Towards a nanophotonic nose: a compressive sensing-enhanced, optoelectronic mid-infrared spectrometer

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Abstract

Infrared (IR) spectroscopy has been a central tool for chemical analysis for decades, useful in a wide range of fields for the detection and quantification of molecules based on their unique vibrational resonances. Conventional IR spectroscopy relies on bulky, dispersive optics, however, making portability and miniaturization a substantial challenge. Here we demonstrate a micron-scale IR spectrometer where spectrally selective detection is performed optoelectronically, based on the wavelength-dependent mid-IR photocurrent responses of an array of Al grating-based detectors fabricated on a doped Si substrate. Compressive sensing techniques extend our resolution, enabling spectral features to be identified with a remarkably small number of detectors. This work demonstrates a CMOS-compatible, readily scalable approach for the fabrication of compact, room-temperature IR spectrometers capable of use in fieldable applications.

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The field of miniature integrated spectroscopic devices is an extremely active research area due to the continual drive to reduce size, decrease cost, and increase portability while maintaining functionality. A wide range of novel devices has been investigated in this pursuit including colloidal quantum dots,\textsuperscript{1,2} pyroelectric devices,\textsuperscript{3} metal-insulator-semiconductor detectors,\textsuperscript{4} plasmonic lab-on-a-chip devices,\textsuperscript{5} graphene photodetectors,\textsuperscript{6} pixel arrays for chemical imaging,\textsuperscript{7} plasmonic filter arrays,\textsuperscript{8} and a variety of other designs.\textsuperscript{9–15} These types of devices have demonstrated that some, if not all, functional components of a spectrometer can be effectively miniaturized across a broad spectral range, spanning the ultraviolet to the infrared (IR). The mid-IR “chemical fingerprinting” region, from 2-12 µm, is of particular interest due to the presence of molecular vibrational resonances. These resonances provide signatures for the detection, identification, and quantification of almost any molecular structure. A detector capable of direct, wavelength-dependent detection in the mid-IR, with room-temperature operation, would be highly useful in a wide range of applications including environmental,\textsuperscript{16} geological,\textsuperscript{17} and food safety\textsuperscript{18} monitoring. In particular, there has been substantial effort invested to design a “nanophotonic nose” – an optical device with direct electrical readouts capable of performing gas-phase molecular detection and identification.\textsuperscript{19–23}

In this work we demonstrate an optoelectronic room temperature micron-scale mid-IR spectrometer fabricated using CMOS-compatible materials. Leveraging a recently developed approach to mid-IR sensing,\textsuperscript{24} we show that by multiplexing the response from as few as 20 detector elements we are capable of accurately reproducing test input spectra with a resolution of 40 nm. Previous work using nanoscale filter elements in the mid-IR have used both a direct peak-envelope\textsuperscript{7} or a least-squares\textsuperscript{8} method for reconstructing an unknown input spectrum. By using techniques from the field of compressive sensing\textsuperscript{25–28} we are able to enhance our resolvable
resolution well below the Shannon-Nyquist limit even with a small number of detectors.\textsuperscript{29} We also use our spectrometer for contact-free molecular detection of a polymer film placed in the excitation path of the spectrometer, demonstrating a proof-of-principle device capable of performing as a “nanophotonic nose”. Finally, we use simulations to estimate the optimal number of gratings for a complete vibrational spectral reconstruction using this type of optoelectronic spectrometer.

\textbf{Results}

\textbf{Device fabrication and photodetection}

Our spectrometer, schematically shown in Figure 1, utilizes plasmonic (high purity) aluminum gratings on highly p-doped silicon substrates. The photodetection mechanism used here has been previously reported,\textsuperscript{24} but, in brief, the detector response is based on the change in device resistance due to free-carrier absorption in the doped silicon substrate. By applying a small voltage and measuring the change in current, a photoresponse due to incident light can be detected. The aluminum gratings here serve a dual purpose: first as plasmonic gratings that filter which wavelengths of light are transmitted through to the substrate, and second, as electrical contacts for wire-bonding to an external chip carrier. This simple combination allows for a spectrometer composed of many individual gratings to be treated in an ensemble.
The gratings were fabricated using standard electron-beam lithography and clean room procedures (full details in Methods). The gratings in this work range in pitch from 1250-3600 nm with a gap of 20% of the total pitch (e.g. for a 1250 nm pitch there is a 250 nm gap) and a thickness of 300 nm. Following fabrication, they were wire-bonded to a chip carrier to ensure a consistent electrical connection through the device. The unique responsivity of each grating as a function of wavelength (e.g. Figure 1(e)) was characterized consecutively by measuring the current through

