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High temperature LGS SAW gas sensor

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Abstract

Novel surface acoustic wave (SAW) devices using the recent langasite (LGS) family of crystals have been designed, fabricated, and tested for high temperature (up to 750 °C) gas sensor applications. The devices were fabricated using platinum (Pt) and palladium (Pd) thin film technology as electrodes and sensing films to withstand temperatures in excess of 1000 °C. Combinations of platinum and platinum/tungsten trioxide (Pt/WO_3) films have been used in the detection of C_2H_4 in N_2 (C_2H_4/N_2). Original all palladium metallic structures have been employed in the detection of H_2 in a N_2 carrier gas (H_2/N_2). SAW resonator structures using these films have been fabricated and continuously cycled from 250 to 750 °C for periods up to 3 weeks, with degradations in the $|S_{21}|$ response up to 7 dB. Constantly held at 250 °C for 12 weeks, the maximum degradation in the $|S_{21}|$ observed dropped to 3 dB. In order to perform the required gas testing, a high temperature, air sealed, stainless steel, dual chamber has been designed and fabricated to house these devices. The two-port SAW resonator structures fabricated with Pt, Pt/WO_3, and the original all-Pd films mentioned above have been tested with exposures to H_2/N_2 and C_2H_4/N_2 in the temperature range of 250–450 °C. Frequency variations up to 10 kHz for the 167 MHz high temperature SAW sensors were measured. The high temperature LGS SAW devices and experiments reported in this work show the capability of these devices to withstand prolonged exposure to temperatures ranging from 250 to 750 °C and to perform as high temperature gas sensors.

Keywords: High temperature; Langasite; Gas sensor; Hydrogen

1. Introduction

High temperature gas sensors capable of operating in harsh environments are needed for safety, aerospace, and space exploration industries [1]. Specifically, high temperature sensors are critical for safety in early stages of fire detection, in the detection of fuel leaks in jet engines, and in explosive conditions from combustion engines. For example, monitoring the levels of oxygen (O_2) and hydrogen (H_2) warns of explosive conditions. Monitoring the levels of carbon monoxide (CO) and carbon dioxide (CO_2) can be used in early fire detection alarms. In addition to the safety issue, the detection and monitoring of combustion cycle gases have environmental and economical appeals [1]. Emission control can be performed by watching the levels of nitric oxides (NO_x) and hydrocarbons (C_xH_y). In particular, the development of a high temperature gas sensor for aerospace industry applications will help to increase fuel efficiency in the combustion cycle, reduce environmental pollution, and decrease travel costs.

In recent years, significant research has been devoted to the development of high temperature nitrides (gallium nitride, GaN; indium nitride, InN; aluminum nitride, AlN; boron nitride, BN) [2–4]. These materials have been experimentally shown to operate up to 400 °C; however, extended operation at higher temperatures is compromised due to junction breakdown and electromigration. Silicon carbide (SiC) and diamond materials are being developed and have been shown to operate at temperatures up to 600 °C for short periods of time. Stable long-term operation is limited to a few days by measurand and temperature permanently induced modifications in the materials [3,5,6]. Fiber optic structural sensors to detect material flaws and cracks have been designed for use up to 1100 °C in airplane environments [7,8]. For high temperature gas and pressure sensors, fiber optic technology is limited to temperatures below 500 °C due to the materials

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used to fabricate the light sensor. Semi-conducting metal oxides (SMO) films, such as zirconium oxide, gallium oxide, and tungsten oxide, have also been the focus of intensive research for high temperature (up to 800 °C) sensors [1,9–12]. The response, stability, and sensing properties of these films are highly dependent on the fabrication process.

