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Growth and Characterization of Gold-Palladium Thin-Films on a Silicon Substrate

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***Abstract*—this paper is concerned with the growth of Gold-Palladium thin films on a Silicon wafer by the process of Magnetron Sputtering, and the subsequent characterization of the thin films encompassing contact angle measurements, scanning electron microscopy, and Energy Dispersive spectroscopy. The results are then correlated with each other to shed light on the self-cleaning properties of the Gold-Palladium thin films.**

Keywords—thin-films; gold-palladium target; Magnetron Sputtering; contact angle; scanning electron microscopy; energy dispersive spectroscopy; self cleaning properties (key words)

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I. INTRODUCTION

Thin films are layers of material of size ranging from nanometers to several micrometers. They are deposited and processed to have properties unattainable in bulk materials. Generally, we can classify thin film deposition techniques into two broad categories: Physical Vapor Deposition (PVD) and Chemical Vapor Deposition (CVD). Although the demarcation line between PVD and CVD is not distinct, in PVD, materials comprising the thin films are delivered onto the substrate by physical means, such as evaporation, sputtering, or Laser ablation while in CVD, thin films are formed on the substrate surfaces by chemical reactions.Evaporation occurs by supplying thermal energy to the atoms of the evaporant. Alternatively, we can bombard the target surface with energetic ions, transferring momentum to the atoms and removing them from the target. This process is known as sputtering. Therefore, thin-film deposition can be performed by sputtering, without deliberately heating the target (although in actual practice, ion bombardment raises the temperature of the target, so the target is equipped with a water cooling system which cools it down during sputter deposition). In actual sputter deposition setup, the energetic ions are provided by gas discharge. The sputter gas environment depends on the materials to be deposited. Sputter deposition is normally performed at a pressure between 0.133 to 1.33Pa.The voltage applied to the target is typically around -500V.

II. THEORY

A. *Magnetron Sputtering*

To increase the deposition rate, the plasma discharge is intensified by incorporating magnets around the target. Two magnets are behind the target so that a strong magnetic field (0.2 to 0.5 tesla) within a few millimeters of the target surface is established. Electrons are trapped by the magnetic flux lines and execute oscillatory motion around the target. This increases the probability of ionization and hence the plasma density (i.e., electron and ion concentration) in the vicinity of the sputter target. The ions then accelerate towards the negatively biased target. The ion bombardment energy is approximately equal to the target potential. For example, if the target potential is -500V, the ion bombardment energy is about 500eV per singly charged ion.The total number of atoms sputtered from the target is given by two quantities: ion current and sputter yield. The ion current gives the number of ions striking the target surface per second.The sputter yield is defined as the number of atoms removed from the target surface per incident ion. Sputter yield initially, increases proportionally with ion current, but decreases after a certain limit. The trend can be understood as follows: as the incident ion energy increases, more energy is available to transfer to the target atoms, resulting in an increased sputter yield. As the ion energy increases further, incident ions penetrate deeper into the target, so that momentum transfer occurs deeper within the solid. It becomes difficult for the sputtered atoms to escape the target, resulting in a decreased sputter yield. Since sputtering involves the removal of atoms, it is related to the heat of vaporization. For Argon gas, higher heat of vaporization results in lower yield. In the sputtering process, Substrates are placed into the vacuum chamber and are pumped down to their process pressure. Sputtering starts when a negative charge is applied to the target material (material to be deposited), causing a plasma glow or discharge.

B. *Scanning Electron Microscope*

In the Scanning Electron Microscope (SEM) and electron micro-probe, the sample is bombarded by a focused beam of electrons. Most incident electrons, rather than penetrating the sample in a linear fashion, interact with specimen atoms and get scattered.Scattering may be elastic or inelastic. Inelastic interaction produces a lot of effects: secondary electrons, back scattered electrons, cathodoluminescence, continuum x-ray radiation, and phonons. Heating effect stays inside the sample; x-rays come out from deep within the penetration volume, back scattered electrons from shallower levels, and secondary electrons and cathodoluminescence from the incident point.

The volume of material analyzed, by an SEM, depends upon many factors. The interaction volume is limited by the energy loss through inelastic interaction. A single electron may be scattered many times. The depth of electron penetration of an electron beam and the volume of sample with which it interacts are a function of its angle of incidence, the magnitude of its current, the accelerating potential, and the average atomic number of the sample. In a standard SEM, the electron gun shoots a beam of electrons under a fixed accelerating potential (7 to 10 kilo electron volts in our case). The back scattered electrons are detected by a detector. These are then used to form the image. The whole SEM chamber is vacuumed during operation, and filled with nitrogen gas when not in use. Since the energy and intensity of secondary electrons depends upon the surface morphology, and topography, we can get a clear image of them. The X-rays are also analyzed by the Energy Dirspersivespectroscope (EDS), for the elements present in the interaction volume. We have used a field emission scanning electron microscope (FESEM). In this, tunneling effect of quantum mechanics is observed. This is the source of the electrons from the electron gun. For this, we need at least 1μPa pressure. Electron gun alignment is allowed to maximize the electron penetration. The secondary electrons are refocused by magnetic lenses before they are passed through the detector.

