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Abstract

Film Bulk Acoustic Resonators for Nitrogen Monoxide Detection at 250 °C †

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Abstract: Exposure to hazardous gases like NO_x can have a negative impact on our health. High-performance sensors 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 demonstrate NO detection below the ppm level by employing an AlScN-based FBAR covered with a sputtered WO_3 active layer. FBARs are potential candidates as they have also proved operation and stability in harsh environments as well as temperature and humidity discrimination with a single sensor.

Keywords: FBAR sensor; high temperature; gas detection; metal-oxide

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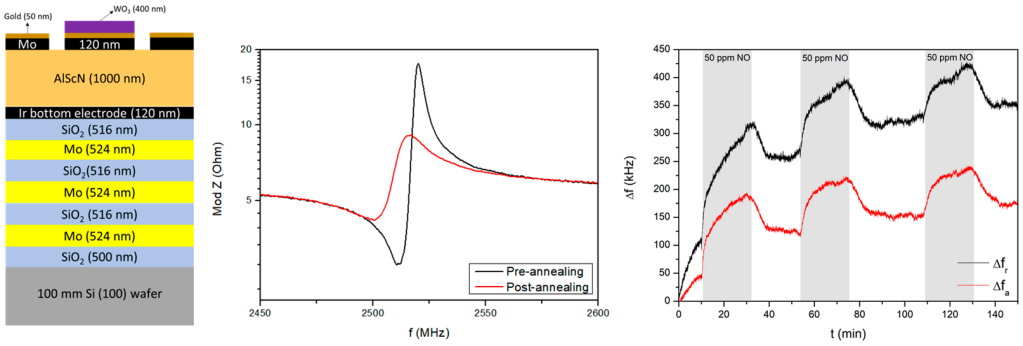
1. Introduction

The size of the global sensor market will pass the 300 billion USD mark by 2028, indicating that this is a sector with strong growth which has no intention of stagnating. Within the sensor market, gas and particle sensors arise as some of the most interesting ones, since the need to monitor the gaseous composition of different atmospheres and environments has become more than evident in recent times. The potential applications of these kinds of sensors can go beyond this. For instance, an appropriate gas sensor could tell us whether the combustion processes inside vehicle engines are working correctly and thus help us to reduce gas emissions. For these types of applications, we need to build a suitable sensor capable of not only capturing the chemical species we are seeking, but also of withstanding exposure to harsh environments while maintaining low costs and high performance. Electroacoustic technology has proved to be adequate for operation in such environments. In particular, thin film bulk acoustic resonators (FBARs) have shown endurance at up to 1000 °C [1]. These devices are capable of detecting gases at the ppb levels, and offer advantages such as low cost, miniaturization, and IC integration. It is, therefore, pivotal to extend the studies on their application in harsh environments, particularly for the most harmful contaminants present in cities, such as CO_2, CO, CH_4, NO_x, or SO_x, among others. To be selective, they need a functional active material that can adsorb the targeted gases. In this study we present the detection of ppm/ppb levels of nitrogen monoxide (NO) at 250 °C using an AlScN-based FBAR coated with WO_x. These conditions simulate the ones present in different industrial fields.

2. Materials and Methods

We manufactured solidly mounted resonators (SMRs) with the structure shown in Figure 1a. SMRs are based on a piezoelectric material (in this case AlScN) sandwiched between two metallic electrodes (Mo/Au and Ir). Upon applying an AC signal, this structure can resonate at a frequency directly related to the thickness of the AlScN; in our case, 2.8 GHz. To prevent acoustic energy dissipation to the Si substrate, the piezoelectric capacitor was grown on top of an acoustic reflector composed of a stack of alternating layers of high and low acoustic impedance materials (SiO_2 and Mo, respectively). The

Mo/Au electrode defining the active area of the device was covered with a 400 nm thick WO_3 selective layer grown at low power by DC-pulsed magnetron sputtering from a W target in a 50/50 Ar/O_2 gas mixture. After deposition, the devices were annealed in air at 350 °C for 12 h to activate the WO_3. The devices were then introduced to the gas chamber and placed on a heater, allowing us to control and monitor their temperature. For the NO detection experiment, the SMR sensors were exposed to NO diluted in synthetic air at a constant gas flow of 700 sccm. During exposure, their frequency response was monitored using an RF-probe connected to a network analyzer. This allowed extracting their electrical impedance and monitoring their frequency shifts when exposed to NO.



(a) (b) (c)

Figure 1. (**a**) Cross section of an SMR; (**b**) frequency response of the SMRs before and after the annealing; (**c**) frequency shifts upon 50 ppm of NO exposure cycles for resonant (black line) and antiresonant frequency (red line).

3. Discussion

Figure 1b shows the behavior of the SMRs before and after the thermal annealing, which is a key step in making the WO_3 layer sensitive towards the analyte. Although the annealing process induces some degradation of the resonance, the resonant (f_r) and antiresonant (f_a) frequencies can still be tracked for sensor characterization. Figure 1c shows the change in frequency upon cycles of NO gas exposure at a concentration of 50 ppm at 250 °C. Both f_r and f_a frequencies experience similar shifts for each exposure cycle, decreasing from 125 kHz to 100 kHz, which implies a sensitivity of 2–3 kHz/ppm. In this type of device, a limit of detection of 1 kHz can be set (optimized readout systems can improve it), hence resolutions below ppm levels can be achieved. The final sensitivity can be improved by tuning the sputtering conditions and the thickness of the WO3 layer, and/or by changing the temperature of operation.

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