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A single input-single output coupled microresonator array for the detection and identification of multiple analytes

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This work reports the experimental demonstration of single input-single output, multianalyte detection and identification using a coupled array of microresonators. A prototype sensor with four frequency-mistuned microbeam sensors, each coupled to a common shuttle mass resonator, is presented. Tailored localized modes of vibration in this coupled system are exploited to embed all requisite resonance shift information into the response of the common shuttle. Four standard polymers are applied to the microbeams to functionalize them for vapor detection. Toluene and methanol vapors, as well as toluene/methanol mixtures, are detected and identified using a single input signal and a single output signal. © *2008 American Institute of Physics*.

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Due to their inherently low power consumption, small size, and high sensitivity, microresonators are attractive for a myriad of chemical and biological sensing applications.[1–8] In typical implementations, microscale resonant mass sensors consist of a single-degree-of-freedom resonator with functionalized, specific, or partially specific surfaces. During the course of operation, target analytes bond to these functionalized surfaces, inducing resonance shifts which can be used for analyte detection.[9] Recently, multi-degree-of-freedom sensor architectures have also garnered some attention, but largely for the stated purpose of increasing device sensitivity.[10–12]

Currently, to identify multiple analytes an array of microresonators is required. Having a large array of isolated sensors, however, requires an equally large number of sensor outputs, which increases hardware and signal processing requirements thereby limiting, in part, the utility of the array approach.[13] To circumvent this complexity, single input-single output (SISO) detection can be achieved by exploiting a coupled microresonator array.[14] This work is believed to represent the first experimental demonstration of SISO, multichemical detection, and identification using a coupled array of microresonators.

A prototype sensor (Fig. 1), composed of four microcantilever sensors S_1, S_2, S_3, and S_4, each attached to a common shuttle mass SM resonator, has been designed and fabricated for the detection of multiple organic vapors. A detailed account of the modeling and analysis of this type of device has been presented in previous work.[14] A critical component in the design of the coupled system is that the frequency response contains tailored localized modes wherein the vibration energy is largely confined to a single microbeam sensor. The system was designed such that the resonances corresponding to these localized modes can be tracked through the shuttle mass using a single output signal. In practice, this was achieved by mistuning the lengths of the microbeam sensors and making their effective masses ($\approx 10^{-9}$ g) small relative to that of the shuttle mass ($\approx 10^{-7}$ g). The shuttle mass is approximately 334 μm long, 310 μm wide, and 5 μm thick. The microbeam sensors are approximately 20 μm wide and 1.3 μm thick and they have mistuned lengths of 129.4, 123.4, 118.2, and 113.5 μm (S_1, S_2, S_3, and S_4, respectively).

The device was fabricated on a silicon-on-insulator wafer with a 5 μm device layer. A deep reactive ion etch (DRIE) was used to define the device and a second DRIE step was used to define the thickness of the microbeam sensors. The underlying silicon substrate and silicon dioxide layers were removed using a wet potassium hydroxide back-

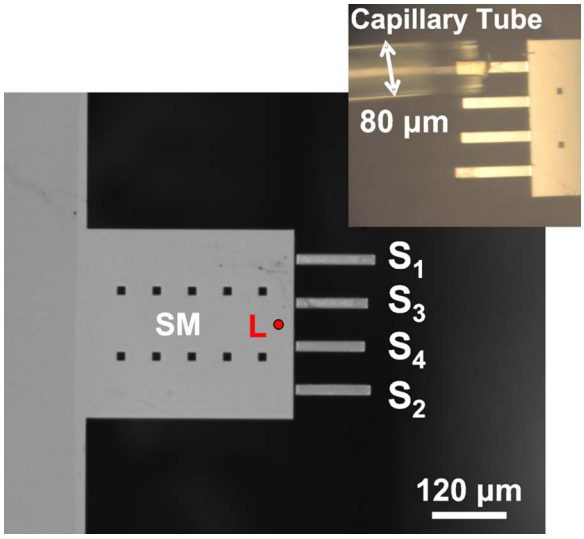

FIG. 1. (Color online) Microscope image depicting the sensor with labeled shuttle mass SM, microbeam sensors S_1, S_2, S_3, and S_4, and detection laser location L. The inset depicts the capillary tube method used to functionalize the microbeam sensors.

