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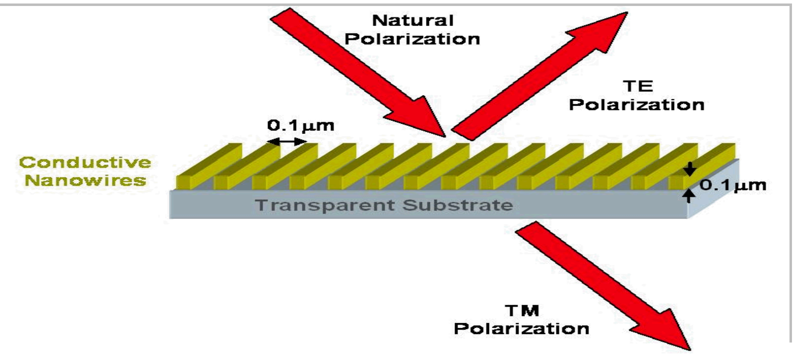

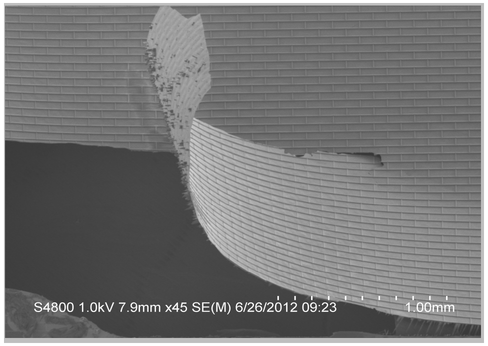

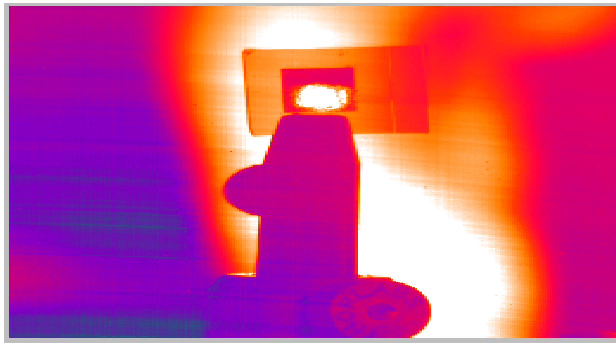

Flexible conductive polymer polarizer designed for a chemical tag

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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Outline

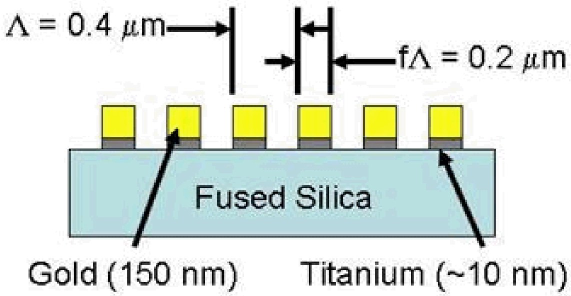
- Background/Objective
- Polymer composite based polarizer design
- Material investigation for HF sensing
- Engineering the flexible polarizer
- Optical test bench
- Results
- Conclusion

