

Electrical Impedance Response of a Thick-Thin film Hybrid Anodic Nanoporous Alumina Sensor to Methanol Vapors.

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Abstract

The response of an anodic aluminum oxide (AAO) nanoporous humidity sensor to an organic vapor such as methanol is presented in this paper. Sensors constructed at Northern Illinois University were exposed to various organic vapors. The characteristic response of the sensor to various vapor dilutions was analyzed using real and complex impedance parameters.

Keywords: Nano-pores, hybrid thick film, sensors, methanol, AAO, Electrical Impedance Spectroscopy, Nyquist Plot, Cole-Cole Plot, Impedance

1 Introduction

Anodic anodized oxide (AAO) thin films for humidity sensing have been demonstrated by Dicky et al [1] and Haji-Sheikh et al [2]. Recent work to discover the sensitivity of AAO to other gases was performed by R. J. Lazarowich et al. [3] using a quartz microbalance. The main hypothesis in this work is to demonstrate that AAO sensors are not only sensitive to water condensation but also the condensation of higher vapor pressure liquids such as methanol and other organic species.

Researchers in Pennsylvania have demonstrated surface acoustic wave (SAW) devices made from self-ordered thin film porous alumina [4] for the sensing of ammonia and humidity. The AAO film used by Varghese et. al. [4] shows only local order and limited inter-domain alignment. The aluminum film used in this approach was produced using thermal evaporation and did not include a conductive adhesion layer. This experiment demonstrates the use of a conductive adhesion layer to produce a nanoporous sensor.

2 Experimental Procedure

To produce the thin-film nanopores, the starting substrate material is ninety-nine percent alumina. This substrate material was chosen for its low cost and ease of use. A



Figure 1. Sputter deposition system. This system is capable of depositing on substrates, which are as large as 6 inches. This system is cryo-pumped with a base pressure measured at 1.1×10^{-7} torr.

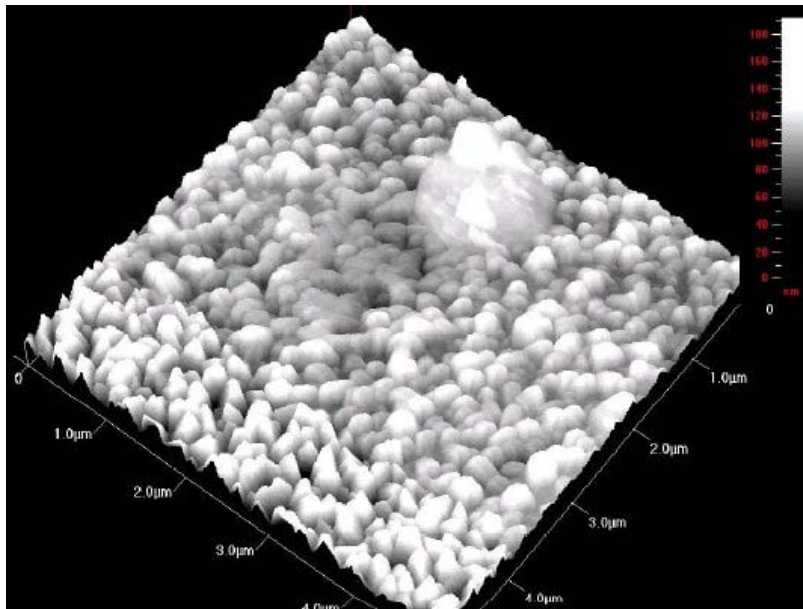


Figure 2. As-deposited nanocrystalline aluminum film. The average grain size is around 200 nm.

interdigitated capacitor was then patterned on the surface of the AAO using a screen printed conductor and fired on a belt furnace. This thick-thin film sensor had leads soldered on in a dual pin configuration that allows for easy handling and measurement.

3 Results

Organic vapors were created using a computer-controlled vapor generator. This device mixes a carrier gas (dry nitrogen) with saturated vapor, at 15C, to produce a prescribed dilution level. The impedance of the sensor, exposed to the various diluted samples, was measured using an Agilent 4980A LCR meter (Inductance-Capacitance-Resistance). The impedance model selected was the parallel resistor with a parallel capacitor. Figure 5 and Figure 6 show the results of measurements taken using the AAO based sensor when it was exposed to methanol and to water. The frequency range shown in this graph is 200 Hz to 200 kHz, though data was taken up to 2 MHz but didn't present any valuable data. .

