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Design and Construction of an ALD Reactor by Growth of Al_2O_3 Nanostructure Films

Diseño y construcción de un reactor ALD por crecimiento de películas de nanoestructuras de Al_2O_3

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Abstract

Objective: This project focuses on designing, building and commissioning work the atomic layer deposition (ALD) reactor for Al_2O_3 ultrathin film, which it will be contain specific components and a system's own control unit. Methodology: The ALD reactor was designed under a system to minimize components, flow lines and connections; to reduce manufacturing costs, volume of precursors, among others. Currently, ALD reactors are expensive to sell, maintain and replace parts. The design and manufacture of the ALD reactor manufactured at the University of Sonora (UNISON) is based on the state art with sequential binary reactions of the precursors, for the proposal for the manufacture of solar cells. Contribution: It was possible to build and commission the ALD reactor for the deposition of ultra-thin films, with the characteristics of being reproducible and scalable, which makes it attractive for commercialization. The homemade ALD reactor at UNISON is considered a very interesting equipment for the semiconductor research area, since it is possible to combine different types of materials in the form of films such as oxides and nitrides in the order of Angstroms (Å).

ALD, Reactor, Thin films

Resumen

Objetivo: Este proyecto se centra en el diseño, construcción y puesta en marcha del reactor de deposición de capas atómicas (ALD) para película ultrafina de Al2O3, que contendrá componentes específicos y una unidad de control propia del sistema. Metodología: El reactor ALD fue diseñado bajo un sistema para minimizar componentes, líneas de flujo y conexiones; para reducir los costos de fabricación, volumen de precursores, entre otros. En la actualidad, los reactores ALD son caros en cuanto a su venta, mantenimiento y sustitución de piezas. El diseño y fabricación del reactor ALD fabricado en la Universidad de Sonora (UNISON) se basa en el estado del arte con reacciones binarias secuenciales de los precursores, para la propuesta de fabricación de celdas solares. Contribución: Se logró construir y poner en marcha el reactor ALD para la deposición de películas ultrafinas, con las características de ser reproducible y escalable, lo que lo hace atractivo para su comercialización. El reactor ALD casero de UNISON se considera un equipo muy interesante para el área de investigación de semiconductores, ya que es posible combinar diferentes tipos de materiales en forma de películas como óxidos y nitruros del orden de los Angstroms (Å).

ALD, Reactor, Películas finas

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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 state of the art of the ALD process involves a series of precursors which are released sequentially under controlled conditions of surface saturation of molecules available for anchoring in the substrate. 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).

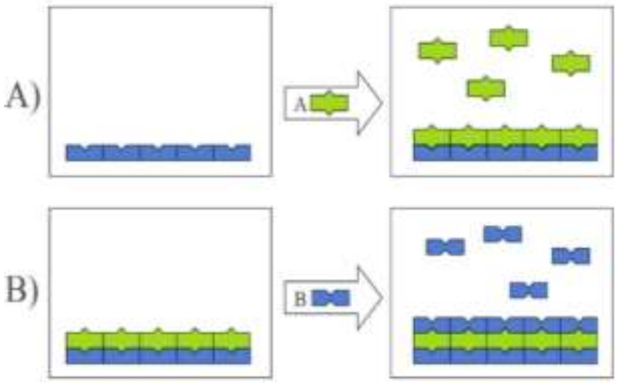


Figure 1 Atomic layer deposition (ALD) diagram

The figure 1 show the ALD diagram where is appreciable two precursors A and B creating two layers. First is deposited one layer with precursor A and when is saturated the remained is purged, the second layer is deposited with precursor B, and the same way the remained of precursor B are purged.

In recent years, the ALD technique has grown exponentially using in all semiconductor industries such as the manufacture of integrated circuits, sensors, micro and nanostructured electromechanical systems (MEMS and NEMS), optoelectronics, mechanics and renewable energy. Some examples of ALD applications are presented in protection against corrosion, energy production in solar cells, coatings of biological compatibility for medical devices and implants, water purification, OLED lighting devices, among others (H. Kim, et.al., 2009; Leskelä, M. and Ritala, M. 2003).

The ALD technique has advantages over other depositing techniques such as CVD or sputtering, having the ability to create ultrathin films with the ability to coat surfaces following irregular topographies (R. Johnson, et.al., 2014). The present work focuses on the design and construction of an ALD reactor with the characteristic of being reproducible, scalable and assembly for various chamber types, using coordinated ALD valves under a controller programmed with LabView2015.

Methodology to be developed
Materials and methods

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)

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. (See figure 2).