Regarding acoustic wave (AW) technology, bulk and surface acoustic wave devices (BAW and SAW) have been used as sensitive gas and liquid sensors due to their wide dynamic range, and benefits from the microelectronic technology such as small size, and reduced cost when produced in large quantities [13]. Most commonly quartz, lithium niobate ($LiNbO_3$), and lithium tantalate ($LiTaO_3$) crystals have been used as substrate materials. However, these crystals cannot be used above 500 °C. Quartz has an alpha–beta (α–β) transition at 573 °C, which causes the loss of piezoelectricity, and thus results in a non-operable sensor. $LiNbO_3$ has been used in the development of a SAW ID-tag up to 400 °C [14]. However, the acoustic wave properties in $LiNbO_3$ are highly dependent on temperature. Both $LiNbO_3$ and $LiTaO_3$ are pyroelectric materials, and therefore the electric charge or voltages, which are generated by temperature changes, limit the operation at elevated temperature.

More recently new piezoelectric crystals, which also operate at high temperatures, have been introduced for acoustic wave filters, frequency control, and sensor applications. The langasite family of crystals (LGX) and gallium orthophosphate ($GaPO_4$) can operate up to 1470 and 933 °C, respectively, and therefore be used for high temperature sensor applications [15–18]. The LGX family of crystals refers to three crystals, namely langasite (LGS, $La_3Ga_5SiO_{14}$), langanite (LGN, $La_3Ga_{5.5}Nb_{0.5}O_{14}$), and langatate (LGT, $La_3Ga_{3.5}Ta_{0.5}O_{14}$), and is part of a larger Ca-gallogermanate family of crystals with more than 100 compounds [15]. Langasite was first grown at the Moscow State University in 1980 using the Czochralski crystal growth method, and these crystals soon started to have their optic, acoustic, and piezoelectric properties researched. As a result of these fundamental material properties studies, the LGX crystals have shown to exhibit the following desirable acoustic wave properties: (i) up to about six times higher electromechanical coupling than quartz, (ii) existence of temperature compensated cuts with zero power flow angle and minimal diffraction, and (iii) up to 26% reduction in phase velocities with respect to quartz, which allows the fabrication of smaller devices. In addition to these desirable properties, LGX substrates do not have crystal phase transitions up to the melting point around 1470 °C, when compared to the α–β transition of quartz at 573 °C and $GaPO_4$ at 933 °C [15–17]. This last property makes LGX very attractive for SAW and BAW high temperature applications, a topic which has been recently investigated by different groups [19–22]. In a recent work [21], the authors have experimentally verified the expected loss of piezoelectricity for quartz between 570 and 580 °C due to the α–β transition, and the capability of LGS SAW devices to operate up to 750 °C for periods of at least 3 weeks, with less than 7 dB decrease in the magnitude of the transmission coefficient, $|S_{21}|$, and a reduction of the quality factor by approximately 25% (Fig. 1). The results obtained qualified the LGS crystals for high temperature SAW gas sensors applications.

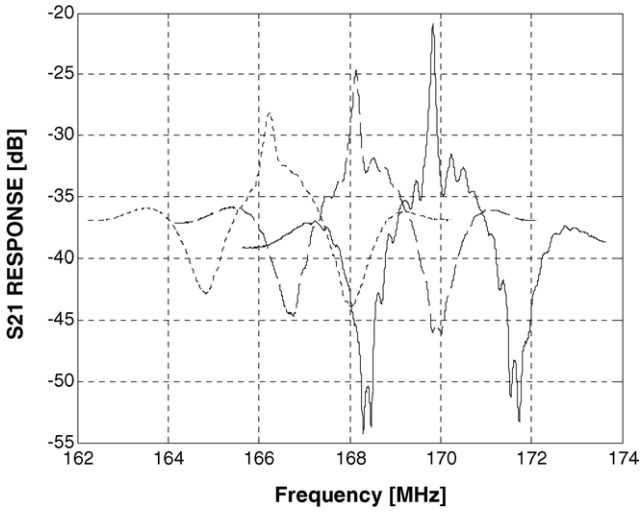

Fig. 1. Measured |S21| for the LGS two-port SAW resonator: solid, 25 °C; dashed, 500 °C; dotted, 750 °C.