C. *Contact angle measurement*

The contact angle of a liquid on a surface means the angle made by the tangent to the surface of the droplet at the point of contact between the liquid and the surface. It helps to study the hydrophilic or hydrophobic nature of the films, i.e., water repellent, or water wettable. It is a surface energy phenomenon. If the liquid-solid surface energy is greater than the liquid-gas surface energy, then the shape of the liquid droplet is such that to minimize the Liquid-Solid surface and thus the film is hydrophobic. If it is the other way, then the film is hydrophilic. It is dependent on the surface morphology and topography as well.

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III. EXPERIMENT

The experiment has three stages: (i)Cleaning, (ii)Sputter deposition, and (iii)Characterization.Each of the steps is described as follows:

A. *cleaning of the substrate*

The cleaning involves the following steps: First the silicon substrates were cleaned with soap to remove oil. Secondly, they were put in distilled water with the help of tweezers, and sonicated by an ultrasonic cleaner for ten minutes.The above procedure was repeated by replacing the distilled water by acetone.The same process is again repeated in distilled water, but for five minutes.Then the samples were blown dry by a compressed air blower, and the substrate cleaning process was over.

B. *Sputtering process*

- The cleaned samples were kept on the stage of the magnetron sputtering machine, and the vacuum chamber was closed.
- Then the vent and leak knobs were closed tight and vacuum pump was started till the pressure dropped to 2 Pascal.
- The plasma was then set by letting in the argon gas till 8 Pascal pressure. The plasma glows pink.
- Then the sputtering process was initiated.
- The process was repeated for 600, 900 and 1200seconds for separate sets of the silicon substrates, and then the deposited thin films were taken for characterization.

C. *Characterization of the depositied thin films*

Characterization of the thin films was done in three steps: (i) The water contact angle measurement of the thin films, (ii) The S.E.M. analysis of the thickness and morphology of the three sets of the thin films, (iii) The EDS plot obtained by the spectrometer attached to the FESEM.

IV. OBSERVATIONS AND CONCLUSIONS

A. *Observations:*

The experimental conditions, measured thickness (by SEM analysis) and observed contact angle values are as given below in table.1.SEM and contact angle images (inset) are shown in Figure.1(a) (top left), (b) (top right) and (c) (bottom). Figure.2 shows the EDS plot.

TABLE I. EXPERIMENTAL CONDITIONS, THICKNESS AND CONTACT ANGLE

	EXPERIMENTAL CONDITIONS, THICKNESS AND CONTACT ANGLE				
	pressure	***current***	***time***	***thickness***	***Contact angle***
Sample no.	(pa)	(mA)	(s)	(nm)	(degrees)
01	8	4	600	44.854	105.34
02	8	6	900	73.180	117.34
03	8	6	1200	172.638	111.34

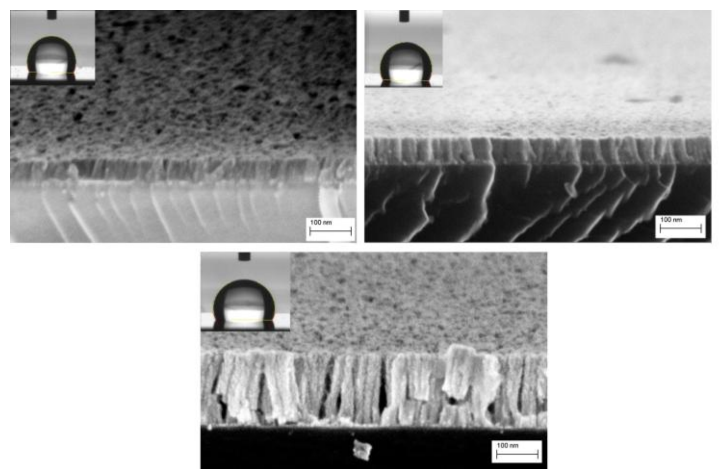

Fig. 1. (a) top left image:44.854 nm thickness sample (cross section view). Inset: contact angle image. (b) top right image: 73.18 nm thickness sample (cross ection view). Inset: contact angle image. (c) bottom image: 172.653 nm thickness sample (cross section view). Inset: contact angle image

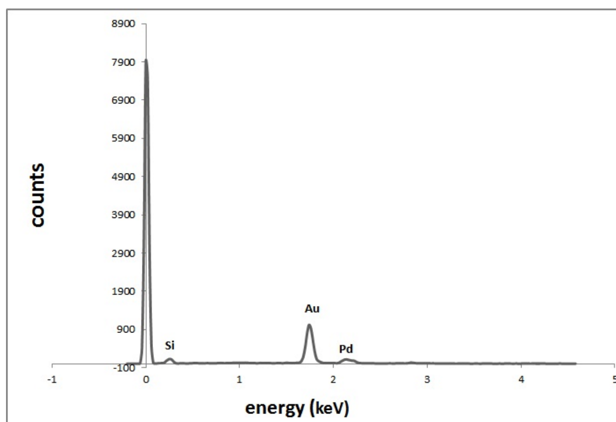

Fig. 2. Energy Dispersive Spectroscopy Plot for the Thin Films.

