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Title: Design and Construction of an ALD Reactor by Growth of Al2O3 Nanostructure Films

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Introduction

The atomic layer deposition (ALD) technique has its origin in Finland in 1974 by Toumo Suntola, et.al. and it is used for the growth of ultrathin films with high uniformity for multiple applications. (T. Suntola, et.al. 1977)

The process is repetitive and sequential; therefore it is possible to control in more detail the thickness of the films with atomic precision. The incorporation of different precursors in the ALD system, favors the possibility of alternating several films of different materials (G. Steven M., 2010).

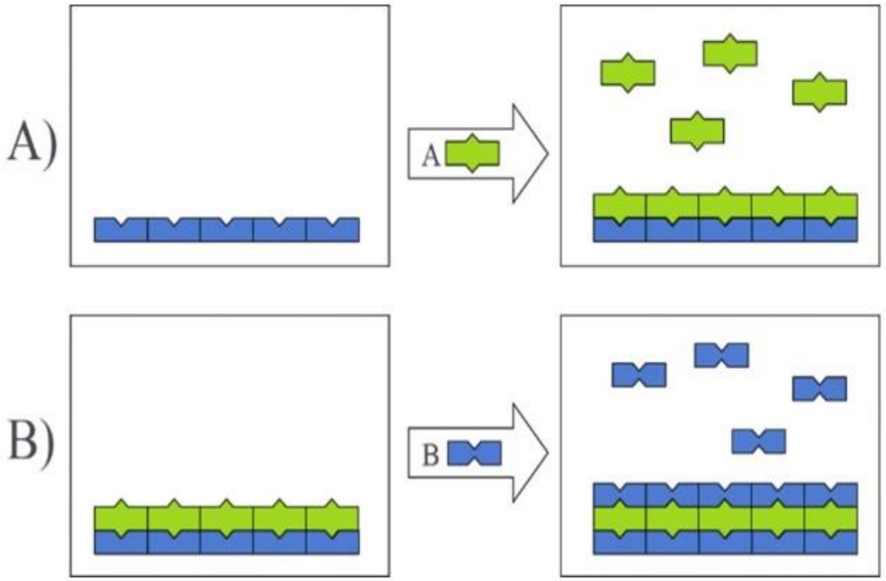


Figure 1 *Atomic layer deposition (ALD) diagram*

Methodology

a) Materials

2-way and 3-way ALD valves

Bellows Valve

Cylinders for sampling

Thermal Tapes

Vacuum Sensor and Gauge

AALBORG GFC17 Mass Flowmeter

EDWARS RV3 Vacuum Pump

Temperature controllers

Computer

Arduino / Genuine Uno Hardware

12V 5A power supply

Bank of relays 5V, 10 A

LabVIEW 2015 programming software

Front panel

Precursors and reagents (TMG, TMI, TMA, NH_3 and Ar)

Methodology

b) Method

The ALD reactor was first designed under the specifications based on the state of the art of the art. The design was made in Solid Works to be presented and describe the operation from the flow of precursors to their incorporation into the surface of the substrate.

