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Investigation and Analysis of Zinc Phthalocyanine films for Resonant Gas Sensor Applications

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Abstract

Thin films of Zinc Phthalocyanine (ZnPc) with a range of different substitutes have been investigated as chemical active layers for sensing selected organic vapour such as (Ethanol, Toluene) by a standard Quartz Crystal Resonator (QCR) based sensor with fundamental resonance 10MHz. Adsorption of vapour onto the films surface has been realized by monitoring resonance spectra. An equivalent circuit (BVD circuit) has been used to extract parameters related to film viscosity and thickness by fitting experimental admittance spectra of QCR around resonance frequency. Moreover, film properties and characterization was obtained from QCR measurements in conjunction with suitable data analysis. Validation of film characteristics has been determined using complementary methods such as Ellipsometry, UV- Visible absorption Spectrophotometer and AFM.

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1. Introduction

Phthalocyanine films have become an increasingly important field of study, with several research groups investigating phthalocyanines in sensor applications utilizing a range of techniques and transduction mechanisms;

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investigating areas such as chemical interaction with gases and the performance of sensing devices. This can be attributed to the properties of the resultant sensitive absorption layers such as high stability, sensitivity, selectivity and the ease of which they be applied as a thin films through traditional coating methods[1,2,3]. Additionally, variations in sensing/detection properties can be realised by the addition of different substituent in the phthalocyanine film [4,5].

In this work, the impedance analysis approach was performed to explore vapour adsorption of organic solvents by several ZnPc substituents. The technique allows additional information about film parameters to be extracted as compared standard oscillator measurements and presents an insight into changes appearing in the film as a result of vapour adsorption [6,7].

2. Experimental

2.1. Sensitive materials

Zinc phthalocyanines with four different substituents were studied and characterized for their gas sensing performance. Table 1 shows the specific materials used; an identification code has been assigned to each substituent pattern. All phthalocyanines (Pcs) used in this work were obtained from Sigma-Aldrich.

Table 1. Materials and their codes and substituting pattern.

Chemical name	Substituting pattern	Code	Empirical formula (Hill Notation)
Zinc 2,3,9,10,16,17,23,24octakis(octyloxy)-29H,31H phthalocyanine	Zn-oct-oct-Pc	a	C ₉₆ H ₁₄₄ N ₈ O ₈ Zn
Zinc 1,2,3,4,8,9,10,11,15,16,17,18,22,23,24,25-hexadecafluoro 29H,31H-phthalocyanine	Zn-hex-Pc	b	C ₃₂ F ₁₆ N ₈ Zn
Zinc 1,4,8,11,15,18,22,25-octabutoxy- 29H,31H-phthalocyanine	Zn-oct-Pc	c	C ₆₄ H ₈₀ N ₈ O ₈ Zn
Zinc 2,9,16,23-tetra-tert-butyl-29H,31H-phthalocyanine	Zn-tp-tb-Pc	d	C ₄₈ H ₄₈ N ₈ Zn

2.2. QCR transducer & Sensor Preparation

The QCR transducers used in this work were 10MHz fundamental resonant frequency AT-Cut quartz crystals. Spin coating was used for deposition of the ZnPcs on the transducers. Solution of sensitive material was prepared by dissolving the ZnPc in analytical grade chloroform at a concentration of 10mg/ml at room temperature. A micropipette was then used to drop a known amount of solution onto the rotating (2000rpm) substrate surface; the solvent subsequently evaporates leaving the desired film. No further chemical or physical treatment of sample was performed after deposition. Impedance/Admittance measurements at a range of frequencies around crystal resonance were then taken and resultant spectra fitted to the BVD equivalent circuit. Two common organic solvent were used as test analytes: (Ethanol and Toluene). Concentrations in the range of Lower to Higher Explosion Limit (LEL-HEL), where used for each analyte.

2.3. Vapour exposure

A typical measurement protocol is as follows: The sample measurements were undertaken in a Teflon chamber with an internal volume of approximately 0.0636 m³ and the inlet pipe was located on the top of the chamber. Measurements were taken using a Keysight E4990A impedance analyser controlled via PC running LabVIEW software to continuously record the spectra during exposure. Specific amounts of liquid test analyte relating to the required concentration are deposited into the chamber through a micro syringe and allowed to evaporate. The chamber was flushed with air after every exposure in order to test if full recovery of the QCR sensor response is observed. The Admittance (reciprocal of impedance) spectra obtained by measurement were then fitted to the BVD equivalent circuit by using LabVIEW program and parameters (Δf and ΔR related to L_{load} and R_{load}) extracted [6].

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