ADVANCED MATERIALS

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Optical-Fiber-Mounted Porous Silicon Photonic Crystals for Sensing Organic Vapor Breakthrough in Activated Carbon**

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Fiber-optic-based sensors have been applied to a variety of remote sensing problems: measurement of pressure, humidity, vapor-phase chemicals, and aqueous biomolecules are leading examples.[1–6] With a width of only a few tens to hundreds of microns, these sensors require little power to operate, are impervious to electrical interference, and can be multiplexed together into distributed sensor configurations.[2,4,5] Fiber sensors may be extrinsic or intrinsic, depending on how the measured parameter is optically transduced;[4] intrinsic sensors, like interferometric and Fabry–Perot cavity devices, leverage the fiber's inherent optical properties to transform a parameter such as mechanical stress into an optical signal.[3,7,8] Extrinsic sensors, such as bead-based arrays[1] and polymer caps,[6] immobilize an indicator or label on the distal end of the fiber, using only the light-carrying capability of the fiber to transduce the sensor signal. In this work, an extrinsic sensor for volatile organic compounds (VOCs) is created by adhering a 0.5 mm-diameter porous Si photonic crystal to an optical fiber. Remote sensing with such a device is demonstrated by detecting breakthrough of VOCs in an activated carbon filtration bed.

There is a growing need for sensors that can monitor the residual adsorption capacity of activated carbon filtration cartridges in gas masks and other personal protective equipment.[9] Typically, these sensors operate by detecting organic vapors as they break through a filter bed of activated carbon. Despite advancements in end-of-service-life sensor technologies,[2,10,11] the need persists for a very small, low-power, and cost-effective sensor. In the United States, government health and safety regulations require the detection of VOCs prior to depletion of the carbon bed's adsorption capacity, yet these regulations have not yet been enforced due to a lack of suitable sensing devices.[12,13]

The porous Si photonic crystal was prepared by electrochemical dissolution of boron-doped p++ Si in a solution of aqueous hydrofluoric acid and ethanol. The current density applied during this process affects the pore size and porosity of the resulting structure.[14–16] A photonic crystal generated by application of a periodic current waveform yields a porous layer with spatially varying porosity in the z-direction (normal to the wafer surface). In the present work, a sinusoidal waveform is used, and the resulting samples display a single sharp spectral reflectivity peak (Fig. 1), at a wavelength determined by the periodicity of the refractive index in the porous layer.[17,18]

The porous Si photonic crystal film was removed from the Si wafer using an electropolishing reaction.[15,16] It was rinsed gently with ethanol, dried at ambient temperature with a stream of pure N_2, and affixed to the end of a cleaved silica-

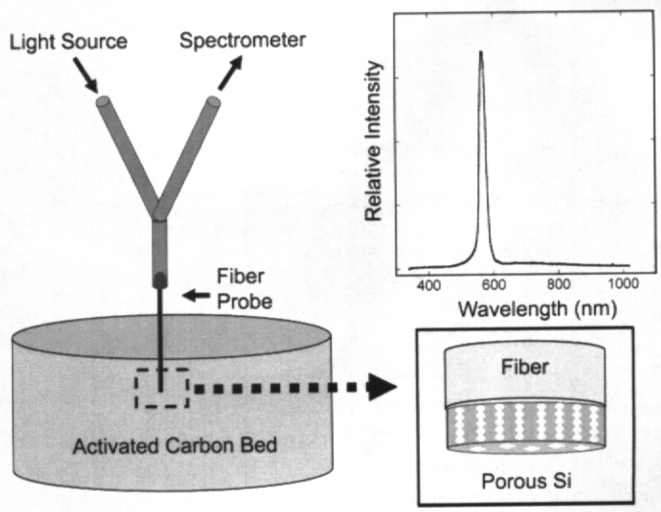

Figure 1. Diagram depicting the experimental setup for the in-situ monitoring of VOC breakthrough in an activated charcoal filtration bed. A porous Si photonic crystal is attached to a silica-core optical fiber (bottom right) and embedded in an activated carbon bed (left). A spectrometer monitors the reflected light spectrum from the photonic crystal (upper right). A red-shift in the spectrum is indicative of the presence of free organic vapors in the carbon bed.

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