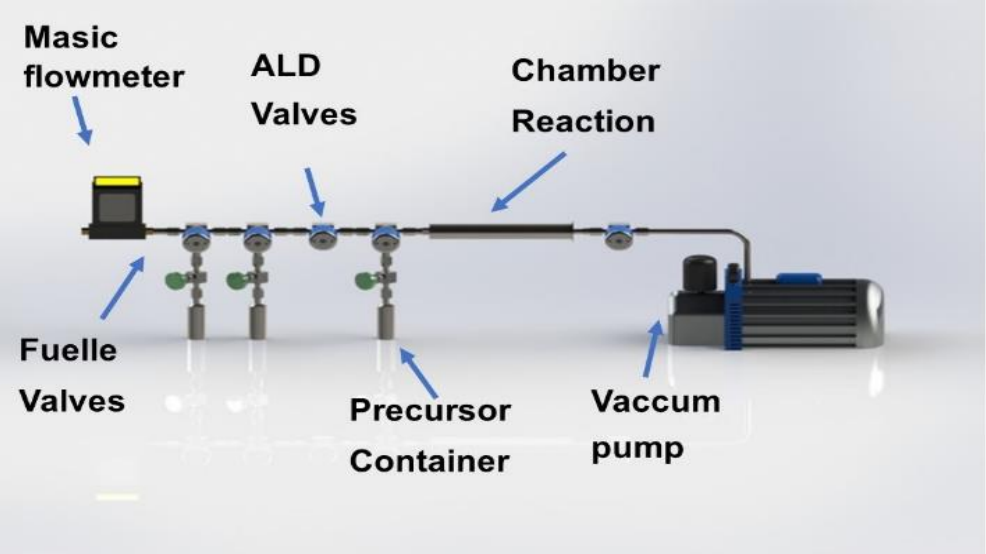


Figure 2 *ALD system designed by SolidWorks.*

Methodology

The ALD reactor has a series of assemblies ranging from a 100 sccm mass flow meter, bellows valves, ALD valves, several precursor container cylinders, the reaction chamber and a mechanical vacuum pump.

Subsequently, in figure 3 the electro-pneumatic and control design was carried out, which are in charge of controlling the work of opening and closing the ALD valves, both to release the precursors and for cleaning.

