Influence of substrate on properties of gold nanolayers

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ABSTRACT

Gold layers were prepared by sputtering on polyethylene (PE) and glass. Sheet resistance, thickness, surface morphology and crystal structure of the layers were studied by two-point technique, gravimetry, AFM and XRD. Under the same conditions the growth rate of the layer is higher on the glass. On the glass a shorter sputtering time is needed for creation of an electrically continuous layer. Roughness of the underlying substrate affects roughness of deposited gold layer. From XRD analysis it was found that the gold layers deposited onto glass and PE digger by biaxial strength, size of crystallites and number of micro-deformations.

1. Introduction

Bulk gold is known as a shiny, yellow, noble metal that does not tarnish. It has a face centered cubic structure, non-magnetic properties, melting point at 1336 K and density of 19.320 g cm⁻³. The melting temperature decreases dramatically as the size of gold particles goes down to nano-meter dimension [1]. Gold in the form of nano-meter sized particles ceases to be noble; 2–3 nm nano-particles are excellent catalysts and exhibit considerable magnetism [2]. Gold nano-particles may also exhibit insulating properties. Nowadays, gold in the form of thin films is used in a vast range of applications, e.g. in electronics [3–5], photonics [6], nano-electromechanics or biosensors [7], devices for Surface-Enhanced Raman Scattering [8], conducting electrodes [9], and catalysis [10,11].

In this work gold layers were sputtered on glass and PE substrates. The effects of substrate properties on the electrical resistance, thickness, surface morphology and crystal properties of gold layers were studied by electrical measurements, gravimetry, AFM and XRD methods.

2. Experimental

2.1. Substrate and Au deposition

We sputtered gold (gold target of purity 99.99% was supplied by Goodfellow Ltd.) layers in diode sputter-coater Balzers SCD 050 device.
using a DC Ar plasma. The sputtering times up to 500 s were used. Other conditions of the sputtering procedure were: chamber volume ca 1000 cm$^3$, gas purity of 99.995%, discharge power of 7.5 W, Ar flow approximately 0.3 l s$^{-1}$, pressure of 5 Pa, and electrode of 48 cm$^2$ in area at the distance of 50 mm. The deposition was performed onto high density polyethylene (PE, Granitol, 40 μm thick) and glass (Marienfeld, 2.5 × 2.5 cm and 1 mm thick).

### 2.2. Diagnostic techniques

The sheet resistance of the layers at room temperature was examined by a two-point technique with a pico-amperemeter KEITHLEY 487. The measurements were performed on the air at a pressure of about 10 kPa. The mean sheet resistance was determined with a typical uncertainty of ±10%. The thickness of gold layers was determined by weighing with Mettler Toledo UMX 2 microbalance of the samples before and after the layer deposition. The thickness was calculated from the weight difference using gold bulk density of 19.320 g cm$^{-3}$. The effective thickness of the gold layers on glass was obtained by “scratch” technique using AFM (Digital Instruments CP II Veeco) [12], working in tapping mode with silicon P-doped probes RTESPA-CP and with the spring constant of 20–80 N m$^{-1}$. The scratches were done at ten different specimen positions (for each examined layer thickness) by nano-indentation tip and the layer thickness was determined from AFM scan in contact mode [13].

The surface morphology and average roughness ($R_a$) of the substrates and substrates with deposited gold layers were examined using AFM. The mean roughness value ($R_a$) represents the arithmetic average of the deviations from the centre plane of the sample calculated from ten independent measurements. XRD analysis was performed by automatic powder refractometer Panalytical X'Pert PRO using copper X-ray lamp ($\lambda_{CuK\alpha_1} = 0.1540598$ nm) equipped with ultra-fast semiconductor detector PIXcel. Measurement has passed on a symmetric Bragg–Brentano geometry. Diffractograms were registered at the angle range of $2\theta = (10–85^\circ)$. Lattice parameter $a$ of cubic face centered lattice of gold was calculated from diffraction lines location and its intensity, using Rietveld’s method.

