Resonant Dust: IR Targets for Tagging and Identification

Marc L. Simpson I&C, Phillip Britt CTD,
John Caughman FED, John T. Simpson I&C,
Chris Rouleau SSD

Oak Ridge National Laboratory
6/21/01

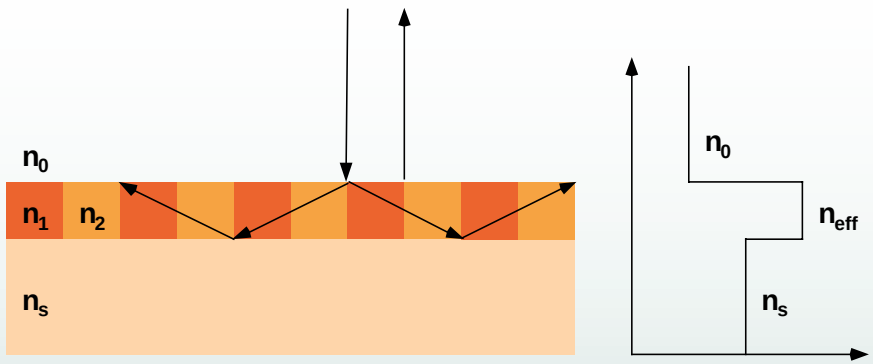
Sponsor: DOE Special Technologies Program

Presented at JASON Summer Study, La Jolla, CA

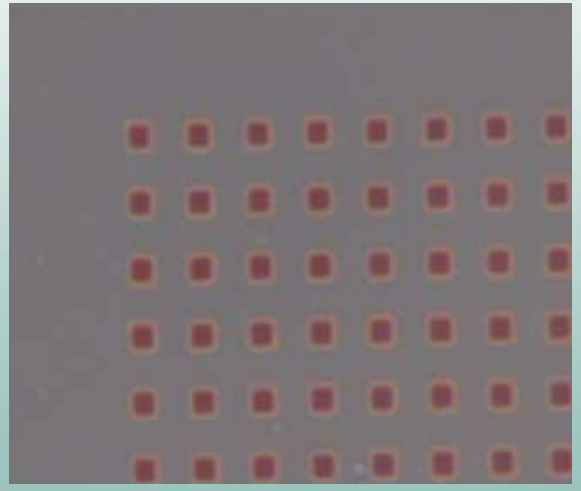
Outline of Talk

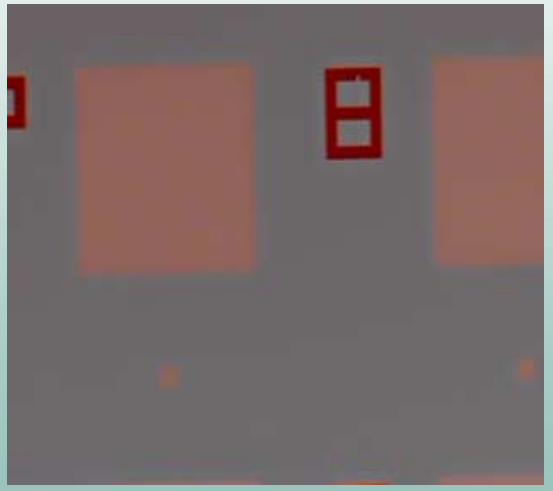
- Overview photonic bandgap (PBG) and subwavelength structures (SWS)
- Embedded structures
  - Photonic bandgap crystals (small holes)
  - Subwavelength structures (small posts)
- Guided mode resonant filters (surface 1D subwavelength gratings)
- Resonant Dust (2D structures…how small??)
- Detection/interrogation
- Future areas of interest (smarter dust)

All-weather IR Tag: Resonant Dust

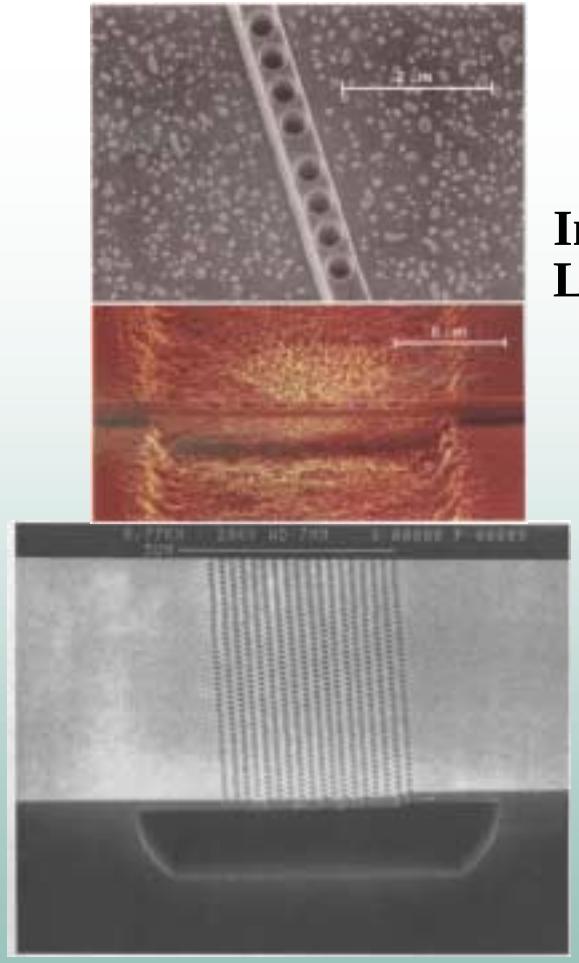


- Small "dust-like particles
- Customized to be sensitive at single wavelengths (LWIR)
- Possible sensitivity to presence/absence of various substances

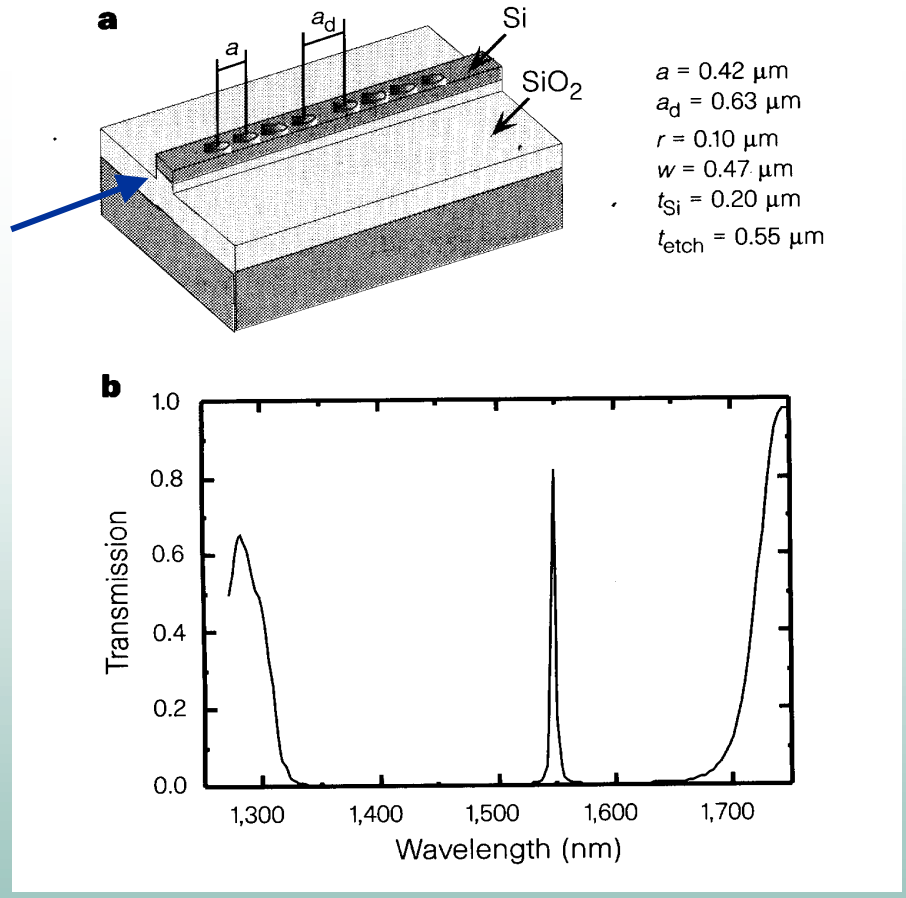




Photonic-Band-Gap (PBG) Devices Based on Arrays of Holes in a Waveguide can Guide and Filter Light.

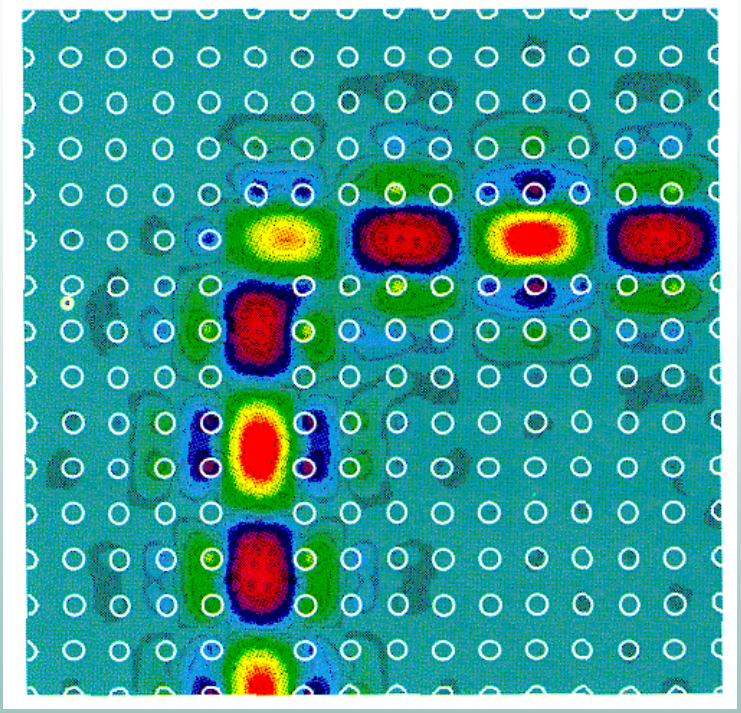


Incident Light

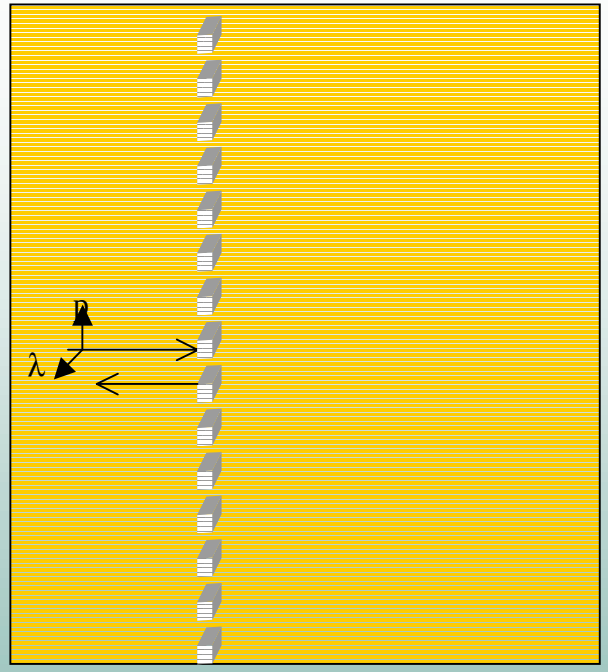


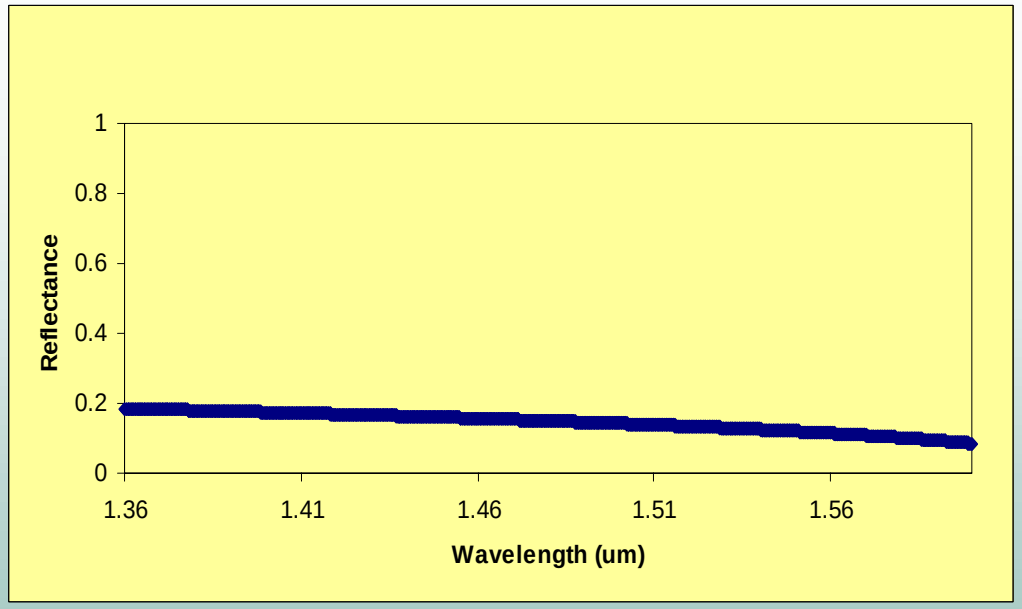
Research in Photonic Bandgap Crystals Involve Features Smaller than a Wavelength

- Periodic structures of holes with “defect”
- Results in “Bandgap” or wavelengths not allowed outside defect region
- Low loss transmission or high efficiency filters in waveguides