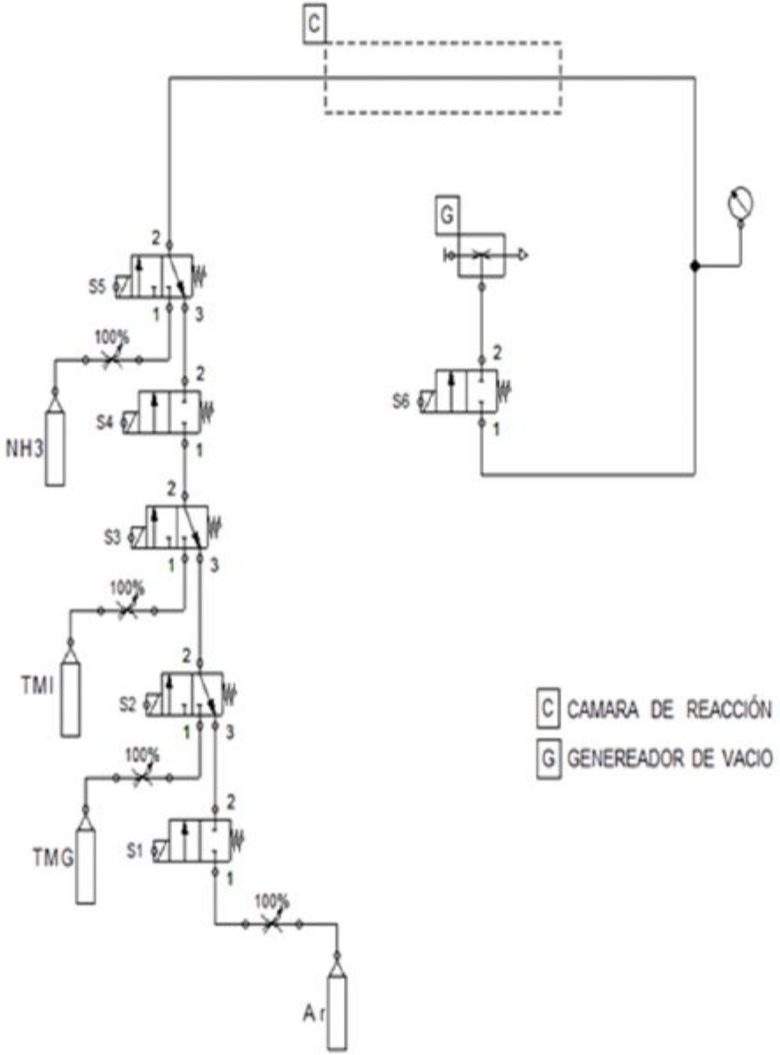


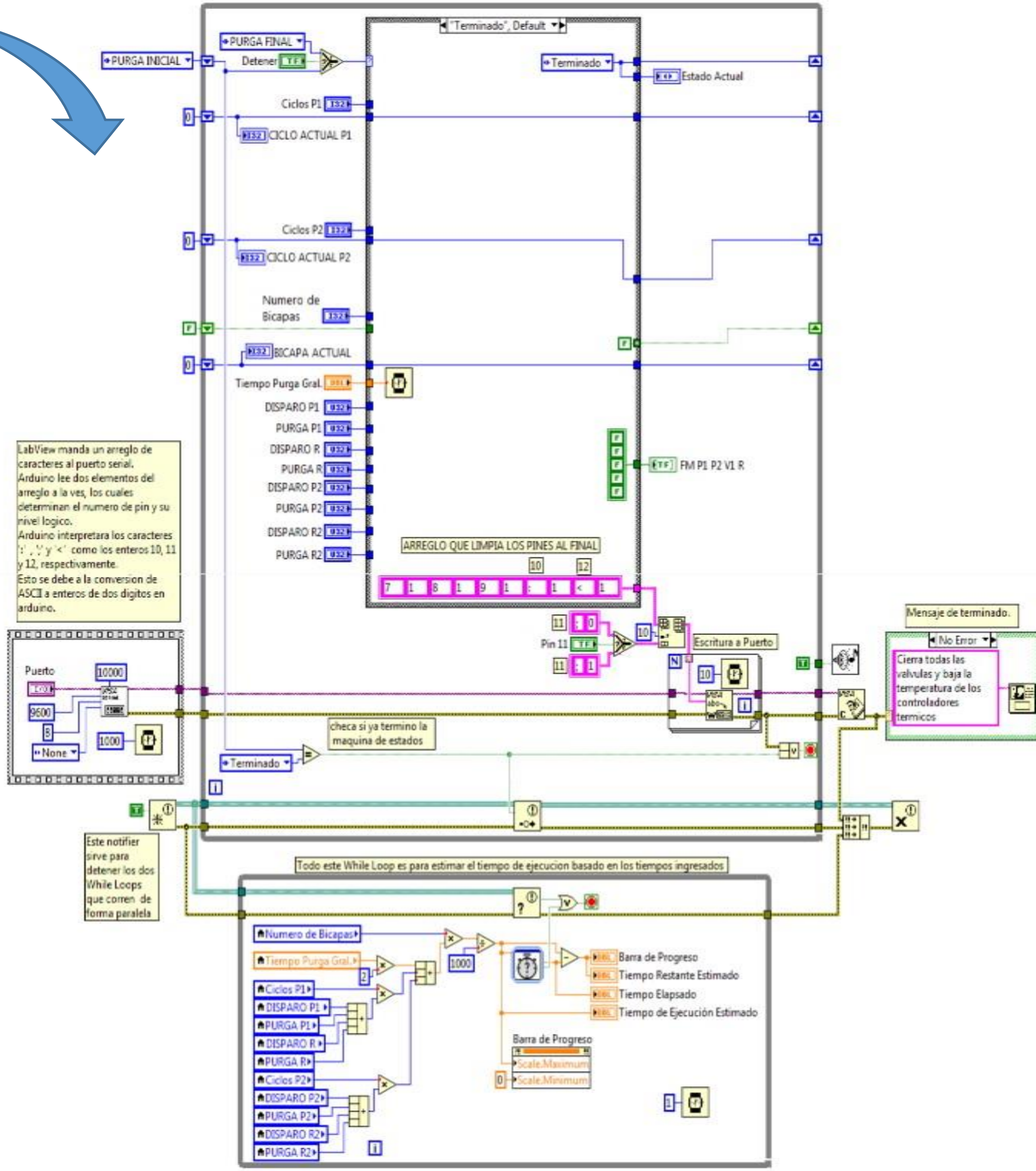
Figure 3 *Electro-pneumatique designee of ALD system.*

Methodology

The cabinet contains temperature controllers for thermal tapes, a microprocessor as an interface with LabVIEW that sequentially actuate the ALD valves, under set trip times with a bank of relays connected to a DC voltage source which feeds all the components.