B. Conclusions:

Figure 1 clearly shows perfect columnar growth of the thin films on the Silicon substrate wafer. The EDS plot as given in Figure 2 and the contact angle versus Film Thickness Plot as shown in Figure.3 below, conform to established theories. The SEM morphology images, shown in Figure 4, and Figure 5 show a typical island nucleation and growth mechanism of the development of the thin film. The contact angle values clearly indicate the hydrophobicity of the Gold-Palladium Thin-films on silicon substrate. the morphology is similar for all samples, however the compactness, and hence the roughness of the films vary with the thickness of the film deposited. Thus, the contact angle values depend on both the roughness and thickness of the thin film. hence, the hydrophobicity (which is a measure of the self-cleaning properties of the film) varies both with the thickness and roughness of the film. further investigation is required with the help of Atomic Force Microscopy to determine the surface roughness and correlate the same with the contact angle values.

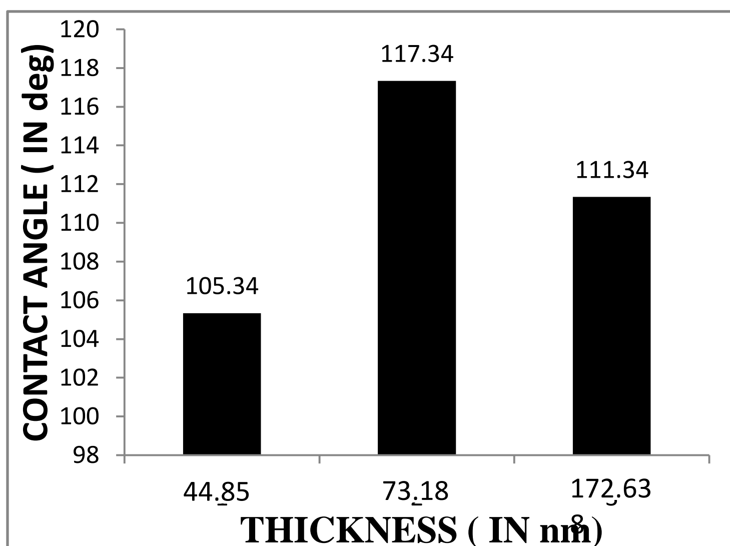

Fig. 3.Contact angle versus thickness of the films.

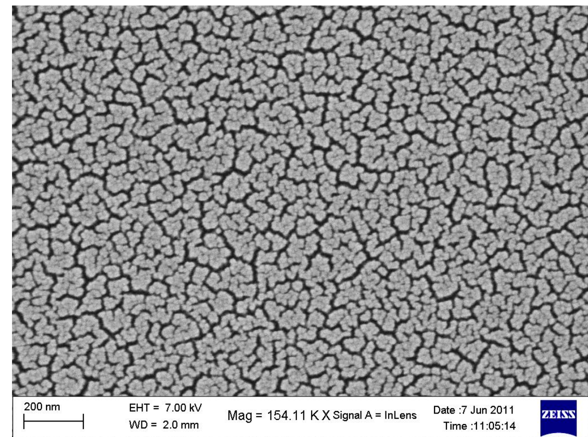

Fig. 4. Morphology of the Gold-Palladium thin film (same for all three samples)

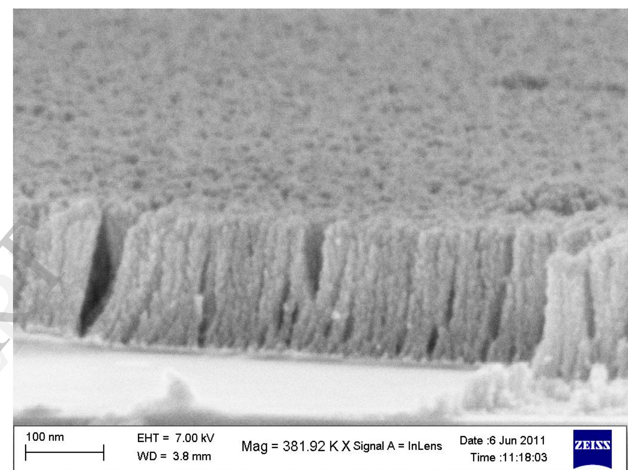

Fig. 5. Cross sectional View of the 172.683nm thickness sample.

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