This paper reports on high temperature LGS gas sensors fabricated and tested from 250 to 450 °C for periods up to 12 weeks. Original all palladium (Pd) two-port resonators have been used for the detection of hydrogen in nitrogen (H_2/N_2), and platinum (Pt) two-port resonators with and without an overlay sensing film of tungsten trioxide (WO_3) have been utilized in the detection of ethylene in nitrogen (C_2H_4/N_2). Zirconium (Zr) has been originally used as adhesion layer in the fabrication of the SAW devices to avoid the migration of the commonly used titanium (Ti) adhesion layer into the Pt film, which results in the loss of film adhesion [22–24]. An original stainless steel gas chamber and a gas delivery system have been designed and fabricated to introduce the target gases to the sensors at elevated temperatures (0–1000 °C). Section 2 discusses the design, fabrication, and material selection for the high temperature LGS SAW devices. Section 3 describes the originally designed high temperature radio frequency (RF) test and measurement system capable of withstanding temperatures of 1000 °C. Section 4 reports results on the Pd, Pt, and Pt/WO_3, two-port SAW high temperature gas sensors exposed to H_2/N_2 and C_2H_4/N_2, respectively. Section 5 is dedicated to the conclusions, followed by the acknowledgments.

2. High temperature SAW resonators

LGS substrates oriented along the propagation direction Euler angles (0°, 138.5°, 26.6°) have been used in the fabrication of two-port SAW resonators with the following characteristics: 80 electrodes interdigital transducers (IDTs),

500 electrodes short-circuited reflectors, 50 wavelength, λ, aperture, finger width 4 μm, with a 1:1 mark to space ratio.

The IDT electrode material used to excite, receive, and manipulate the SAW signal is of major concern for high temperature applications. Aluminum (Al), which is the most common electrode material used for SAW devices, cannot be employed at temperatures significantly above 300 °C due to the electro-migration phenomenon, well known in the semiconductor and SAW industries [25]. For high power and high temperature devices, a sandwich film technique has been devised, where multiple and distinct layers of metal are used to improve the device's temperature performance [25,26]. However, this technique has been reported to operate adequately only up to 200 °C. Therefore, Pt and Pd (melting points 1769 and 1554.9 °C, respectively) have been selected for the fabrication of the IDTs electrodes, in order to permit the SAW gas sensors capable of withstanding temperatures above 250 °C for long periods of time without significant metal film degradation or failure. Both metal films were deposited by electron-beam evaporation over a zirconium (Zr) adhesion layer at University of Maine. Titanium (Ti), a commonly used adhesion layer material for Pt and Pd, has been shown to migrate into Pt after prolonged exposure to high temperatures [24], causing the loss of film adhesion, which results in poor and unpredictable SAW transducer and resonator performances. Zr, which belongs to the same class as Ti, provides similar adhesion properties and does not migrate into the Pt layer [24].

Three different two-port SAW resonator metallization schemes were fabricated and tested for the detection of hydrogen in nitrogen (H_2/N_2) and ethylene in nitrogen (C_2H_4/N_2). The three different metallization schemes consisted of an all Pd two-port SAW resonator, an all Pt device, and a Pt device with a WO_3 sensing film.

The original all palladium (Pd) film SAW two-port resonator was fabricated for the detection of H_2/N_2. Pd has been widely used as a sensing film for the detection of H_2, due to the fact that Pd can absorb up to 900 times its own volume of H_2 at standard temperature and pressure [27]. Previous work with SAW delay lines by D'Amico et al. [28] has indicated that a thick Pd sensing layer has to be used in order to produce significant changes in the SAW delay line response. Therefore, all the Pd SAW resonators reported in this work were fabricated with a 200 Å Zr adhesion layer and a 3000 Å Pd electrode/sensing film. The two-port all Pt resonators used in the detection of C_2H_4/N_2 were fabricated with a 40 Å Zr adhesion layer and a 500 Å Pt electrode/sensing film. These films were deposited by electron-beam evaporation, and patterned with a lift-off photolithographic technique. Finally, WO_3 has been shown to optimally detect C_2H_4/N_2 at 350 °C in semi-conducting metal oxide film technology [29]. Therefore, the WO_3/Pt two-port SAW resonators were fabricated as the Pt devices previously mentioned with an additional 200 Å layer of WO_3 RF sputtered over the entire device including the non-metalized surface.