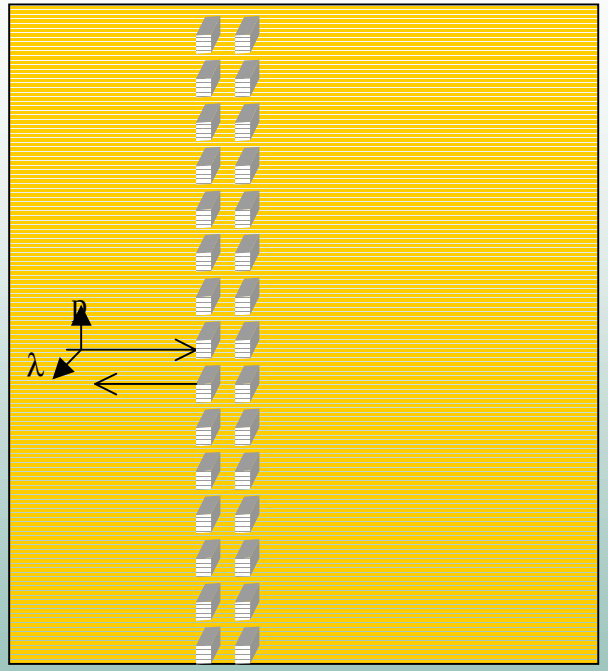


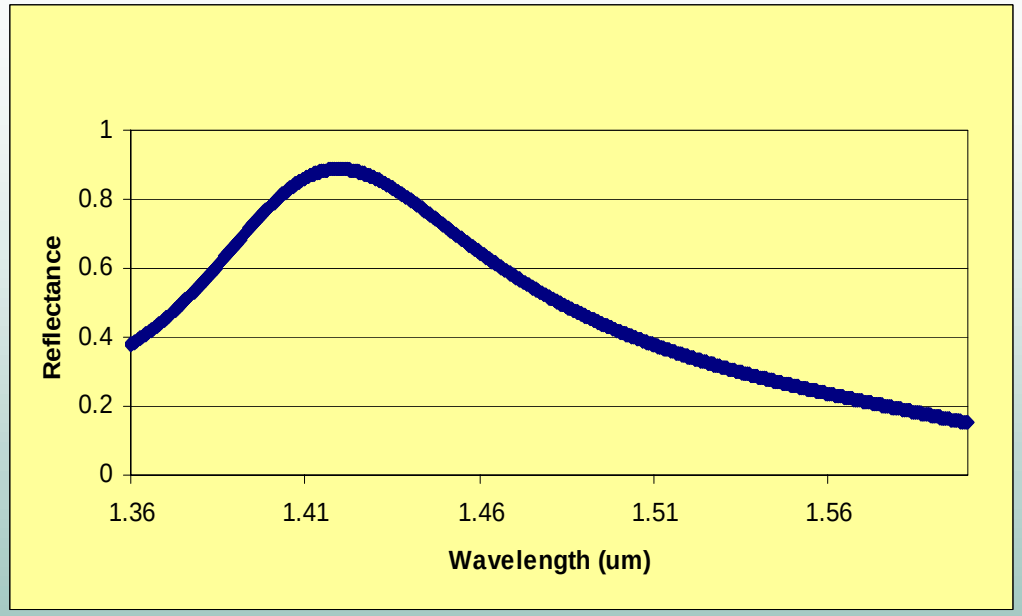
Reflectance from an Infinite Row of Holes (silicon waveguide) Modeled with Rigorous Coupled Wave Analysis



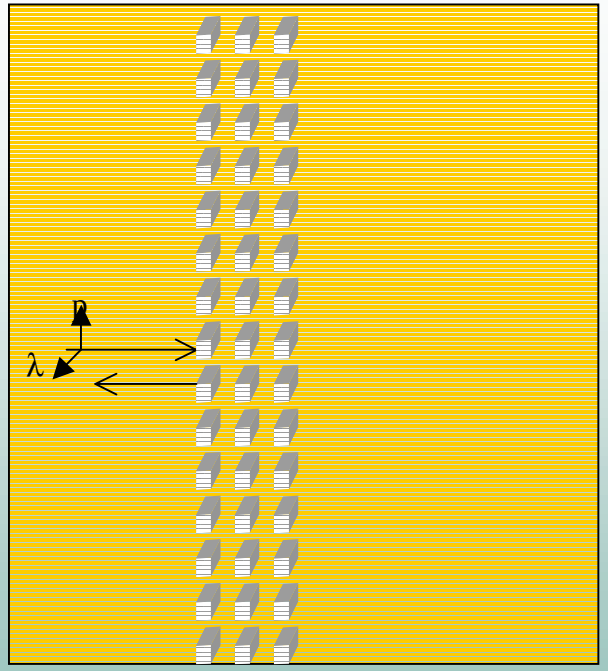


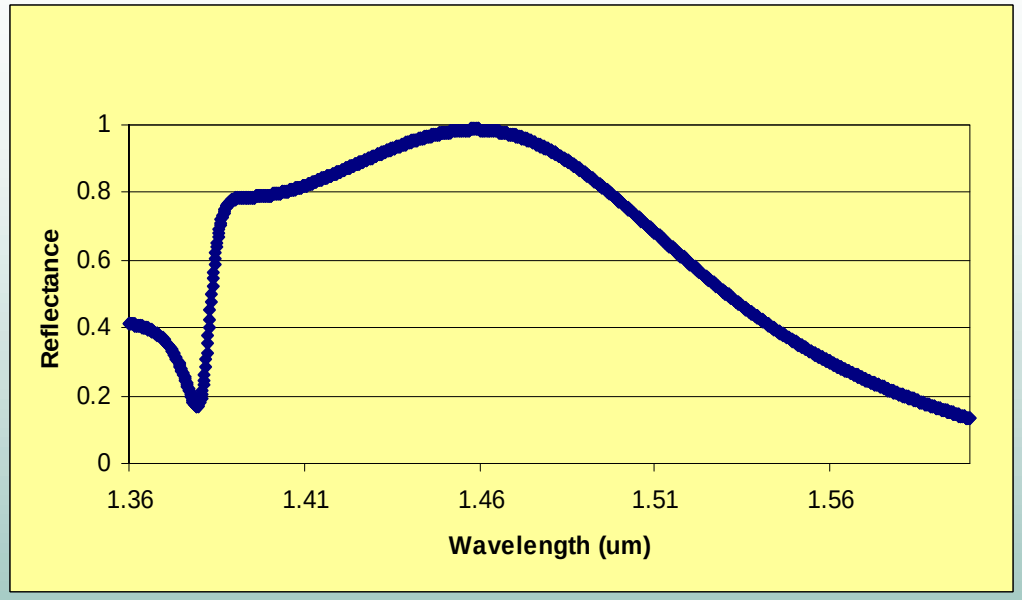
Reflectance from Two Infinite Rows of Holes



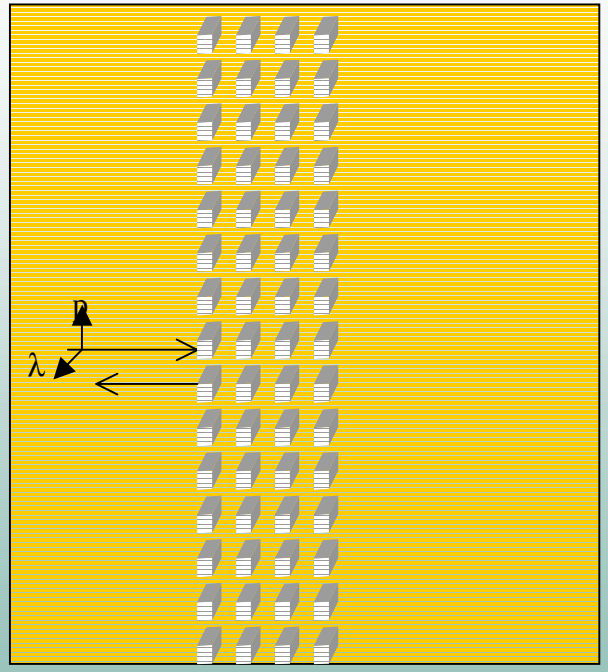


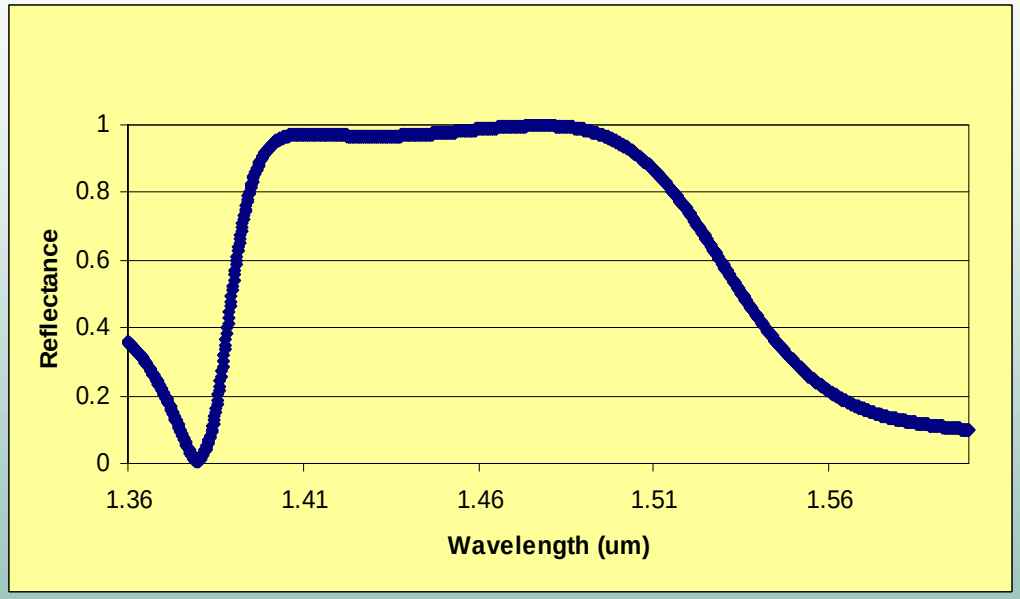
Reflectance from Three Infinite Rows of Holes



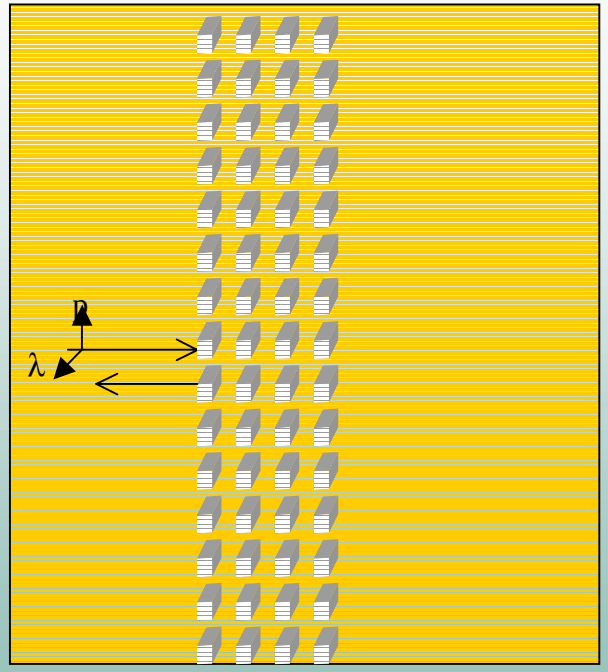


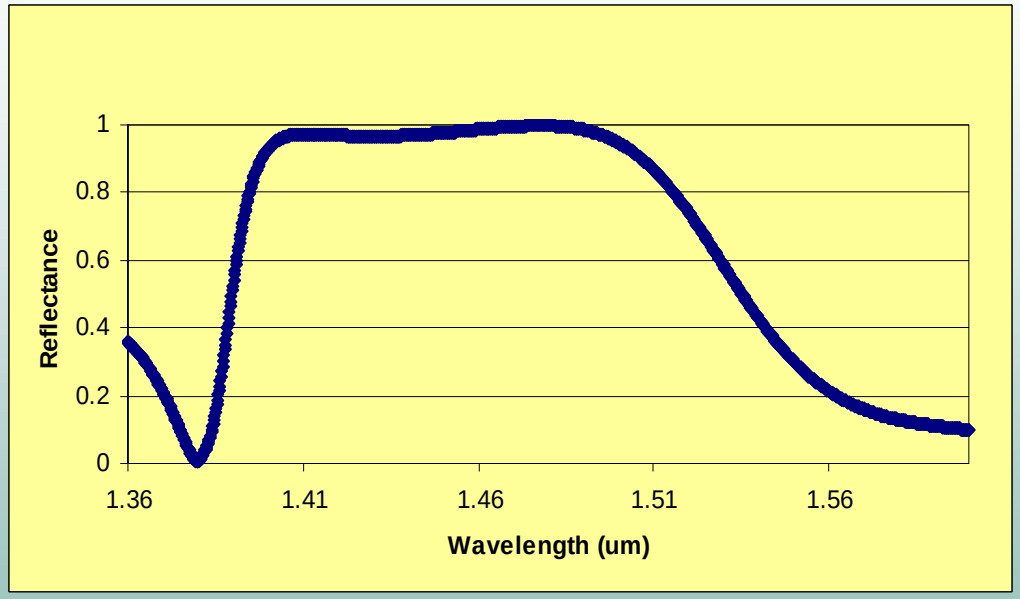
Reflectance from Four Infinite Rows of Holes



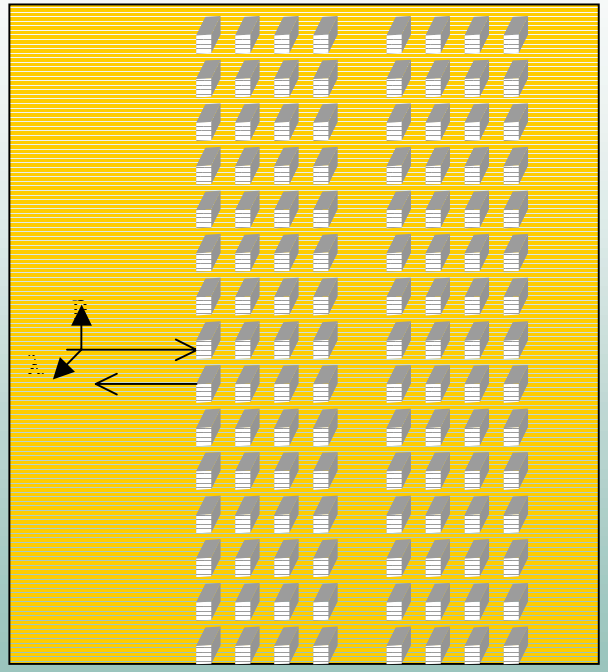


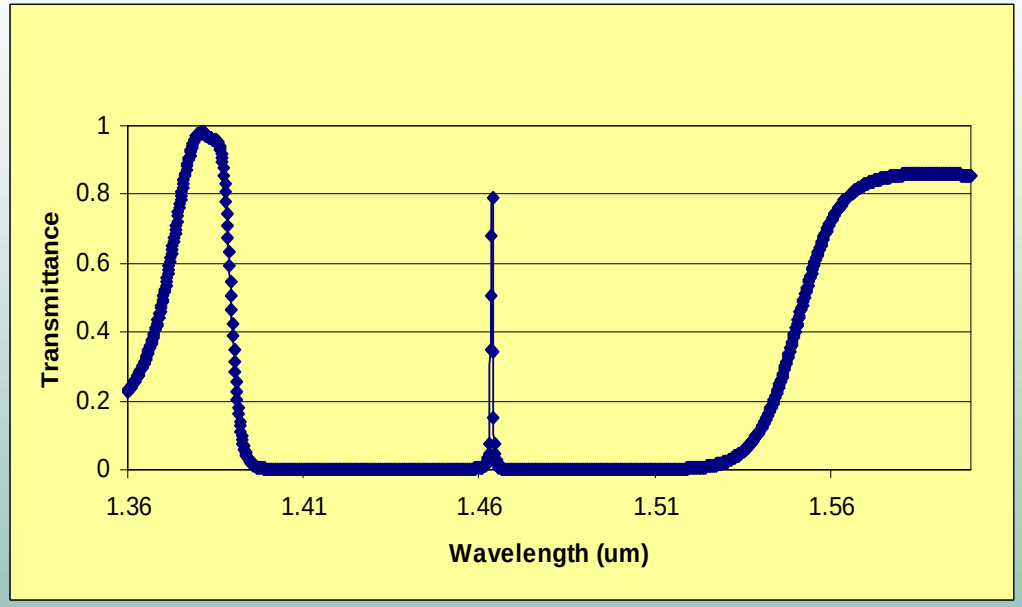
Reflectance from Four Infinite Rows of Holes