Figure 1: Layout and schematic of the optoelectronic spectrometer used in this work. (a) Rendering of the gratings showing that all gratings are oriented in the same direction and how a measurement is taken with the input light focused onto each grating individually (red beam). (b) Schematic representation of the grating array showing the polarization of the incident light and how the gratings change in pitch when going around the array. (c) Optical image of the grating array used in this work (gratings range from 1250–3600 nm in pitch). (d) SEM image of the grating array. (e) Representative responsivity curves for four gratings with pitch 2000 nm (red), 2375 nm (blue), 2625 nm (purple), and 3000 nm (green), as indicated in (b). The curves are normalized relative to the largest recorded responsivity for any of the gratings.
each grating with a lock-in amplifier while the grating was illuminated with a focused tunable mid-IR laser polarized perpendicular to the grating (for all responsivity curves see Figure S1). As each grating has a different pitch, they all possess different responsivity curves as a function of wavelength. Once the response curve as a function of wavelength for each grating was measured, we found we were then able to create a proof-of-principle mid-IR spectrometer using as few as 20 gratings.

**Spectral reconstruction**

Reconstructing an unknown input signal directed onto a detector element (or elements) requires that the response from the detector be correlated to a particular input intensity at a given wavelength. This requires either a single detector element coupled with a mechanism for wavelength selection, such as a grating or prism, or a large number of detector elements each with
different wavelength-dependent response characteristics. The approach taken here is to use the readout from a number of detector elements, each with a unique response as a function of wavelength.

To use a collection of detector elements as a spectrometer, the mathematical problem posed is the following: how do we extract an unknown input signal, \( \tilde{x} \), given the measured response of each device as a result of this input signal, \( \tilde{y} \), and the previously measured responsivity curves stored in the matrix \( \mathbf{D} \)? This process is schematically presented in Figure 2. First, an unknown input spectrum is directed onto a single grating and the current through that grating is recorded.

![Figure 2: Reconstruction process for an input signal. An unknown input spectrum, \( x \), is directed on to each grating. Each grating has a unique responsivity function contained in the matrix \( \mathbf{D} \), and generates a current \( y \) in response to the incident light. These currents are then used to estimate the original input spectrum.](image)

This process is then repeated for all of the gratings (20 in this work). Finally, all of the current measurements are combined to reconstruct the original input spectrum. Mathematically, this corresponds to the equation
\[ \mathbf{D}\tilde{x} - \tilde{y} = 0 , \] 

(1)

where \( \tilde{x} \) is a representation of a continuous input spectrum \( x(\lambda) \) sampled at \( m \) points and \( \tilde{y} \) is our set of \( m \) current measurements, making both of size \( m \times 1 \). \( \mathbf{D} \), of size \( m \times m \), contains the measured responsivity curves and so, ideally, this equation can be thought of as integrating our input signal times the response function of each of the gratings at a discrete number of wavelengths. To find \(-y=\theta\),

(1),

\[ \Rightarrow \tilde{x} = \mathbf{D}^{-1}\tilde{y} , \]

assuming that \( \mathbf{D} \) is invertible. Physically, the condition that \( \mathbf{D} \) is invertible corresponds to the responsivity functions of the different detector elements being distinct from one another. This is simply a mathematical restatement of what was postulated above, that the detector elements must all have unique response functions in order to be able to reconstruct arbitrary input signals.