3. High temperature gas testing and RF measurement system

In order to test the SAW gas sensors at high temperature (up to 1000 °C), an appropriate gas chamber and gas delivery system had to be designed and implemented. The materials used in the gas chamber fabrication had to be carefully selected to permit continuous operation at high temperatures and to ensure that no significant contamination disturb the gas sensing, since most materials emit chemicals when heated to elevated temperatures. For this reason, stainless steel was selected for the gas chamber material, since outgassing of chemicals generally occurs around 600 °C [30]. Stainless steel gas compression connectors ensured an airtight gas seal, and an 1/8 in. ceramic paper seal guaranteed closure from air contaminants. The high temperature chamber consists of two isolated airtight chambers to house a reference and sensor device, as indicated in Fig. 2. Also shown in Fig. 2 are the SAW devices mounted to a sapphire substrate to allow for minimal disturbance to the system set-up when changing the testing device. The sapphire holding wafers used Pt film for the 50-Ω coplanar transmission line. The gas delivery system and gas chamber are capable of withstanding temperatures in excess of 1000 °C due to the materials used in the system. The experimental set-up shown in Fig. 2, including the stainless steel chamber has been designed and fabricated at UMaine.

Fig. 2 also depicts the stainless steel tubing used to introduce the target gases to the high temperature gas chamber, and the two-high temperature coaxial transmission lines (Meggitt Safety Systems) used to provide the RF access to the devices under test. These coaxial transmission lines are capable of withstanding 1000 °C due to the usage of SiO_2 as dielectric.

In order to bring the SAW gas sensor system to the required high temperatures, a Thermolyne 6000 Furnace capable of heating up to 1200 °C was employed. Tylan Mass Flow Controllers were used to precisely deliver 10 ppm C_2H_4/N_2 and 1000 ppm H_2/N_2 target gases and O_2 and N_2 baseline gas flow rates to the high temperature test chamber. An HP 8753ES network analyzer and a PC were used to save and analyze the measured data. Fig. 3 shows a block diagram of the experimental set-up implemented.

4. Experimental results

The experiments for the detection of H_2 in a N_2 carrier gas (1000 ppm H_2/N_2) with the all Pd two-port SAW resonators used alternatively O_2 or N_2 as a baseline gas. Figs. 4 and 5 plot the response of the H_2/N_2 exposure to the all Pd resonator with an O_2 baseline at 250 °C. Fig. 4 shows the actual frequency variation (Δf) as a function of time, with the (∗) indicating the turning on of the H_2/N_2 mixture and turning off of the O_2 gas, and the (▲) representing the turning off of H_2/N_2 and the reintroduction to O_2 gas. Fig. 5 shows the