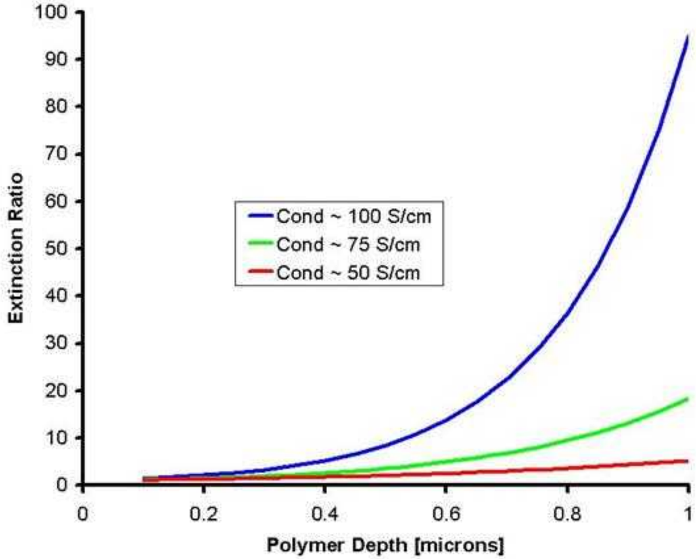
Background/objective

- Classic metal wire-grid polarizer
- Polymer’s
  - π–conjugated materials (polythiophene, polyaniline, polypyrrole)
- Nano-materials
  - Carbon nanotubes, metal nano-particles, and graphene.
- Percolation thresholds range from as low as 1% doping using some of these techniques.
- <u>Baseline:</u> Develop an optical polarizer which is sensitive to vapor phase hydrogen fluoride (HF) by changing the conductive mechanism of the material backbone.
- <u>Stretch Goal:</u> Flexible substrate.

Polymer composite based polarizer design

- Criteria:
  - Conductivity ~ 100 S/cm
  - Optically and electrically respond appreciably in the MWIR (3.39 μm HeNe source).
  - Modeling indicates a material thickness of ~ 2.0 – 2.5 μm.
  - Period of ~1.15 μm (line/space) on mask dimension.

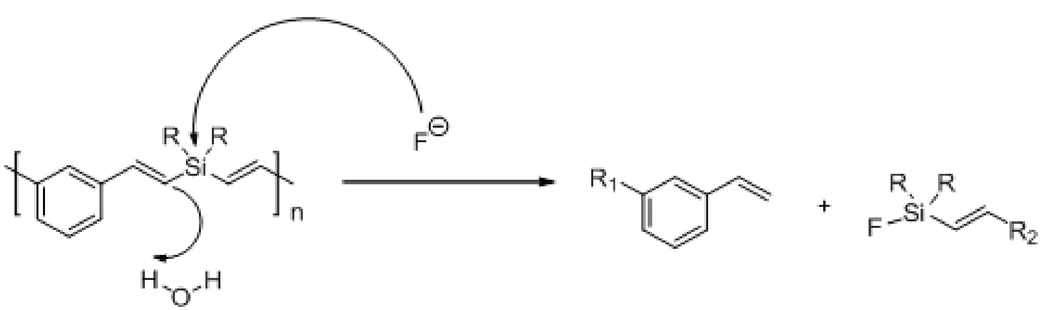




Effect of conductivity on extinction ratio

Material investigation for HF sensing

- Develop a polymer nano-composite material with three requirements
  - CONDUCTIVITY, level determined by optical device modeling
  - SENSITIVITY, change of conductivity upon exposure to analyte
  - SELECTIVITY, tailor response to specific analyte
- Analyte chosen – HF
- Two basic approaches
  - Chemo-selective polymers doped for conductivity, e.g. silanes and boranes
  - Nano-composites alter sensitivity (Group 1,2 elements, transition metals and semiconductors).



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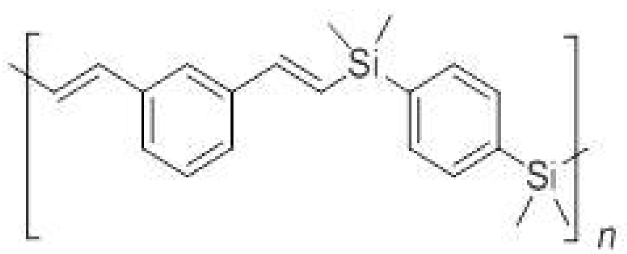
Material investigation for HF sensing

- Down select of various polymer and blends through empirical efforts.
  - Molecular weight
  - Water stability (co-habitation)
  - Dopants – nearly 60 wt.% for gold nano-particles, where as ~15-18 wt.% for MWCNT's for the same conductivity.
  - Sensitivity to HF vapor
  - Selectivity to HF vapor