In practice, since the noise of an experimental measurement typically prevents an exact solution, the most common numerical approach to solving this problem is through non-negative least-squares regression,\(^{30}\)

\[
\min_{\tilde{x}_{i} \geq 0} \| \mathbf{D}\tilde{x} - \tilde{y} \|_2^2 .
\]

(2)

In this numerical approach we are looking for the vector, \( \tilde{x} \), which minimizes the squared value of the \( L^2 \)-norm,

\[
\| \mathbf{D}\tilde{x} - \tilde{y} \|_2 = \| \tilde{z} \|_2 \overset{\text{def}}{=} \sqrt{(z_1^2 + z_2^2 + \cdots )} \\
\tilde{z} = \begin{pmatrix} z_1 \\ \vdots \\ z_m \end{pmatrix} ,
\]
subject to the constraint that every element of \( \tilde{x} \) is greater than or equal to 0, which holds here since the input spectrum intensity is intrinsically positive or 0. Although least-squares minimization is computationally straightforward and efficient, this technique is limited in spectral resolution by the length of \( \tilde{y} \), that is, the number of unique current measurements.

To keep the solution tractable, our final spectrum, \( \tilde{x} \), can only have as many data points as there are current measurements in \( \tilde{y} \). If we tried to increase the number of points in our reconstructed spectrum beyond the number of current measurements, we would end up with an underdetermined system that is unsolvable (we would have a system of \( m \) equations and \( n \) unknowns, with \( n > m \)). This means that \( \tilde{x} \) and \( \tilde{y} \) are both of length \( m \), and \( D \) is \( m \times m \), where \( m \) is the number of grating elements, since each grating produces a single current measurement. In our system, which spans the spectral range 2200-3800 nm and has \( m = 20 \), this corresponds to a final reconstructed resolution of 80 nm \( \left( \frac{\text{spectral window}}{\text{number of points}} \right) \) as displayed in Figure 3. If we try to distinguish different input spectra with peak positions closer together than this limit, we are unable to tell them apart using this method of spectral reconstruction, as shown in Figure 3(d). Thus, the most obvious method to improve the spectral resolution is to simply increase the number of
gratings (increasing the length of $\mathbf{y}$). The improvement in resolution is linear with the increase in number of gratings, and simulations demonstrating this improvement are presented in the next section. However, another mathematical approach may be employed to obtain an increase in spectral resolution such that we are able to achieve sub-Nyquist resolution without increasing the number of gratings.

![Figure 3: Spectral reconstruction using both L^1 and L^2 methods with comparisons to ground truth. Input signals centered at 2506 nm and 3358 nm (a), 3105 nm (b), and 3145 nm (c). While L^2 methods are unable to distinguish the peaks in (b) and (c), L^1 reconstruction methods are easily able to tell them apart as seen in (d).](image)

In the previous analysis, we ignored the fact that our responsivity matrix, $\mathbf{D}$, can be sampled with higher resolution than our final reconstruction is capable of producing and that this extra information is lost when we restrict $\mathbf{D}$ to a size of $m \times m$. We can re-cast our system of equations to be an underdetermined set by noting that $\mathbf{D}$ is in fact of size $m \times n$, where $n > m$. 
corresponds to the resolution with which we sample the responsivity curves that compose D. Not only can we measure D at a higher resolution, but we can reasonably assume on physical grounds that the responsivity curves are smooth and then interpolate between points to further increase the effective resolution of D. In a standard least-squares regression, such as the one above, this extra information is wasted since it must be discarded when performing the fitting to avoid the accumulation of errors.31 However, following the work of Oliver et al.,25 an adjustment to our reconstruction algorithm allows us to make use of this additional data to substantially increase our reconstructed spectral accuracy. By incorporating a few assumptions about the nature of our input signal, we are able to use this additional information to improve the accuracy of our spectral reconstruction.

The improved reconstruction method is based on a technique from compressive sensing for solving underdetermined problems by incorporating the L1-norm,

\[ \| \mathbf{z} \|_1 \equiv |z_1| + |z_2| + \cdots, \]

as opposed to only L2 (or least-squares) norm minimization in Equation

\[ \min_{x_i \geq 0} ||D \mathbf{x} - \mathbf{y}||_2^2. \]

By incorporating the L1-norm in our numerical algorithm we can exploit the extra information contained in D rather than discarding it without making our system unsolvable. The inclusion of the L1-norm is analogous to the following pair of assumptions: 1) that there exists a set of basis curves that can accurately reproduce our input spectrum and 2) that in this new basis of curves only a relative few coefficients will be nonzero when describing our input spectrum. Together, these assumptions are referred to as the assumption of sparsity (meaning most elements are zero) in a domain (for a rigorous mathematical formulation see Ref. 32). As a point
of reference, for the spectrometer presented here the number on nonzero coefficients was ~3 out of several hundred, though the technique would still work even for more complicated input signals.