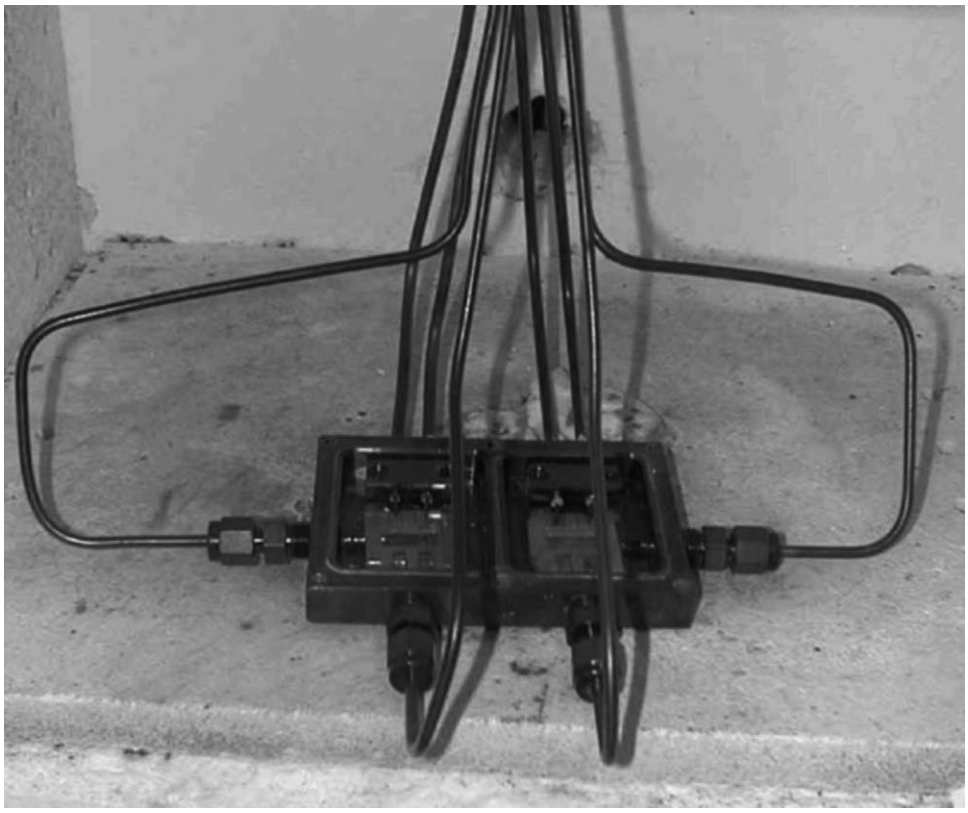

Fig. 2. SAW two-port resonator gas sensors mounted on sapphire substrates, and air tight high temperature gas chamber with the lid removed.

frequency variation for each test run, indicating the change in frequency from subsequent H_2/N_2 exposure cycling at 250 °C. As observed from Fig. 5, there is a consistent decrease in frequency with exposure to H_2/N_2 in the 5–8 kHz range. The recovery with the reintroduction of O_2 and removal of H_2/N_2 ranges from approximately 2–6 kHz. A constant drift in the center frequency response can be observed from Fig. 4. One possible explanation for this constant frequency decrease is that the surface is being cleaned of carbon atoms, by a reduction–oxidation cycle.

Figs. 6 and 7 plot the response of the H_2/N_2 exposure to the all Pd resonator with a N_2 baseline at 250 °C. Fig. 6 shows the actual frequency variation as a function of time, with the (✳) indicating the turning on of the H_2/N_2 mixture and turning off of the pure N_2 that was establishing the baseline, and the (▲) representing the turning off of H_2/N_2 and the reintroduction to N_2 gas for the baseline. Fig. 7 shows the frequency variation for each test run, indicating the change in frequency from subsequent H_2/N_2 exposure cycling at 250 °C. The constant decrease in frequency with exposure to H_2/N_2 is in the range of 5–6 kHz. The recovery is on the same order of magnitude, 4–8 kHz, after the H_2/N_2 gas is turned off, and the N_2 baseline is turned back on. As seen from Figs. 6 and 7, the

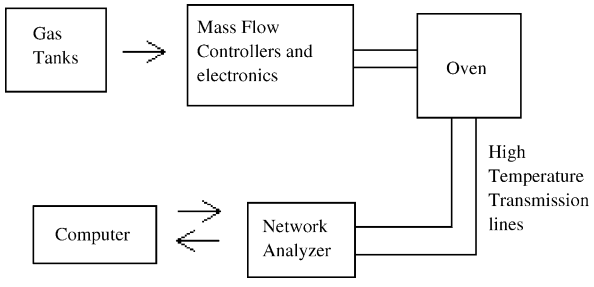

Fig. 3. Block diagram of measurement set-up.

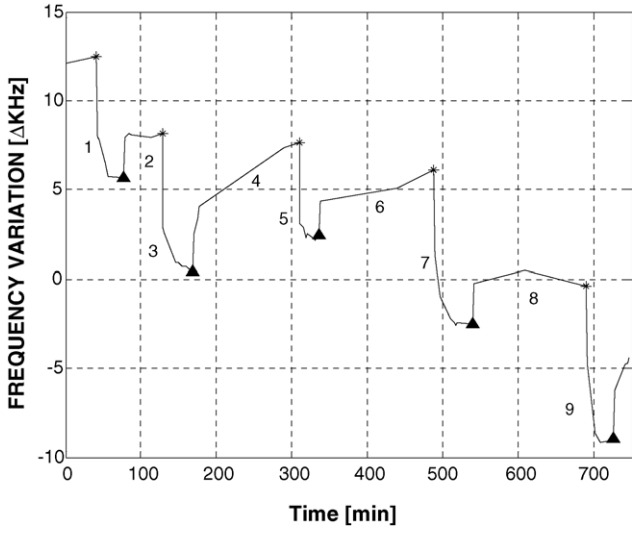

Fig. 4. Cycling exposure to hydrogen with an oxygen baseline: (✳) H_2 on and (▲) H_2 off (f_O 167.205 MHz).

