Silicon on Sapphire Chip Based Mid-Infrared Optical Spectroscopy for Detection of Chemical Warfare Simulant Triethyl phosphate

Parker Wray, Yi Zou, Swapnajit Chakravarty, and Ray T. Chen

1Microelectronics Research Center, Electrical and Computer Engineering Department, University of Texas at Austin, Austin, TX, 78758, USA
2Omega Optics Inc., Austin, TX, 78759, USA

Author e-mail address: parkerwray@utexas.edu, yzou@utexas.edu, swapnajit.chakravarty@omegaoptics.com, raychen@uts.cc.utexas.edu

Abstract: Triethyl phosphate (TEP), a chemical warfare simulant, has absorption peaks in the mid-infrared. Using a single mode slot waveguide we were able to detect TEP, with a detection limit down to 75 ppm. This provides enhanced sensitivity while simultaneously achieving device miniaturization.

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With terrorists’ use of nerve agents, it is becoming increasingly more important to develop technology for rapid detection and identification of hazardous chemicals in a manner that has both high sensitivity and mobile form. Infrared (IR) absorption spectroscopy is widely accepted as the ideal technique for chemical sensing due to the unique capability to distinguish analytes of interest based on unique molecular vibration signatures [1,2]. Its methods outshine those that depend on sensing changes in refractive indexes. The principle of operation of our device is governed by the Beer–Lambert law. According to this law, the transmitted intensity $I$ is given by

$$I = I_0 \times \exp(-\gamma \alpha L)$$  \hspace{1cm} (1)

where $I_0$ is the incident intensity, $\alpha$ is the absorption coefficient of the medium, $L$ is the interaction length, and $\gamma$ is the medium-specific absorption factor determined by dispersion-enhanced light–matter interaction. For various applications, $L$ must be large to achieve high sensitivity since $\gamma = 1$. In addition, from perturbation theory

$$\gamma \propto f \times \frac{c ln}{n_c}$$  \hspace{1cm} (2)

where $c$ is the speed of light in free space, $v_g$ is the group velocity in the medium, and $n$ is the refractive index of the medium [3]. The term $f$ is the filling factor denoting the relative fraction of optical field residing in the analyte medium. Group velocity $v_g$ is inversely proportional to the group index $n_c$. Hence, theoretically, the optical absorbance by a waveguide on a same chip increases in order as follows in silicon: (a) strip waveguides, ($n_c \sim 3$), (b) slotted strip waveguides ($n_c \sim 3, f \sim 10$) since the intensity of light in a low-index slot is significantly enhanced compared to strip waveguides [4]. A comparison of power drop due to absorption of TEP for a single mode strip waveguide and our slotted strip waveguide is shown experimentally in Fig 2. The fundamental vibration signatures in mid-infrared are stronger by two to three orders of magnitude than their corresponding overtones in the near-infrared. Hence, due to an increase in $\alpha$ in Eq. 1, together with the device enhancements from Eq 2, mid-infrared spectroscopy can be much more sensitive to concentration changes than near-infrared spectroscopy.

Our slot waveguide device is fabricated on a silicon on sapphire (SOS) substrate using standard e-beam lithography and inductively coupled plasma (ICP) etching. A SEM image of the slot waveguide is shown in Fig 1. The wavelength chosen was 3.4 μm, a strong absorption peak for TEP, shown in Fig 4. Light was emitted from an Interband Cascaded Laser (ICL) on an air-cooled heat sink, the wavelength being stabilized by an external temperature controller. The light passes through single mode mid-infrared optical fibers and coupled into and out of our SOS chip through sub-wavelength grating couplers. The light was then collected by an Indium antimonide (InSb) detector. A reference InSb detector measured any possible variations in the source laser. A 1 KHz mechanical chopper was used in order to improve the signal-to-noise ratio. The detected signals from the InSb detector and reference were then demodulated by a lock-in amplifier and data was processed using LabView code and gathered in real time.
For gas sensing, a heated glass chamber was used where liquid TEP was vaporized then flowed onto the waveguide. Gas concentration was measured using the following calculation,

$$
\text{Concentration} = \frac{\text{Grams}_{\text{TEP}}}{\text{Grams}_{\text{air}} + \text{Grams}_{\text{TEP}}} \quad (3)
$$

Where Grams_{air} and Grams_{TEP} are the number of grams of air and TEP inside the closed chamber, respectively, before administering the vapor onto the chip. Since the TEP was administered in a gaseous state at nominal pressures and temperatures it is assumed to behave as an ideal gas. Based on these calculations we were able to experimentally verify that our slot waveguide could measure concentration down to 75 ppm, TEP. Time scanning of Normalized Transmission change is plotted in Fig. 3 when TEP is introduced. The TEP was administered to the chip at 400 seconds and clearly shows an absorbance spectra between 400 to 700 seconds, when administration was stopped. With Pressure (1atm), volume (6500 cm^3), and temperature (50°C) held constant throughout the experiment. We feel that the low pressure and temperature used in the experiment justifies our use of the ideal gas approximation and rules out the possibility of sample decomposition. As it has been experimentally shown that acquiring spectra of these compounds (TEP) at temperatures above 1100 °C with either low or atmospheric pressures caused significant sample decomposition and combustion [5].