Improving the structural characterization of supported on glass gold nanoparticles using Atomic Force Microscopy at vacuum

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ABSTRACT

Surface characteristics of gold nanoparticles [1] at ambient conditions and in vacuum of the order of 10^{-4} Pa are compared using Atomic Force Microscopy topographic images in tapping mode. The results show the improvements of the images got at lower pressure than that obtained to normal laboratory conditions. The image got at lower pressure shows more clearly the like spheroid shape characteristics of the particles as predicted by the SPR experiments and theory [1]. Profiles and RMS roughness of the particles are also measured.

Keywords: Atomic Force Microscopy, Gold nanoparticles, Tapping, Vaccum Techniques.

1 INTRODUCTION

It is a well known fact that as deposited over quartz slides (or glass) of gold thin films of thicknesses <5 nm are discontinuous and form islands [2]. When this gold thin films are thermally treated at 600 °C during 5 minutes, the formation of like spheroids shape nanoparticles occurs [2]. Warmack and Humphrey in reference [2] and the references therein, discuss optical excitation of surface plasmons in such spheroids shape gold nanoparticles. The generation of surface plasmons in the gold nanoparticles is revealed as a peak in their optical absorption spectrum. When the gold nanoparticles are immersed in different media a red shift in their absorption spectrum typically occurs. Therefore, the absorption spectra can be used as a tool for sensing dielectric changes in the media surrounding the particles [1].

On the other hand, besides the multiple applications of these gold nanoparticles [1], a complete surface characterization using Microscopy tools has not been carried on yet. For example, P. Royer et. al. [3] reported a Scanning Electron Microscopy (SEM) image of like spheroids shape silver particles viewed at 60 degree from normal. The disadvantage of this SEM image is that the length of the minor semi-axe can not precisely be measured. For gold particles, F. Meriaudeau et. al. [4] reported an Atomic Force Microscopy (AFM) image taking it at ambient conditions and never talks about the length of their semi-axes. Obviously, when AFM images are taking at ambient conditions the size and distribution of the particles is not real because of the humidity layer over the surface [5]. That means the measurement of the semi-axes and the size distribution of the like spheroids gold particles has not been reported yet. In this work, using AFM images at vacuum conditions, the length of the semi-axes and the average size of the like spheroids gold particles are measured. The advantage of the AFM images taken at vacuum with respect to ambient conditions is also discussed.

2 EXPERIMENTAL

Similar to the procedure followed for the researching in reference [1], high-purity gold thin films of 5 nm thicknesses were evaporated on cleaned corning 7059 glass substrates in a thermal evaporator system. The operating pressure was 2.5×10^{-4} Pa. A crystal thickness monitor (Mastek Inc.) was used to control the thickness of the evaporated films with a precision of 1Å. The glass substrates were carefully cleaned in solvent steps of microcleaning solutions [6] starting with distilled water and followed by acetone, ethanol, and finalized with isopropanol, then dried with high purity nitrogen. For each solvent, except water, the substrate was immersed in the cleanser and set in an ultrasonic bath for 15 minutes. After deposition, the gold films were annealed at 600 °C during 5 min in order to get a distribution of spheroidal gold particulates [1]. A JSPM-5200 Scanning Probe Microscopy (SPM) from JEOL Inc. was used to get the surface characterization. The surface characterization was made at laboratory ambient (23 °C and 40 % of humidity) and vacuum of the order of 1×10^{-4} Pa conditions.

3 RESULTS

Figure 1 shows Atomic Force Microscopy images got by means of tapping mode for as deposited 5 nm thickness thin film (top) and after it was annealed to 600 ^{o}C during 5 minutes (buttom). The scanned area was 500 × 500 (nm). The images were taking at laboratory ambient conditions about of 23 $^{\circ}$C and 50 % of humidity. It can be observed from the top image of this figure the irregular topography of the as deposited film surface. From the buttom image well defined spheroids shape particles are observed besides the humidity layer present over the surface. Obviously, more defined spheroidal shape of the gold particles can be got if the image should be obtained at lower humidity conditions.

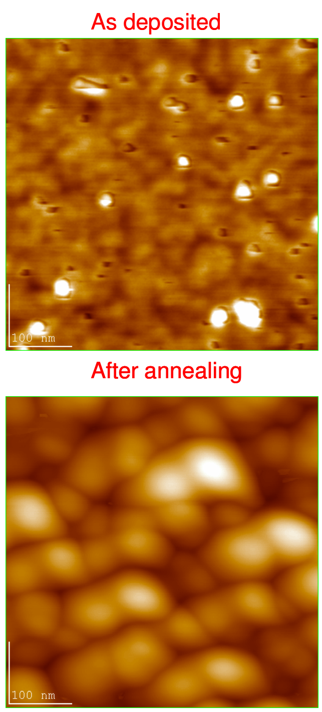

Figure 1: Atomic Force Microscopy images for as deposited 5 nm thickness thin films (top) and after it was annealed at 600 ^{o}C during 5 minutes (buttom). The scanned area is 500 × 500 nm ogt at laboratory ambient conditions.