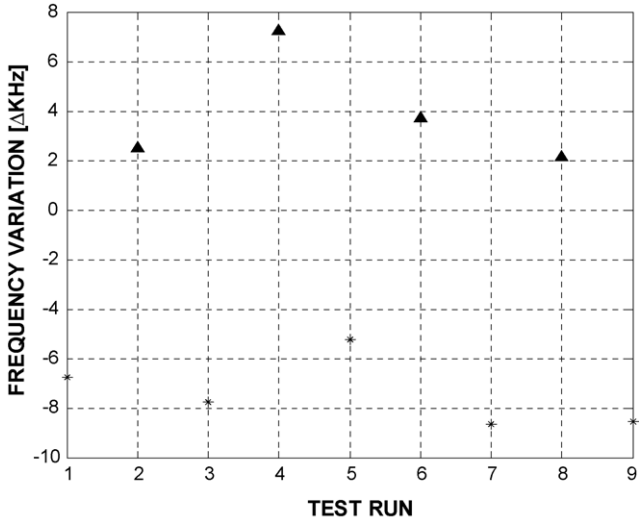

Fig. 5. Δf (kHz) of multiple exposures and recoveries of hydrogen/oxygen cycling: (✳) H_2 on and (▲) H_2 off.

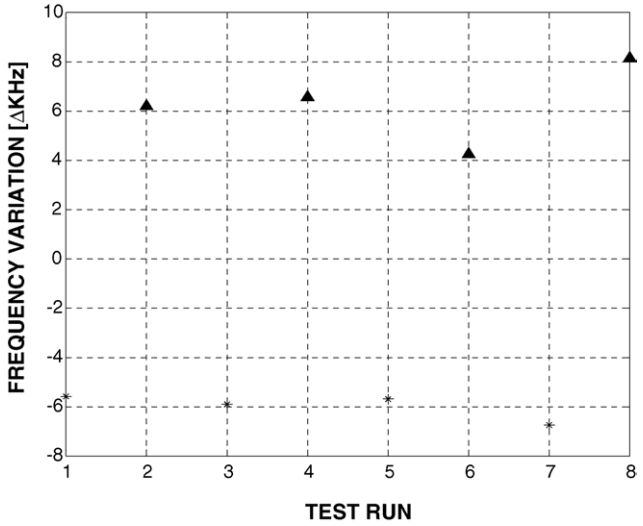

Fig. 7. Δf (kHz) of multiple exposures and recoveries of hydrogen/nitrogen cycling: (✳) H_2 on and (▲) H_2 off.

frequency shifts due to the exposure to H_2/N_2 and the recovery to the N_2 baseline are consistent and repeatable, with the magnitude of both changes around 6 kHz. The results shown in Figs. 4–7 indicate that N_2 provides a more stable baseline when compared to O_2 as the gas used to establish the sensor baseline.

Long-term exposure to high temperatures is a necessity for high temperature sensors. All the Pd LGS two-port resonator sensors were continuously tested and operated at 250 °C with an overall $|S_{21}|$ degradation of less than 3 dB after a 12-week period.

Feasibility tests have also been performed for the detection of C_2H_4/N_2 using all Pt and Pt with WO_3 overlaying film. In executing these tests, the Thermolynne 6000 furnace was at the target temperature for more than 24 h to ensure that the two-port resonators were at the operating temperature. The Pt resonators were exposed to O_2 for 30 min to oxidize the Pt film, followed by an exposure of 10 ppm C_2H_4/N_2 for 15 min. Through the moderately high temperature range selected (250–350 °C), the dominating physical phenomenon was the removal of surface-bound O_2 from the resonator by the reaction with C_2H_4/N_2 to form CO_2 and H_2O.

