On-Chip Dispersion Spectroscopy of CO₂ Using a Mid-Infrared Microring Resonator

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Abstract: We demonstrate on-chip molecular fingerprinting by measuring the refractive index dispersion of gas in the mid-IR using a thermally tuned suspended silicon microring resonator. We show CO₂ sensing down to 1000 ppm at 4.23 μm wavelength. © 2020 The Author(s)

Absorption spectroscopy relies on the measurement of a transmission spectrum through a sample of interest to determine its composition and the concentrations of its components. Using mid-infrared (mid-IR) light allows molecular identification with high specificity, since the mid-IR spectral range contains the sharp absorption lines associated with the fundamental vibrational and rotational modes of small molecules. For this reason, it is known as the “fingerprint region”. This method, applied at a single or a few wavelengths, is widely used by conventional low-cost sensors of trace gases such as CO₂, with important applications in agriculture, industry, and climate science. However, absorption-based gas measurements rely on the determination of optical intensity and are thus sensitive to fluctuations in the light source emission, coupling, and detection.

In contrast, refractive index sensing based on tracking the resonance wavelength of integrated optical resonant cavities is much less sensitive to intensity variations, and very low detection limits in small sensing volumes (below 1 μm^3) are regularly reported for sensors operating in the near-IR [1]. However, this near-IR detection is typically not specific, since the refraction is a broadband effect and not molecule specific.

Here, we present a gas sensor that combines the selectivity of absorption spectroscopy with the robustness of refractive index sensing, by leveraging the physical link between absorption and refractive index described by the Kramers-Kronig relations. The sensor exploits that the sharp molecular absorption peaks in the mid-IR also give rise to strong variations (dispersion) of the real part of the refractive index of gases. We demonstrate this sensing principle by performing on-chip dispersion spectroscopy of CO₂ using a mid-IR silicon (Si) microring resonator. To our knowledge, this has not been shown before, although absorption-based sensing of nitrous oxide has been shown using a silicon ring on sapphire [2].

Our racetrack-shaped microring resonator (Fig. 1 (a,e)) consists of a 935 μm long Si rib waveguide on a Si membrane suspended in air, 3 μm above the handle substrate (Fig. 1 (b–d)). The waveguide is 220 nm thick and 2 μm wide, and the supporting membrane is 70 nm thick. The bus waveguide has the same dimensions, is separated from the ring by a coupling gap of 1 μm , and terminates in a diffractive surface grating coupler. The waveguide is single mode at 4.23 μm wavelength, and we use its fundamental quasi-TE mode (Fig. 1 (d)), which features an evanescent field ratio (EFR) of 67.1 % and an external confinement factor Γ [3] of 50.0 %.

Fig. 1: SEM images of (a) the fabricated racetrack resonator and bus waveguide; (b) close-up view of the suspended ridge waveguide, and (c) in-coupling waveguide facet. (d) Cross-sectional FEM simulation of the waveguide, displaying the electric field profile of the deconfined fundamental quasi-TE mode. (e) Schematic of the setup used for characterization of the resonator and dispersion spectroscopy of CO₂.

Fig. 2: (a) Thermal tuning of the ring resonance. The inset shows a linear tuning rate of 0.18 nm/K. (b) The observed ring resonance wavelength shifts when switching from pure N₂ to diluted CO₂ in N₂, as a function of the initial spectral distance from the fixed CO₂ absorption line, for the three tested CO₂ concentrations.

The device was fabricated from a commercial silicon-on-insulator (SOI) substrate with a 220 nm Si device layer and a 3 μm SiO₂ buried oxide layer. The waveguide was patterned by electron-beam lithography and a timed partial dry etching of the Si device layer. A second lithography step and through dry etching of the Si layer formed holes through which the buried SiO₂ was removed by hydrofluoric acid wet etching. Before this final step, we cleaved the substrate to form a waveguide facet for coupling light in from the edge.

We characterized the ring resonator using the setup in Fig. 1 (e). The mid-IR light source is a tunable CW DFB quantum cascade laser (MLQD4232, Thorlabs, USA) focused onto the input facet of waveguide. The chip was placed inside a steel case with a Peltier cooler for temperature regulation, a gas inlet and an outlet, and a mid-IR-transparent window for visualization. We imaged the surface of the chip with a calibrated FILR A6700sc mid-IR camera, to quantify the ring transmission at the bus waveguide output and to measure the chip temperature. The measured free spectral range (FSR) of the microring resonator is 6.6 nm, which agrees well with finite element (FEM) eigenmode simulations of our ring waveguide. The coupled quality factor Q is 8000.

Fig. 2 (a) shows the thermal tuning of the ring resonance. In addition to the resonance dip, three dips due to CO₂ absorption are visible. By varying the temperature of the resonator chip using the Peltier element, we scan the ring resonance over the CO₂ absorption dip at 4234.7 μm and, thus, vary the overlap between the two dips. The resonance wavelength varies with temperature at a tuning rate of 0.18 nm/K (Fig. 2 (a) inset), and thus agrees well with the expected tuning rate of 0.17 nm/K based on the reference thermo-optic coefficient of Si of $1.7 \times 10^{-4} \text{ K}^{-1}$ at 4 μm and the simulated internal confinement factor $\partial n_{\text{eff}}/\partial n_{\text{Si}} = 0.7$ and group index $n_g = 2.9$.

We sensed CO₂ concentrations of 0.1 %, 0.5 %, and 1 % diluted in N₂ by alternatively injecting each CO₂ dilution and pure N₂ in 1 min intervals. The free-space optical paths before and after the chip box were continuously flushed with N₂. For each gas concentration and Peltier temperature setting, we measured the transmission spectra of the device, and the measured spectra were then fitted using a double-Lorentzian curve to track the ring resonance position relative to the CO₂ absorption dip. We highlight the very small gas volume sampled above the ring in this measurement, below 2000 μm^3 .

Fig. 2 (b) displays the observed ring resonance wavelength shifts when switching from pure N₂ to diluted CO₂ in N₂, as a function of the initial spectral distance from the fixed CO₂ absorption line, for the three tested CO₂ concentrations. We observe that our measured shifts closely follow the behavior predicted from the theoretical effective index change for our waveguide geometry, based on finite-element method simulations.

In summary, we have demonstrated on-chip dispersion spectroscopy in the mid-IR by sensing CO₂ down to 1000 ppm at 4.23 μm wavelength in a sensing volume below 2000 μm^3 . Our approach uses commonly available SOI substrates and provides a route for trace gas sensing in very small volumes, paving the way towards new and exciting applications in life-science and chemistry.

References

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