Parameter Optimization of a Microfabricated Surface Acoustic Wave Sensor for Inert Gas Detection*

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ABSTRACT

This work is related to designing, fabricating, and testing a surface acoustic wave sensor to be used for detecting metastable inert gases, particularly helium. The assembly consists of two microsensor configurations: (a) a reference device with no deposition at the delay line and (b) a sensing device with an Au-activated TiO₂ e-beam-deposited thin film on the delay line. The interdigitated transducers and delay lines are fabricated by photolithography techniques on a single Y-cut LiNbO₃ substrate oriented for Z-propagation of the acoustic waves. Variation in electrical conductivity of the Au-activated TiO₂ film due to exposure to metastable He is translated as a frequency change in the assembly. Various characteristics of the surface acoustic microsensor have been studied to better understand and optimize the variation of acoustic wave velocity and the operating frequency of the microdevice. Methods for the TiO₂ thin-film deposition are discussed.

INTRODUCTION

Surface acoustic wave (SAW) sensors have been used in many fields because of their wide applicability. The technology of varying the SAW velocity by some physical variable is attractive due to the ease in measurement and quick sensor turnaround time during manufacturing. SAW sensors have been tested for robustness and have proven to provide repeatable results with good sensitivity and resolution.

Many SAW sensor applications, e.g., sensing of vapors, flow, optical orientation, force, pressure, mechanical positioning, voltage, temperature, acceleration, viscosity, density, and thermal conductivity have been thoroughly investigated [1-8]. Most of these applications rely on mass loading on films deposited on a delay line intermediate between the transmitting and receiving transducers. This mass loading is propagated into a change in a frequency shift or phase change of the SAW either of which is easily detected.

The basic principle of the SAW sensor is that a frequency signal is applied to the transmitting interdigitated transducer (IDT). The SAW undergoes a variation in the delay line due to interaction with a physical variable most commonly realized by employing a sensing film. This variation in the SAW is transmitted to the receiving IDT. Applying signal amplification to the received wave allows accurate detection of a change in the operating frequency of the device.

Metastable He atoms (1s2s¹S- or ³S-states) belong to electron-excited states that are spin-prohibited from returning to their ground state. A large number of metastable He atoms exist in ionized plasma because of the long lifetime of metastable He atoms. In the past, electrical

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conductivity of ZnO has been found to have a direct relationship with the presence of metastable atoms. Kupriyanov et al. [9, 10] employed activating thin-film surfaces with microcrystals of metals that interact with metastable atoms (thus demonstrating electron coupling with semiconductors) but the results did not demonstrate the direct correlation of a parametric change with He pressure. Much other work has metastable He but none has reported the use of a microsensor that was fabricated specifically for detection of metastable He [11, 12].

Surface interaction in the gaseous phase is due primarily to the high density of metastable atoms, and a robust method of inert gas detection is available through metastable He detection. This paper discusses a micromachined SAW sensor that detects metastable He. The sensing system used in this device consists of an Au-activated TiO₂ film on an LiNbO₃ piezoelectric substrate. A method to deposit the TiO₂ film has also been proposed. The unique aspect of the present research is the development of a microsensor with the potential for detecting a frequency change of the SAW with respect to a variation in the electrical conductivity of an Au-activated TiO₂ thin film deposited on the delay line of the SAW microsensor when exposed to metastable He atoms.

EXPERIMENT

Figure 1 shows the configuration of the He SAW microsensor, which consists of (a) a sensing device that uses a pair of transmitting and receiving IDTs with a delay line coated with an Au-activated TiO₂ film capable of sensing metastable He and (b) a reference device configured in the same manner but without the sensing film on the delay line [13]. By comparing the outputs from the sensing and reference devices, we investigated the exposure and reactivity of the sensing film on the delay line to metastable He atoms. An Au-activated TiO₂ thin film was deposited by using an Edwards Auto 306 reactive electron beam evaporator. Reactive electron-beam evaporation was utilized in thin-film deposition because the metal transforms directly from vapor to solid and the thin films are rapidly cooled ($\approx 10^{13}$ K/s), leading to ultrafine structures [14].

TiO₂ thin films are chemically resistant and have high temperature stability. Several researchers have studied different starting materials such as TiO₂, Ti₃O₅, and TiO [15]. The present investigation was oriented toward utilizing Ti₂O₃ as a source material due to its uniform evaporation rate. Ti₂O₃ has not been explored greatly in terms of surface roughness, and a successful attempt was made to deposit consistent-quality thin TiO₂ films by introducing O₂ into the chamber. Oxygen chamber pressure was altered until good-quality TiO₂-rich films were produced.

To begin the deposition, we placed the Ti₂O₃ powder in an Mo crucible inside the evaporator. The background chamber pressure of 5×10^{-6} torr was used with a deposition rate of 0.2 nm/s. When a stable deposition rate was achieved and Ti₂O₃ began evaporating, oxygen was leaked into the chamber such that the chamber pressure did not exceed the threshold of 3×10^{-4} torr. Electron dispersion spectroscopy (EDS) studies determined the film composition with a Hitachi S-4500 scanning electron microscope. Scans were performed with a working distance of 15 mm, an EDS distance of 50 mm, and an accelerating voltage of 5 kV to detect lower-energy Ti L-lines. An oxygen pressure of $1.2\text{--}2.9 \times 10^{-4}$ torr was necessary to produce stoichiometric TiO₂ thin films.

