THERMALLY ISOLATED MICROMACHINED QUARTZ RESONATOR ARRAY FOR DIFFERENTIAL MICRO-CALORIMETRIC MEASUREMENTS

David Gaddes¹ and Srinivas Tadigadapa^{1,2}

¹Department of Biomedical Engineering, The Pennsylvania State University, USA

²Department of Electrical Engineering, The Pennsylvania State University, USA

INTRODUCTION

We have reported on the concept and performance of a calorimetric biosensor using a micromachined Y-cut quartz resonator as sensitive thermometer and a physically decoupled but closely located microfluidic reaction chamber[1]. Using layer by layer urease enzyme immobilization and combining with microfluidic pumping of the analyte we were able to demonstrate continuous measurements of urea with resolution of ~ 2 mM [2]. Micromachined bulk acoustic wave resonators with frequencies in few hundred MHz allow for temperature resolution of tens of microKelvin and better SNR as compared to thin film thermistors and thermocouples [1]. Proximity coupling of the thermal energy via conduction through thin film of air and radiation from the reaction chamber to the resonator thermometer, allows for complete separation of the biology and the physical sensor/electronics. In this paper we report on over 18 times improvement of this calorimetric sensor design achieved through improved thermal isolation of the resonators and differential sensor configuration to remove common mode temperature artifacts.

RESULTS AND DISCUSSION

8-resonator arrays of monolithic, micromachined quartz resonators of thickness $10\text{ }\mu\text{m}$ (167 MHz) were fabricated using RIE etching of quartz [3] as schematically shown in Fig. 1. The inverted mesa design of the resonators resulted in large thermal coupling and cross-talk between the resonators. Focused ion beam etching was used to create a thermally isolated resonator pixel (Fig. 1). The thermally isolated resonator was used as the reference sensor which provides a sensitive reference for background temperature changes as well as cancels out any spurious noise. For these measurements a 1 mm diameter Kapton[®] tubing with wall thickness of $25\text{ }\mu\text{m}$ was used. A nichrome wire of $4\text{ }\Omega$ resistance was wound and placed upstream in the tube and the temperature of the water downstream from the heater was measured by inserting a thermocouple into the tube. Two such Kapton[®] tubes (channels) are placed above the resonator chip (Fig. 2). Although only channel 1 was exposed to heated water (6.05 mW for 20 s), in the case where the resonator is not thermally isolated, heat is conducted through the quartz substrate and results in the temperature of both resonators to rise and results in poor compensation in the differential mode (Fig. 3(a)). For the thermally isolated resonator chip, when only channel 1 was exposed to heat ($450\text{ }\mu\text{W}$, 20 s), excellent compensation in the differential mode is achieved (Fig. 3(b)). Similarly, when a common mode thermal signal (3.0 mW , 20 s) is applied simultaneously to both channels, excellent compensation is achieved in the thermally isolated sensor chip (Fig. 3(c)). In this case the reference sensor effectively cancels both thermal drift as well as the large $\sim 175\text{ Hz}$ common mode response. Allan deviation noise characteristics of similar

frequency Y-cut and AT-cut resonators are compared in Fig. 4. The noise in Y-cut resonator is 10 times larger mainly due to radiatively and conductively induced thermal response. Fig. 5 shows the thermal sensitivity of various Y-cut resonators, showing 2.25 times higher sensitivity for the 165 MHz resonator in comparison to a 68 MHz resonator. Furthermore, the 167 MHz thinner and thermally isolated resonator with IR reflective gold electrode has 1.76 times lower noise than 68 MHz Y-cut resonator thereby improving the overall SNR by 4.73 times. Combining the improved differential measurement achieved by the thermally isolated sensor with the improved SNR of the 167 MHz resonator, an improved resolution of $110\text{ }\mu\text{W}$ from the previous limit of 2 mW measured for a non-isolated 90 MHz resonator is realized. The improved resonator design is expected to enable quantification of urea down to $275\text{ }\mu\text{M}$ where previously only 5 mM was possible.

Figure 1: (a) Schematic 8-pixel Y-cut quartz resonator array (b) Fabricated device with thermal isolation cuts along the marked lines

Figure 2: Schematic of the dual channel calorimetric sensing system.

Figure 3: Differential measurement based compensation: (a) Resonator array (without thermal isolation). Heated solution in channel 1 and channel 2 acts as a reference. (b) Resonator array with thermally isolated resonator under channel 2 acts as a reference. Heated solution in channel 1 (c) Common mode thermal signal compensation using the thermally isolated resonator array.

Figure 4: Allan Deviation of Y-cut resonators at 68 MHz (red), a 70 MHz AT cut quartz resonator (blue), a 165 MHz resonator (black) and an isolated 165 MHz resonator (green).

Figure 5: The sensitivity of a non-isolated 68 MHz resonator (red) and 167 MHz resonator (black) are compared with that of a thermally isolated 165 MHz resonator (blue).

References:

- [1] K. Ren, P. Kao, M. B. Pisani, and S. Tadigadapa, "Monitoring biochemical reactions using Y-cut quartz thermal sensors," *Analyst*, vol. 136, no. 14, pp. 2904–2911, 2011.
- [2] D. E. Gaddes, M. C. Demirel, W. B. Reeves, and S. Tadigadapa, "Remote calorimetric detection of urea via flow injection analysis," *Analyst*, vol. 140, no. 23, pp. 8033–40, 2015.
- [3] Hatipoglu, Gokhan, and Srinivas Tadigadapa. "Experimental studies in magnetically induced transverse force-frequency effect in thin quartz microresonators." *Journal of Applied Physics* 118.3 (2015): 034508.