In essence, these conditions are a statement about the nature of a signal being measured and how it can be represented. As an example, consider a sine wave of frequency \( \omega \) and unit amplitude. According to the Shannon-Nyquist theorem, we need, at minimum, \( 2\omega \) samples in order to uniquely determine this signal.\(^{29}\) With fewer samples than this we run the risk of aliasing, or having different sine waves appear the same due to our sampling rate. An example of how this could happen is presented in Figure S2. Alternatively, we can express this signal in the domain consisting of sine waves with varying frequency \( (\omega, 2\omega, 3\omega...) \). In this basis, our reconstruction only needs a single point corresponding to a sine wave of frequency \( \omega \), while all other values will be 0. Thus, our signal can be described using a sparse representation in this new basis, since it will only have a single nonzero term. This is the central concept of compressed sensing: carefully selecting the domain in which an input signal is represented can allow for the amount of data needed to describe it to be dramatically reduced, or compressed. We note however that the approach taken in this work has several differences from “traditional” compressed sensing, as discussed in the Supplemental Information.

In this work our input is a Gaussian spectrum from a laser source with a known width. If we choose our basis curves to be a set of Gaussian curves, with approximately the correct width, spanning the mid-IR, there is (theoretically) only a single coefficient needed to uniquely determine our input signal (i.e. the coefficient corresponding to the center wavelength of our laser). Now, instead of searching for one coefficient value per wavelength in our reconstruction, we are only looking for the magnitude of a single coefficient and are assuming that the rest are zero (or
negligible). This dramatically reduces the solution space possible in our system of equations and turns our previously intractable problem into one that is eminently solvable numerically.

In general, the steps for performing this type of reconstruction are as follows: first, define a basis set of curves that can be summed with varying magnitude to form any potential input signal of interest – this is called our dictionary, \( \Psi \) (see Figure S3). Then, assume that our input signal, \( \tilde{s} \), is sparse in the basis defined by the dictionary. Now, we are able to apply a second condition on the solution to our minimization problem – that the solution should be sparse in this new basis. Without this additional constraint there would be no way to select a single solution from the many possible solutions to such an underdetermined problem.

Mathematically, these steps correspond to: 1) defining an \( n \times n \) dictionary matrix, \( \Psi \), consisting of a set of basis curves; 2) expressing our reconstructed input signal \( \tilde{x} \), as a new vector, \( \tilde{s} \), of length \( n \) multiplied by this dictionary set,

\[
\Psi \tilde{s} = \tilde{x} ;
\]

3) retaining the high-resolution information about the response of each detector in \( D \), allowing it to be of size \( m \times n \); and 4) incorporating the new \( L^1 \) constraint into our fitting algorithm,

\[
\min_{\tilde{s} \geq 0} ||D\Psi \tilde{s} - \tilde{y}||_2^2 + \lambda \|\tilde{s}\|_1 .
\] (3)

Here we have introduced \( \lambda \) as a thresholding parameter to select for sparsity in the final solution (it will not be used to represent wavelength at any later point in the text). That is, we are looking for the vector \( \tilde{s} \) that has the fewest number of nonzero elements possible, while still minimizing our original equation and setting values smaller than \( \lambda \) to 0.
This type of problem, commonly referred to as basis pursuit denoising (BPDN), has been well studied in the convex optimization literature, and is known to be effective at reproducing input signals from a relatively small number of measurements in a wide range of applications. (MATLAB code for numerically solving Equation \( \min_{s \geq 0} ||D\Psi \tilde{s} - \tilde{y}||^2 + \lambda ||s||_1 \). (3) is provided in the Supplemental Information).

Using this more sophisticated fitting algorithm, the achievable spectral resolution is doubled, and we are able to resolve peaks that were indistinguishable using an \( L^2 \)-norm reconstruction. As can be seen in Figure 3(e) (solid lines), the \( L^1 \)-norm based spectral reconstruction is able to accurately reconstruct two input signals separated by only 40 nm using the same experimental data as the \( L^2 \)-norm reconstruction. This is well below the Shannon-Nyquist limit imposed by the limited number of data points and is a clear demonstration of the achievable improvements when using more advanced reconstructive methods and some general assumptions about the nature of the input signal. We stress that this type of analysis has been demonstrated to be both robust against noise and well-suited for a wide variety of input signals, not just those that are explicitly members of the dictionary matrix as in this work. In any number of signal-acquisition problems the use of such a reconstruction algorithm has the potential for improvements in signal reconstruction accuracy.

