Sensors for Manufacturing Efficiency Workshop

DOE Sensors & Automation
2005 Annual Portfolio Review
Chicago, IL
June 6, 2005

Broadly Tunable Mid-Infrared Hydrocarbon Sensor

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Supported by the U.S. Department of Energy under Contract No. DE-FG02-03ER83851. Such support does not constitute an endorsement by DOE of the views expressed herein.

Outline

- **Summarize project**
- **Summarize barriers, pathways, and metrics**
- **Review progress to date (especially past year)**
- **Describe future plans (coming year)**
- **Describe commercialization plans**

Project Description

VG05-154-3

- **Need: Improved petrochemical manufacturing cost and energy efficiency**
- **Goal: Develop a prototype broadly tunable mid-infrared laser spectrometer for petrochemical industry process control analyzers**
- **Core Technology: novel mid-IR laser source based on Difference Frequency Generation (DFG) suitable for tunable diode laser absorption spectroscopy (TDLAS)**
- **Novel/Transformational Element: A waveguide periodically-poled lithium niobate (PPLN) chip providing acceptable power conversion efficiency over a broad range of wavelengths**
- **Initial Industry for Application: petrochemical production**
- **Key Project Deliverable: portable prototype gas analyzer**

Application Example – Hydrocarbon Cracking

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- **Furnace conditions change because of fouling of furnace elements, changes in feedstock, etc.**
- **Processes cannot be adjusted in real time to compensate for these changes because measurements are too slow; this leads to conservatism (operating well below capacity)**
- **With real-time process control offered by mid-IR TDLAS analyzers, furnace can be operated closer to full capacity, increasing the yield of the desired end products**
- **This leads to a more efficient process (energy and cost savings) and a product mix with higher market value**

Barriers, Pathways, and Metrics

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Barriers	Pathways	Critical Metrics
Inadequate tuning range of commercially-available laser sources	Produce custom-made laser source with a wider tuning range	Tuning range >200 cm^{-1}
Slow response time of existing gas analyzers (remote location)	Perform measurement near the process stream	Portable instrument capable of measuring at least three hydro-carbons to a precision of ~1% with a response time of a few seconds

Project Tasks

VG05-154-6

1. **Design, fabricate optimized waveguides for efficient MIR generation**
2. **Design and fabricate portable prototype gas analyzer**
3. **Demonstrate speciation of calibrated gas mixtures in the laboratory**
4. **Demonstrate speciation on process gases in a petrochemical facility**

Progress to Date (Task 1)

1. **Design, fabricate optimized waveguides for efficient MIR generation**
   - improved computer model of waveguide fabrication process developed, shown to make accurate predictions of phase-matching conditions and waveguide mode profiles
   - tapered waveguide devices designed, fabricated, and tested, leading to solution of the “multimode excitation” problem
   - broad tuning, well centered on hydrocarbon absorption bands, demonstrated using tunable 1300 nm telecom laser as the tuning element
   - fabrication of optimized waveguide devices (incorporating optimized tapers for single-mode excitation and optimized fabrication recipes for broad tuning) is underway

Progress to Date (Tasks 2-4)

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2. Design and fabricate portable prototype gas analyzer

- fiber “pigtailing” (permanently attaching fiber to waveguide) demonstrated
- signal-to-noise calculations performed to guide the choice of infrared detectors and detection schemes
- application requirements specified and incorporated into design of portable prototype gas analyzer
- makes and model numbers for most key components of gas analyzer (lasers, optics, electronics) have been identified
- fabrication of gas analyzer will begin shortly, after preliminary design review

3. Demonstrate speciation of calibrated gas mixtures in the laboratory

- target species identified, FTIR spectra studied to identify best wavelength region
- laboratory DAQ system for scanning lasers, acquiring spectra developed

4. Demonstrate speciation on process gases in a petrochemical facility

- site visit to petrochemical facility conducted, measurement location identified, commitments to allow the testing obtained

Major Accomplishments in Past Year

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- **Tapered waveguide devices developed to overcome one of the biggest problems with previous tunable mid-IR DFG laser sources: multimode excitation**

- **Broad tuning centered near 3000 cm^{-1} demonstrated using a tunable 1300 nm telecom laser as the tuning element; this allows us to "piggyback" off telecom industry engineering to increase the ruggedness and compactness of the mid-IR laser source**