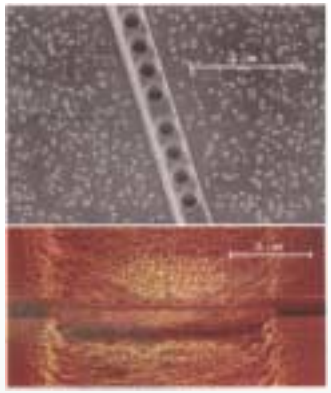


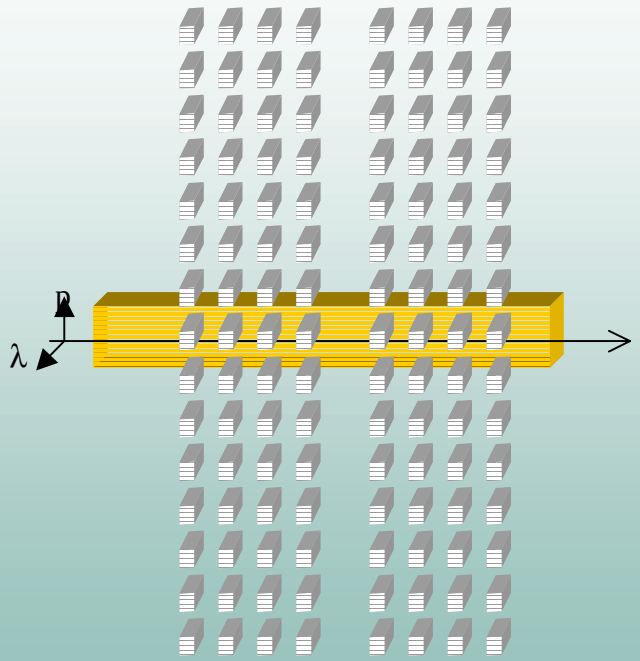
Transmittance from Two Sets of Four Infinite Rows of Holes (i.e. 2 mirrors - Fabry-Perot "Defect")

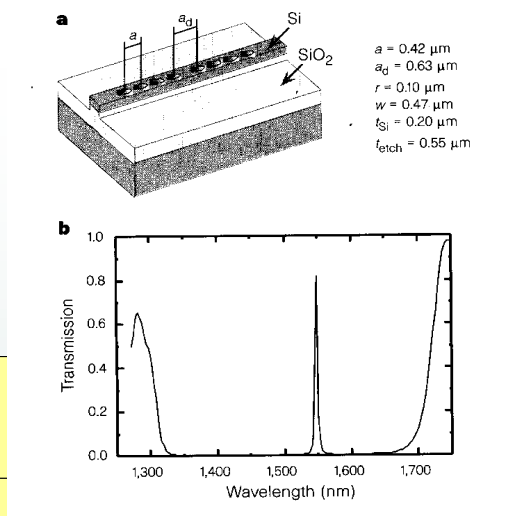


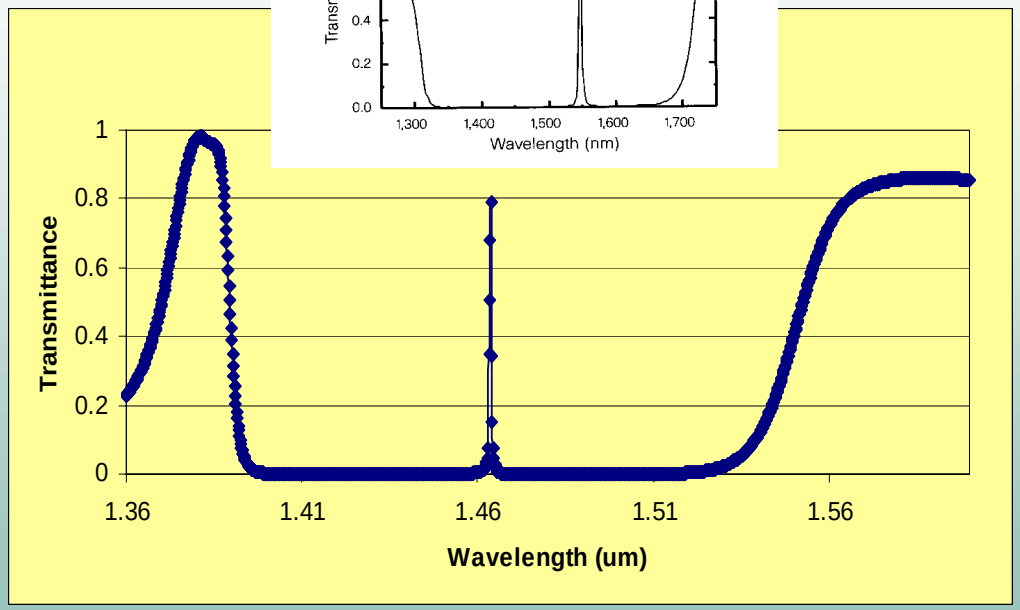


Transmittance from Two Sets of Four Holes in a Narrow Channel Silicon Waveguide (i.e. Joannopoulos et. al., Nature 386)









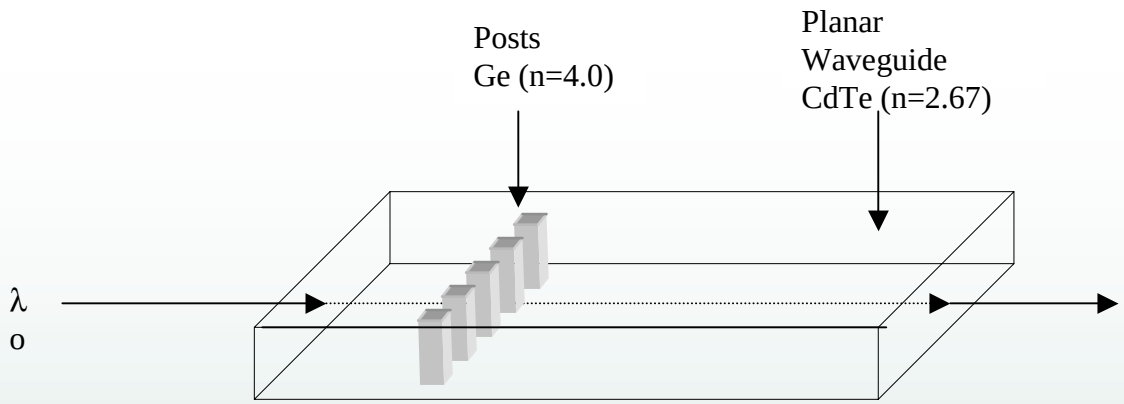
Implications

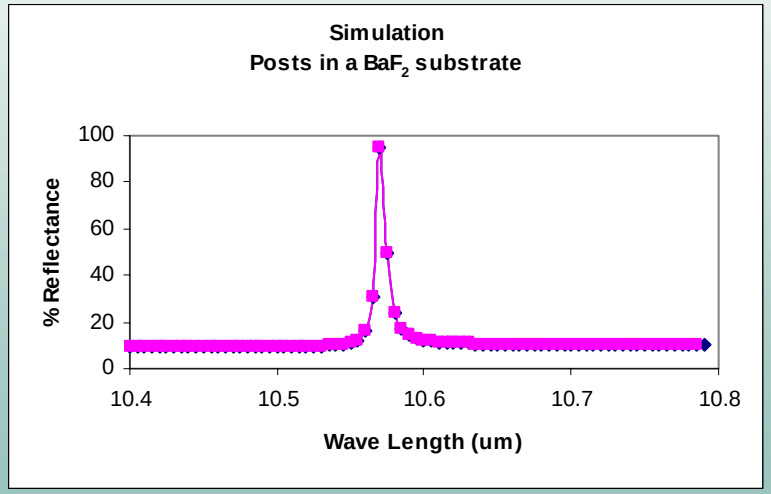
- A finite extent (1D) waveguide defect structure (holes or posts) can be described as a 2D PBG infinite extent structure (a much simpler calculation).
- A high "Q" Fabry-Perot device can be constructed by a simple 1D planar waveguide (small, highly functional PBG devices possible).
- Boundary conditions (finite extent) may be critical in making realizable devices

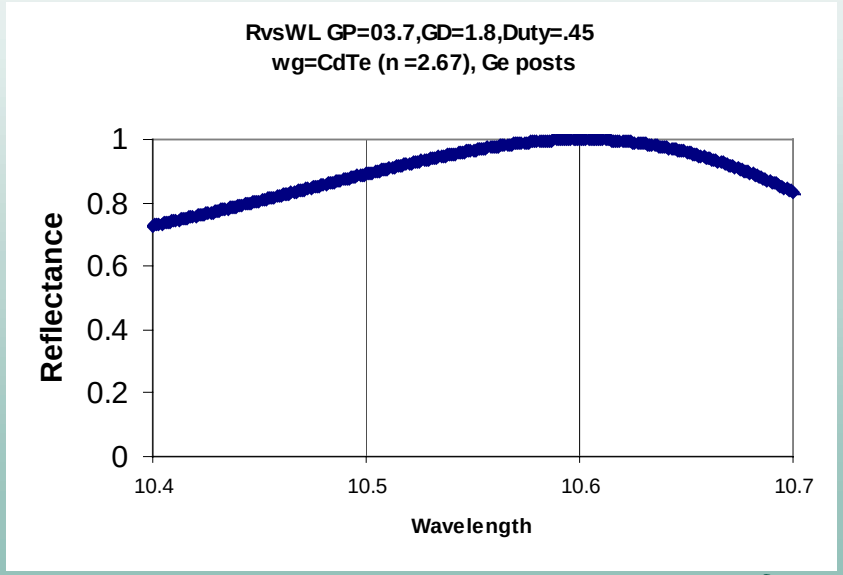
ORNL is Investigating High Index Features "Subwavelength Structures" (SWS)

- **Single linear array of high index <u>posts</u> replaces multiple arrays of holes**
- **Customize wavelength and polarization by materials, shape, and periodicity of posts**
- **Applications to datacom, telecom, and sensing**

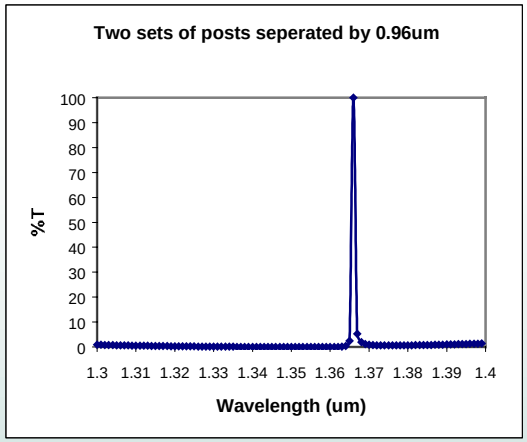
Depending on the materials used, periodicities and geometries, embedded high refractive index posts can product narrow band, broadband, or no reflectance

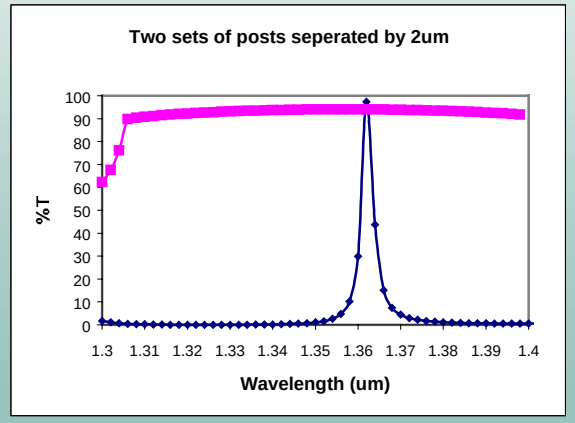


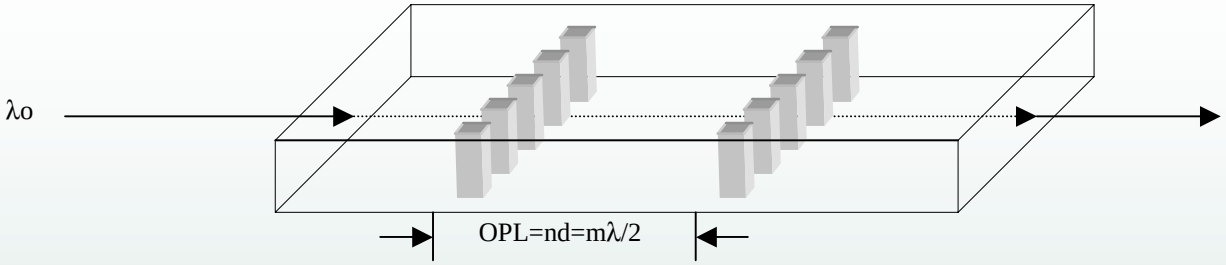




Resonant Cavities using Embedded Subwavelength Structures (SWS)