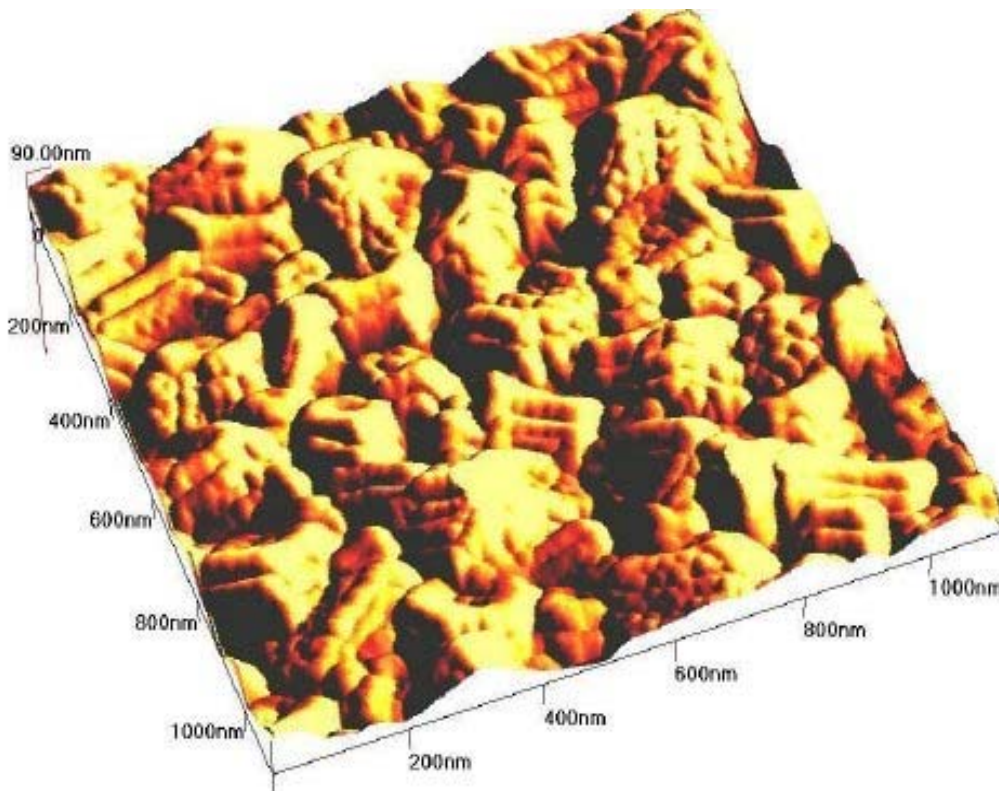
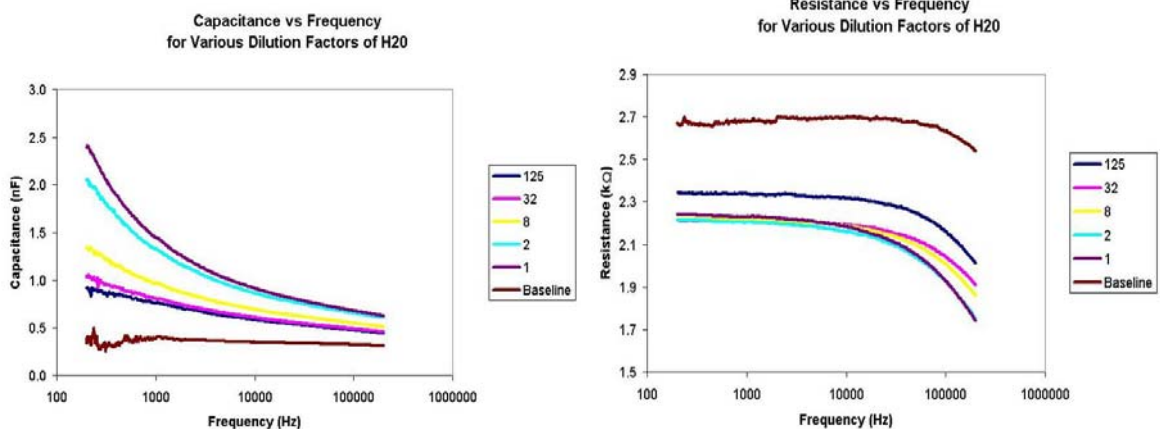
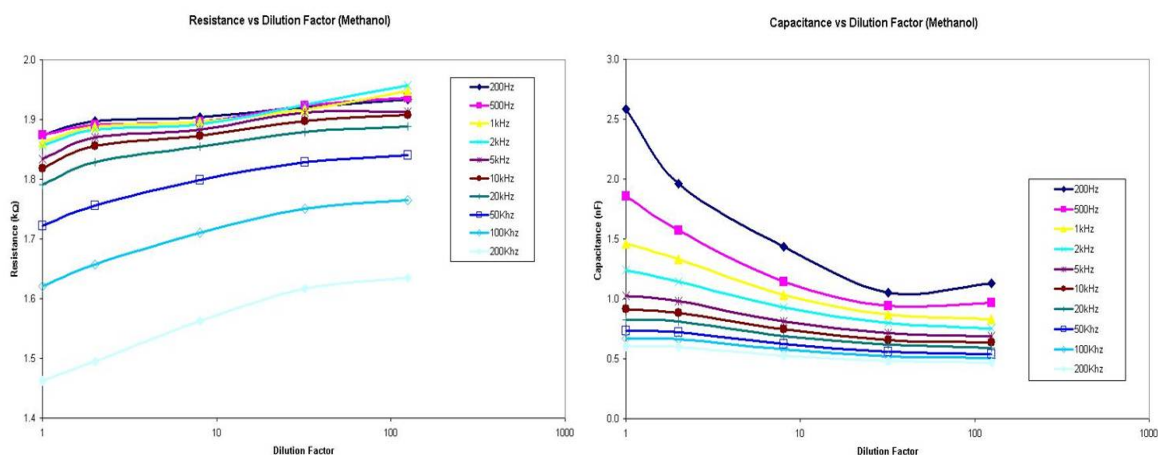