In figure 4 show the LabVIEW programming was carried out at the user's disposal and is divided into two parts: a) control panel and b) block diagram. The block diagram develops the programming code for the manipulation and execution of the valve operation (number of cycles, exposure time of the precursor, reagents, purge gas, vacuum and an emergency button). An Arduino is integrated into the flow diagram as a data acquisition card.

Figure 4 *Block diagram developed with LabVIEW for the ALD reactor.*



Results

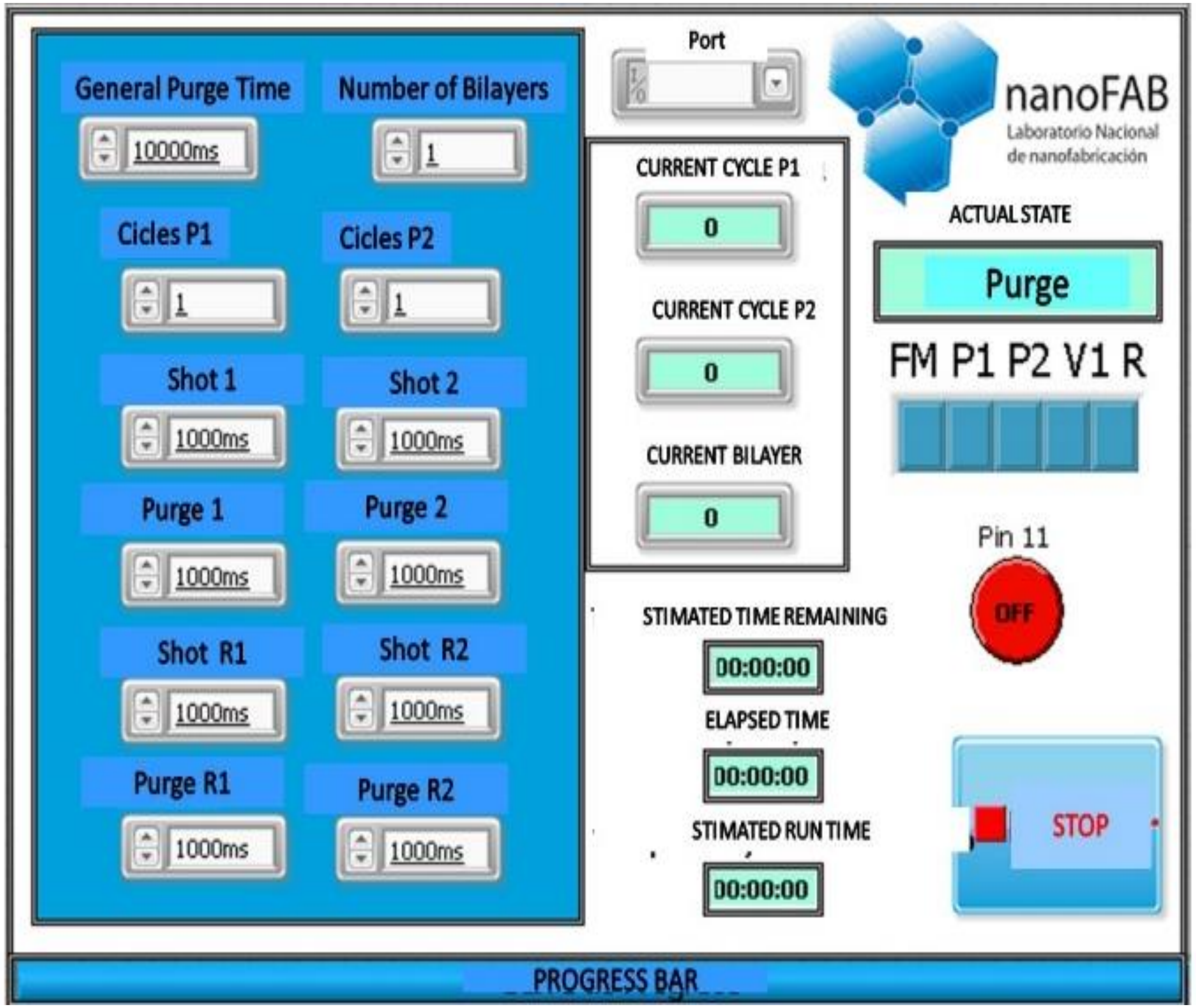
The ALD reactor was assembled from the mechanical parts, the electrical connections, gases and controllers, as well as the computer with which it has the software designed to send command programming and data capture. Figure 5 shows the ALD reactor with all its assembled components.