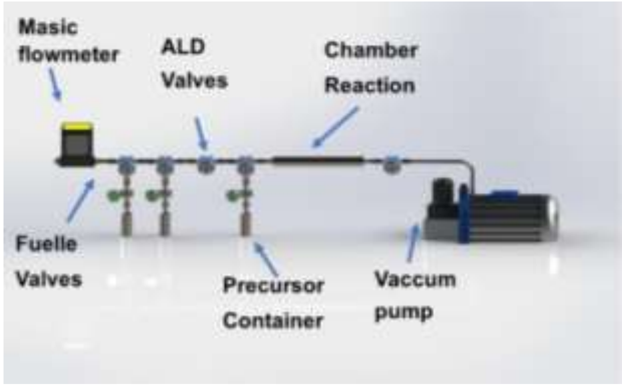


Figure 2 ALD system designed by SolidWorks

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.

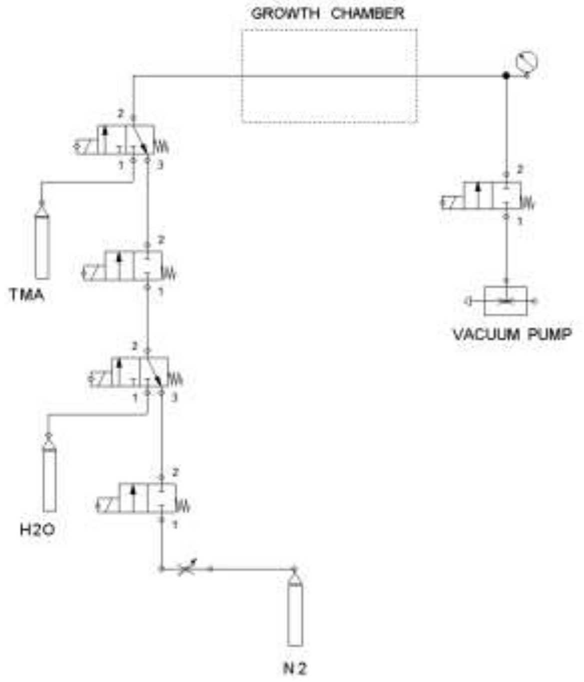


Figure 3 Electro-pneumatique designee of ALD system

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.

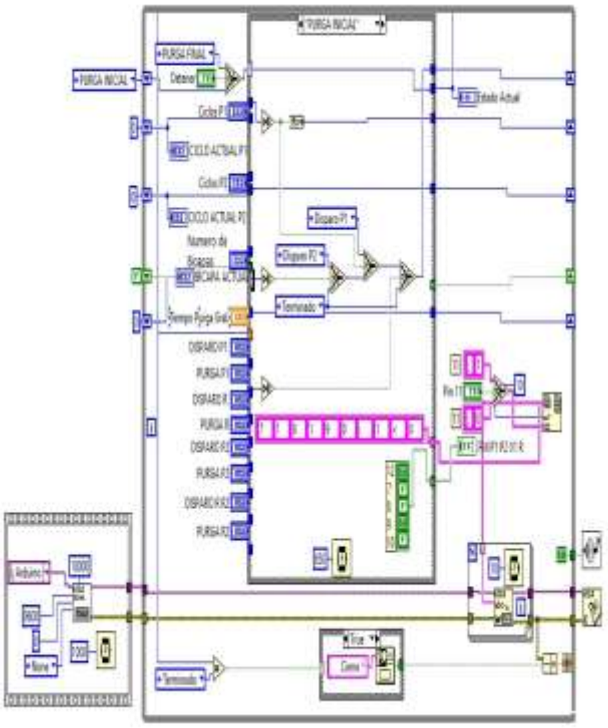


Figure 4 Block diagram developed with LabVIEW for the ALD reactor

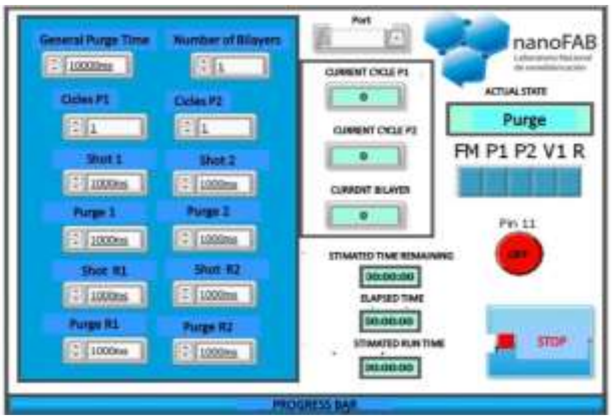
Results

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

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. (See figure 6).



