Report on the outcomes of a Short-Term Scientific Mission

Action number: CA17107

Grantee Name: Kamran Ali Syed

Details of the STSM

Title: Low-temperature Atomic layer deposition (ALD) of ultra-thin ZnO film as an active Gas Sensing layer on flexible Gas Sensor for textile application.

Start and end date: 25/04/2022 to 20/05/2022

Description of the work carried out during the STSM:-

Atomic Layer Deposition involves a cycle of 4 steps that is repeated as many times as necessary to achieve the required deposited thickness. Figure 1 shows ALD system for deposition of ZnO using ZnEt2.

One of the primary challenges in Atomic layer deposition (ALD) is the design of precursors.The precursors must be volatile to enter the gas phase as well as thermally stable so as not to decompose resulting in an inhomogeneous growth rate. The precursor surface chemistry is vital to the ALD process in that the precursor must interact strongly with the surface functional groups in order to bind effectively. Hydroxyl functional groups have been reported to be important for MOx ALD. The overall reaction chemistry of the precursor with the surface functional groups should ideally have a strong thermodynamic driving force with large negative ΔG values.

In metal oxide (MOX) ALD, a typical reaction scheme exists where the precursor of interest (typically an organometallic) is chemisorbed onto a surface substrate with the elimination of one or more ligands. A purge gas is applied to remove residual precursor followed by the introduction of an oxygen source which reacts with the chemisorbed species to produce the metal oxide. All volatile by products of the ALD reaction are removed by a second purge gas cycle.

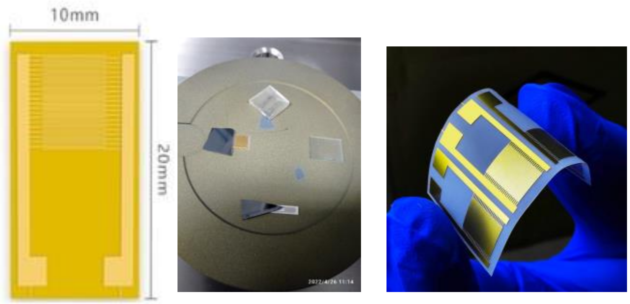
The two different substrates for for gas sensing has been used in this work, with the use of the same IDE and the use of the same fabrication technique Atomic Layer Deposition (ALD) for the ZnO as an active layer of both types of Sensors.

- A flexible Polyimide Interdigitated sensor. (wearable smart sensor)
- A rigid Alumina Interdigitated sensor

❖ Polyamide flexible IDE Sensor: -

The flexible PET electrode (IDE) is a smart wearable sensor. The substrate is polyethylene terephthalate (PET), with a thickness of 60um, size 10mm*10mm. The line width is 100um, line spacing is 50um, and finger length is 7.7mm. The metal layer structure Cu/Ni/Au, the thickness of 12um, 3um, 1um respectively. The electrode temperature range: is -50℃~120℃.

The ALD system uses an inert carrier gas, nitrogen, to deliver precursor to the substrate and also minimize reactant condensation on internal system components. These functions require a continuous flow of gas at a pressure of ~10-3 Torr. At that pressure, there is a mixture of nominally 2.4×10^{13} molecules/cc of carrier and precursor gases. In addition, the substrate is typically heated to promote a complete chemical reaction. Unfortunately, the deposition of ZnO on the flexible PET electrode substrates was unsuccessful due to a non-ideal chemical process. For non-ideal chemical processes, the exact method of surface functionalization may not be clear. One considerable challenge is operating a QCM at the temperatures required for ALD; another is the functionalization of the QCM's surface so that it attracts precursors and undergoes the same chemical reactions as the substrate. In addition, the reactor developed a leak whilst we were doing some of the ZnO depositions. While gas flow in an ALD system is deterministic based on known conditions, it is still not predictable. Mixing of the carrier gas and precursor during the dose cycle can also be an issue.



(a) (b) (c)

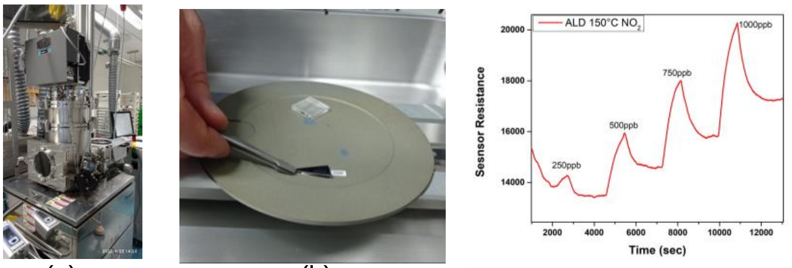
Fig 2(a) The felxible IDE smart sensor configuration (b) Coating of ZnO on different substrates (c) Felxible IDE smart sensor.

One of the keys to achieving dense, defect free, monolayer films is complete and perfect substrate functionalization. This implies the comprehensive siting of hydroxyls on the surface of a PET substrate or engineering the thickness of an oxide layer for PET substrates. Given the chaotic gas flow conditions in a reactor, it is highly possible that regions of a substrate may not see the functionalization gases uniformly, or that functionalized sites might be ablated away by the torrent of carrier gas molecules continuously bombarding the substrate.

Description of the STSM main achievements and planned follow-up activities

❖ Alumina Interdigitated gas sensor: -

The ZnO has grown by the ALD at a low temperature of 100 ºC shown in Figure 2. The results were obtained from the alumina substrates with platinum screen-printed interdigitated electrodes (IDE). It is worth noting that a heating element was placed at the backside of the IDE for increasing the operating temperature of the sensors We applied increasing concentrations of NO_2 (250, 500,750, and 1000ppb) for several cycles. In addition, the sensor shows clear resistance towards nitrogen dioxide detection by applying repeated exposure and recovery cycles to increasing concentrations of the analytic considered.



(a) (b) (c)

Fig 2(a) The ALD system (Picosun R-200 ALD reactor) at UCL (b) ALD coating of ZnO at 100°C thickness of 41nm (c) The resistance response detecting NO_2 annealing treatment at 150 °C

Acknowledgments

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Future collaborations

Development of Rigid gas sensors on commercial alumina interdigitated substrate. Our main focus is on the Atomic Layer Deposition of ZnO at low temperatures and of different thicknesses. The film composition is usually more homogenous but this method can be more challenging as the film stoichiometry is controlled by the concentration of the individual precursors and competition for active hydroxyl sites. The influence of thickness on the Gas sensing properties will be investigated.

Reference

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