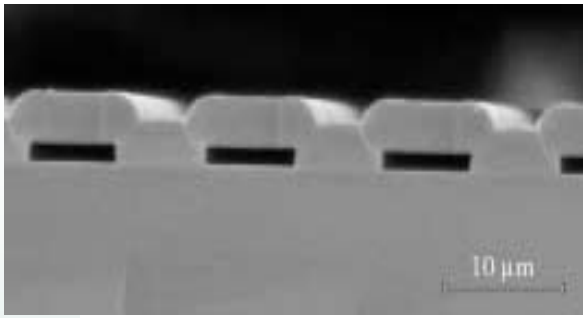


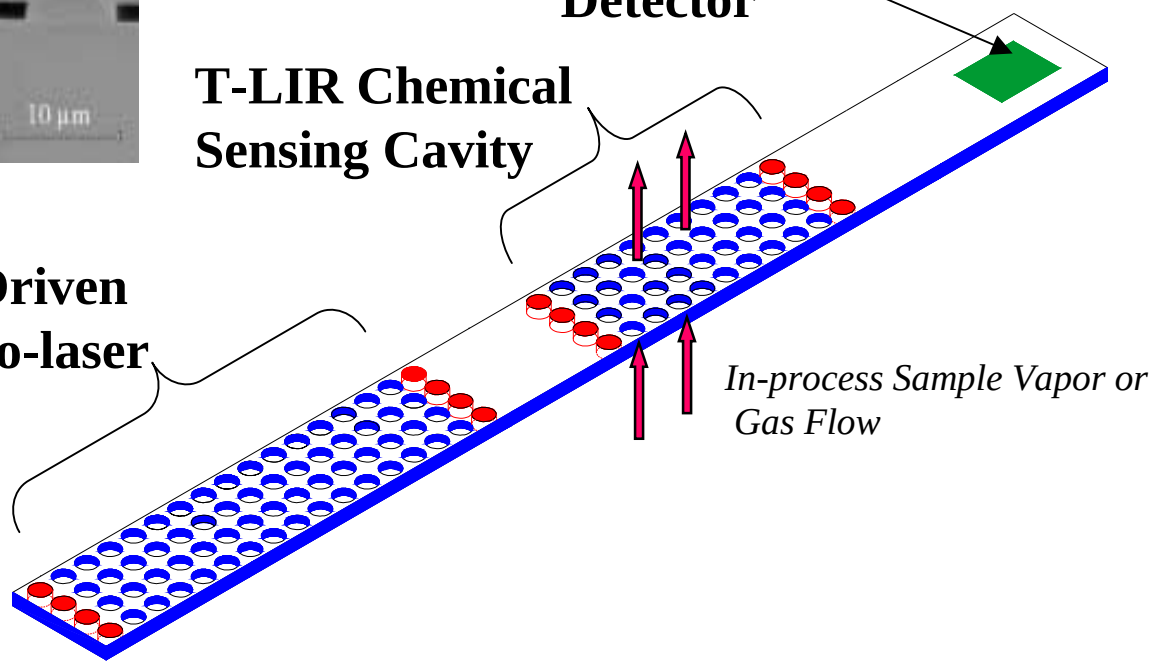




- High index "posts"
- 100 micron Fabry Perot cavity with Q = 1000 provides an effective 10-cm OPL
- Modulation with EO efficient materials
- Present IRAD Project looking at SBN

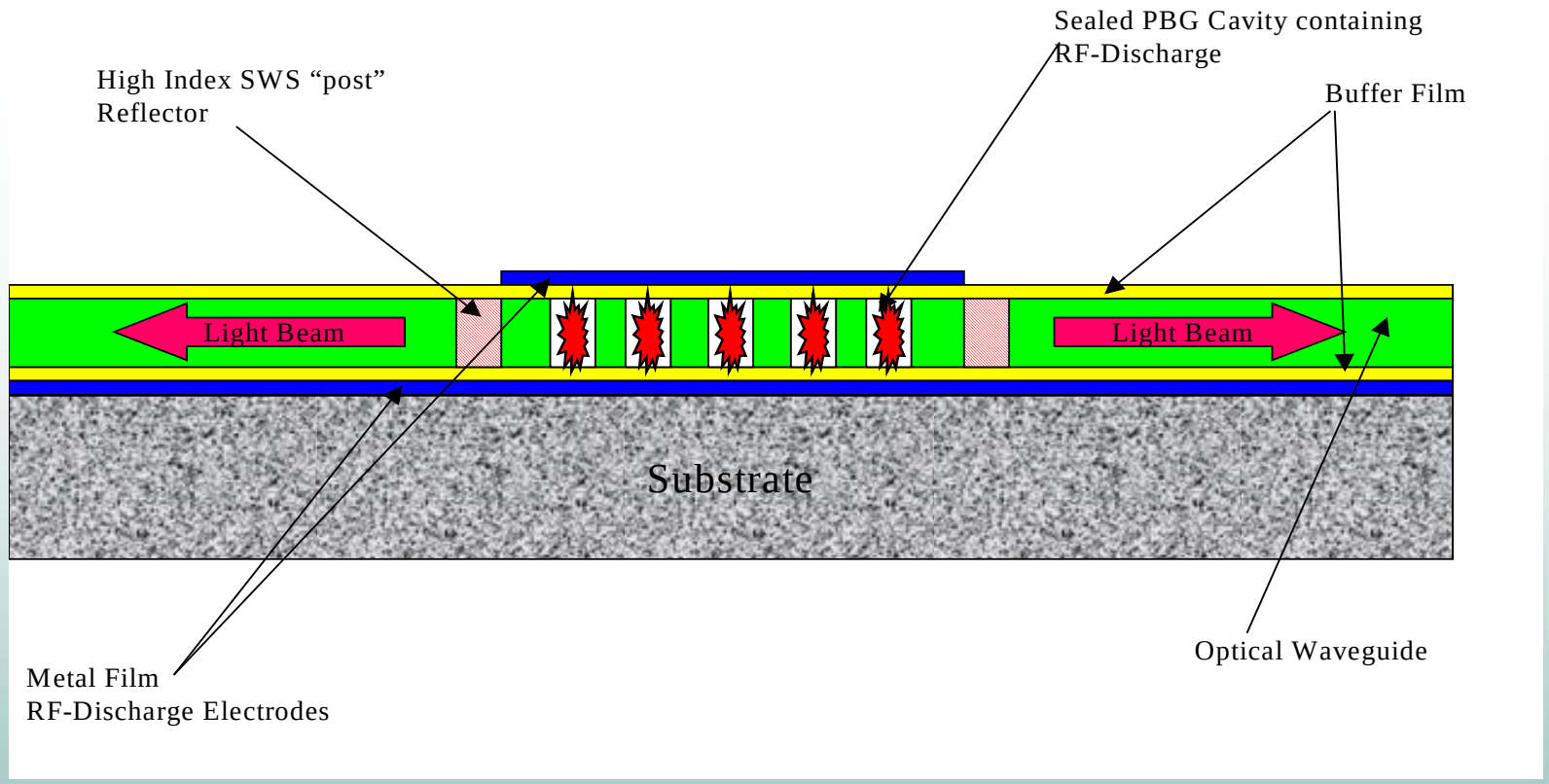
Putting 100 Lasers, Sampling Cells, and Detectors on a Chip Results in a High Resolution LWIR Spectrometer





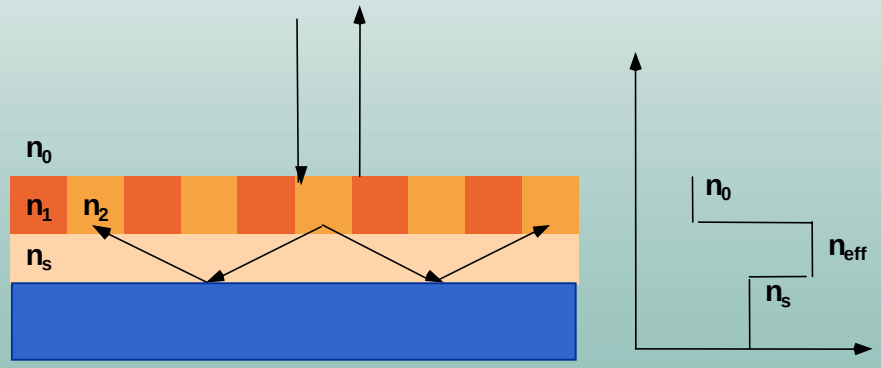
Integrated Optical Waveguide LWIR Chemical Sensor

Using Nanostructures and Resonant Cavities, CO_2 Lasers 30 um X 3 um are Realizable



Research in Guided Mode Resonant Filters (GMRFs)

- Coupling orthogonal to surface
- Linear gratings formed by surface relief
- Waveguide/grating couplers
- Resonant narrow band filters



ORNL Initially Worked with Sinusoidal GRMFs due to Ease of Fabrication

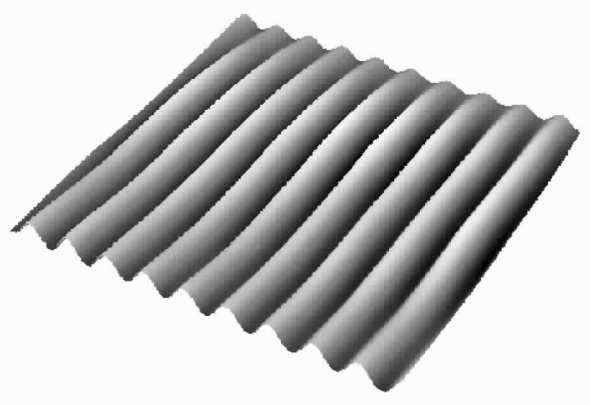
- Bandwidth (FWHM)

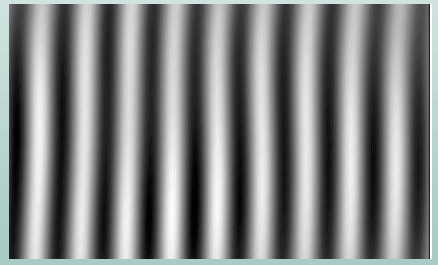
  $\Delta\phi = 0.3^{o}$

  --> $\Delta\lambda = 0.85nm$

- Performance limitations
  - grating nonuniformity
    - ghost interference
    - beam nonuniformity
    - mechanical jitter during exposure

AFM Scan

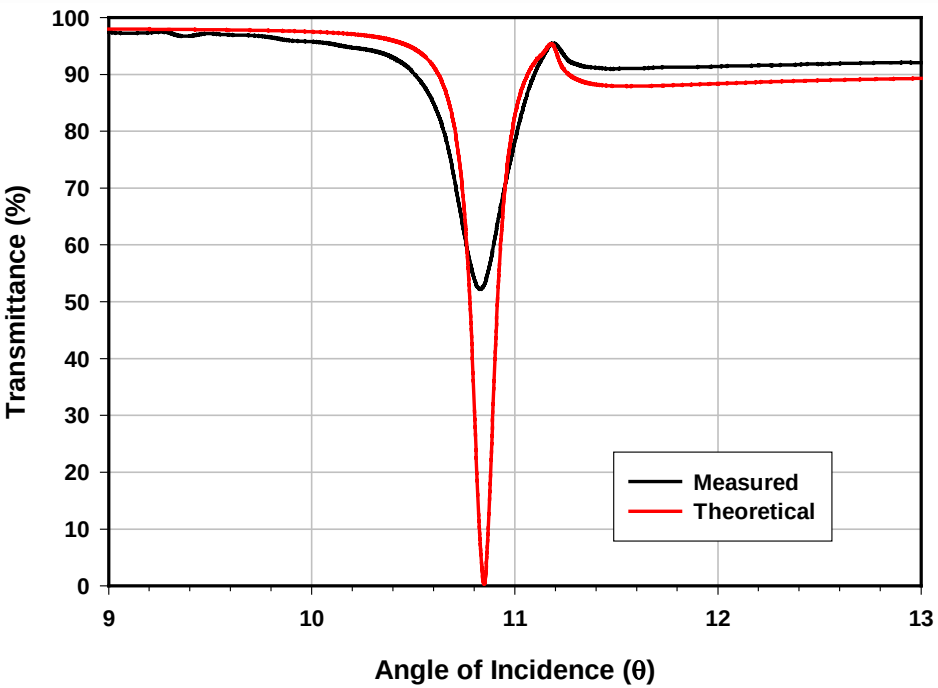




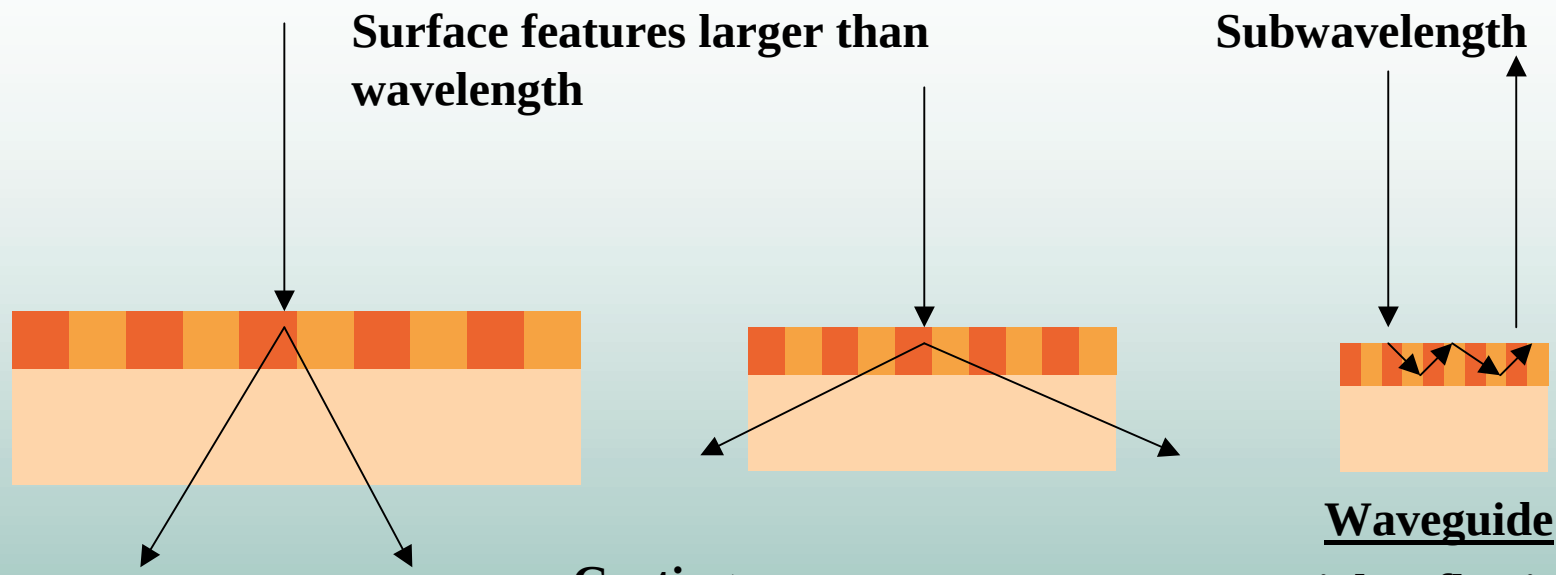
Sinusoidal GMRF Performance

Grating Specs

TE polarization
$\phi = 0^o$
$\lambda = 632.8nm$
$n_o = 1.0$
$n_s = 1.516$
$n_c = 1.64$
$d_c = 145nm$
$d_m = 270nm$
$\Lambda = 370nm$