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TABLE I. Polymers and solvents used for microbeam functionalization. Solution concentrations shown as mg polymer per mL solvent.

	Polymer	Solvent	Concentration (mg/mL)
S_1	Polymethylmethacylate	Toluene	1.5
S_2	Polystyrene	Toluene	1.2
S_3	Polyurethane	Dichloromethane	0.6
S_4	Poly(4-vinylpyridine)	Ethanol	5.0

side etch followed by a wet hydrofluoric acid etch.

Polymers readily absorb organic compounds such as solvents and fragrances.[15] To functionalize the microbeams for the detection of organic vapors, different polymer coatings (from Sigma Aldrich) were used (see Table I). Polymer solutions with appropriate concentrations were prepared by mixing the polymers with solvents. The functionalization procedure involved using a precision three axis stage to accurately position the sensors and dip their tips into microcapillary tubes (80 μm outer diameter) containing the corresponding polymer solutions (Fig. 1).

Once functionalized, the device in Fig. 1 was base excited by a piezoelectric actuator and the motion of the shuttle mass was sensed with a Polytec laser vibrometer. The dot labeled L in Fig. 1 denotes the laser location. By applying a band-limited white noise signal to the piezoelectric actuator, all modes were simultaneously excited. The chamber pressure was held at 200 Torr for all experiments in this work. Figure 2 depicts the shuttle's frequency response after applying polymers to the microbeams. Note that the device has a bulk out-of-plane mode B and four higher frequency localized microbeam modes M_1–M_4, which are used for sensing (see Fig. 2 for experimental mode shapes). Also note that due to the localized nature of these modes, the mass responsivities (i.e., resonance frequency shifts per unit mass addition) associated with the resonances corresponding to M_1–M_4 are slightly less than the responsivities of an identical array of isolated uncoupled microbeams.[14] For the system of interest here, the mass responsivities are approximated to be between 11 and 31 Hz/pg. This, provided a frequency resolution of approximately 1 Hz, a metric which is easily obtained with the current setup, renders subpicogram mass sensitivities.

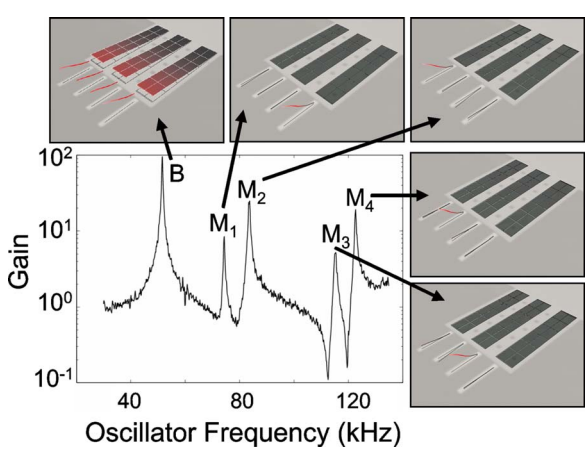

FIG. 2. (Color online) The frequency response of the device obtained experimentally by base exciting it with band-limited white noise and sensing the shuttle mass motion with the laser vibrometer in 200 Torr vacuum. The mode shapes, obtained experimentally using a Polytec scanning laser vibrometer, at resonances B, M_1, M_2, M_3, and M_4, are also depicted.

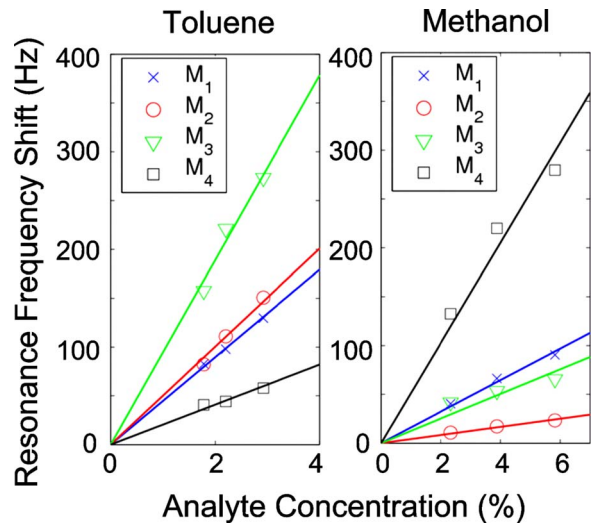

FIG. 3. (Color online) The resonance frequency shift of the localized microbeam modes (M_1–M_4) vs concentration of toluene (left) and methanol (right). Note that the analyte concentration is given by (moles of analyte/moles of mixture)$\times$100. Also note that in this figure, and in Fig. 4, the device was excited with a swept sine wave. Band-limited white noise excitation yielded similar results.