- **Fiber pigtailing demonstrated in laboratory; this is a key step in the manufacturing of a rugged prototype requiring no manual optical alignment**

Tapered Waveguide Devices: Before

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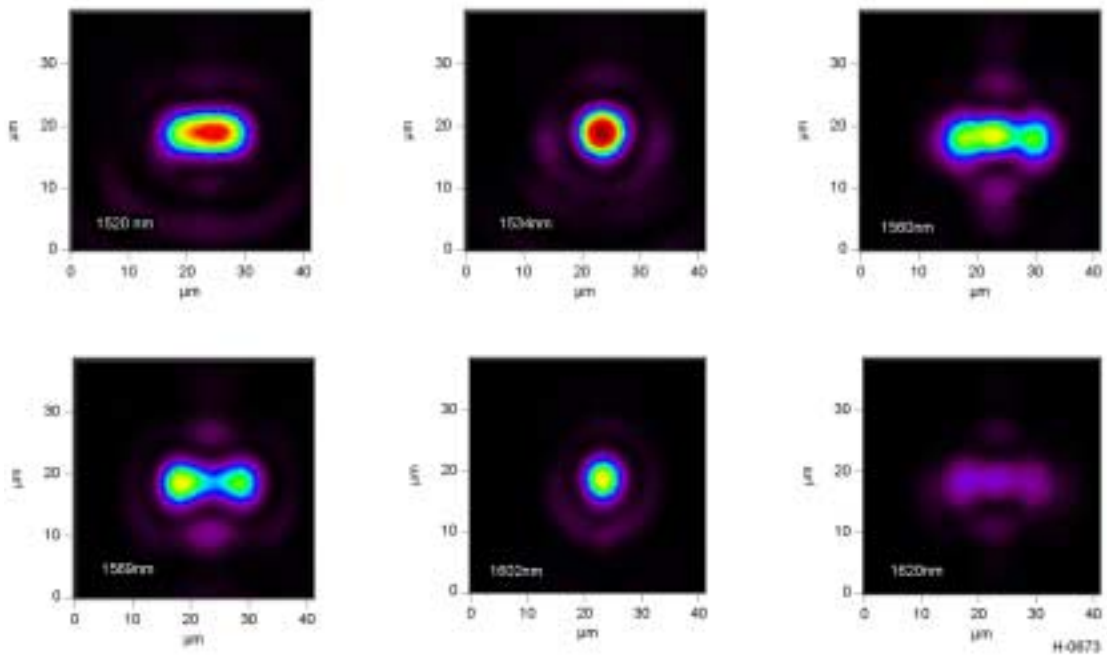
- **Mode profile at waveguide exit changes significantly as laser wavelength is tuned; this is a sign of multimode excitation**



- **Multimode excitation lowers the mid-IR output power, because each pair of pump/signal modes has a different phase-matching condition**

Tapered Waveguide Devices: After

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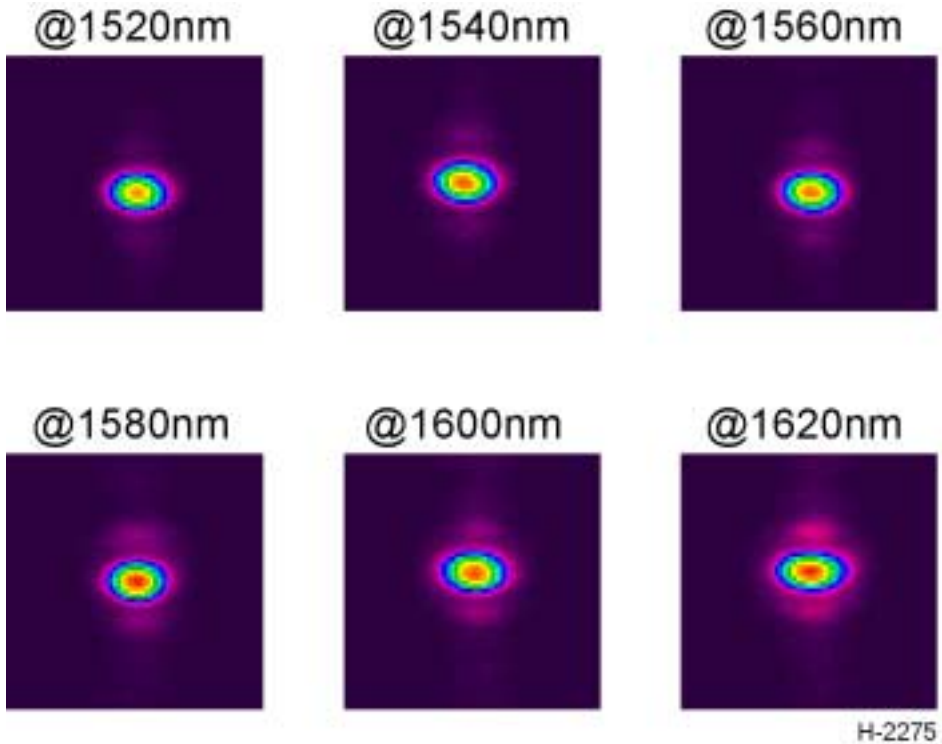
- **Using new waveguide design (developed in Phase II), the mode profile at the waveguide exit does not change when the wavelength is tuned**