As Surface Features Get Smaller than the Wavelength of Incident Light, Layer Becomes a Waveguide



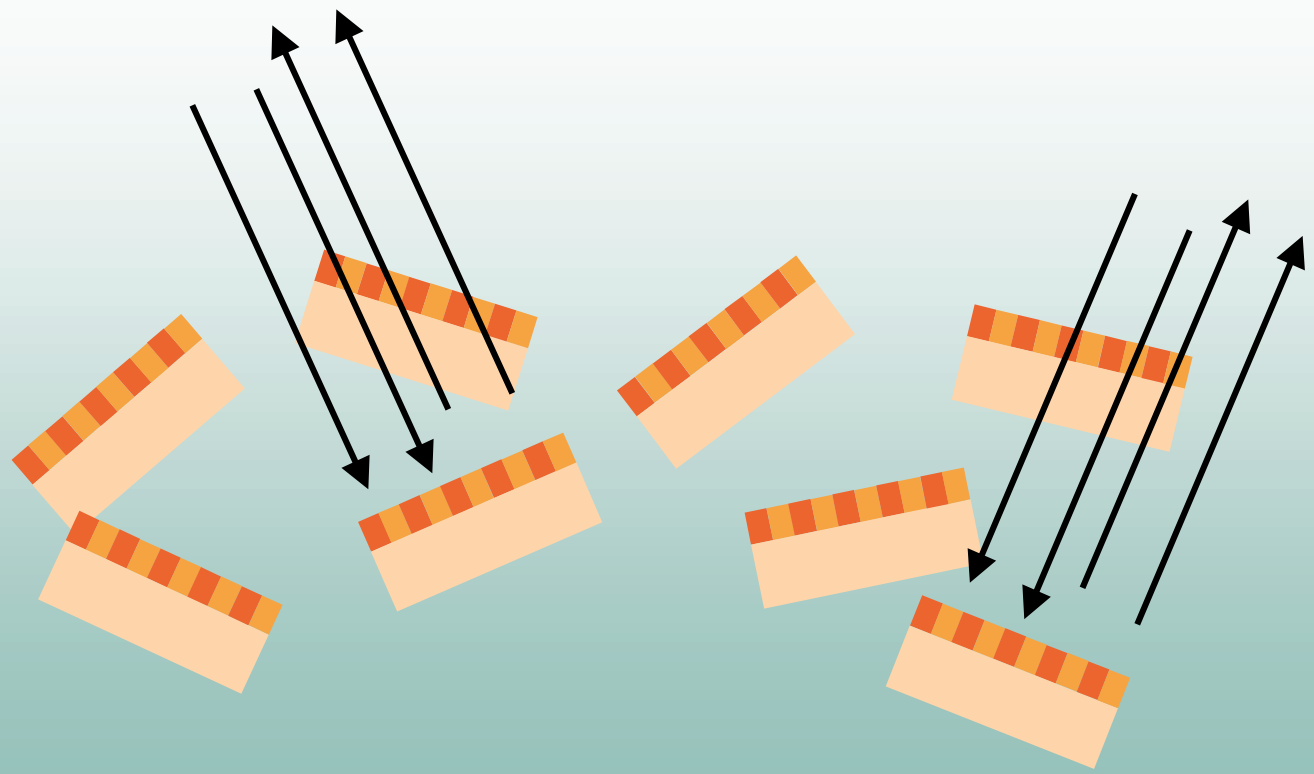
Grating

Low reflectivity:
Multiple diffractive orders transmitted

Waveguide

High reflectivity:
Entrance and exit conditions match

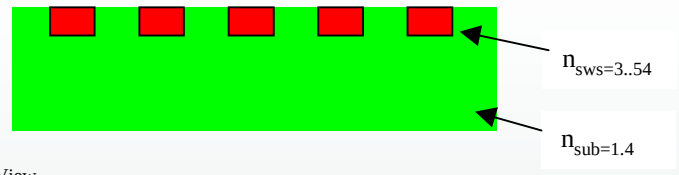
Key to Resonant Dust: Random Orientation Provides Signal Return from Wide Angles



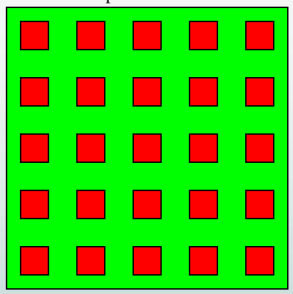
Present Resonant Dust Program R&D

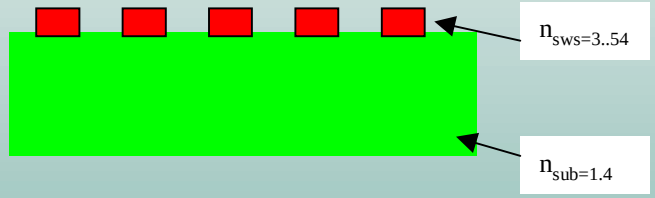
- Proof-of-principle resonant dust concept using 2D subwavelength surface reliefs at CO_2 laser wavelengths (9 to 11)
- Rigorous modeling of structures with finite extent (assisted by University of Arizona)
- Material science issues related to thin films and monolithic structures
- Fabrication Issues

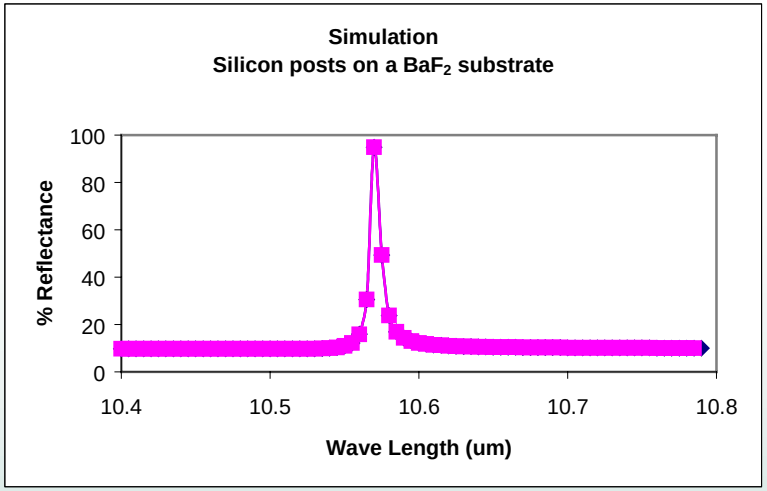
Simulation of Reflectance for 2D SWS Layouts

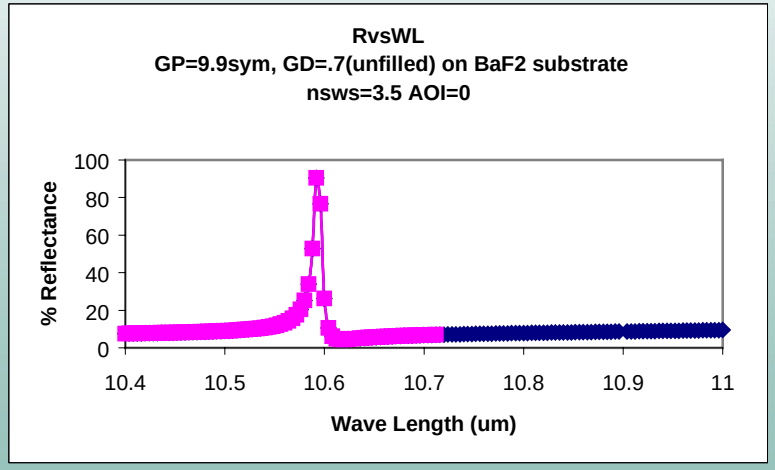


Top View

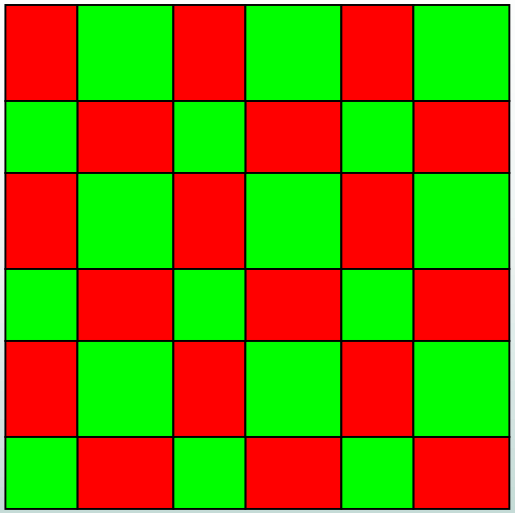




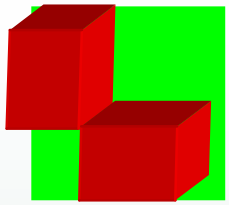




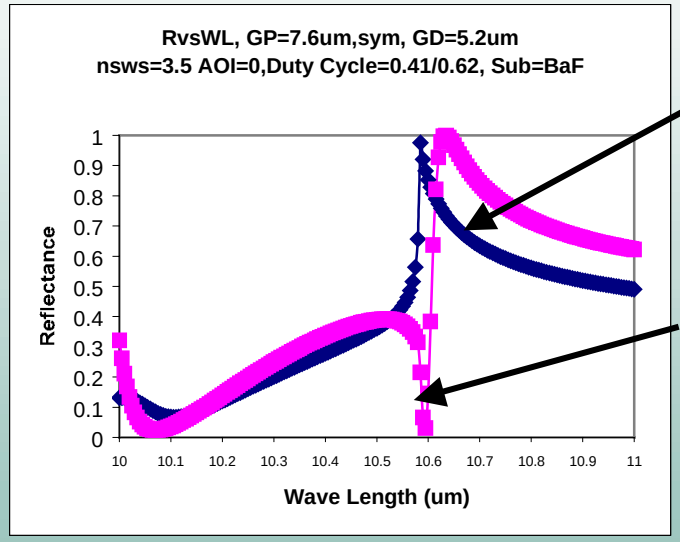
2D- Resonant Dust Checkerboard design - polarization selectivity



Si posts (Red) on BaF substrate (Green)



Unit cell

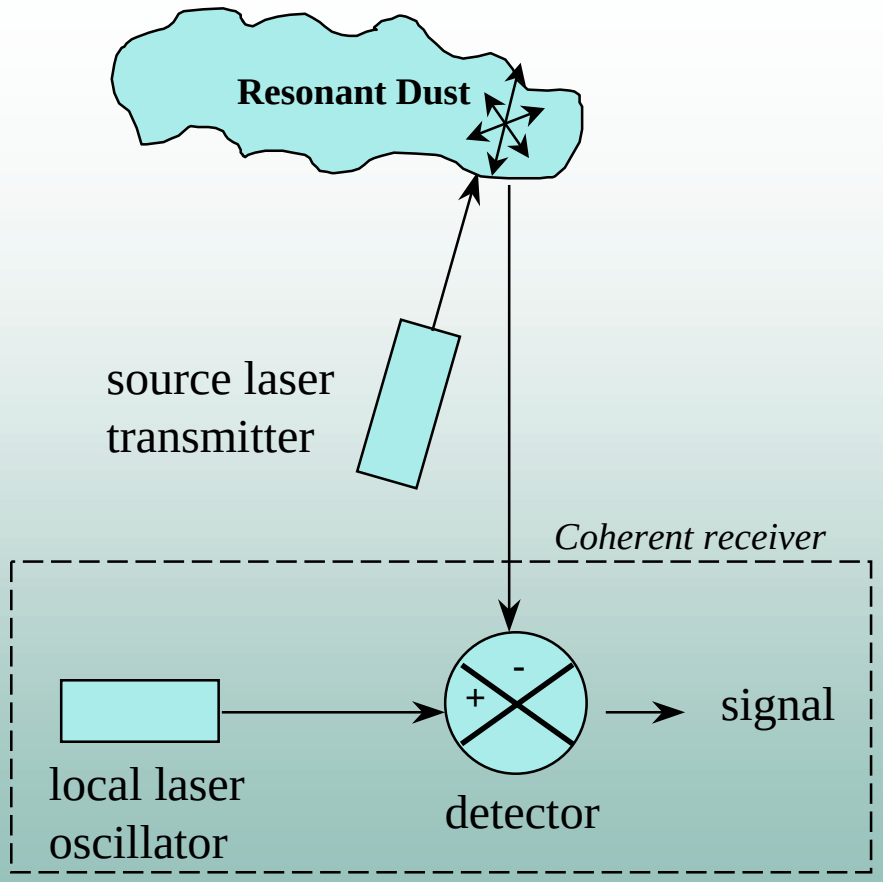


P polarization

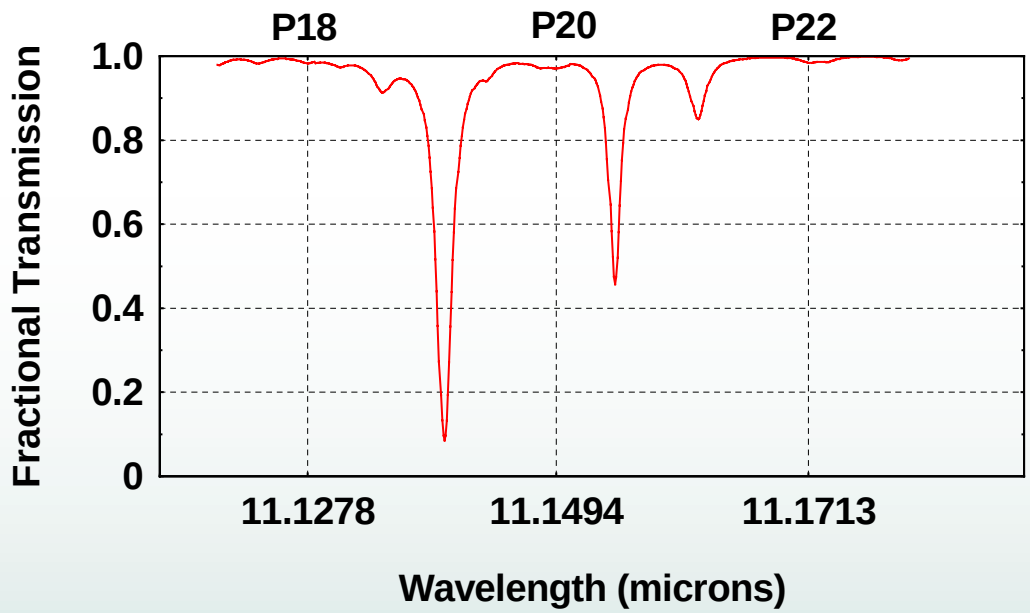
S polarization

Active Systems Measure Return Signals using a Laser Transmitter

- Differential lidar (DIAL) for spectroscopy
- Doppler lidar for velocity or vibration
- Time-of-flight lidar for range

