proceedings

Abstract

Nitrogen Monoxide Detection with Pentacene-Based Film Bulk Acoustic Resonators †

José Manuel Carmona-Cejas , Teona Mirea *, Ricardo Hervás, Jimena Olivares and Marta Clement

CEMDATIC-ETSIT, Universidad Politécnica de Madrid, Av. Complutense 30, 28040 Madrid, Spain; jm.carmona@upm.es (J.M.C.-C.); ricardo.hervas@upm.es (R.H.); jimena.olivares@upm.es (J.O.); marta.clement@upm.es (M.C.)
* Correspondence: teona.mirea@upm.es
† Presented at the XXXV EUROSENSORS Conference, Lecce, Italy, 10–13 September 2023.

Abstract: Exposure to hazardous gases like NOx can have a negative effect on our health. High-performance sensors that are also able to sustain harsh environments are needed to control high levels of these contaminants, which in most cases, are generated by automotive exhaust or industrial factories. In this work, we prove NO detection in the ppm level, employing an AlN-based FBAR with pentacene as the active layer. FBARs are potential candidates as they have also proven operation and stability in harsh environments as well as temperature and humidity discrimination with a single sensor.

Keywords: FBAR sensor; SMR sensor; pentacene

Citation: Carmona-Cejas, J.M.; Mirea, T.; Hervás, R.; Olivares, J.; Clement, M. Nitrogen Monoxide Detection with Pentacene-Based Film Bulk Acoustic Resonators. *Proceedings* **2024**, *97*, 226. https://doi.org/10.3390/proceedings2024097226

Academic Editors: Pietro Siciliano and Luca Francioso

Published: 2 July 2024

1. Introduction

Many human diseases and physical conditions have been linked to long-term exposure to contaminated environments. There are several contaminants present in many cities: CO_2, CO, CH_4, NO_x, or SO_x, among others. These gases can be generated by several means, including the exhaust of automotive engines or different industrial factories. Hence, monitoring systems is extremely important. The main blocks in these systems are the sensing elements. Different technologies have proven their ability to detect hazardous gases, including resistive sensors or transistor-based technologies. Thin-film bulk acoustic resonators (FBARs) are a mature technology that we can nowadays find in our cellphones acting as RF filters. Nevertheless, FBARs have also shown potential as highly sensitive gas sensors with resolutions in the ppm and even ppb levels. Besides their low cost and micrometric size, they can also operate at extreme temperatures and discriminate between temperature and relative humidity without the need for a reference sensor [1]. FBARs act like gravimetric sensors, which can detect mass attached to their surface. To be selective, they need a functional active material that can adsorb the target gases. In this work, we present the detection in the ppm levels of nitrogen monoxide (NO) using an AlN-based FBAR coated with pentacene. Pentacene is a carbon-based material that has shown responses to NO_x gases in different technologies. In this study, we have optimized its deposition in nanometric layers. Its integration into FBAR showed no influence in terms of the device performance.

2. Materials and Methods

We fabricated FBARs following a solidly mounted resonator (SMR) structure (Figure 1a). SMRs are based on a piezoelectric material (in this case, AlN) sandwiched between two metallic electrodes (Mo and Ir). Upon applying an AC signal, this structure can resonate at a frequency directly related to the thickness of the AlN, in our case, 3 GHz. To prevent acoustic energy dissipation to the Si substrate, in SMRs, we deposit these structures on an acoustic reflector based on high and low acoustic impedance materials (SiO_2 and

AlN, respectively). A 50 nm thick pentacene was deposited on top of the Mo active area by Joule evaporation. For the NO detection experiment, the SMR sensors were placed inside a gas chamber and monitored using an RF probe connected to a network analyzer. This enabled the extraction of their electrical impedance and monitored their frequency shifts when exposed to NO. Inside the gas chamber, a commercial sensor monitored the temperature and relative humidity levels. We used synthetic air as the diluting gas and for humidity generation.

