Study of the Structural Properties and Morphologies of Gold Thin Films Deposition by Annealing

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Abstract
In this paper, the effect of annealing temperature on properties of gold thin films has been investigated. Au thin films have been deposited on glass substrates by the electron beam coating technique and after subjected to annealing in a mixed ambient of air and oxygen at 575, 650, 725, 800 and 875 K for 1 h and then cold slowly. The crystallographic structure of Au thin films was studied as a function of the annealing temperature. X-ray diffraction was used to estimate the crystallographic texture, grain size. All the films were found to have crystalline structure. The films morphologies were studied by scanning electron microscopy (SEM). The surface topographies were studied using atomic force microscopy (AFM). The surface topographies were studied using atomic force microscopy (AFM). The surface topographies were studied using atomic force microscopy (AFM).

Keywords: Thin film; Deposition; X-ray diffraction (XRD); Scanning electron microscopy (SEM); Atomic force microscopy (AFM).

1. Introduction
Thin films have two important features: (i) thickness of under micron and closer to nanometer; (ii) extremely large surface to thickness of layer. These properties because that ratio surface to bulk is large and are created certain features. Thin films of noble metal nanostructures have been of an attractive research field because of their size and shape-dependent properties and optical properties [1-5]. Nan crystalline thin solid films nowadays present enormous scientific interest, mainly due to their attractive novel properties for technological applications [6, 7]. The most important prerequisite for the preparation of high quality film is an understanding of its growth dynamics and structure in different phases of deposition. In the course of the twentieth century, the theory of size-dependent effects in metal thin layers was further developed by numerous scientists, and various approaches to the problem were proposed. For isolated metal particles’ behavior at exiguous dimensions (1D and 2D), quantum size effects are decisive, whereas for ultrathin metal layers both surface effects and quantum size effects must be considered [8, 9].

Gold is known as a shiny, yellow noble metal that does not tarnish, has a face-centered cubic structure, is non-magnetic, melts at 1,336 K, and has density a 19.320 g cm$^{-3}$ [10]. Gold in the form of thin films is nowadays used in a vast range of applications such as microelectro mechanical and nanoelectromechanical systems [11, 12], sensors [13], electronic textiles [14], bioengineering [15], generator of nonlinear optical properties [16], or devices for surface-enhanced Raman scattering [17].

In this work, Au alloy thin film was created on glass substrate, and various heat treatments was conducted. These films were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) and atomic force microscopy (AFM).

2. Experimental
Au used for deposition was Merck grade. Au thin films were prepared by electron beam coating method. The films were deposited on 25×75 mm glass substrates. Prior to gold deposition process, the substrates were cleaned with ethanol, acetone and ultrasonic apparatus for 30 min. Film thickness were measured with a gauged quartz crystal microbalance. Typical coating parameters were cathode voltage 9 kV, pressure 5×10$^{-4}$ mbar, substrate temperature 32 C, coating rate 1 Ås$^{-1}$ and film thickness about 370 Å. The obtained films were annealed for 1 h. The annealed temperature was chosen in the range 575-875K in 75K step (Table 1). The structure characterization of the thin films were obtained by X-ray diffraction. The Au nanoparticles formed and the fine structure of the Au were observed by using SEM. The surface topographies were studied using atomic force microscopy (AFM).
3. Results and Discussion

3.1. X-Ray Analysis

The structures of the Au thin films were studied using X-ray diffraction (XRD) technique. The diffraction spectra were measured at 20 scanning mode ranging from 5 to 90 degrees diffraction angle. The XRD spectra of Au thin films with the same thicknesses on glass substrates and annealed different temperatures are shown in Fig. 1. These patterns show effect of annealing on layers crystallite properties. All the diffraction peaks can be assigned to (111), (211) planes respectively for gold. Miller indexes of planes of cubic Au are confirmed. In the patterns no impurity phases can be detected. The particles average size were calculated using the Debye-Scherer formula, \( D = \frac{0.89 \lambda}{\beta \cos \theta} \), where \( \lambda \) is the x-ray wavelength (1.5406 Å), \( \theta \) is the Bragg diffraction angle and B is the peak width at half-maximum (FWHM). The calculated crystallite sizes of samples can be seen in Table 2. Generally the crystallite size depends on the substrates temperature, deposition rate, film thickness and annealing temperature [18]. XRD result shows, the structure of the layers are crystalline with a preferred (111) orientation and invigorate crystalline structure with increasing annealing temperature (by increasing annealing temperature increased maximum peak intensity from 68.67 to 1566.9 (a.u)).

3.2. SEM Observation

The structure and morphology of obtained coating were studied by SEM. Fig. 2 shows the scanning electron micrographs for thin films gold in difference annealing temperatures. Present SEM images show a grain structure for Au films, and the size of grains is higher with increasing of annealing temperature. Moreover these results are in agreement with XRD results.

3.3. AFM Observation

The structure and topography of obtained coating were studied by AFM. Fig. 3, a – c and Fig. 4, d – f shows the two-dimensional (2D) and three-dimensional (3D) AFM images for Au thin films in without annealing temperature and annealing temperature in 575 k, 650 k, 725 k, 800 k, 875 k, respectively. The 2D images show that the films are uniform and the substrate surface is well covered with grains that are almost uniformly distributed over the surface. The 3D images exhibit large separated conical columnar grains in all thin films and the average grain size increases with annealing temperature. This may be due to the bigger clusters formed by the coalescence of two or more grains [19]. The values of the average surface roughness increases with increasing annealing temperature from about 0.85 nm to 46 nm. (AFM) analysis showed the gold layer growth to be running over isolated islands.

4. Conclusions

We have shown that Au thin films can be formed on glass substrates by electron beam coating technique. The changes in structural properties of the Au films were investigated with the annealing temperature using XRD, SEM. Results of different analyses on the gold films shown that they maintained the crystalline structure even after annealing at 875 k and the increasing of annealing temperature could increase the Au nanoparticles size.

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### Table 1. Demarcation the Samples

| Samples        | Annealing Temperature | The Name of Samples |
|----------------|-----------------------|---------------------|
| Au 370 Å       | without annealing     | Au 370 Å            |
| 575            | 575 Å 575 K           |                     |
| 650            | 650 Å 650 K           |                     |
| 725            | 725 Å 725 K           |                     |
| 800            | 800 Å 800 K           |                     |
| 875            | 875 Å 875 K           |                     |

### Table 2. The Crystallite Size of Samples in Au Thin Films at Various Annealing Temperatures

| The Name of Samples | Crystallite size of Samples (nm) |
|---------------------|---------------------------------|
| Au 370 Å            | 19                              |
| Au 370 Å 575 K      | 28                              |
| Au 370 Å 650 K      | 41                              |
| Au 370 Å 725 K      | 43                              |
| Au 370 Å 800 K      | 43                              |
| Au 370 Å 875 K      | 47                              |

### Figure 1. The XRD Spectra of the Au Thin Films
**Figure 2.** SEM Images of Au thin Films in Without Annealing Sample & at Different Annealing Temperatures
Figure 3. AFM Images of Au thin Films at a) Without Annealing & b) at 575 k c) at 650 k

Figure 4. AFM Images of Au thin Films d) at 725 k e) at 800 k f) at 875 k