### 3. Results and discussion

The measured sheet resistance $R_s$ as a function of the deposition time is shown in Fig. 1. It is seen that for short deposition times discontinuous layers are formed. For longer deposition times the layers become continuous [14]. The transition, manifested by a rapid decrease of the sheet resistance $R_s$, occurs for 30 and 45 s deposition times for glass and
PE substrates respectively. More rapid decrease of the sheet resistance on glass substrate is due to its lower initial surface roughness (see also below). For deposition times above that corresponding to formation of continuous gold layer the sheet resistance $R_s$ remains unchanged within experimental errors. The observed difference between sheet resistance $R_s$ of continuous gold layer and the resistance of bulk gold may be explained by low thickness of the layers. The resistance of thin gold layers, deposited onto different substrates, is higher than that of bulk gold, the difference being due to the size effect according to Matthiesen rule [15,16].

Dependence of the thickness of sputtered Au nanolayers on deposition time is shown in Fig. 2. The thickness of the layers deposited onto glass substrate was measured by “scratch” method with the aid of AFM device and also by gravimetry. This technique cannot be used for the layers deposited onto the PE substrate because of the PE softness. In this case only gravimetry was used. It is seen from Fig. 2 that the thickness grows linearly with increasing deposition time. Under the same deposition conditions, thicker layers are formed on glass substrate. The difference may be due to different initial roughness of both substrates or due to their different surface energies. It is known that lower roughness facilitates surface diffusion of the deposited atoms and in this way their nucleation. So that, lower roughness of the glass substrate as determined by AFM method (see Fig. 3) may be the reason for formation of thicker layers. It is further seen from Fig. 2 that the layer thicknesses determined from the AFM measurement are higher than those determined by gravimetry. The difference is probably due to the well known fact that the density of thin gold layers decreases with decreasing thickness [12,17]. Since the determination of the real density of the gold layers with thicknesses below 10 nm is extremely difficult [12,17], gold bulk density was used for the calculation of the layer thickness from gravimetry.

As was mentioned before the initial roughness of the substrate affects initial phases of the layer grow. The AFM images of pristine and gold coated glass and PE are shown in Fig. 3. The images show that the roughness of the glass substrate is lower compared to the PE substrate. After gold deposition onto glass substrate the surface roughness decreases in contrast to PE substrate where the gold deposition results in a roughness decrease. In later case significant change in the surface morphology is also observed. It may be concluded that the initial morphology of the substrate affects the morphology of the deposited gold layer significantly.

Gold layers deposited for 500 s deposition time were investigated by XRD method and the results are summarized in Table 1. The sputtered gold layers are polycrystalline with preferred orientation of their grains in the (111) direction. Lattice parameter of the gold layers does not depend on the substrate. Tensile stress is higher for the layers deposited onto PE, so that these layers exhibit higher strain. Larger grains (exactly size of the coherently diffracting domains) are observed in the layers deposited on PE substrate as compared with those deposited onto glass. These grains exhibit larger microstrain. These findings lead to conclusion that more stable gold layers are formed on glass substrate.

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Lattice parameter (nm)</th>
<th>Grain size (nm)</th>
<th>Microstrain $\times 10^{-3}$</th>
<th>Lattice stress (MPa)</th>
<th>Texture $T_{200}/T_{111}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass</td>
<td>0.40741</td>
<td>25</td>
<td>1.7</td>
<td>16.2</td>
<td>0.219</td>
</tr>
<tr>
<td>PE</td>
<td>0.40743</td>
<td>36</td>
<td>2.7</td>
<td>278.8</td>
<td>0.282</td>
</tr>
</tbody>
</table>

### 4. Conclusions

Thin gold layers were deposited onto PE and glass substrates by sputtering technique for different sputtering times. Present results show that, under the same deposition conditions, gold layers grow faster on the glass substrate and electrically continuous coverage is formed sooner. AFM measurements show that the initial roughness of the PE surface is significantly higher than that of glass. The surface roughness affects not only speed of the layer grow but also the layer thickness and its stability. According to XRD analysis, Au layers are polycrystalline with preferred orientation of grains in the (111) direction. The layers formed on the glass substrate are more stable. The layers deposited on the PE substrate contain larger grains.

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