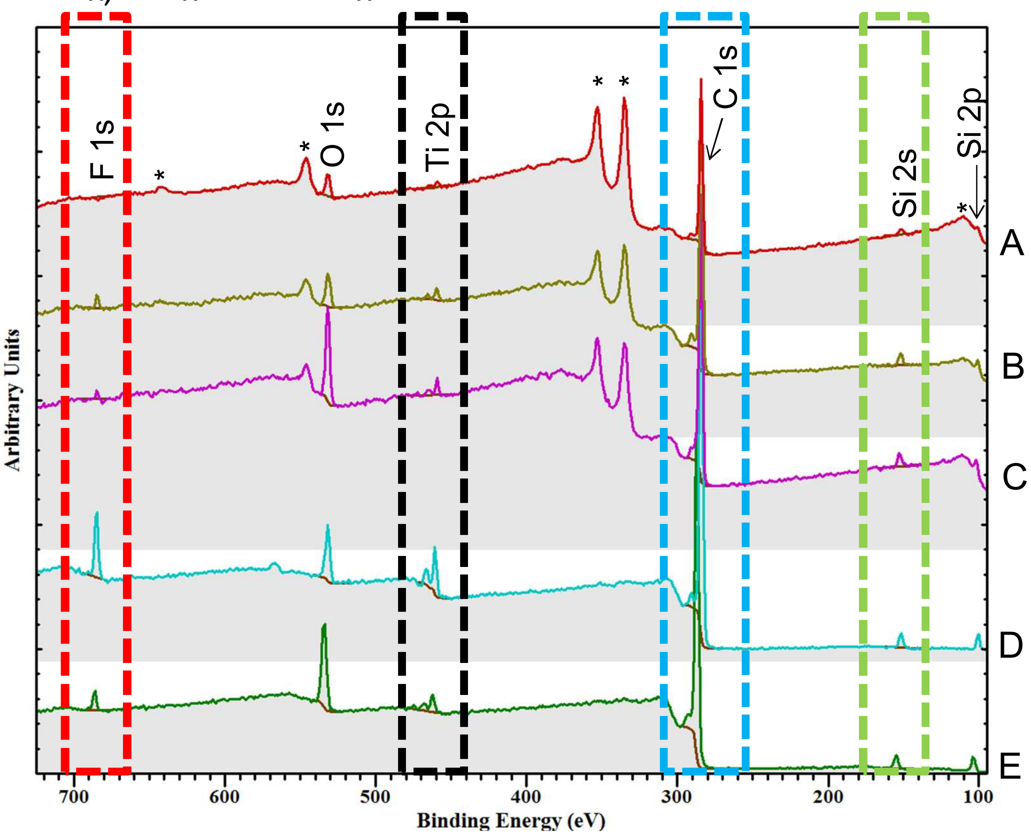
Material investigation for HF sensing

- Di-phenyl silane polymer
- ~18 wt.% multi-walled carbon nanotubes (1nm x 0.5-5 micron in length).
- ~30 wt.% 100nm Titania (TiO_2) used an F^- getter in the composite mix.
- XPS matrix of individual components versus total composite describe surface bond arrangements. CF_x, SiF_x, or TiF_x - irreversible binding event.