**Molecular detection**

To demonstrate the viability of our microscale IR spectrometer for molecular detection, we placed a quartz window coated with a thin layer (~2-3 \( \mu \)m) of poly(methyl methacrylate) (PMMA) in the optical excitation path of the spectrometer. Similar to the spectral measurements above, we
directed a fixed wavelength (centered at 3387 nm to overlap the CH$_2$ and CH$_3$ stretching region) from a tunable mid-IR laser onto each grating, sequentially, and measured the response with and without the PMMA layer for 60 seconds each (full details in Methods). After reconstructing the resulting spectra, we were then able to detect the change in optical intensity due to the vibrational absorption of the PMMA, as compared against an uncoated quartz window, Figure 4(a,b) (solid-symbol lines). As can be seen, there is a clear drop in measured intensity due to the presence of the thin molecular layer. To verify that the measured change in intensity we observed was due to absorption by the molecules in the beam path and not some other effect, we also performed simulations of the spectral reconstruction process (dash-dot lines) accounting for the separately measured absorbance of the PMMA (see Figure S4). We hypothesize that the discrepancy seen between the simulated and experimental curves (Figure 4(b)) is due to experimental noise.
producing a larger shift in the measured spectrum than was theoretically predicted. However, the peak heights match exceptionally well, indicating that the reduction in amplitude is due to the presence of the PMMA layer.

Briefly, these simulations were performed as follows (see Supplemental Information for sample code). The predicted output of the laser at a given wavelength was modulated by either the measured absorbance due to a blank quartz window or a window with a layer of PMMA. These simulated spectra were then used as inputs to the measured responsivity curves to generate a series of ideal current values for the gratings. These idealized currents were then fed into the spectral reconstruction algorithm to generate simulated reconstructed spectra. Finally, these simulated reconstructions were scaled by a constant factor to correlate their intensity with that of the experimental reconstructions. The relative peak heights match very well between the simulated and experimental spectra, demonstrating that the spectrometer is accurately measuring the reduction in light intensity due to the molecular absorption. This is particularly evident in the difference between the reconstructed spectra with and without the molecular layer, Figure 4(b), where we see a prominent feature that lines up very well with the molecular absorption of PMMA (at ~3350 nm). Based on the observed noise in the measurements, we estimate that the minimum detectable layer would be a factor of ~4 smaller than what was measured here, enabling the detection of a molecular layer approximately 0.6 µm thick without increasing the integration time of the measurements.

To demonstrate the achievable resolution with this type of spectrometer, we performed simulations with increasing numbers of gratings until we were able to completely reproduce the vibrational structure of PMMA, Figure 4(c), with a blackbody source. These simulations were performed by generating random responsivity curves, using those curves to generate current
values, and then finally using those currents to do spectral reconstruction. As can be seen, by increasing the number of gratings from 20 to 500 we are able to fully capture the vibrational fingerprint of PMMA.

**Discussion**

In this work we have demonstrated a fully integrated spectrometer capable of reproducing spectra with resolution below the Shannon-Nyquist limit using algorithms adopted from the convex optimization literature. By using such algorithms many of the miniature spectrometers in the literature could substantially increase their achievable resolution. Additionally, we showed that this spectrometer can be used directly for molecular detection without any modification. Finally, we performed simulations to demonstrate that an optoelectronic spectrometer of this type is capable of directly measuring the vibrational modes of a sample. Since this spectrometer is CMOS compatible, straightforward to fabricate, inexpensive, and operates at room temperature, we believe that fully-integrated spectrometers such as this one provide an approach towards developing a compact “nanophotonic nose” capable of gas, liquid, or solid-phase molecular sensing in a miniature package with a direct electrical readout. This type of spectrometer shows promise in an expanding range of applications where compact, fieldable IR spectrometers are of increasing need, including biosensing, environmental monitoring, and chemical imaging.

