Supplementary information

A Monolithic Photonic Microcantilever Device for in-Situ Monitoring of Volatile Compounds.

Konstantinos Misiakos a, Ioannis Raptis a, Annamaria Gerardino b, Harry Contopanagos a, & Maria Kitsara a

(a) Institute of Microelectronics, NCSR “Demokritos” 15310, Athens, Greece,
(b) Istituto di Fotonica e Nanotecnologie (IFN), CNR, 00156 Roma, Italy

Monolithic Optocoupler Fabrication.

The optocoupler process flow consists of six lithographies, three low pressure chemical vapor deposition (LPCVD) steps and two implantations (phosphorus and boron) for the formation of the avalanche diode and the photodetector (boron) on the n-type substrate. The LPCVD steps included two for SiO₂ (field oxide and spacers) and one for Si₃N₄ (waveguide). The spacers were created next to the field oxide vertical walls by SiO₂ LPCVD and anisotropic plasma etching (Fig. 1a). Total field oxide thickness is 3 μm. The nitride film was then deposited (LPCVD) and lithographically patterned to create the waveguides. Then boron was implanted through the nitride film to form the self-aligned avalanche junctions by phosphorus compensation, as shown in Fig. 1a. Then a rapid thermal annealing of the boron implant followed by Al metalization and patterning completed the device (Fig. S1).
Figure S1: Microphotograph of the emitter. It shows the light emitted by the LED (white segment shown by the arrow), the nitride waveguide leading to the left and the Al contact to the emitter.

Narrow Trench Lithography.

PMMA (M_w=350K) resist was spun on the completed optocoupler wafer and prebaked at 160 °C for 60 min on a hot plate, with a film thickness of 700 nm. The narrow trenches were written by e-beam lithography with a Vistec EBPG 5HR working at 100KeV by beam current of 0.4nA and dose 1300μC/cm². The development was performed in isopropanol: deionized water 7:3 mixture for 30sec. After anisotropic plasma etching in CHF₃ for 11 min. the PMMA thickness was reduced to 450 nm.

Fig. S2 shows the photocurrent drop as a function of the etch depth of the unreleased cantilever fingers.
Figure S2: Detector photocurrent for the various stages of the waveguide patterning and dry etching. The LED excitation current was set at 10 mA which corresponds to a bias of 13 V. The photocurrent drops from the 715 pA level of the uncoated optocouplers to 542 pA following PMMA coating (red circles). This is caused by the bending losses over the spacers (Fig. 1a) which increase when the core-cladding refractive index step drops. The development of the comb pattern on PMMA causes another drop to 498 pA while the dry etch step reduces photocurrent to a minimum of 236 pA at complete nitride layer removal. Further dry etching of underlying silicon dioxide causes an increase to 279 pA.

Packaging and Measurement Set-up.

After the controlled cantilever release, the dies were diced and then mounted on a dual inline package and wire bonded. The package was placed on a printed circuit board and a thermocouple in contact with chip edge was monitoring the die temperature. The entire cartridge was inserted in a gas flow module connected to the gas lines while the board provided the electrical connections to the excitation current source and the current meter. Vapors were supplied in controlled concentrations by the use of mass flow controllers and by mixing pure nitrogen with nitrogen bubbled through water and methanol vessels. The gas delivery chamber was kept at temperature of 28.0 °C.
Independent verification of the Microcantilever bending

In Fig. S3, a tilted side-view SEM image of a defected cantilever device having completely released as well as unreleased fingers is illustrated. The two fingers at the bottom have not been released, while the top six fingers are released and bent. By using the unreleased fingers as a reference (red line is drawn parallel to them) and by taking into account the viewing angle, 60°, the cantilever edge deflection is measured 494nm. This deflection corresponds to zero vapor concentration and is in very good agreement with 468nm deflection calculated from eq. 1 at 25°C.

Figure S3: SEM image showing the bended upwards cantilevers. The red line shows the reference of the unreleased fingers.

To point out the change in microcantilever bending using external excitation, we focused white light on the released cantilever area and we monitored the reflectance spectrum by an Ocean Optics QE65000 spectrometer through an optical fiber mount in the place of the eyepiece of an optical microscope. Two cases are presented in Fig. S4, one with dry nitrogen
and another with 70000ppm methanol vapors. At the high methanol condition we clearly observe Fabry-Perot resonance which disappears at the dry nitrogen condition. The Fabry-Perot resonance indicates cantilever arms parallel to the silicon substrate (zero deflection) while the bending of the arms at dry nitrogen condition destroys the resonance.

**Figure S4:** White light reflectance spectra monitored through an optical fiber mount in the place of the eyepiece of an optical microscope while a light beam was focused on the released cantilevers. Methanol vapors were supplied at controlled concentration and the spectra of the reflected light was monitored through an Ocean Optics QE65000 spectrometer.

**Unreleased cantilevers response**

To point out that in figure 8 the monitored response is result of microcantilever and not an effect of a change in the PMMA refractive index, a measurement with unreleased cantilevers was carried out. Here the optocouplers had a similar geometry to the cantilever devices but with the notable difference that now the fingers have not been released (wet etching step skipped). In Fig. S5, the normalized photocurrent for a wide methanol vapor concentration range is plotted. The photocurrent increases by a fraction of 0.5% when the methanol concentration goes from 0ppm to nearly 100000ppm. This is to be compared with an order of magnitude photocurrent increase of the released cantilever device displayed in Fig. 8 of the manuscript for a similar change of methanol concentration.
Figure S5: Normalized photocurrent of unreleased cantilevers for a wide methanol vapor concentration range (from 0ppm to nearly 100000ppm)