Fig. 8 plots frequency variation between the exposure to O_2 for about 45 min and the subsequent exposure to C_2H_4/N_2 for about 15 min as a function of temperature between 250 and 350 °C. The graph shows a positive Δf from 0.3 kHz at 250 °C to 1.1 kHz at 350 °C, indicating the removal of mass from the Pt film of the all Pt resonator and that at higher temperatures, within the mentioned range, a more efficient removal of the O_2 takes place.

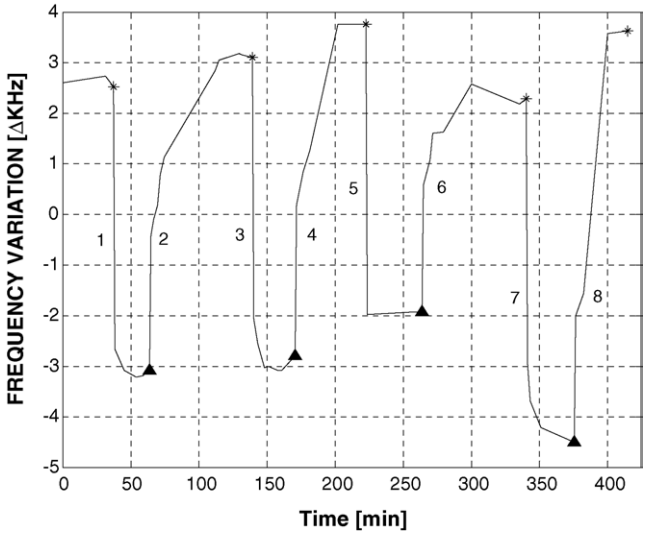

Fig. 6. Cycling exposure to hydrogen with a nitrogen baseline: (✳) H_2 on and (▲) H_2 off (f_O = 167.1475 MHz).

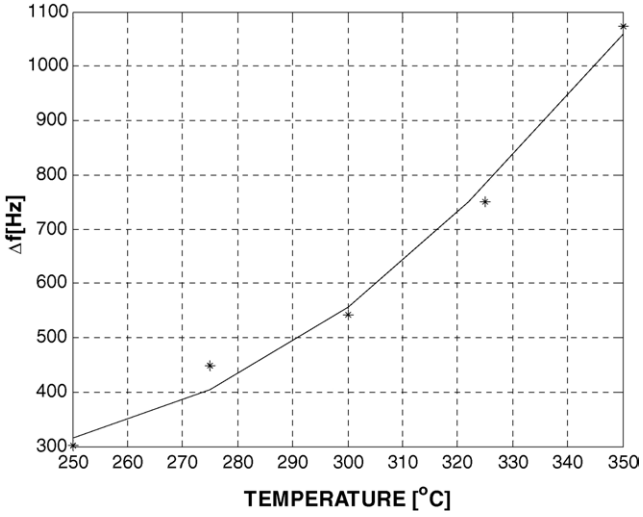

Fig. 8. Δf vs. T (°C) plot indicating the removal of O_2 from Pt and formation of CO_2 and H_2O after C_2H_4/N_2 exposure.

Table 1
Measured frequency variations (Δf) with exposure to C_2H_4/N_2 on a Pt/WO_3 gas sensor

Temperature (°C)	Δf (kHz)
300	1
350	1.5
400	1.2
450	0.8

In an attempt to increase the device's response to C_2H_4/N_2 at high temperature, WO_3 film has been deposited over the entire two-port SAW LGS Pt resonator. In the experiments conducted, the WO_3/Pt resonator was exposed to O_2 for 30 min, followed by the exposure to C_2H_4/N_2 for 20 min, over the temperature range of 300–450 °C. The selection of this temperature range was based on previous SMO work [29], which indicated strong variations in conductivity around 350 °C in the detection of C_2H_4/N_2 using WO_3. For the SAW sensors tested, the frequency variations using the WO_3 film improved by 85% at 300 °C and 39% at 350 °C when compared to the bare Pt resonator SAW device, as can be inferred from Fig. 8 and Table 1.