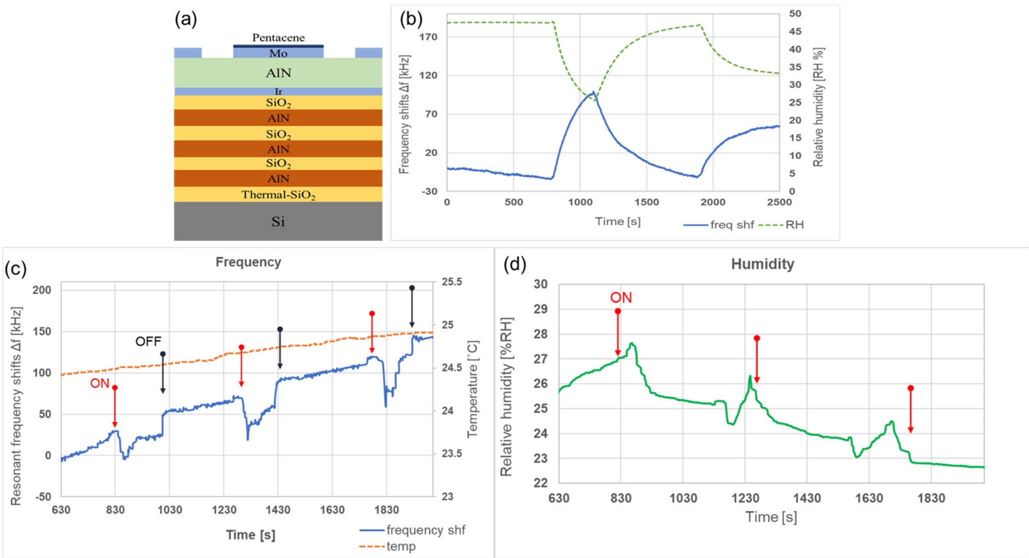

Figure 1. (**a**) Cross−section of an SMR. (**b**) Frequency shifts due to RH variations in the gas chamber. (**c**) Frequency shifts and temperature variation of the SMR with 33.3 ppm NO exposures. Red and black arrows show when NO flow starts and stops. (**d**) Relative humidity variation for the same experiment.

3. Discussion

We first tested the SMR's relative humidity (RH) sensitivity so we could extract afterwards their pure mass sensitivity to NO detection. Figure 1b shows how the frequency increased when RH decreased with a sensitivity of 3.9 kHz/%RH. Figure 1c shows, as proof of the concept, how the sensor responds to 33.3 ppm of NO with a downwards shift in a frequency of 34.2 ± 2 kHz. The graph shows a positive slope of the frequency curve due to slight temperature increases during the experiment. To discard the humidity effect, Figure 1d shows humidity variations at each NO cycle (ON/OFF) of a maximum of 1.4% RH. This RH variation implies 5.7 kHz shifts, which can be translated to a pure NO sensitivity of ~1 kHz/ppm. This sensitivity can be improved by chemical treatments of the pentacene layer since in this experiment, pristine layers were used. Moreover, experiments at high temperatures (400 °C) are ongoing since NO detection can be useful in industrial harsh environments.

Author Contributions: Conceptualization, T.M. and J.M.C.-C.; methodology, T.M., J.M.C.-C., and R.H.; software, T.M.; validation, J.M.C.-C.; writing—original draft preparation, T.M. and J.M.C.-C.; review, J.O. and M.C.; funding acquisition, J.O., M.C., and T.M. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the R&D National Plan of Spanish Government (PID-2020-118410RB-C22) and Comunidad de Madrid through the call Research Grants for Young Investigators from Universidad Politécnica de Madrid, ACUEX, APOYO-JOVENES-21-TUGJDU-108-WNZK0H.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are contained within the article.

Conflicts of Interest: The authors declare no conflicts of interest.

Reference

1. Carmona-Cejas, J.M.; Mirea, T.; García-Herván, R.; Moreno, M.; De Miguel-Ramos, M.; Olivares, J.; Clement, M. Decoupling of Humidity and Temperature Effects with a single Solidly Mounted Resonator Sensor. In Proceedings of the 2022 IEEE International Ultrasonics Symposium (IUS), Venice, Italy, 10–13 October 2022.