Figure 2 at the buttom shows the topography of the particles got at 10^{-4} Pa of pressure. The image at the top of the figure 2 was got at laboratory conditions. The image at the top in figure 2 is the same as the buttom image of Figure 1, which means the scale is 500 × 500 nm. It can be observed a better quality of the image scanned at vacuum. Also, the definition in size and shape of particles is better as shown by the cluster identified by the white circle and line. Letters show the corresponding particles of the cluster. Unfortunately, it was not possible exactly scanning the same area after taking the vacuum. The cantilever suffers a friction force when it is approaching to the surface in air, and almost cero friction force when it is approaching to the surface in vacuum, which means the position of the tip over the surface is not the same. The image at buttom of the figure 2, shows clearly two average particle size of the spheroids.

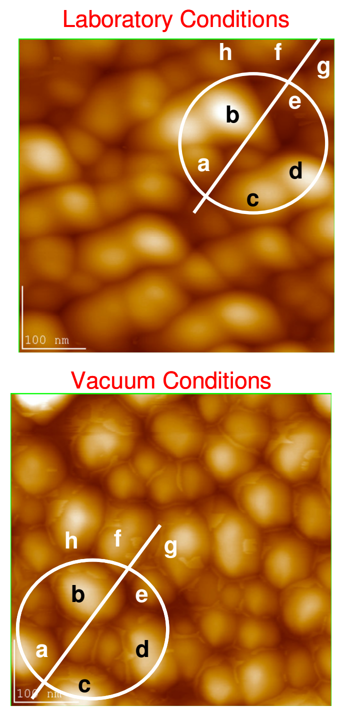

Figure 2: Comparison of images got at laboratory (top) and vaccum (bottom) conditions. The scale of both images is 500 × 500 nm.

Figure 3 shows the resulted length of the axes for these two kinds of particles. The green color profile show a mayor axe about 90 nm of average length and a minor axe about 8 nm of average length. The red and blue profiles show a mayor axe length approximately of 60 nm and 6 nm averagelength of the minor axe.

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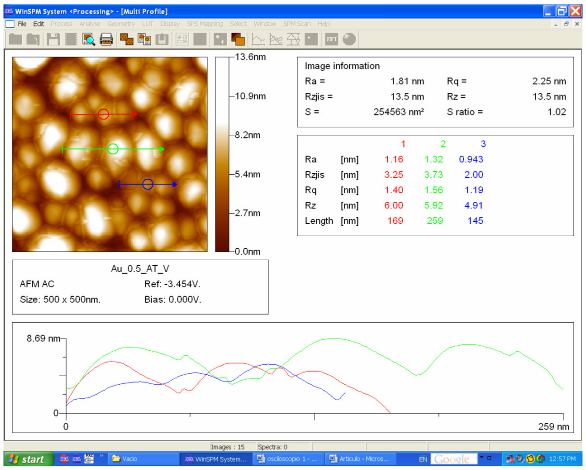

Figure 3: Profiles in three different zones identifying the two principal averaged lengths of the axes of the particles.

4 CONCLUSIONS

A study of the humidity effects on the Atomic Force Microscopy images of supported on glass gold nanoparticles was made. It was observed better images got at lower humidity (or at vacuum) than those got at laboratory conditions. It was very well determined the size and shape of the gold nanoparticles resulting in two sizes of spheroids. The averaged lengths of the axes of the first type of nanoparticles are 90 nm of major axe and 8 nm of minor axe. The second type of particles has 60 nm of major axe and 6 nm of minor axe. The shape and size got for the gold nanoparticles will be of great utility for the determination of the normal vibrational modes of the surface plasmons at the collective oscillations of the conduction electrons of the metal.

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REFERENCES

[1] N. Muñoz Aguirre, E. Lopez Sandoval, A. Passian, C. Vazquez López, L. Martínez Pérez and T. L. Ferrell, Sensors & Actuators B 99, 149, 2004.

[2] R.J. Warmack, S.L. Humphrey, Observation of two surface-plasmon modes on gold particles, Phys. Rev. B 34, 2246–2252, 1986.

[3] P. Royer, J.P. Goudonnet, R.J.Warmack, T.L. Ferrell, Substrate effects on surface plasmon spectra in metal-island films, Phys Rev. B 35, 3753–3759,1987.

[4] F. Meriaudeau, A. Wig, A. Passian, T. Downey, M. Buncick, T.L. Ferrell, Gold island fiber optic sensor for refractive index sensing, Sens. Actuators B 69, 51–57, 2000.

[5] Ernst Meyer, Scanning Probe Microscopy: The Lab on a Tip. Springer-Verlag Berlin Heidelberg New York 2004 (IBBN 3-540-43180-2).

[6] Micro Concentrated Cleaning Solution, International Products, Burlington, NJ, USA.