- **Waveguides on appropriate substrates incorporating this design should produce mid-IR powers >1 μW when excited with near-IR diode lasers**

Phase II Broad Tuning Demonstration

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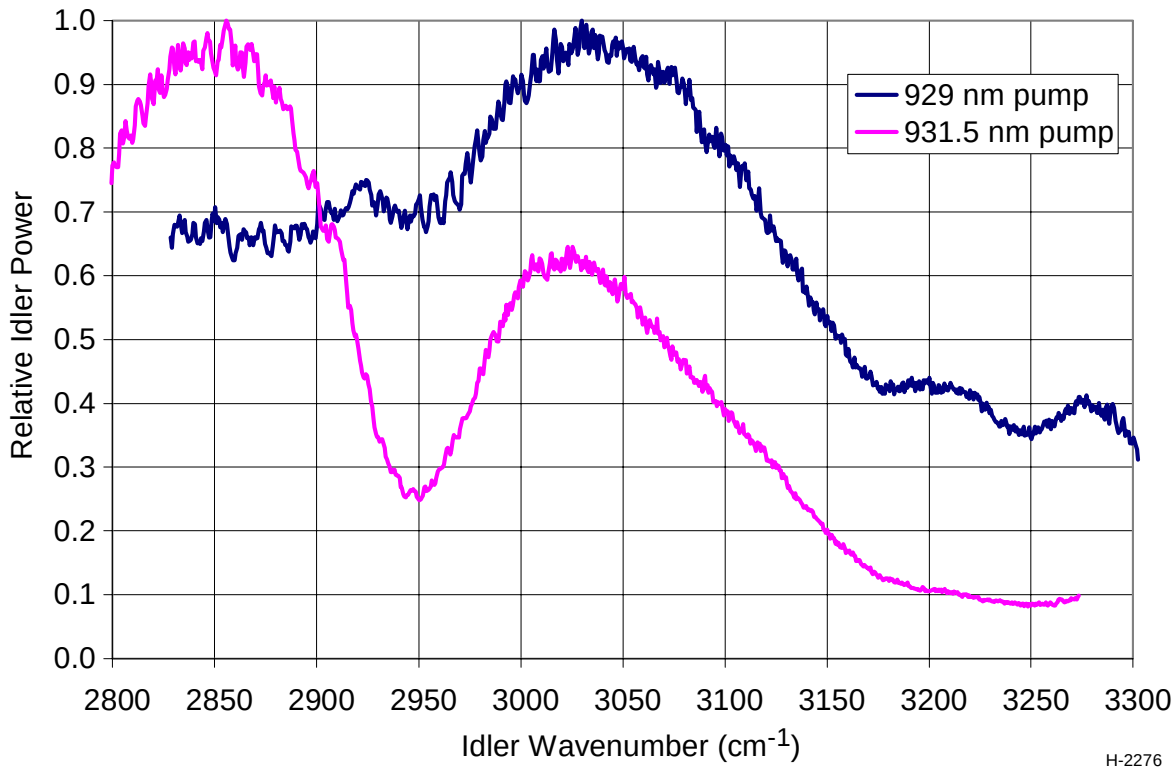
- **Pump λ fixed near 930 nm, signal λ tuned over 1260-1340 nm**
- **Waveguide temperature fixed at 65° C**