To pattern the IDTs, photolithography was employed by using a positive photoresist to coat the substrate, exposing the photoresist-covered substrate to UV light through the IDT pattern by using a photomask, and developing and rinsing the photoresist to expose the substrate

Fig. 1. SAW microsensor configuration.

Fig. 2. Optical photomicrograph of a portion of interdigitated transducer of microsensor showing consistent finger width (50x).

specifically patterning the IDTs. 1800 Å Cr and 200 Å Au films were deposited by electron beam evaporation on the substrate, patterning the IDTs of the reference and the sensing device. The Au layer reduced the contact resistance between the sensor external electronics. After deposition, unwanted metal was removed by liftoff. Figure 2 is a magnified photomicrograph of the IDT, showing the consistent thickness of the fingers. With the same deposition and liftoff procedure, the sensing film of Au/TiO₂ was applied to the delay line of the sensing device. The contact pads of the SAW microsensor were interfaced to the external electronics with gold wire. With conductive epoxy, 3-mil gold wires were adhered to the contact pads, which were then connected to a 24-pin integrated circuit socket. The pluggable sensor, shown in Fig. 3, was connected directly to external electronics for testing.

Fig. 3. SAW microsensor mounted on a 24-pin socket.

The IDTs of the sensors were designed to operate at 108.9 Mhz with an impedance of $50\ \Omega$ at the transducers. A voltage-controlled oscillator was used to provide an output frequency of 108.9 MHz controlled by a tuning voltage applied externally to the circuit [15]. The integrated electronics were attached to the IC socket of the SAW microsensor, which was enclosed in a sensing chamber in the test setup. Because the sensing film detects metastable He, a plasma chamber was incorporated into the gas flow system to excite stable He to the higher-energy metastable state.

RESULTS

The as-fabricated SAW microsensor was used to detect metastable He and the mechanism was found stable due to the one-to-one correspondence of metastable atoms exposed during surface interaction. Variation in the frequency at the receiving ends of the IDTs of both the sensing and the reference devices provided the frequency change with respect to exposure to metastable He. To ensure consistent film thickness, a Tencor P-10 Surface Profiler was employed to determine surface roughness and film thickness with profile lengths of $100\ \mu\text{m}$.

Figure 4(a) shows the effect of the oxygen chamber pressure to the root-mean-squared surface roughness (R_{rms}) of the thin film and Fig. 4(b) shows how the R_{rms} varies with film thickness. A consistent roughness of 2 nm was observed, with an uncertainty of $\pm 0.4\ \text{nm}$ due to equipment limitations. These values are much lower than the film thickness employed for TiO_2 and therefore do not affect the results significantly. Figure 5 shows variation of the frequency difference with increasing He pressure; on-set of plasma is at an He pressure of 10 kPa. Because both the sensing and the reference devices experience identical environments, the frequency shifts are independent of most parameters. Due to possible change in processing conditions, and also to dust trapped in the IDT fingers, small variations in the sensing behavior were found. These small changes were investigated and provided a baseline frequency difference of $\approx 4\ \text{kHz}$, noticeable when experiments were run in vacuum for long periods.

From Fig. 5, when production of metastable He atoms began, a rise in the frequency shift was observed due to activation of the Au atoms overlying the TiO_2 thin film. The metastable He collided with the sensing film, thereby imparting its decay energy to the film. With the bombardment of He atoms with Au, additional energy was imparted and the Au impurities in the semiconducting TiO_2 film were activated as deep traps, varying the electrical conductivity of the film. The change in electrical conductivity was translated as a change in the frequency of the sensing device and therefore as a change in the frequency difference between the sensing and the

Fig. 4. (a) Effect of O_2 pressure on surface roughness (R_{rms}) of film; (b) surface roughness variation for different film thicknesses.

Fig. 5. He pressure vs. frequency difference between sensor and reference devices.

reference devices. Due to the limited number of Au atoms, a constant variation was observed in the curve where increased pressure of He did not increase the electrical conductivity of the sensing film. Due to stripping of the Au atoms by metastable He atoms, the variation becomes abrupt at certain He pressures. This abrupt variation may be attributed to sensor vibration in the chamber. When the plasma was shut off, no metastable He atoms were produced and, after a momentary time lapse for evacuation of the leftover metastable He, the change in frequency shift decreased dramatically, as expected, eventually becoming constant at a level equal to the baseline difference.

CONCLUSIONS

We have developed a microsensor for the detection of metastable He. The microsensor assembly consisted of two SAW configurations, one a reference with no deposition at the delay line and the other sensing with an Au-activated TiO_2 e-beam-deposited thin film (sensitive to

electrical conductivity changes) on the delay line. The sensitivity of the device was limited to the number of Au atoms present in the Au-TiO₂ sensing film. As the Au atoms interact with the metastable He, a change in the electrical conductivity of TiO₂ was observed as a change in the frequency difference between the sensing and the reference devices of the microsensor. Due to bombardment of the metastable He atoms with Au atoms, some of the Au atoms were removed from the sensing film and therefore, even after saturation of the sensing film with metastable He atoms, some variation in frequency was observed due to varying numbers of Au atoms in the sensing Au/TiO₂ film. These results indicate the role of metastable He atoms in the detection of surface interaction with Au-atoms. In the future, we will study the width of the delay line, temperature exposure, operating frequency, IDT finger width and separation, and separation between IDTs and delay line to better understand and optimize the variation of acoustic wave velocity with varying operating frequency of the SAW device.

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