Figure 5 *ALD reactor complete.*

Results

The software has the programming commands of:

1. Purge time
2. Number of bilayers
3. Number of cycles
4. Precursor firing time
5. Purge time
6. Working times
7. Off button.



Results

A reactor workbook was described ALD, where its execution is summarized in 14 steps to process sample, which are the following:

A) Sample introduction

1. Fix substrate to sample holder
2. Introduce it to the camera
3. Reach the desired vacuum
4. Opening of material valves

B) Parameter settings

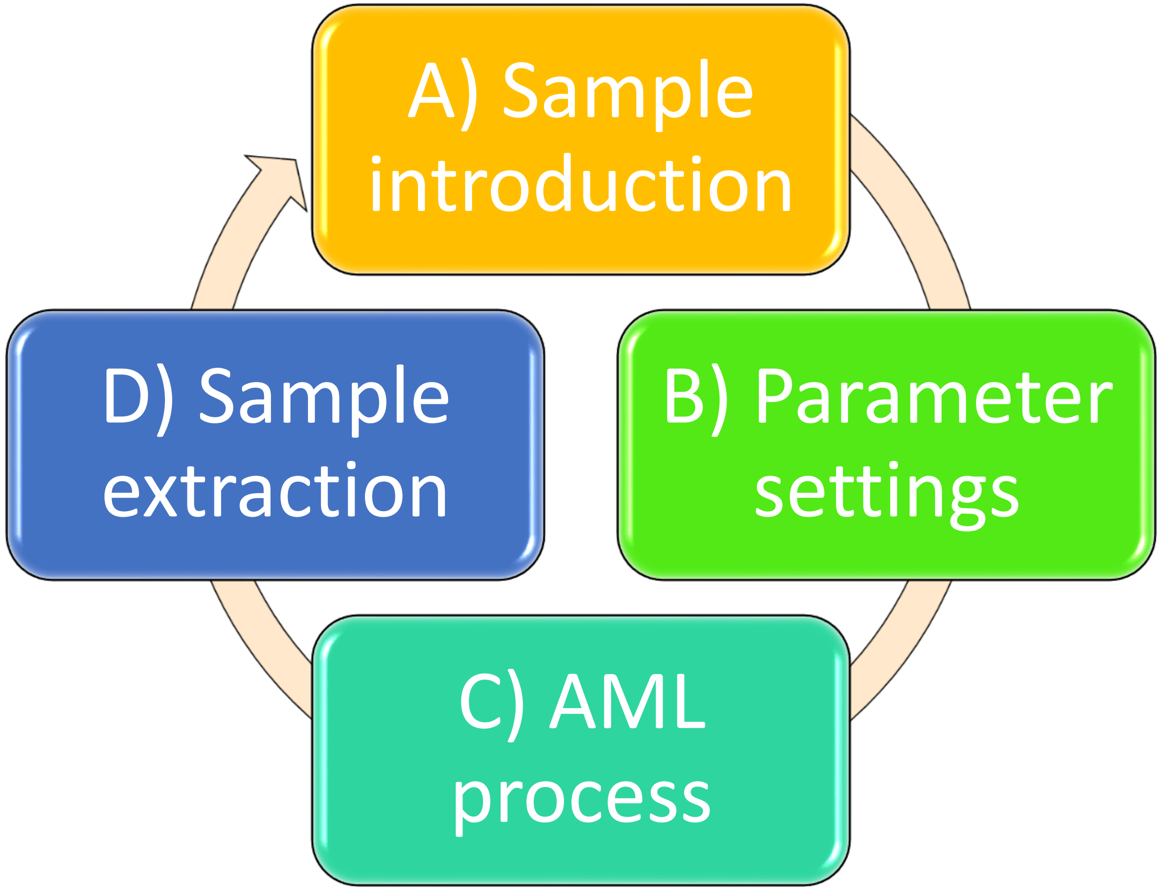
5. Temperature in the 4 zones
6. Number of cycles
7. Shooting time