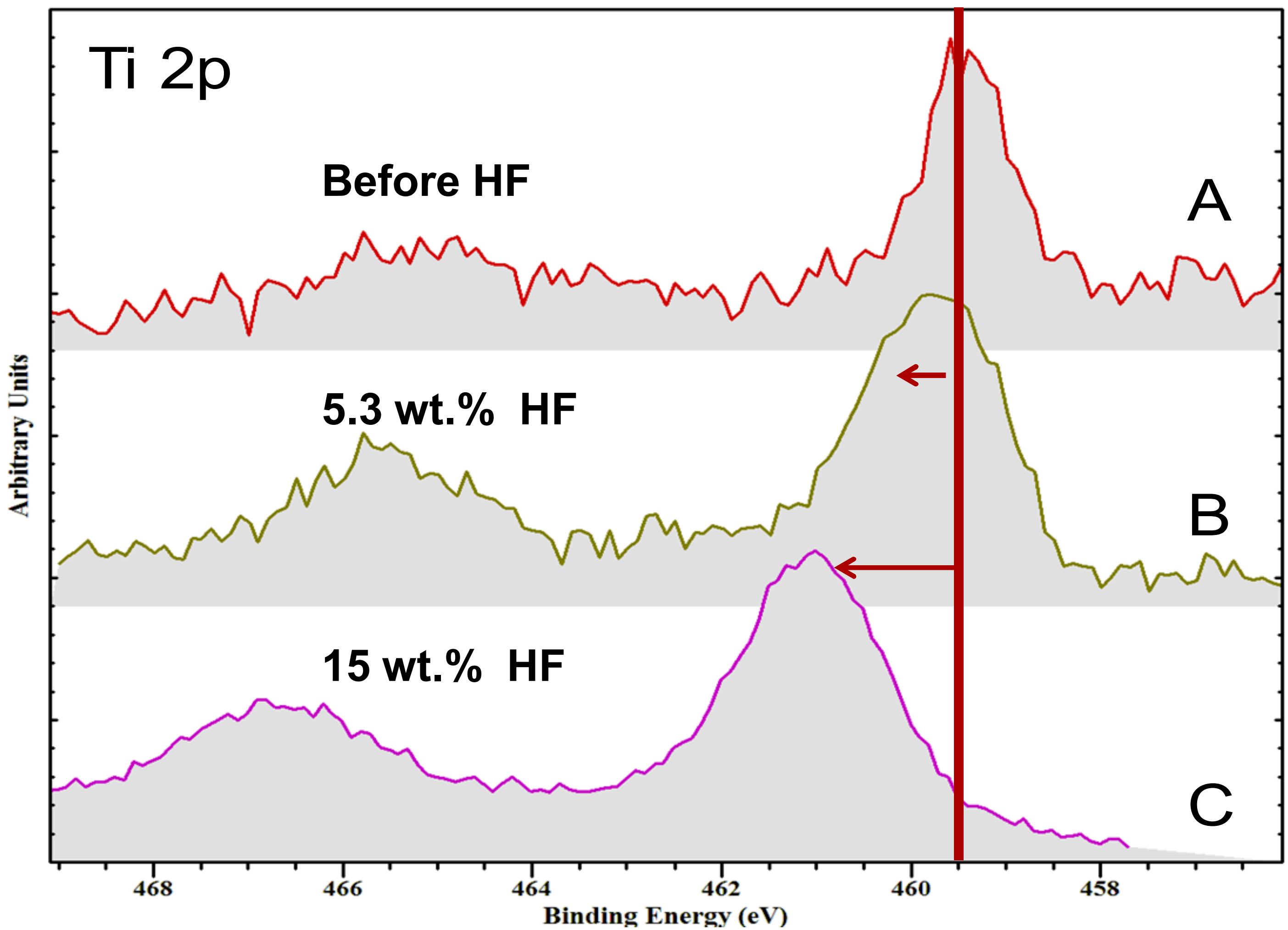


Energies required to make bond

<u>CF:</u> 544 kJ/mol
<u>SiF:</u> 552.7 +/- 2.1 kJ/mol
<u>TiF</u>: ~130-150 kJ/mol