- **Broad tuning (~300-400 cm^{-1}) is observed, in agreement with predictions of the PSI computer model of waveguide fabrication**

Overlap Between Laser Tuning Range and Hydrocarbon Absorption Bands

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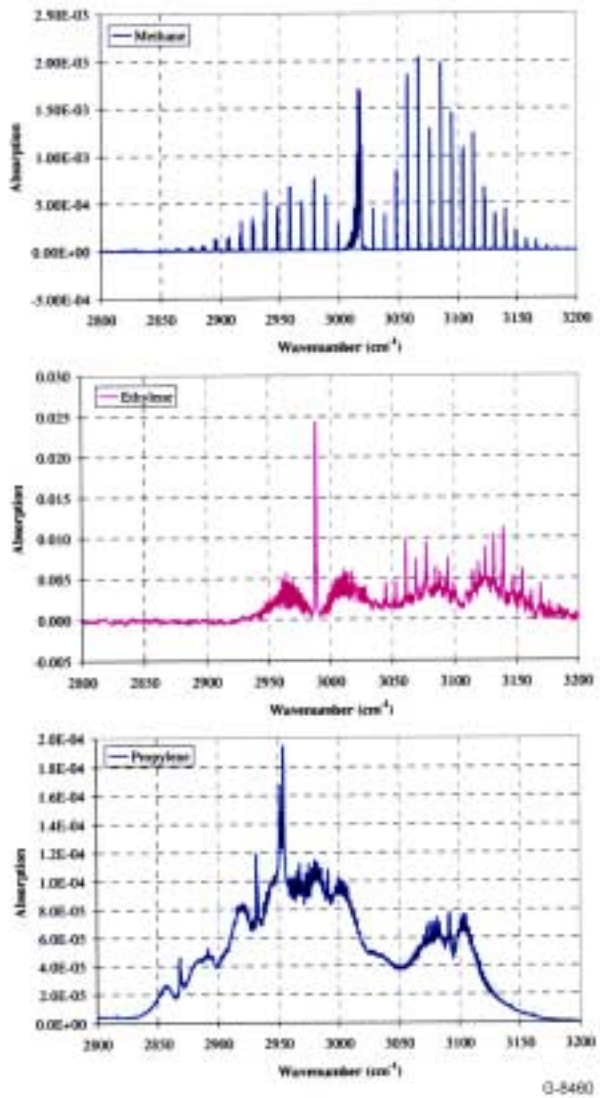
- **Laser tuning range 2800-3200 cm^{-1}**
- **Very good overlap with characteristic absorption features of small hydrocarbon molecules**