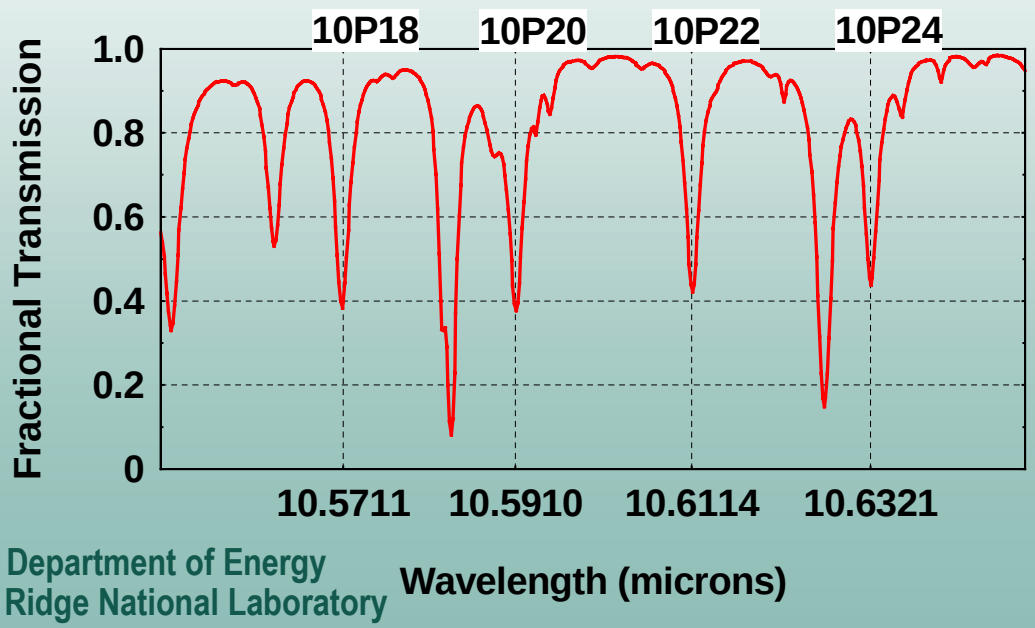


Atmospheric Absorption for 6-Km Path Length



$^{13}C^{16}O_2$

Alternate isotopes of CO_2 can be used to improve atmospheric transmission if an appropriate modulator gas can be found.



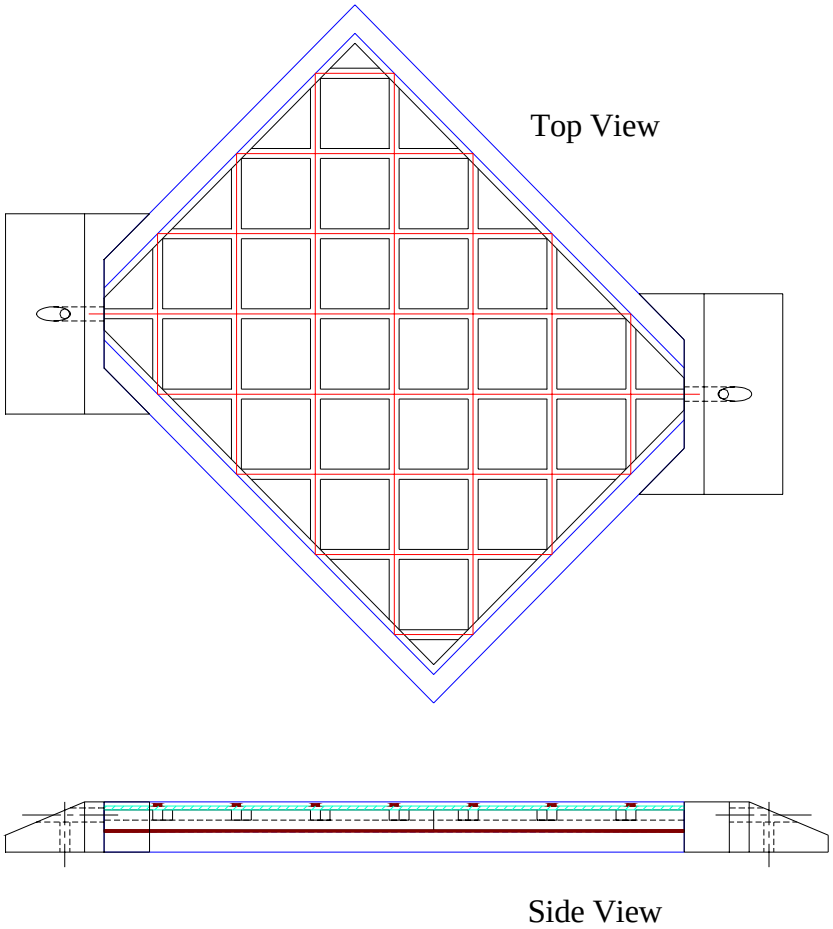
$^{12}C^{16}O_2$

10 Micron Technology is Moving toward Smaller Lasers and Room Temperature Detectors and Arrays

- Folded cavity and waveguide CO_2 lasers with 0.1 m^2 footprints and >5W output power
- Quantum well infrared photodetectors (QWIP) arrays to 640 X 480
- HgCdZnTe and QWIP room temperature detectors

Mini-CO_2 Laser – "Wristwatch" size

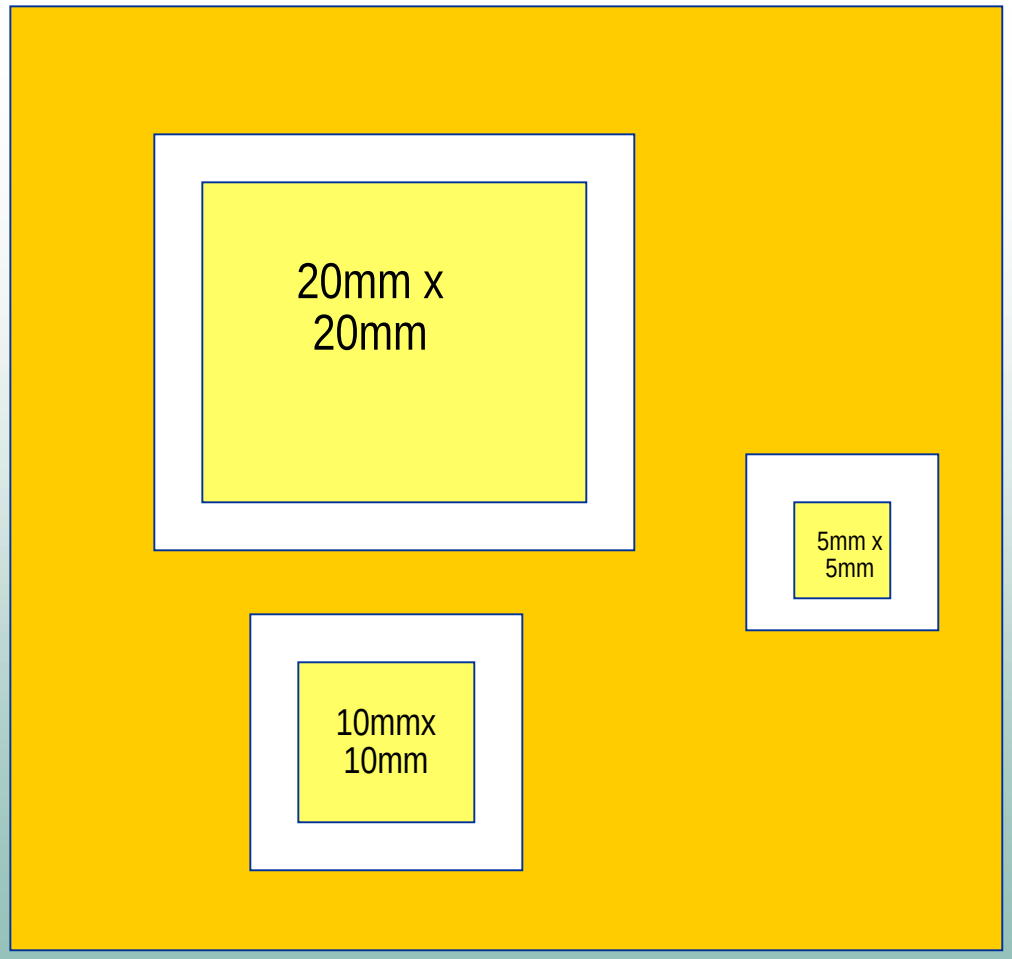
- RF driven--20 watts
- Laser Power--1-2 watts
- 0.5 x 0.5 mm glass or ceramic folded waveguide structure
- Pressure--100-300 torr
  - –sealed operation
- Gain length~10 cm
- Thickness~3 mm
- Size--2.5 x 1.8 cm



Resonant Dust Fabrication and Testing Milestones

- Deposition of Silicon on BaF_2 substrate
- Control of deposition rate
- Photolithography (mask, imaging, and developing)
- 10 reflectivity measurements and surface metrology (SEM, AFM, optical microscopes)

Resonant Dust Lithography Mask (testing different extents)

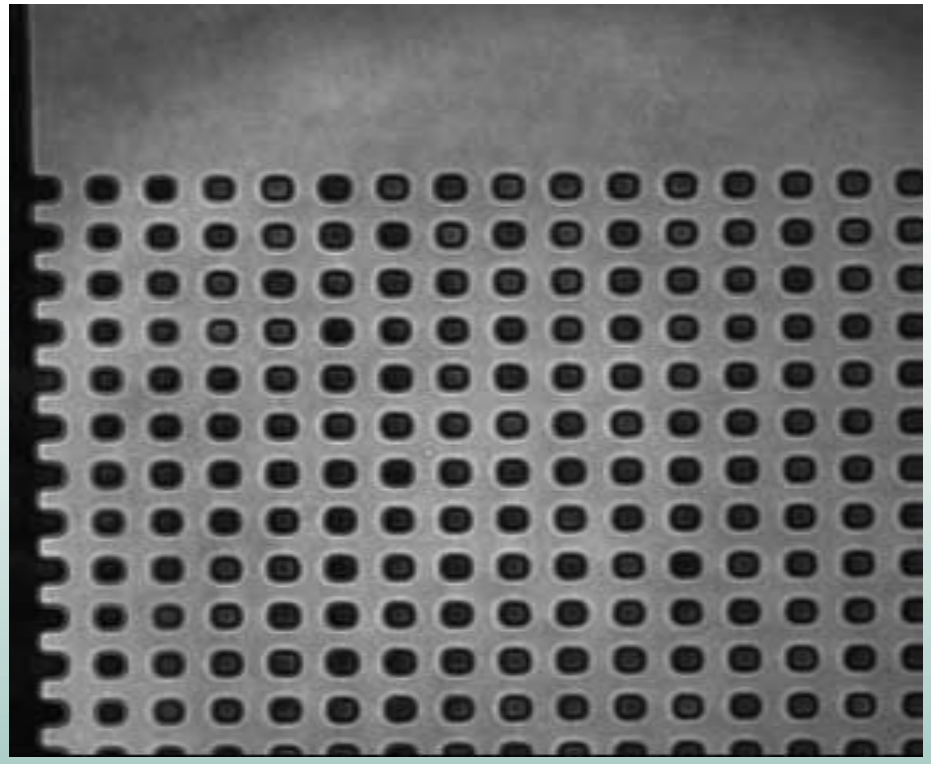


Crome

2-D SWS

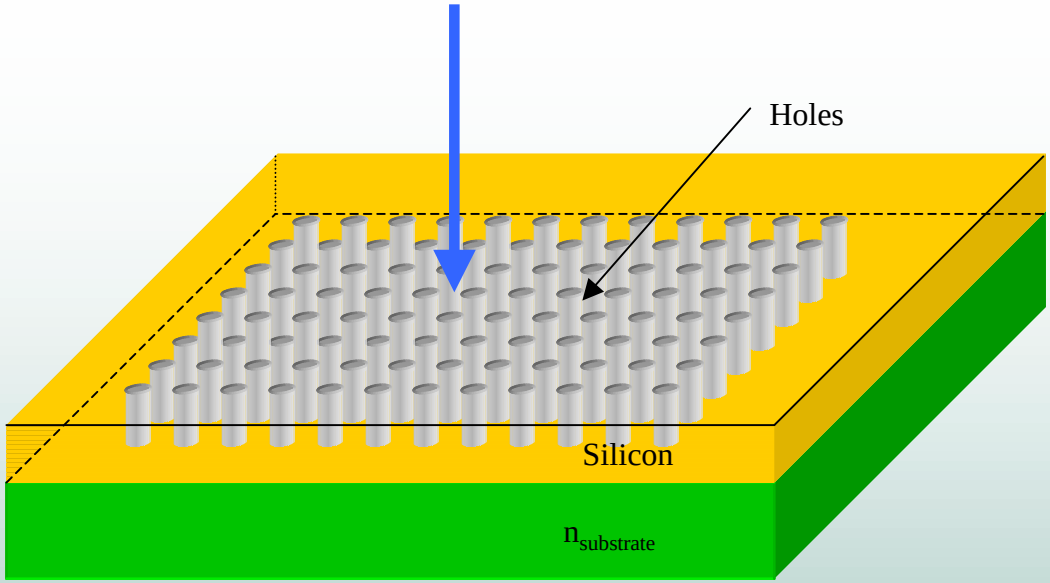
Clear Glass

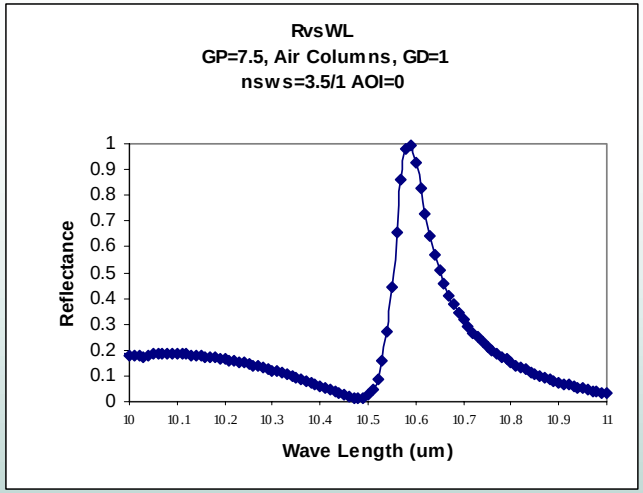
Lithography Mask 2-D Subwavelength Structure Image



