Obtaining Au thin films in atmosphere of reactive nitrogen through magnetron sputtering

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Obtaining Au thin films in atmosphere of reactive nitrogen through magnetron sputtering

J H Quintero¹, R Ospina² and A Mello²
¹Universidad de Medellín, Medellín, Colombia.
²Centro Brasileiro de Pesquisas Físicas (CBPF), Rio de Janeiro, Brasil.

E-mail: jhquintero@udem.edu.co

Abstract. 4d and 5d series of the transition metals are used to the obtaining nitrides metallic, due to the synthesis of PtN, AgN and AuN in the last years. Different nitrides are obtained in the Plasma Assisted Physics Vapour Deposition system, due to its ionization energy which is necessary for their formation. In this paper a Magnetron Sputtering system was used to obtain Au thin films on Si wafers in Nitrogen atmosphere. The substrate temperature was varied between 500 to 950°C. The samples obtained at high temperatures (>500°C) show Au, Si and N elements, as it is corroborated in the narrow spectrum obtained for X-Ray Photoelectron Spectroscopy; besides the competition of orientation crystallographic texture between (111) and (311) directions was present in the X-Ray Diffraction analysis to the sample heated at 950°C.

1. Introduction

Actually, the developments in the binaries nitrides are in the inclusion of element as: Al or Si, the which are calls ternary nitrides of the Me-X-N form (where Me: Transition Metal, N: N and X: B, C, Al or Si), due to that improve the hardness and thermodynamic stability when are compared with binary metal correspondingly [1,2]. Particularly, TiN/Si₃N₄ present high elastic modules, high hardness and high resistance to the oxidized, because, the Si₃N₄ amorphous phase is found as nanocrystals into TiN phase [3]. Besides, different studies has shown the relation between Si content with the hardness in the coatings [4]. On the other side, CrN/Si₃N₄ has been reported with 26 GPa [5], and 34GPa [6] hardness and low friction coefficients due to the presence of Si (9%) [7]. AuN thin films has been produced for: Arc Pulsed and Reactive Ion Sputtering [8-10] and only it has been characterized for N1s narrow spectra, while in the structural part it has shown small widenings in the diffraction pattern to Au-fcc phase, therefore, the research are heading to the obtaining of some change in the Diffraction Patterns [11-13]. In this work we present the growth of Au films with N and Si in the surface through a sputtering reactive system and we show the competition of orientation crystallographic texture between the directions (111) and (311) in the Au-fcc phase and through the narrow spectres we show the Au, N and Si in the surface of the samples.

2. Experimental setup

To produce the films a Magnetron Sputtering Reactive (AJA International) was used. The films were deposited on Si wafers of 10mm² in Argon and Nitrogen environment. The substrate temperature was varied from 500 to 950°C with a step size of 100°C, only is shown the samples of 500°C to 950°C, because they were the samples that presented Au, N and Si. The total discharge conditions are shown...
in Table 1. A X-Ray Photoelectron Spectrometer - XPS (Specs) and a X-Ray Diffractometer (XRD, PANalytical) were used to obtaining narrow spectra and diffraction pattern respectively.

**Tabla1. Growth conditions for the films.**

<table>
<thead>
<tr>
<th>S</th>
<th>Current (mA)</th>
<th>Pressure (mTorr)</th>
<th>Time (S)</th>
<th>Temperature (°C)</th>
<th>% Nitrogen/Argon</th>
<th>Power supply (Watts)</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1</td>
<td>50</td>
<td>30</td>
<td>180</td>
<td>500</td>
<td>20/80</td>
<td>400</td>
</tr>
<tr>
<td>M2</td>
<td>50</td>
<td>30</td>
<td>180</td>
<td>600</td>
<td>20/80</td>
<td>400</td>
</tr>
<tr>
<td>M3</td>
<td>50</td>
<td>30</td>
<td>180</td>
<td>700</td>
<td>20/80</td>
<td>400</td>
</tr>
<tr>
<td>M4</td>
<td>50</td>
<td>30</td>
<td>180</td>
<td>800</td>
<td>20/80</td>
<td>400</td>
</tr>
<tr>
<td>M5</td>
<td>50</td>
<td>30</td>
<td>180</td>
<td>950</td>
<td>20/80</td>
<td>400</td>
</tr>
</tbody>
</table>