Fiber Pigtailing

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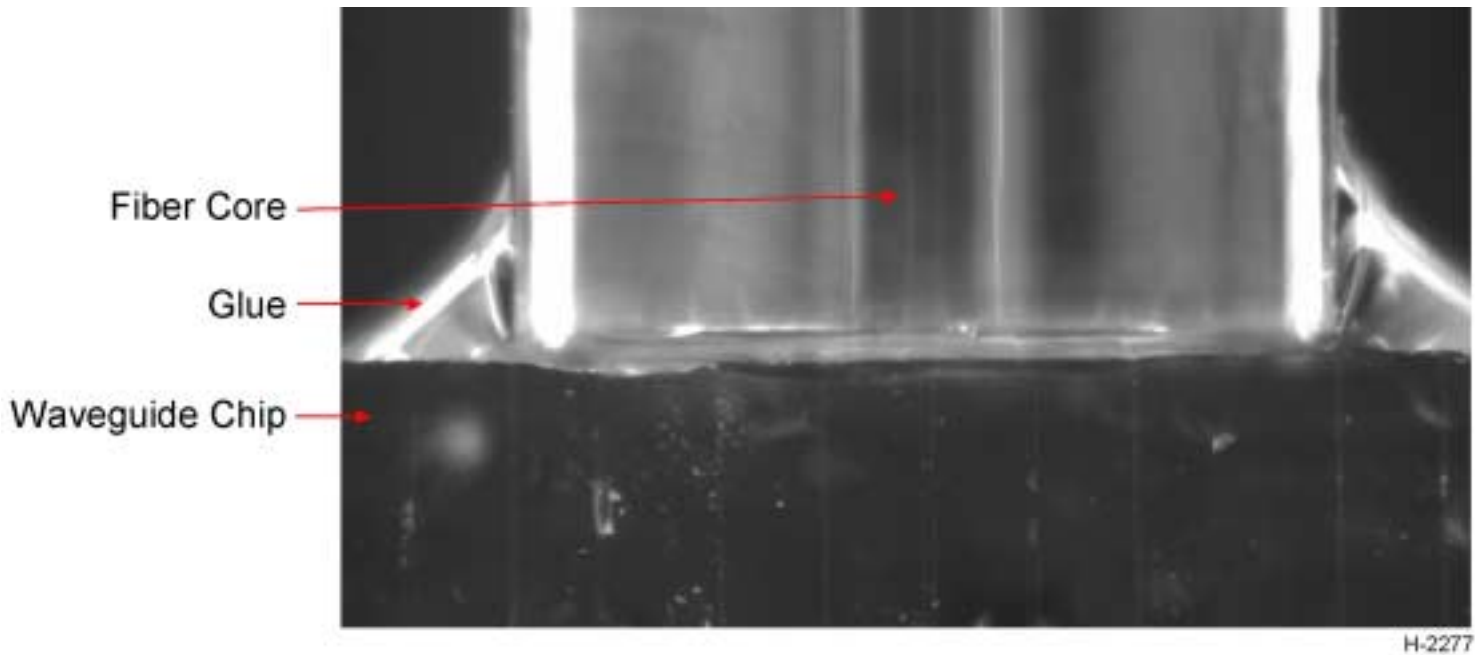
- **Optical fiber assembly (core, cladding, and ferrule) attached to chip containing waveguides**
- **40-50% coupling from fiber into waveguide**



- **Better coupling possible when mode sizes are better matched**

Future Plans: Upcoming Milestones

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Milestone	Completion Date
Iterate Design, Fabricate, and Testing to Produce Optimized Laser Source	10/1/05
Design and Fabricate Portable Prototype Gas Analyzer	11/1/05
Use Portable Prototype Gas Analyzer to Measure Calibrated Gas Mixtures in the Laboratory	12/1/05
Use Portable Prototype Gas Analyzer to Measure Species Concentrations in a Working Petrochemical Facility	2/15/06
End of Contract	2/28/06

Current Commercialization Plan

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- **PSI has established a relationship with Analytical Specialties Inc. (ASI)**
  - ASI is supporting development and testing of the Phase II prototype analyzer
    - Testing to be done at Dow Chemical
  - Pending Phase II success, ASI may invest in development of commercial product
  - PSI will supply laser source modules as part of commercial product
  - ASI will manufacture, market, sell, and service analyzer systems and services

Continued R&D Recommendations

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- **After Phase II, PSI recommends continued R&D investment for:**
  - Ruggedization for permanent field installation
  - Engineering for manufacturability and cost
  - Enhanced DFG efficiency
  - Measurement multiplexing, maximizing use of the laser source at several measurement sites

- **PSI envisions a combination of government, industry, and commercial investment to achieve these objectives**

Summary and Conclusions

VG05-154-18

- **Phase II program is designed to produce a portable prototype gas analyzer based on difference-frequency generation in waveguides**
- **Three major accomplishments in past year:**
  - improved waveguide taper designs have solved “multimode excitation” problem which limited conversion efficiency in the past
  - tuning range of laser source increased to 300-400 cm^{-1}, and better centered on the absorption bands of the target hydrocarbons, by changing waveguide design and wavelengths of near-infrared input lasers; tuning provided by a telecommunications laser in 1300 nm band
  - fiber pigtailing, a key manufacturing step, demonstrated
- **Design and construction of portable prototype gas analyzer are now underway; testing planned in petrochemical facility**
- **Project completion expected in February 2006**

Backup Vugraph

Energy Savings

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- **With real-time process control, furnaces which produce small hydrocarbons can be operated less conservatively (closer to full capacity)**
- **Energy savings come from greater efficiency of the furnaces (less energy consumed for a given amount of end product)**
- **When fully implemented, potential annual energy savings in U.S. are 5×10^{12} BTU (1.5×10^{9} kW-hour)**