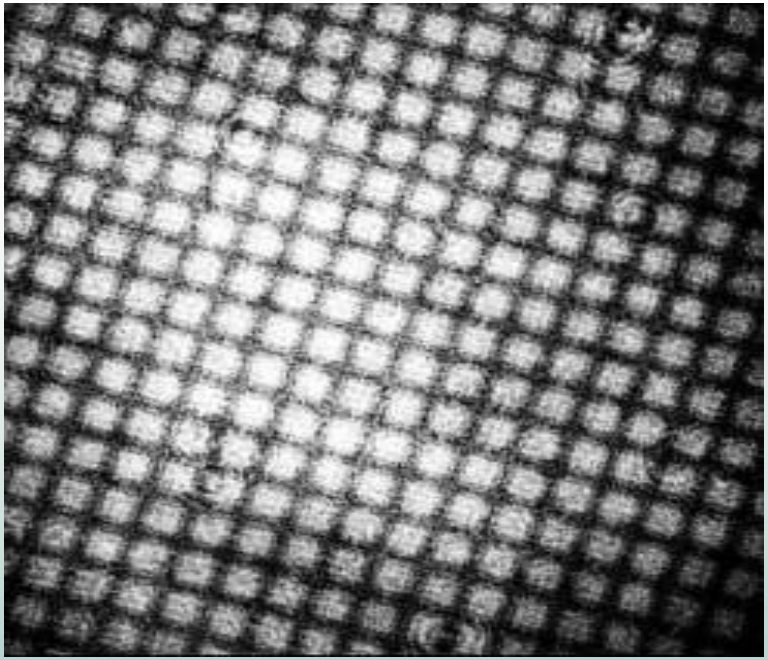
30 um

Current Design /Simulation

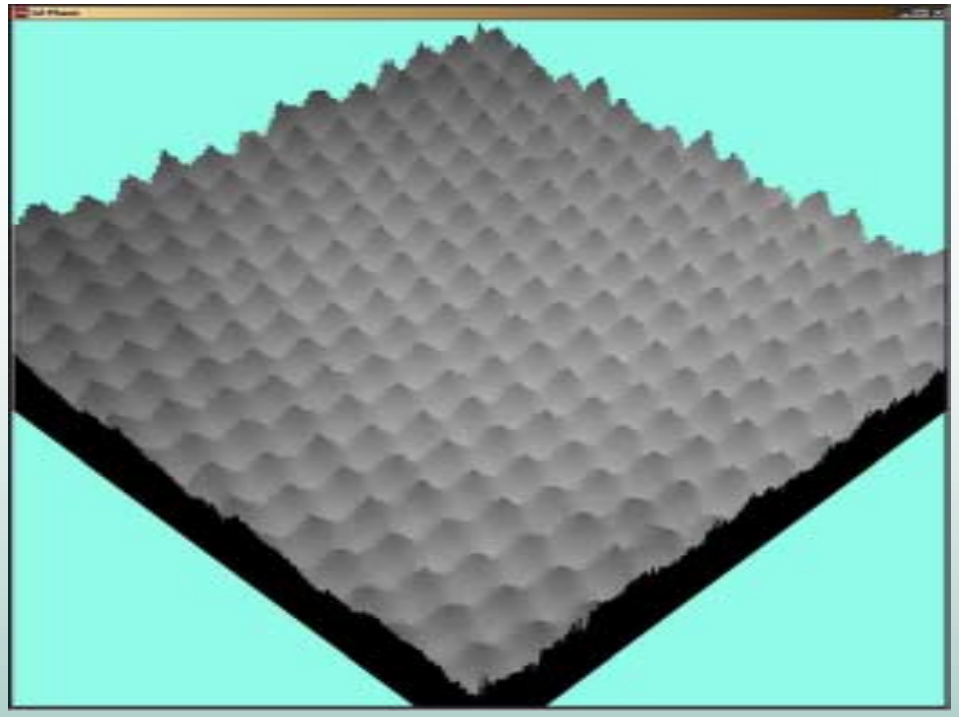




Current Resonant Dust Structure



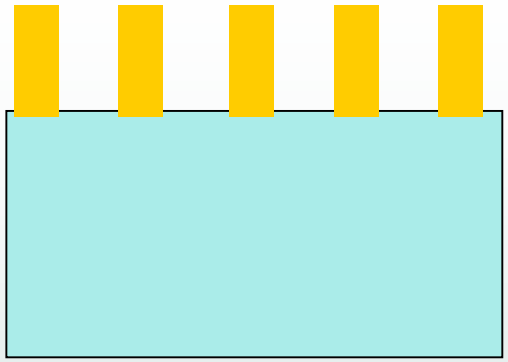
Optical Intensity Image

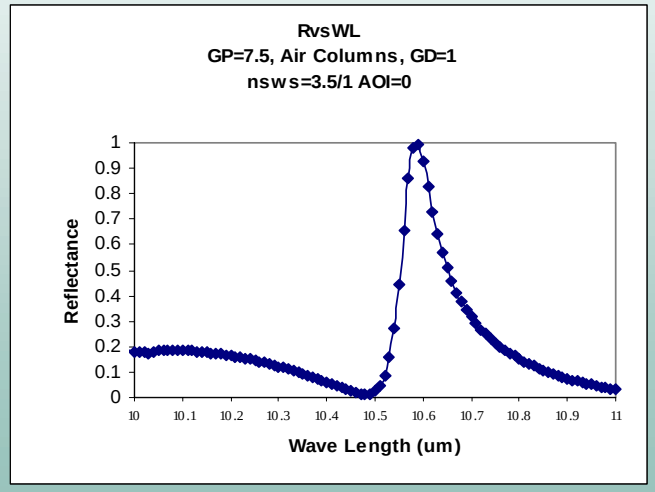


Optical Phase (3-D) Image

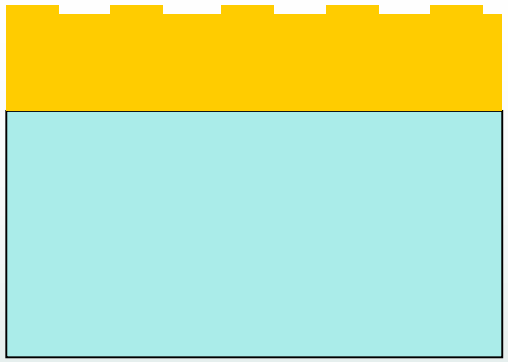
Current Structure Cross-section using Photolithographic Process

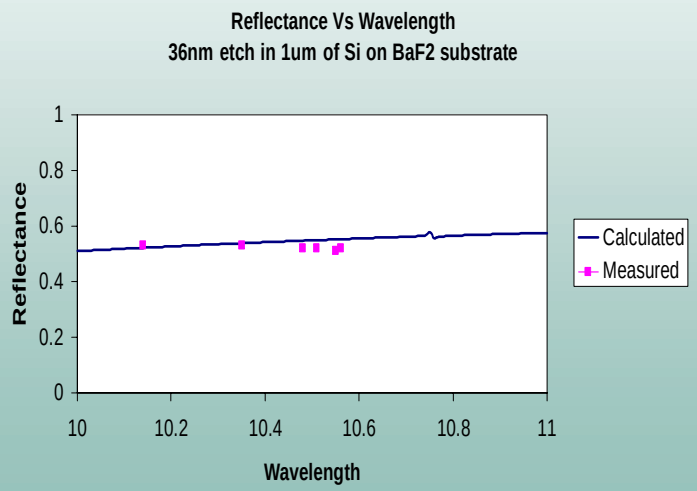
Design/Simulation





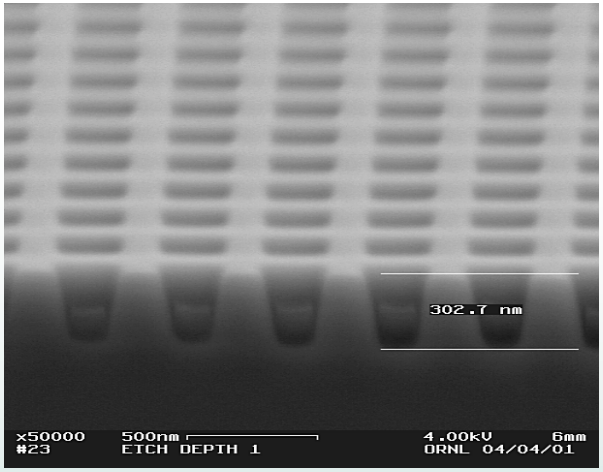
Fab'ed/Measured

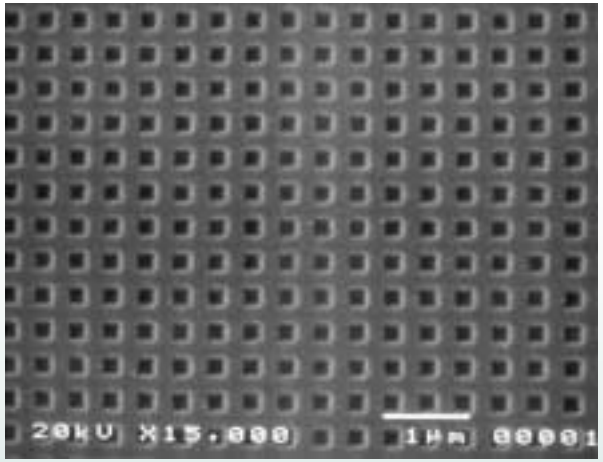


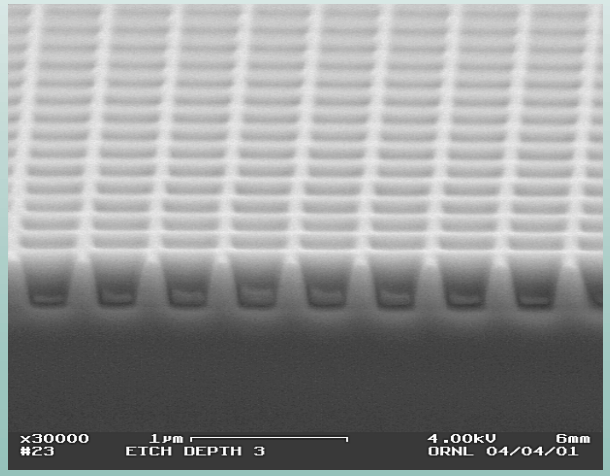


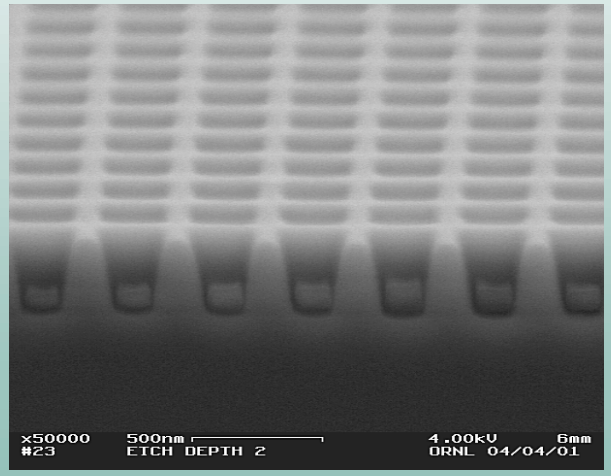
Alternate Microfab Process at ORNL: Focused Ion Beam Milling

a-Si on a-SiO_2



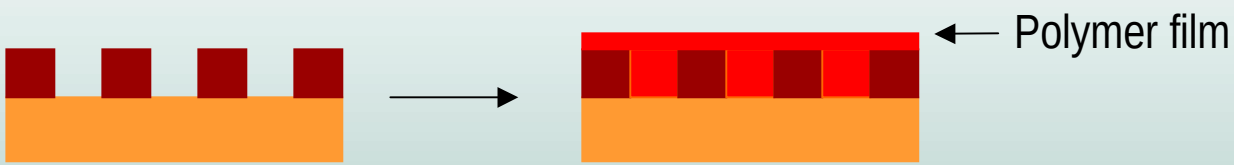






Chemically Selective Resonant Dust

- **Polymer film covalently bound on the surface of the resonant dust.**
- **Adsorption of organic vapors into the polymer film:**
  - **Changes the polarity of the polymer film (i.e., its dielectric constant) which alters the refractive index of the polymer.**
    - **For an example see: *Nature* 1996, *382,* 697 and *Acc. Chem. Res.* 1998, *31*, 267,**
  - **Swells the polymer film which changes the optical pathlength.**