**Materials and Methods**

**Device fabrication**
The detectors were fabricated on <100> boron-doped silicon substrates with resistivity of 0.01-0.02 Ω-cm. PMMA A4 (Microchem) was used as an electron-beam resist, followed by development in 1:3 methyl isobutyl keytone (MIBK)/isopropyl alcohol (IPA) (Microchem). After development, the oxide layer on the substrate was etched in a 10:1 solution of buffered oxide etchant (NH₄:HF, Microchem) followed by a 45 second rinse in deionized water to grow a uniform thin oxide layer. The sample was then dried using N₂ gas and immediately placed into the evaporation chamber. 300 nm of pure aluminum (99.99%, Ted Pella) was deposited at 5×10⁻⁷ Torr and a rate of ~5 Å/sec as determined by a quartz crystal microbalance. Lift-off was accomplished by soaking the sample in room-temperature acetone overnight, followed by a brief sonication and IPA rinse. A large (~2 mm radius) ground connection was made to the silicon substrate using indium solder after scratching through the oxide layer with a diamond-tipped scribe. Every grating on the chip, as well as the ground connection, was then wire-bonded with 1 mil gold wire to an external chip carrier (Kyocera) to facilitate consistent repeated measurements.

IR measurements of molecules

FTIR transmission spectra were taken on a Hyperion 80v spectrometer (Bruker) using a silicon carbide globar source; the background for all measurements was an empty beam path. The molecular samples were prepared by spin-coating PMMA A4 on a quartz window (Ted Pella) before heating at 180° C for approximately 5 minutes to cure and dry.

Electro-optical characterization

An optical parametric oscillator (OPO, Coherent) pumped by a Ti:Sapphire laser (Coherent) was used to generate mid-IR light for this study. The OPO idler outputs mid-IR light
from roughly 2-4 µm with a pulse frequency of 80 MHz, a pulse width of ~200fs, and an average power of ~5 mW at the device. The beam was chopped to allow photocurrent signals to be recorded using a lock-in amplifier (Signal Recovery) in “current mode” which applies a small voltage (~0.15 mV) in opposition to the open-circuit potential of the device. The laser was split using a CaF₂ 50/50 beamsplitter (Thorlabs) so that its power could be monitored in parallel with the current through the devices. The laser was then focused onto the grating portion of each device individually using a reflective objective (Thorlabs, 15x, 0.30 NA). For responsivity curves, the laser scanned its spectral range in 25 nm intervals while both laser power measurements and current values were recorded. For spectral measurements, the idler was set to a target wavelength and then current and laser power values for each grating were recorded at a constant wavelength for 60 seconds at a rate of 1 Hz. For molecular detection measurements, the beam was additionally passed through either a blank quartz window, or a quartz window coated with a target molecule as described above. For the molecular detection measurements, the laser was centered at 3387 nm to overlap with the CH stretching region of the PMMA.

Spectral reconstruction

L²-norm spectral reconstructions were performed using straightforward linear programming least-squares minimization. L¹-norm spectral reconstructions were calculated using a gradient-descent method with a fixed step size based on the Lipschitz condition to ensure convergence. Pseudocode for both fitting algorithms and all simulations is supplied in the Supplemental Information. Simulated reconstructions for molecular detection were based on the measured IR spectra of bare quartz and PMMA-coated quartz windows. To determine the number
of gratings necessary for direct vibrational spectral readout, random strength and width responsivity curves with peak positions spanning the range 2.2-3.8 µm were used to approximate physical gratings. These responsivity curves were then used to generate idealized currents for a given input spectrum. Finally, these responsivity curves and currents were used to produce spectral reconstructions just as the experimentally measured values were.

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**Supporting Information** – Responsivity and SEM characterization for all gratings. Introductory compressive sensing images. IR spectra for uncoated- and PMMA-coated quartz windows. Picture of experimental setup. Differences between “traditional” compressive sensing and the approach taken here. Example numerical code for compressive sensing reconstruction.

**References:**


CONTRIBUTIONS

Author contributions: B.C. and N.J.H. designed research; B.C. performed research and analysis; B.C. and N.J.H. wrote the paper.

COMPETING INTERESTS

The authors declare no competing financial interests.