General Purge Time: 10000ms
Number of Bilayers: 1
Cicles P1: 1
Cicles P2: 1
Shot 1: 1000ms
Shot 2: 1000ms
Purge 1: 1000ms
Purge 2: 1000ms
Shot R1: 1000ms
Shot R2: 1000ms
Purge R1: 1000ms
Purge R2: 1000ms
CURRENT CYCLE P1: 0
CURRENT CYCLE P2: 0
CURRENT BILAYER: 0
ACTUAL STATE: Purge
STIMATED TIME REMAINING: 00:00:00
ELAPSED TIME: 00:00:00
STIMATED RUN TIME: 00:00:00

Figure 6 ALD reactor control panel

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

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.

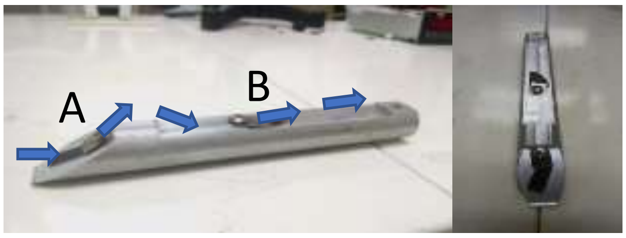


Figure 7 ALD substrate holder. Two locations of substrate A and B are shown. The flow of precursors sticks directly to substrate A and rebounds to substrate B

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

A topographic analysis was performed to analyze the uniformity of the thin film surface by plotting the 16 analysis points taken in ellipsometry using Wolfram Mathematica 9.0 software. (See figure 8).

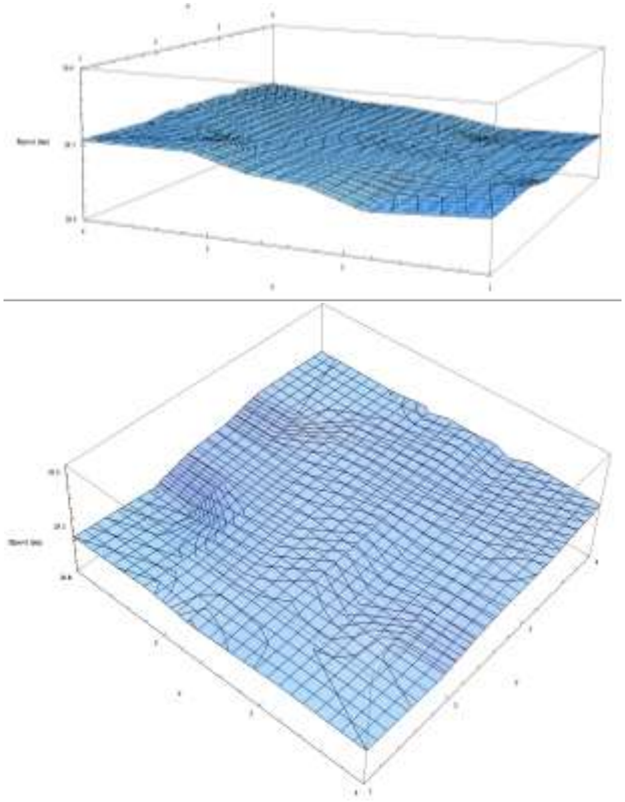


Figure 8 Topography graph of ALD de Al_2O3 deposits

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B) It was possible to deposit thin films of Al2O3 on nanoparticles (NP's) Si on a substrate of Si. The experiment was carried out at 40 cycles, 180°C in the reaction chamber, water and trimethyl-aluminum (TMA) as precursors and swept with N_2 flow.

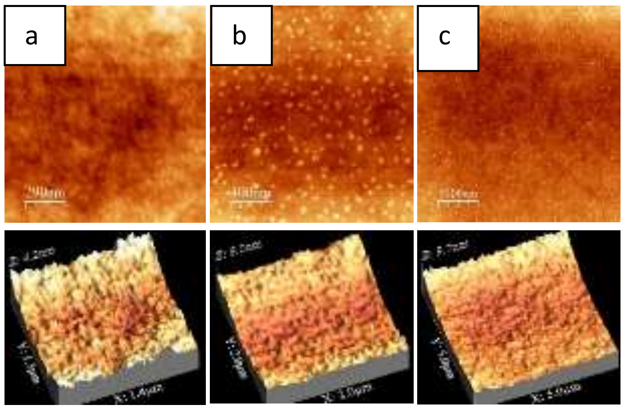

Figure 9 (a) Reference sample without ALD deposit of Al2O3, (b, c) silicon nanoparticle films subjected to 40 ALD cycles of alumina

Figure 9 shows atomic force microscopy (AFM) micrographs where the following samples are described: a) the deposit of Si NP's on a Si substrate. Images b) and c) show results of ALD deposits of Al_2O_3 / Si NP's / Si. In images a) and b) it is observed how the morphology of the Si NPs is maintained in the Al2O3 deposit, in a uniform way.

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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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