Contents lists available at ScienceDirect



Sensors and Actuators B: Chemical

journal homepage: www.elsevier.com/locate/snb



Detection of ppb ozone using a dispersive surface acoustic wave reflective delay line with integrated reference signal



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ARTICLE INFO

Article history: Received 29 August 2012 Received in revised form 23 October 2013 Accepted 23 October 2013 Available online 9 November 2013

Keywords: Surface acoustic wave Ozone Polybutadiene Gas sensor

ABSTRACT

We report the characterization of a dispersive surface acoustic wave (SAW) delay line for the detection of ozone in air at parts-per-billion (ppb) concentrations. Augmenting recent developments in wireless SAW tag technology, we fabricated 435 MHz SAW devices and prepared them with polybutadiene coatings to serve as ozone-specific gas sensors. We measured the frequency dependent one-port electromagnetic scattering parameter, $S_{11}(f)$, at regular time intervals as the gas stream was adjusted using an EPA primary standard ozone calibrator. The temperature and relative humidity were also recorded using an independent commercial sensor. The data allowed us to estimate the sensitivity to ozone and to temperature, and we estimated other parameters useful in personal dosimetry: saturation dose, shelf life, and limit of detection. Finally, the built-in time multiplexed response provided reference data for first order temperature compensation, yielding a chemical limit of detection of 63 ppb-min ozone.

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1. Introduction

Ozone (O_3) is a major component of urban smog and is of particular concern due to its impact upon public health. Regulations set forth by the U.S. Occupational Safety & Health Administration (OSHA) specify a maximum allowed concentration of 100 parts per billion (ppb) averaged over an 8 h period [1]. Further illustrating the concern for ozone levels, in 2008 the United States Environmental Protection Agency (EPA) revised the National Air Quality Standards (NAAQS). The "8-h primary ozone standard" was reduced to 75 ppb. Fig. 1 shows the 8 h average for outdoor data, beginning at midnight (hour 0). This data reveals a metropolitan ozone concentration approaching unhealthy levels for an arbitrary day of the year [2]. In fact the EPA threshold is frequently exceeded, particularly in regions of the southeast United States where, in addition to pollution, ozone levels are elevated naturally.

In 2010, exceedances of the federal air quality standard for ozone began on May 5 and continued at a rate of approximately 4 days per month through September 2010 [2]. In Fig. 1 the use of an 8-h averaging window and the peak concentration near hour 20 (8:00 PM) imply the period of greatest average ozone concentration was during the 8 h prior; i.e. after 12:00 PM. In 2006 the National Institutes of Health (NIH) issued a request for applications titled, "Environmental Sensors for Personal Exposure Assessment" (RFA-ES-06-011). Although the influence of ozone on human health has been widely documented, a key problem remains in the assessment of actual impact to the public – measurements are seldom performed on a personal and real time basis. Such measurements are needed for two primary reasons:

- Location: As an example, there are just 65 air monitoring sites in the state of Georgia [2], and furthermore, ozone is a reactive gas and substantial spatial concentration gradients have been shown to exist within just half a meter of test subjects [3].
- Latency: Studies of personal ozone exposure have relied on passive sampling methods requiring long exposure intervals of 12–24 h [4,5].

Thus the development of inexpensive, portable, and queryable ozone sensors is expected to be of great utility to the occupational and environmental health communities.

Existing technologies for the detection of ozone center around the well-known photometric and gas-sensitive semiconductor methods. Photometric analyzers employ the ultraviolet absorption of ozone to determine the concentration using Beer's law. This conventional approach requires ultraviolet lamps that dissipate heat. Although lower power ultraviolet LEDs are available, they operate

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^{0925-4005/\$ -} see front matter © 2013 Elsevier B.V. All rights reserved. http://dx.doi.org/10.1016/j.snb.2013.10.104



Fig. 1. Outdoor ozone concentration obtained from a government monitoring station in metropolitan Atlanta, GA, USA on April 4, 2010 [2]. The data correspond to a standard 8-h moving average.

in the near-UV, e.g. zinc oxide at 395 nm, and not near the strongest optical absorption peak of ozone, i.e. at 253 nm in the Hartley band [6]. When passive wireless sensing is desired, the power consumption of these optical techniques is prohibitive. Even alternative gas-sensitive semiconductor sensors require significant power to heat metal oxides, e.g. tin oxide (SnO_2), above 100 °C to expel moisture. One handheld ozone sensor (Aeroqual S-200UL) requires 5 W on average; its 9.6 V and 2100 mA-h battery pack only powers the device for 4 h in continuous operation. Such equipment does not seem suitable for routine and scalable personal monitoring. In this paper we instead present results from a passive SAW RFID sensor approach, which was tested using just 1 mW of RF excitation power.

1.1. SAW RFID sensors

Demonstrations of passive SAW tag sensors date back to approximately 1994 [7,8], but they only recently have been extended to gas sensing [9]. The previously reported QCM ozone sensors operated at 10 MHz and were sufficiently sensitive to detect ozone in the relevant ppb range [10,11]. However, there are several advantages of operation at higher frequencies: greater mass sensitivity, greater bandwidth for signal encoding and multiplexing, and smaller antennas in wireless operation.

We note that a challenge encountered at higher frequencies is the inherent mass loading by the film itself. One must functionalize the sensing surface with a chemically selective film without introducing too much acoustic loading and loss at high frequencies. In this paper we demonstrate ozone detection in the ultra high frequency (UHF) range that is convenient for passive backscatter SAW tags.

A fundamental difference between most SAW tag sensors and the more conventional resonant sensors is the measurand: phase delay rather than oscillation frequency. To first order the change of phase velocity is related to mass change at the surface:

$$\Delta \nu = \Delta f \lambda \propto -\Delta \rho, \tag{1}$$

i.e. the effective phase velocity of an acoustic wave linearly decreases as mass density increases. We therefore measure the change in phase of the acoustic wave to detect a change of mass at the surface.

The devices of this study produce multiple delayed reflections of the interrogation signal to provide multiplexed sensing information. This dispersion in space and time allows us to distinguish the perturbations to each part of a sensor. The sensor's identification (ID) sequence, its temperature, and the cumulative effect of ozone attack of the sensitive film are returned in response to a query signal. A schematic diagram is shown in Fig. 2. An incident electromagnetic wave is converted to acoustic waves at the transducer (IDT) and these radiate leftward and rightward along the substrate. In this way half of the energy is eventually reflected from reflectors positioned on the left side and half on the right. The identification (ID) and reference (R) reflectors share one wave path, and the other wave path is entirely devoted to chemical sensing in attempt to receive a strong signal despite the coated surface and possible chemical reaction on that side.

Following common practice in passive SAW tags [8,9,12], we have positioned the reflectors to achieve pulse delays at least 1 μ s after the query pulse. In wireless operation this allows the early background reflections (purely electromagnetic) to be excluded from the prominent acoustic modulated response. In Fig. 3 we show the pulse response of a device, which rapidly decreases until the onset of acoustic reflections at 1 μ s. The acoustic features of interest occur after the corresponding vertical line. For sensing purposes, the prominence of the later peaks ("R," "O₃") is important because we monitor these features and because a lossy film reduces their amplitude.

We used the inverse fast Fourier transform to convert the complex data from frequency domain to the time domain. A time response obtained in this way is a "pulse" response (rather than "impulse") because of the finite bandwidth of the measured data. The simulated response was computed using the delta function



Fig. 2. Labeled optical micrograph of a diced SAW device of the type used in subsequent measurements. "R" indicates the reference reflectors, "ID" the identification reflectors, and "O₃" the ozone-specific reflectors.

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