5. Conclusions

LGS SAW devices have been designed, fabricated and tested at UMaine as high temperature gas sensors. High temperature chambers have been also designed and fabricated at UMaine and a high temperature gas test set-up has been implemented. The devices were initially tested and cycled in temperature from 250 to 750 °C for periods up to 3 weeks, with degradations in the $|S_{21}|$ response up to 7 dB.

For high temperature sensor applications, palladium, platinum, and tungsten trioxide thin films were used as electrodes and/or sensing films on two-port SAW resonators for the detection of hydrogen and ethylene in nitrogen gas over temperatures ranging from 250 to 450 °C. Ethylene has been detected by all Pt SAW resonators and by the deposition of tungsten trioxide over the entire Pt SAW resonator device over the temperature range from 250 to 450 °C. The use of the WO_3 film improved the C_2H_4/N_2 detection up to 85%.

The original all Pd LGS SAW resonators used for the detection of hydrogen in nitrogen gave consistent and repeatable frequency variation readings in detecting H_2 in nitrogen gas at 250 °C, with a degradation in |S21| of less than 3 dB over a test period of 3 months.

The results presented in this paper also verify that LGS is an applicable substrate to be used as a platform for high temperature gas sensor over an extended time period, with minimal degradation in the device's response.

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Biographies

Jeremy Thiele was born in Portland, Maine, on 6 May 1980. He received a BS degree in electrical engineering from the University of Maine in May 2002. He is currently working toward his MS degree in electrical engineering at the University of Maine where his research is focused on high temperature surface acoustic wave device*s*. Jeremy has also done research work in the area of semi-conducting metal oxide sensors at the Laboratory for Surface Science and Technology at the University of Maine. Jeremy is a student member of the *IEEE Society*.

Mauricio Pereira da Cunha, born in Brazil in 1963, received the Bachelor's degree, 1985 and the Master's with Honors in electrical engineering, 1989, from the Escola Politécnica, Universidade de São Paulo. Master's thesis title is *Design and implementation of a 70 MHz SAW convolver*. He received the PhD, Dean's Honor List, from McGill University, Canada, in electrical engineering in 1994. PhD thesis title is *SAW propagation and device modeling on arbitrarily oriented substrates*; Mauricio has worked with the Microwave Devices R&D Group at NEC (Nippon Electric Co.), Brazil, Laboratório de Microeletrônica, Escola Politécnica, Department of Electrical Engineering, Universidade de São Paulo, McGill University, Montreal, PQ, Canada, and SAWTEK Inc., Orlando, FL. He passed a sabbatical year at University of Central Florida, Consortium for Applied Acoustoelectronic Technology (CAAT), Orlando, FL, where he worked in cooperation with Piezotechnology Inc., on the characterization of new piezoelectric materials, namely langatate, langanite, and langasite, and with bulk and surface acoustic wave devices. Mauricio was a professor in the Department of Electronic Engineering, Universidade de São Paulo,until he joined the Department of Electrical and Computer Engineering at the University of Maine in 2001; Dr. M.P. da Cunha is a member of the IEEE, Sigma Xi, and of the Brazilian Microwave Society (SBMO). He was elected to serve on the SBMO Administrative Committee from 1996 to 1999. He is a reviewer for the *IEEE UFFC Transactions*, and he has been a member of the IEEE International Ultrasonics Symposium Technical Program Committee since 1997. He has been recently elected to serve on the UFFC-Society Administrative Committee 2002–2005. At Ultrasonics Symposia,he gave the invited paper, *High velocity pseudo surface acoustic waves (HVPSAW): further insight*, in 1996, and presented the short course, *SAW, PSAW, and BAW materials and devices* in 2000. He wrote the invited paper *Pseudo and high velocity pseudo SAWs*, for the *International Journal of High Speed Electronics and Systems* (IJHSES), also published as a chapter in the book *Advances in SAW Technology, Systems and Application*, vol. 2, Singapore (2001).