Using nitrogen as a carrier gas, analyte (toluene and methanol) vapors were created through the use of bubblers. Predetermined concentrations were created by mixing the analytes from the bubblers with pure nitrogen using mass flow controllers. A single output signal was measured optically from the shuttle mass, using the laser vibrometer, to determine the resonance frequencies M_1–M_4, which correspond to the localized microbeam modes of the coupled system. Detection involved measuring these resonance frequencies in pure nitrogen gas, introducing an analyte and waiting for the absorption to reach steady state, measuring the resonance frequencies again, and determining the resulting frequency shifts. In this manner the frequency shift of each localized microbeam mode was determined for various analyte concentrations. The sensitivity of each microbeam to a given analyte was determined by the polymer coating, and expressed as resonance frequency shift per unit analyte concentration. Figure 3 shows that the polymer coated microbeams exhibit substantially different sensitivities to toluene and methanol. For instance, the resonance of the localized microbeam mode M_4 is much more sensitive to methanol than it is to toluene. Figure 4 shows the resonance frequency shift of each localized microbeam mode due to the presence of 2.9% toluene vapor, 3.9% methanol vapor, and a mixture of 2.3% methanol and 2.3% toluene. Using the shifts of the two most sensitive microbeam modes M_3 and M_4 in Fig. 4(c) and their sensitivities from Fig. 3, the concentrations of methanol and toluene in the aforementioned vapor are estimated to be 2.8% and 2.0%, respectively, which are in good agreement with the actual concentrations of 2.3% methanol and 2.3% toluene. Toluene and methanol show very different signatures, based on the frequency shift patterns shown in Figs. 3 and 4, and therefore are identifiable with the sensor.

In conclusion, the unique ability to detect and identify multiple chemicals using a single input and single output signal is experimentally demonstrated for the first time using a coupled array of microresonators, which utilizes vibration localization. The sensor presented herein was created for

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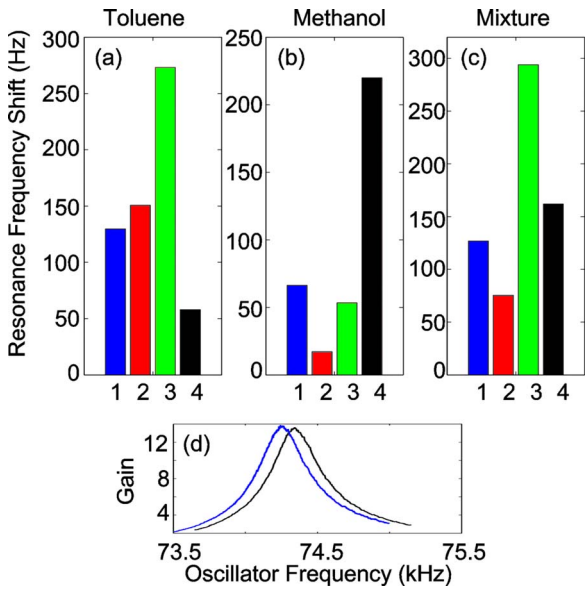

FIG. 4. (Color online) The resonance frequency shift of the localized microbeam modes M_1–M_4, which are labeled 1–4, respectively, due to the presence of (a) 2.9% toluene, (b) 3.9% methanol, and (c) a mixture of 2.3% methanol and 2.3% toluene. (d) Close-up of the downward resonance shift in the shuttle mass response near M_1 comparing pure nitrogen and 5.8% methanol environments.

proof of concept purposes and future architectures are projected to incorporate a larger number of sensors for increased functionality and feature smaller dimensions for increased sensitivity and improved performance in ambient environmental conditions. Additional surface chemistry explorations will also be necessary to address various selectivity and orthogonality issues that arise in the presence of numerous complex vapors.

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