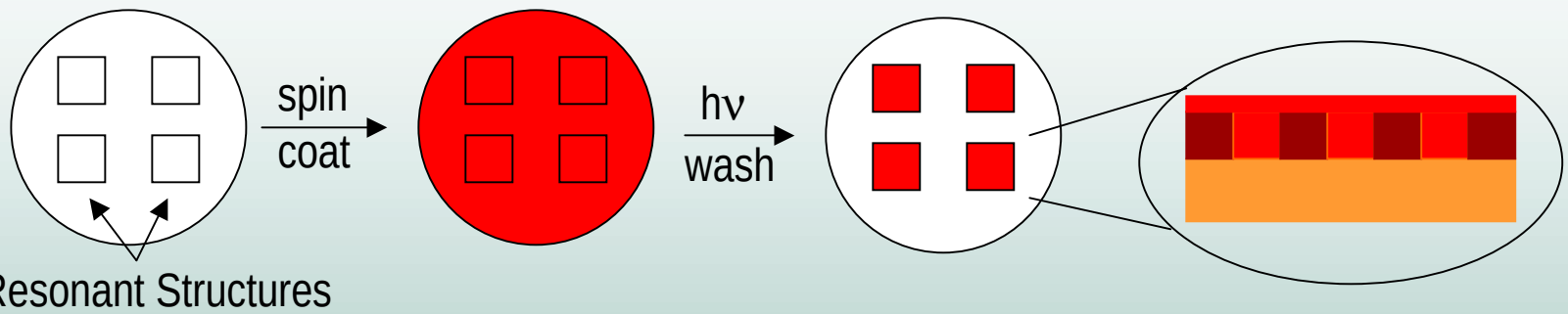


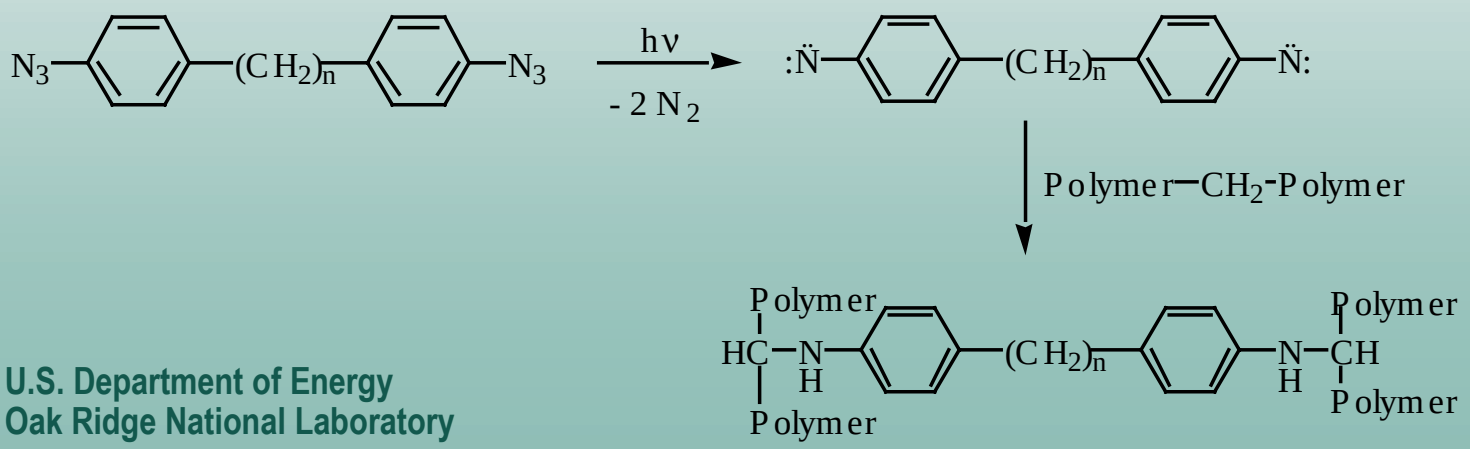
Polymer Properties

- **Polymers should be optimized to selectively absorb the vapor of interest, but binding should be reversible.**
  - **Polymer selection can be guided by using the linear solvation energy relationship (*ChemTech* 1994, *24(9)*, 27).**
    - **Determines the equilibrium (i.e., the partition coefficient K_p) of a vapor between the gas phase and the polymer phase based on polarizability (R_2), dipolarity (π_2^H), hydrogen bonding acidity (α^{2H}) and basicity(β_2^H), and dispersion (L^{16}) interactions.**
    - **$\text{Log } K_p = c + r R_2 + s \pi_2^H + a \alpha_2^H + b \beta_2^H + l \text{ Log } L^{16}$**
- **Polymers must be optically transparent to the probe wavelength.**
- **Polymers should adhere to substrate surface, be thermally stable, and have a small response to humidity.**
- **Best approach which provides the most chemical information is to use multiple polymers (3 to 5) that cover the range of solubility interactions.**

Synthesis Methods

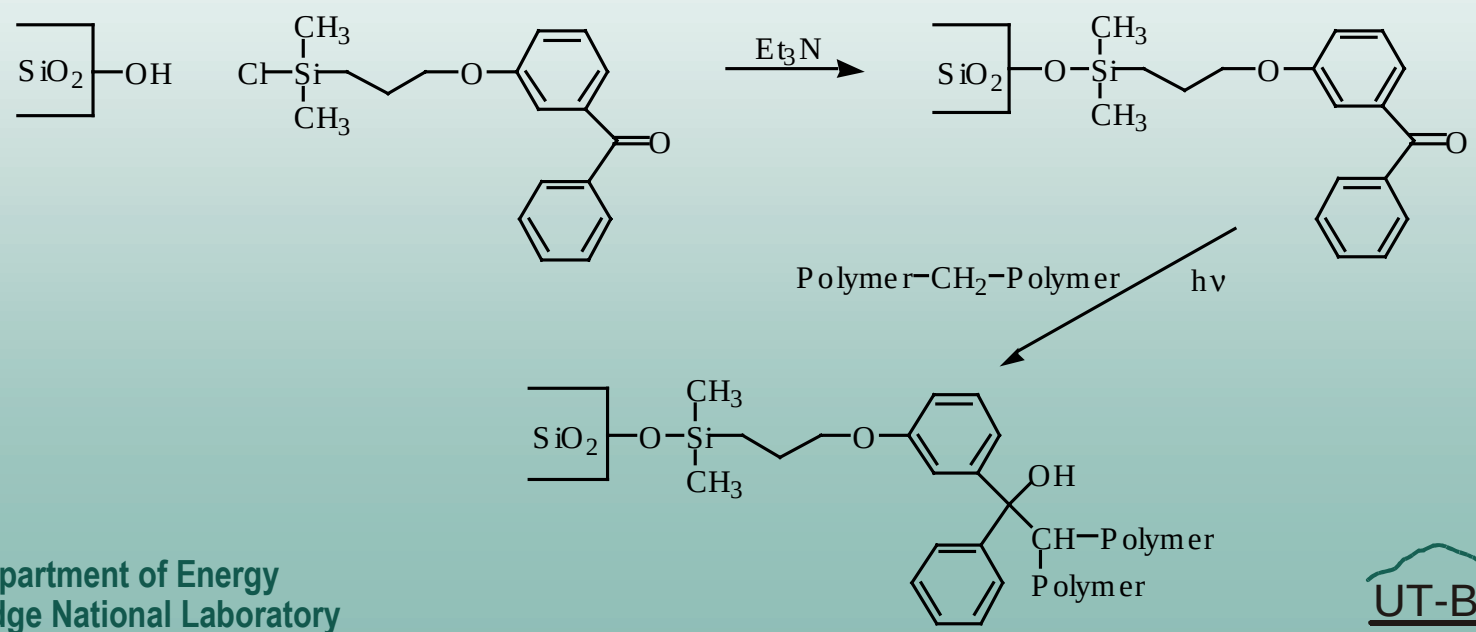
- Photochemical cross-linking of the polymer.
  - Spin coat polymer and photoactive cross-linking agent onto the structure.
    - Surface of substrate might need to be modified to improve polymer adsorption.
  - Irradiate the structure to cross-link the polymer (and reduce its solubility).
  - Wash off excess polymer.





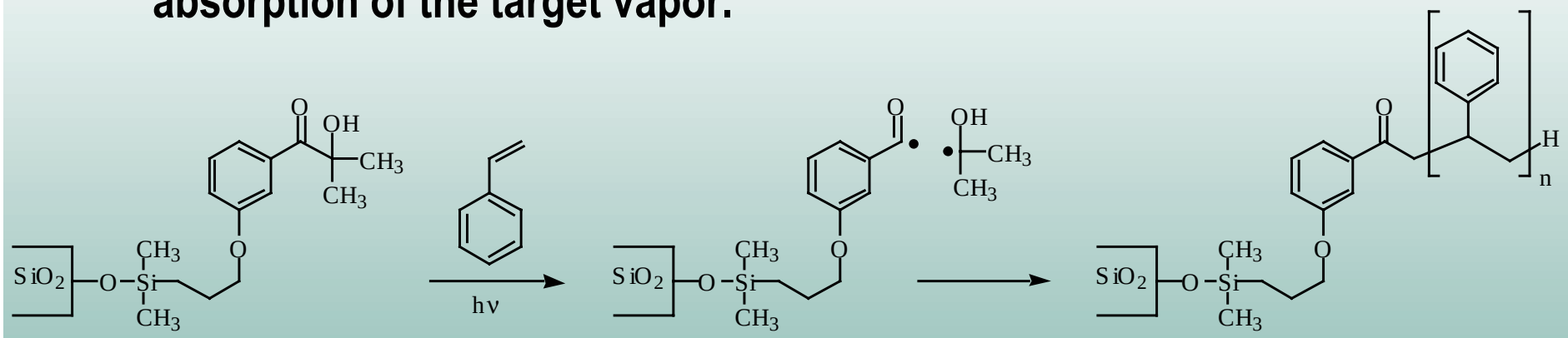
Photochemical Attachment of Polymer Films to Surfaces

- **Covalent attachment enhances stability of the polymer film on surface.**
  - **Silane chemistry can be used to condense cross-linking agents to the surface.**
  - **Illumination (at 350 nm) couples the benzophenone to the polymer by hydrogen abstraction and coupling of the resulting radicals. (see *J. Am. Chem. Soc.* 1999, *121*, 8766)**
  - **Nonattached polymer removed by extraction.**
  - **Thickness controlled by illumination time and polymer molecular weight.**



Polymerization on the Surface

- **Attach photochemical or thermal initiators to the surface and grow the polymer films directly on the surface.**
  - **Surface-initiated polymerizations have been reported with hydroxyacetophenone derivatives (*Adv. Polym. Sci.* 1995, *123*, 128) and azo initiators (*Macromolecules* 1998, *31*, 592).**
  - **Polymers, polymer brushes (*J. Am. Chem. Soc.* 2000, *122*, 1844) and novel block copolymers can be prepared on the surface to optimize the absorption of the target vapor.**

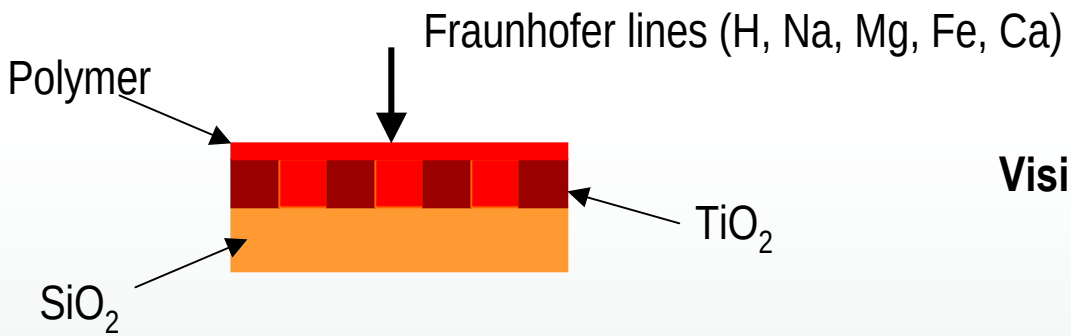


Surface attached polystyrene

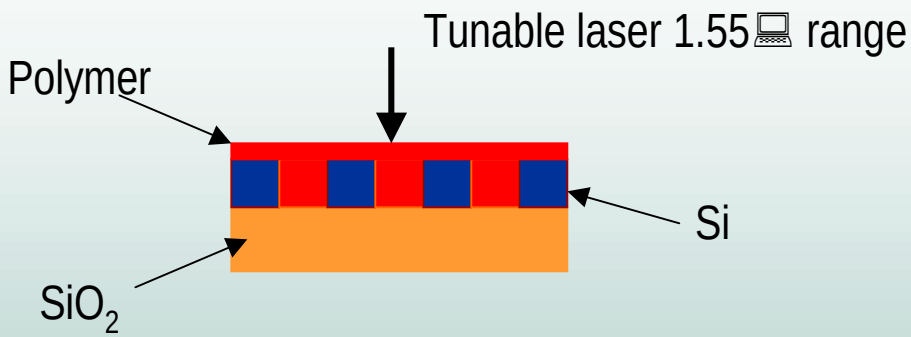
Research at ORNL on Polymer Coated Chemical Sensors

- **Microcantilever chemical sensors:**
  - **Self assembled monolayers (alkanethiols on gold) have been placed on microcantilevers for detection of cesium and chromate in solution. (*Chem. Commun.* 2000, 457 and *Anal. Chem.* 2001, *73*, 1572)**
  - **Polymer films have been coated on microcantilever for vapor phase detection of explosives (TNT and 2,4-dinitrotoluene). Polymers coated on structures by matrix assisted pulsed laser evaporation.**
- **Internally funded project to develop new synthetic methods to grow polymer films on micromechanical devices via surface polymerization techniques.**
- **Molecularly imprinted polymers were synthesized for the specific recognition of a target analytes for sensor applications.**

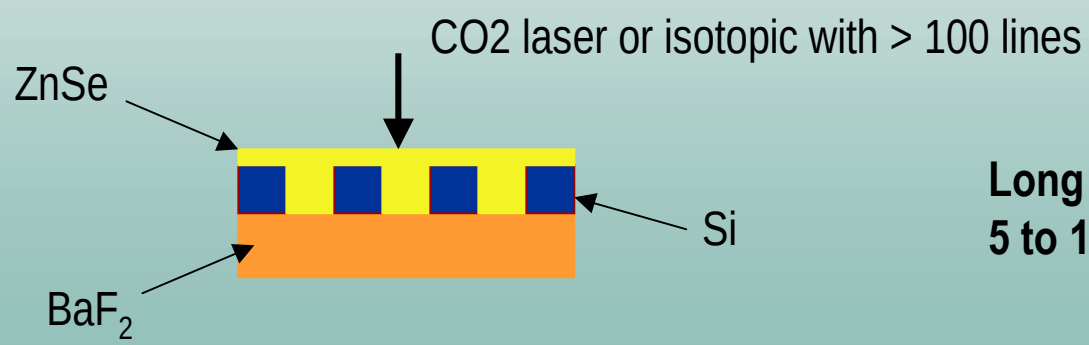
Several Material Systems are Possible for Different Wavelength Ranges, Sources, and Applications



Visible: Passive tags with selectivity



Near IR: Active tag with selectivity



Long wave IR: Active tag for ID over 5 to 10 Km

Research in Micro/Nano-structures is Important to the Future of Semiconductors, Communications, and Sensors

- **R&D in micro optics enabled by MEMS and nanotechnology at National Labs and Universities**
- **Device breakthroughs in the areas of subwavelength structures and photonic bandgap crystals**
- **Material science is very important for realizable devices**

Future Areas for Research in Micro and Nano-structures for Tagging/Tracking: Smarter Dust

- **Anisotropic 2D structures (e.g. polarization sensitivity)**
- **Customized, multiple wavelength/polarization for ID**
- **Substance-specific polymer films for detection**
- **Passive concepts for tags**