brass samples were used as the cathode material. Cu–Zn alloys were chosen since they constitute the main components in various applications such as thin film solar cells, photovoltaic thin films, decorative features providing a bright gold-like appearance, memory and switching devices in computers and antenna cores, and contrast agents in magnetic resonance imaging in the biomedical field [12–15]. The different glow discharge parameters (sputtering/deposition time, axial distance, pressure, and power) were optimized in our previous work [4–6]. In the current study, the deposited thin films were characterized via wavelength dispersive X-ray fluorescence (WDXRF) and X-ray diffraction (XRD).

2. Experimental setup

2.1. Modified Grimm-type glow discharge

The modified DC Grimm glow discharge used in this work has been described in detail elsewhere [4–6]. The glow discharge plasma source, based on the original model of Grimm [16], was modified. In this study, the distance between the hollow anode cylinder 29.4 mm (length) × 9.2 mm (inner diameter) × 13.9 mm (outer diameter) and the sample (i.e., the cathode) was set to 0.2 mm. In contrast to Grimm's model, an 8.85 mm (diameter) × 4 mm (thickness) quartz substrate was mounted inside the anode for deposition of the sputtered atoms from the cathode. A brass material supplied by Egyptian Copper Company, Cairo, Egypt was used as the cathode material and different deposited thin films were obtained. The elemental quantitative analysis of the brass sample was previously performed [17]. Argon gas (99.9% purity), at a flow rate of 0.3 L/min, was used in order to generate the discharge plasma. A discharge power of 0–2.5 kV and 0–250 mA was used at a pressure of 1.5 Torr. The operating conditions, applied voltage, current, and pressure were optimized in our previous study [4–6].

2.2. X-ray diffraction

XRD measurements were used to determine the compositions of the cathode material of the modified Grimm-type glow discharge and the deposited thin films. An X Pert Pro diffractometer was used to scan the surfaces of the brass alloy thin films using Cu-Kα radiation at 40 kV and 40 mA. The scans were typically performed over a 2θ range of 45°–100° at a speed of 0.02°/s, with an aperture slit, an anti-scatter slit, and a receiving slit of 2 mm, 6 mm, and 0.2 mm, respectively.

3. Results and discussion

In our previous work, various parameters of the modified Grimm-type glow discharge were optimized by using gas flow directions 01 and 02; these parameters were the gas pressure, applied voltage, discharge current, sputtering yield (atoms/ion), deposition rate (μg/sec), and deposition time. The gas flow direction 01, the normal one, means that the gas flows through the inner diameter of the anode cylinder to the cathode surface. On the other hand, the gas flow direction 02 means that, the gas flow through the outer diameter of the anode cylinder annularly to the cathode target, Fig. 1. Using gas flow direction 02, the sputtering and deposition rates were increased 16- and 17-fold, respectively, at a gas pressure of 1.5 Torr and deposition time of 21 min. The use of gas flow direction 02 constitutes therefore a promising procedure for controlling the crystalline structure. Owing to its simple structure and use in various applications, this control was demonstrated for the case of a Cu–Zn composite.

3.1. Gas flow direction

The schematic in Fig. 1 shows the two directions of Ar gas flow used for the modified Grimm-type glow discharge. In both cases, the thin films were deposited on a quartz substrate that was placed inside the anode cylinder. The distance between the substrate and the brass samples (cathode) was varied from 1 to 6 mm. The sputtered atoms and ions originating at the cathode surface were deposited on the substrate using both gas directions. Furthermore, the glow discharge was operated in the abnormal glow discharge zone at low power and pressure, and a gas flow rate of 0.3 L/min was used for both directions. Using gas flow direction 01, as proposed by the Grimm setup, resulted in low levels of cathode-surface bombardment by the Ar gas particles; therefore, only a small number of ions and free electrons were ejected from the surface onto the quartz substrate. However, in the case of direction 02, Ar gas is directed tangentially into the outside surface of the anode cylinder towards the target surface (cathode); i.e., the gas is directed as jet-assisted laminar flow across the target surface. The cathode surface is then completely covered by the discharge owing to the high level of bombardment by the Ar gas particles and consequent large numbers of ejected atoms and small clusters of the cathode surface. The depth profile therefore became deeper, than that resulting from the use of direction 01, and many ions and free electrons were generated; this resulted, in turn, in an increase in the electrical current and current density inside the discharge cell. Therefore, the sputtered yield obtained at the optimized physical parameters (gas flow rate, pressure, current, and voltage), was significantly larger than that calculated for gas flow direction 01. In the case of direction 02, a crystalline-deposited thin film can be obtained at various conditions even without an annealing process.

3.2. I–V relation

Fig. 2 shows the I–V characteristics of the modified Grimm-type glow discharge at gas flow directions 01 and 02, for a distance of 2.8 mm between the substrate and the cathode target. The applied voltage is varied from 265 to 430 V and 220–520 V for gas flow directions 01 and 02, respectively. However, the DC current is kept constant in the range 5–100 mA. An optimized applied pressure of 1.5 Torr and a constant gas flow rate of 0.3 L/min were used in both cases. The resulting I–V curves exhibited significant dependence on the direction of the gas flow. In fact, the I–V curve exhibited only weak linearity, with an r-squared value of 0.938, in the case of direction 02. This weak linearity resulted from the variation in the

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