C) AML process

8. Initial purge
9. Execution of ALD cycles
10. Final purge

D) Sample extraction

11. Close material valves
12. Lower temperature
13. Ventilate with N2
14. Take Samples



Results

The results of handling the ALD reactor and the synthesis of ultrathin films are described below:

A) It was possible to deposit aluminum oxide (Al_2O_3) at two points in the reaction chamber. The experiment was carried out under 240 cycles, 200 °C in the reaction chamber, water and trimethyl-aluminum (TMA) as precursors and swept with N_2 flow.

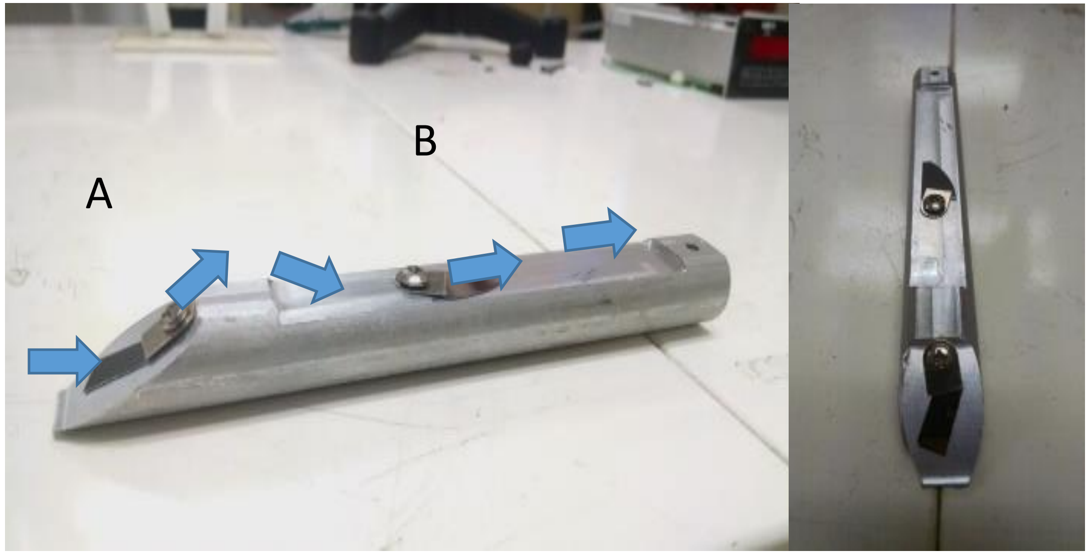


Figure 7 *ALD substrate holder. Two locations of substrate A and B are shown. The flow of precursors sticks directly to substrate A and rebounds to substrate B.*

Results

The films deposited on substrates A and B were analyzed by ellipsometry (Philips PZ2000, HeNe 632.8nm) obtaining the following averages of thicknesses obtained in 16 points

Sample	Thickness (nm)	Growth Range (Å/ciclo)
Sample A	29.4	1.22
Sample B	28.5	1.19
Differences	.9	.03