Figure 4. Atomic force microscopy of the AAO film. The average nanopore is around 40 nm in diameter and the original deposited aluminum grain size was 300 nm.



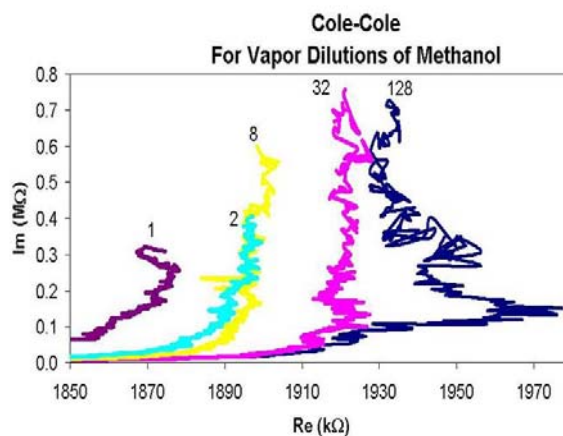
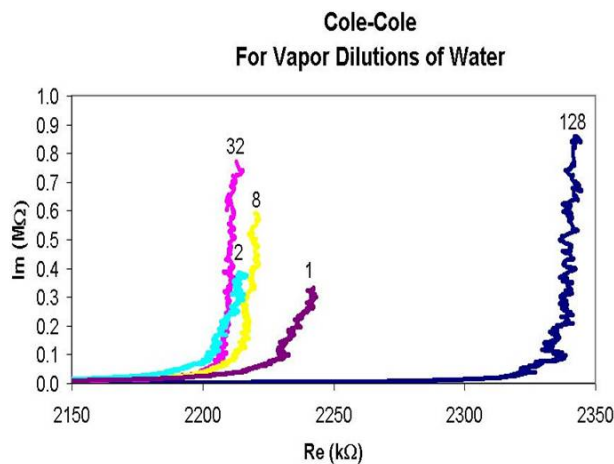
Figures 9 & 10. Capacitance and resistance versus frequency graphs for various dilutions of water

The exponential behavior is frequency dependent and concentration dependent with the highest sensitivity happening at the lowest frequencies. Figures 7-10 show the behavior of the sensor at different frequencies and different dilution factors for water and methanol. The response with varying frequency is significantly different than the response of methanol at various frequencies. Resistance varied at higher concentrations of methanol, especially at higher frequencies, however when sensing water the resistance was changed from baseline but didn't change among different concentrations.



Figures 11 & 12. Capacitance versus dilution factor for methanol.

200Hz initial frequency appearing at the upper right-hand data point terminating with the 2kHz data point at the far left.



Figures 15 & 16. The Cole-Cole plots derived from the capacitance and resistance data. Note the orderly parametric shift in methanol as the dilution factor is decreased (increased MeOH concentration) at the sensor surface.

5 Conclusions

The AAO devices demonstrated in this paper show that the nanoporous material is sensitive to organic molecules. It also can be seen from the data in Figure 5 that methanol can be detected by using an AAO capacitive sensor. From this limited data set it appears that sensor has the ability to discriminate between water and methanol vapor. Future work will be performed to determine the sensor response to other organic vapors and the mechanism responsible for the interaction of the organic molecule with the AAO.

