Morphological Study Of Palladium Thin Films Deposited By Sputtering.

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ABSTRACT
This paper presents a morphological analysis of thin films of palladium (Pd) deposited on a substrate of sapphire (Al₂O₃) at a constant pressure of 3.5 mbar at different substrate temperatures (473 K, 523 K and 573 K). The films were morphologically characterized by means of an Atomic Force Microscopy (AFM); finding a relation between the roughness and the temperature. A morphological analysis of the samples through AFM was carried out and the roughness was measured by simulating the X-ray reflectivity curve using GenX software. A direct relation between the experimental and simulation data of the Palladium thin films was found.

1. Introduction

A seek for clean and renewable energy systems has increased in the last years due to environmental concerns such as emissions of pollutants produced by fossil fuels. This is why the study of phenomenon occurring in the hydride systems have returned to be important. Due to its high hydrogen absorption capacity, palladium turns to be a material of great interest, both for its structural properties, as for its optical [1, 2], physical and chemical [3, 4] properties. These properties change when the palladium films are under hydrogenated environments [5 -8]. Due to the palladium ability to absorb hydrogen, it is considered as a potential hydrogen container, which acts as a membrane separating the hydrogen atoms [9], and reducing its explosive nature. On the other hand, changes in the optical properties of palladium result in increase of the reflectance. It is applicable also to switchable mirrors, in which cases; its use allows considerable energy savings.

The interest in the study of the palladium thin films morphological properties relies in the importance of the morphology of the films in the analysis of surface properties. The main interest is triggered because of the changes generated by the cycles of loading and unloading of hydrogen [10], the possibility of modifying the surface conditions of the films by different methods, the impact on the rate of diffusion of hydrogen [11], and changes in optical properties and reflectance which changes significantly the hydrogen found in the palladium thin films [12]. This paper analyzes the influence of temperature on the roughness of palladium thin films and there is a correlation between the roughness and grain size.
2. Samples

Palladium thin films were deposited by sputtering at a constant pressure of 3.5 mbar of Ar gas, with a thickness of 10 nm, on a substrate of sapphire (Al₂O₃), with a preferential orientation (1 1 1). The Palladium target was deposited with a purity of 99.95%. The deposition process of films was performed at different temperatures: 473 K, 523 K and 573 K.

3. Results And Discussion

The study of the morphology of Pd thin films was carried out by AFM contact mode. Figure 1 presents the different AFM images of the samples in 3D, deposited at a constant Argon pressure of 3.5 mbar and at different temperatures: 473 K, 523 K and 573 K respectively. The scan of the samples was carried out in an area of 2.0 µm x 2.0 µm. The microscopic images of the samples reveal little homogeneous surfaces and its roughness increase within increasing the temperature.

![AFM images of thin films](image)

**Figure 1** AFM images of thin films deposited at 3.5 mbar argon pressure and at temperatures of: a) 473 K b) 523 K c) 573 K.
Fig. 1 shows the morphological differences in palladium thin films as the temperature is varied. The sample grown at 473 K is the most homogeneous surface, with a root mean square roughness, \( R_q \), of 1.21 Å (figure 1a). In Fig. 1b, the surface of the sample grown at 523 K with a higher roughness \( R_q = 2.49 \) Å and a similar form grain is observed. And the surface of the sample grown at 573 K presents the highest roughness \( R_q = 4.25 \) Å and it has a columnar growth as shown in Fig. 1c. It can be seen from Fig. 1a and c, how these surfaces have smaller grain sizes than the surface of sample grown at 523 K (Fig 1b), this is confirmed with the texture profile of the cross-section area of the AFM images, obtained by the Gwyddion software [13], which shown in Fig 2, which allows obtained statistically the grain size of Pd thin films.

![Texture profiles of AFM images of palladium thin films grown at. a) 473 K b) 523 K c) 573 K.](image)

**Figure 2.** Texture profiles of AFM images of palladium thin films grown at a) 473 K b) 523 K c) 573 K.
Fig. 2 shows the texture profiles with a random surface growth of films at low temperatures, which tend to have a define grain growth form, but at higher temperatures, a columnar growth process predominates. Furthermore, these profiles reveal no homogeneous surfaces. The trend in the change of the grain size as temperature increases is not obvious. This is probably due to the effects of pressure which wasn’t enough for the diffusion process of present adatoms, in such a way that a uniform growth couldn’t be obtained. This can be reflected in roughness increase within increasing the temperature, which allows the fast adherence of the atoms during the deposition process.

Table 1. Correlation between the roughness, grain size and variation in temperature.

| Temperature (K) | Roughness (RMS) (Å) | Grain Size (± 4.8 nm) |
|----------------|---------------------|----------------------|
| 473            | 1.21                | 364                  |
| 523            | 2.49                | 504                  |
| 573            | 4.25                | 130                  |

Table 1 shows the correlation between the roughness, grain size and variation in temperature. The simulation of reflectivity curve using GenX software[14] is in well agree with the experimental reflectivity curve of low angle X-ray diffraction as can be seen in the Fig. 3 from the simulations of palladium samples the roughness was obtained. These results are in accordance with the results obtaining by AFM analysis, which demonstrate the exact of the x-ray reflectivity curve measurement. The effects of the Al₂O₃ substrate reflectivity measurements of the Pd thin films were not analyzed in this work, however, this can be found in the literature related work[15].

Figure 2. Simulation of the reflectivity of Pd thin film deposited at 573 K. The result obtained by simulation is in accordance with the experimental data obtained by AFM analyze (Rₖ = 4.25 Å). The experimental data is in blue and red curve simulation.

A PdO layer was identified on the Pd sample surface by the simulation curve, by means of the input information to about the present layers on the sample. This probably was due to the contamination of the samples in the laboratory when they were characterized.
This could be a factor of great interest in the results of subsequent analysis of the optical properties of the films in the presence of hydrogen [16].

4. Conclusions

In this paper shows the influence of temperature on the morphology of palladium films by determining the increase in roughness with increasing temperature, with little homogeneous textures and an unclear trend in the size of grain. There was also a good agreement with the simulation data by the software GenX of the roughness of the films, which identified the formation of a layer of palladium oxide (PdO) due to the contact of the film with the environment.

5. Bibliography

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