3. Results and discussion

In the Figure 1 is shown N1s, Si2p and Au4f narrows spectrum of the sample M5 (950°C), it is not shown the spectrums of the all samples because they present the same behaviour. The presence of N and Si compounds in the surface of the samples can be due to heating of the substrates (N and Si only are present to temperatures above 500°C), because is known that substrate temperature during the deposition process increase the particles mobility and the nucleation sites, the which lead to the densification and the appearing of new stable thermally species [14,15].

![N1s](image1)

**Figure 1.** Narrow spectra of N1s, Si2p and Au4f to samples 2A6 (950°C).

It is observed that the Si atoms act as precursor in the hybridization between N and Au, because they always appear with the presence of Si atoms. The low temperatures of the different compounds that are produced in a film depend strongly of the first nucleation stage, which is present in the interface. In the literature is found that the diffusivity and solubility of the Au atoms on Si substrate is increasing with high temperatures (temperatures near to eutectic temperature) and it improving the presence of bonds [16]. When the surface layer has Si-Au eutectic compounds (how would expect in the PAPVD systems), an alloy of liquid phase is produced, which accelerating the processes of diffusivity and mix to reach the composition saturation [17], which generate small Au-Si polycrystalline islands where is possible that the N atoms arrive to the surface [18] and they can find the best stabilization state to the film growth (called crystallization) [19]. Other authors has found that the Au-Si eutectic compound is based on Si diffusion [20], besides that thermal treatment and structural in the interface can be controlled through a-Si or c-Si compounds [21].

In the Figure 2 is shown the superposition of X-Ray Diffraction Patterns, it is observed the characteristic pattern of the Au-fcc phase for all samples. A small change on the range of 54 to 56° were observed for the samples grown to 800 and 950°C (new peaks appear), see Figure 3. In the sample M5 (950°C) a fighting on the preferential orientation between (111) and (311) direction was observed, because the texture coefficient to the M5 sample (950°C) was of 0.62, while to the other samples were approximately of 0.80 (it was found through of the crystallographic texture coefficient
between directions (111) and (311)) [11-13]. This change is a big advance to this nitrides class due to that in the literature is found only small widening of the Diffraction Pattern, we want to leave it to discussion to future papers.

![Diffraction Pattern](image)

**Figure 2.** Superposition of X-ray Diffraction Pattern.

![Superposition of directions](image)

**Figure 3.** Superposition of direction (111) and (311) and appearing of a new peak in 53 to 56° range.

4. Conclusions
The Au-Si eutectic compounds are highly involved with the N atoms, because the nitrogen appears in the surface when the Si atoms appear. N, Si and Au atoms are favoured for the increase of substrate temperature, because to that, to higher temperatures, the diffraction patterns presented changes in its peaks for example the fighting on the preferential orientation between (111) and (311) peak.

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¹ Universidad de Medellín, Medellín, Colombia.
² Centro Brasileiro de Pesquisas Físicas (CBPF), Rio de Janeiro, Brasil.

E-mail: jhquintero@udem.edu.co

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The authors would like to add an affiliation to this work. The correct list of authors and affiliations for the paper ‘Obtaining Au thin films in atmosphere of reactive nitrogen through magnetron sputtering’ is:

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J H Quintero¹, R Ospina¹²³ and A Mello³

¹ Universidad de Medellín, Medellín, Colombia.
² Centro Brasileiro de Pesquisas Físicas (CBPF), Rio de Janeiro, Brasil.
³ Universidad Industrial de Santander, Bucaramanga, Colombia.

E-mail: jhquintero@udem.edu.co