Table 1 *Ellipsometry measurement of sample of substrate A and B.*

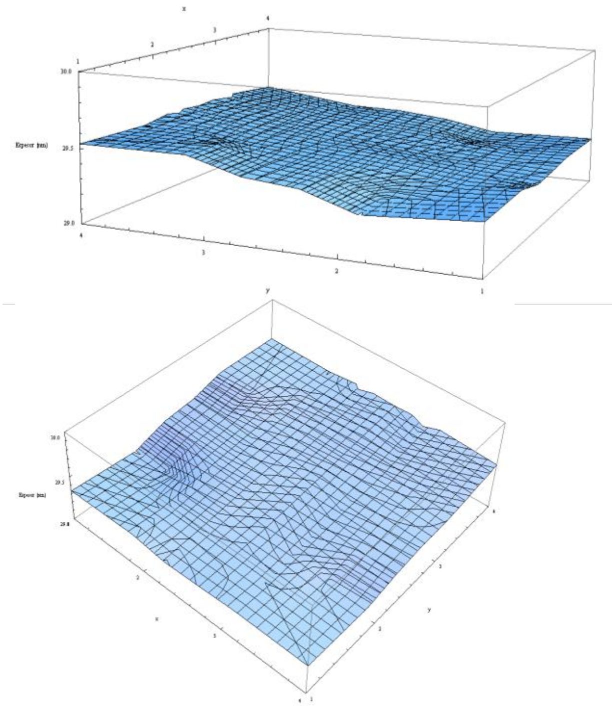


Figure 8 T*opography graph of ALD de Al_2O3 deposits.*

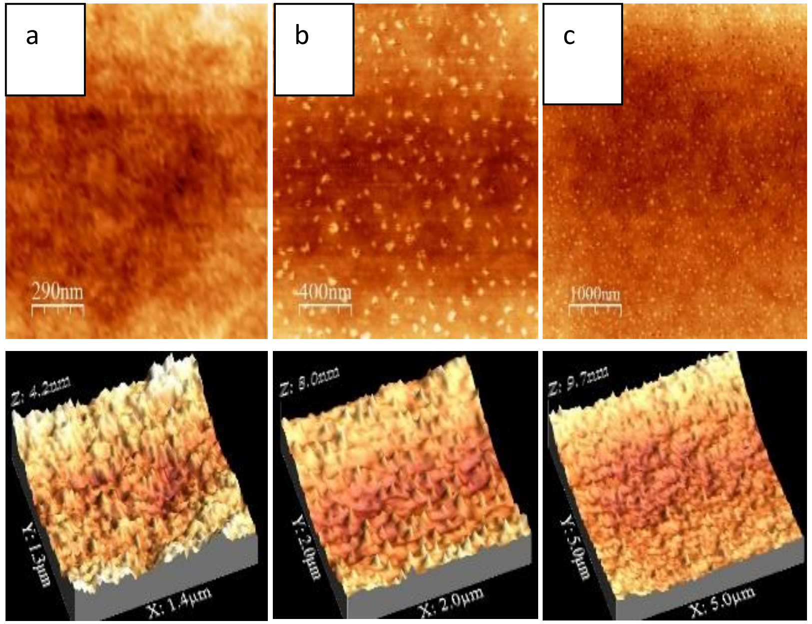


Figure 9 *(a) Reference sample without ALD deposit of Al2O3, (b, c) silicon nanoparticle films subjected to 40 ALD cycles of alumina*

Conclusions

In this project, the objectives in the design, manufacture and start-up of an ALD reactor were met, with an efficient and high-precision system for thin-film deposits. This ALD reactor is fully reproducible and scalable which makes it perfect for commercial and industrial applications. Likewise, the ALD reactor turned out to be an essential tool in the investigation of nanostructured materials by combining layers of thin films of different materials such as oxides and nitrides in the nanometer range and with the possibility of controlling ultrathin film thicknesses in the order of Ångströms.

The characteristics of this ALD system can lead to the discovery of new properties in semiconductor materials for application in optoelectronic devices such as high-efficiency solar cells and solid-state lighting.

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