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Novel Miniature Spectrometer for Remote Chemical Detection

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Research Objective

This research will develop an entirely new class of chemical sensing technology that will enable qualitative and quantitative remote, real-time optical diagnostics of chemical species in hazardous gas, liquid, and semi-solid phases through a completely novel implementation of evanescent wave spectroscopy. The sensor design uses a tiny, solid block (< $1 cm^3$) of ultra-high purity optical material that is fabricated into a regular, planar polygon with a convex facet to form a total-internal-reflection ring cavity. Cavity ring-down detection measures the absorption of evanescent waves by chemical species in contact with the cavity. By interfacing the cavity to a remotely located light source and detection system (e.g., 0.1 to 10 Km away), this new technology will permit remote, high-sensitivity, broadband chemical sensing with a rugged, cost-effective, miniature spectrometer.

Research Progress and Implications

This report summarizes work conducted after 1 year of a 3-year project. In brief, the laboratory program demonstrated the operation and sensitivity for chemical detection of a prototypic, square total-internal-reflection (TIR) minicavity ringdown spectrometer. We confirmed experimentally the theoretical predictions of performance and refined strategies for cleaning the surfaces and for practical, integrated packaging of the TIR spectrometer with its coupling optics. In addition, a simple, solid-state, TIR, non-ring resonator cavity was designed which may speed practical implementation of this technology for EMSP applications. This design is outlined in the propriety section of this report.

The experimental program utilized a prototype square miniature spectrometer comprised of a 7.5x7.5 mm block of high-purity fused silica and two coupling prisms. To form a stable TIR ring cavity, one face of the block is convex. Extremely high reflectivity (~99.9999%) is achieved by superpolishing each face to a surface roughness of ~ 0.05 nm. One fused silica prism is placed close to one facet to inject laser light into the cavity and a second prism extracts a small fraction of the circulating light and transmits it to a photodetector. The coupling efficiency and cavity performance are functions of the alignment and spacing of these coupling prisms. Hence, much time was invested to determine the alignment and gap width that optimizes the spectrometer performance. In the test stand of the prototype, the alignment and gap widths of the coupling prisms were adjusted with piezoelectric driven translation stages and goniometers. To prevent system drift, an active stabilization system used interferometry and Newton's rings to measure and stabilize the gap-widths of the coupling prisms. Extensive experiments have verified that the optimum gap between the minicavity and each prism is 2-3 micrometers when the spectrometer is operated between 520 and 680 nm. These experiments will facilitate the design of integrated modules that use fixed and adjustable gaps.

A series of experiments were conducted that verify the performance of this spectrometer against the predictions of advanced modeling calculations. These calculations predicted that this minicavity should exhibit a characteristic ring-down time of 1.8 microseconds at 620 nm. Indeed, this decay time was experimentally observed by using a commercial 8-bit, 1-Ghz digital oscilloscope. This corresponds to a cavity loss of 50 parts-per-million-per-pass, which yields practical detection sensitivity for absorbance changes of 0.1 parts-per-million.

Experiments have demonstrated that the square cavity is very sensitive to chemical species that adsorb onto a fused silica face. Iodine was selected for these experiments because it has a quantitatively characterized optical spectrum and their vapor pressure and saturation densities upon glasses have been measured previously. During these experiments a cell containing iodine was placed against one facet and the time-dependent extinction of the evanescent wave was monitored as the surface coverage by iodine increased. Measurements were performed between 520 and 680 nm. The measurements showed that the square resonator could detect iodine surface coverage of 0.001 monolayer! Further optimization may yield one to two orders of magnitude increase in sensitivity.

Several minicavities for use in aqueous solution were also explored with advanced computational modeling. The most sensitive design for use in the visible spectrum is octagonal minicavity fabricated from fused silica. Designs based upon other substrates were investigated of which the most intriguing is composed of high quality sapphire. Sapphire should produce very rugged devices that can operate in the very useful fingerprint near-IR and mid-IR spectral regions. Because sapphire has a relatively high refractive index, the critical angle for TIR in aqueous media is reduced and the minicavity geometry is a cheaper-to-produce, hexagonal solid.

Planned Activities

Experiments that measure the sensitivity of the square cavity design for chemical species deposited from vapors will continue. When the prototypes of a new, simplified resonator design (discussed in the proprietary section of this report) are delivered, their performance properties will be characterized. Strategies for fiber-optic coupling of these cavities to lasers and detectors also will be investigated.

A sapphire hexagonal minicavity prototype will be fabricated to permit measurements of solution-phase anions and metal cations. Time permitting, we will also integrate this resonator with an infrared laser source and perform measurements of solution-phase species in the near- and mid-IR spectrum. During the next year we hope to develop interactions with EMSP technical personnel who can help us select realistic performance tests so that interest in field deployment is stimulated.

Other Access To Information

The technical progress of this project is reported at http://www.nist.gov/crds. As appropriate, rigorously detailed descriptions of this work are published in the scientific journals and patent literature. Two patents related to this technology are pending. Two publications on this technology are available in the archival literature:

1) "Evanescent Wave Cavity Ring-down Spectroscopy as a Probe of Surface Processes", A.C.R. Pipino, J.W. Hudgens and R.E. Huie, Chem. Phys. Lett. 280, 104 (1997).
2) "Evanescent Wave Cavity Ring-down Spectroscopy with a Total-internal-reflection Minicavity", A.C.R. Pipino, J.W. Hudgens and R.E. Huie, Rev. Sci. Instrum. 68, 2978 (1997).