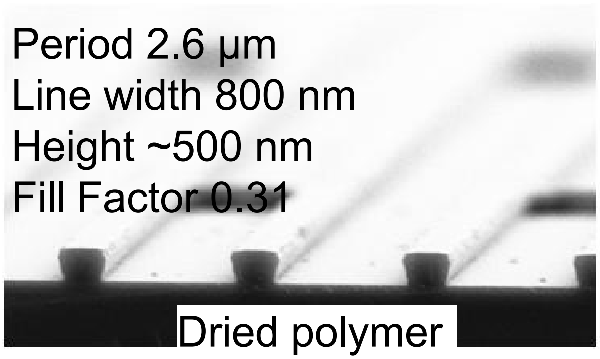


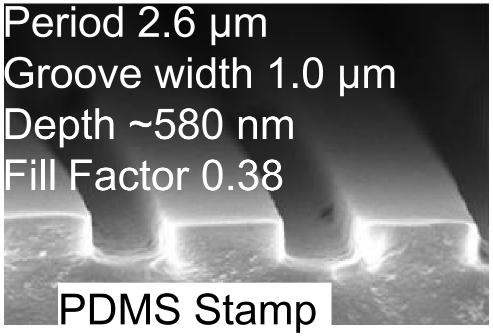
Material investigation for HF sensing

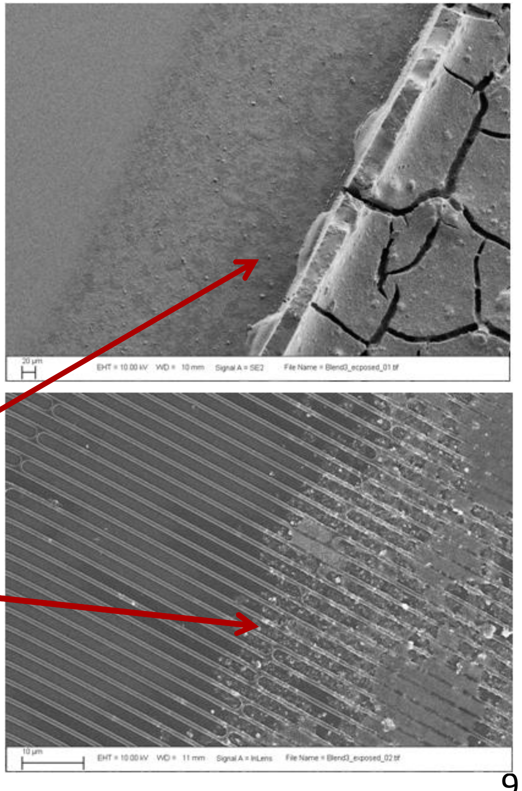


Engineering the flexible polarizer

- Why a template to support the material?
  - Previous work included efforts in:
    - MiMIC, stamping, and aqueous lift off techniques, and dry etch.



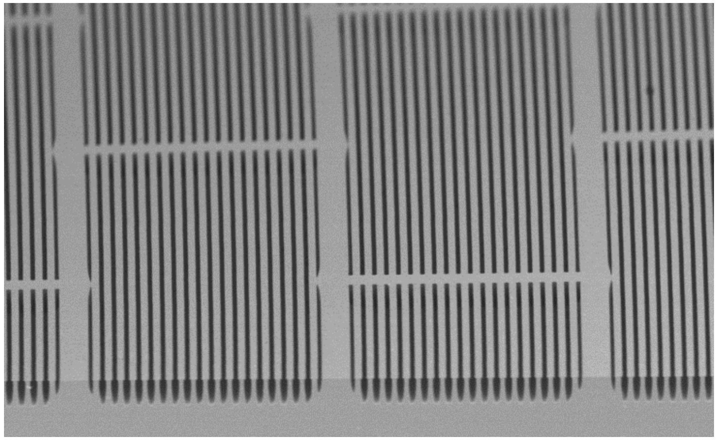


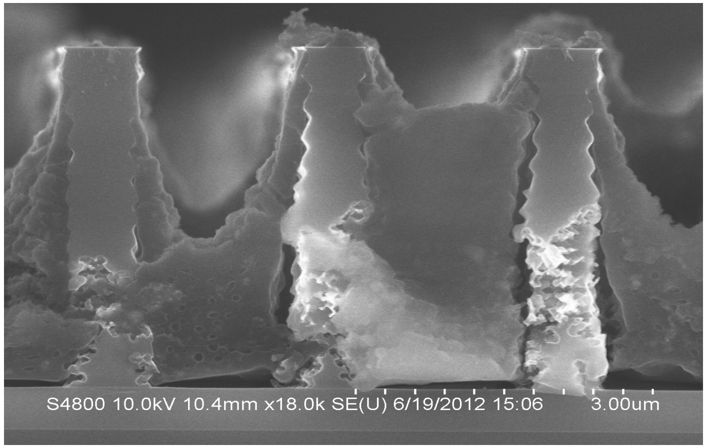


Capillary force and density gradient of materials would cause the solids to be filtered out.

Engineering the flexible polarizer

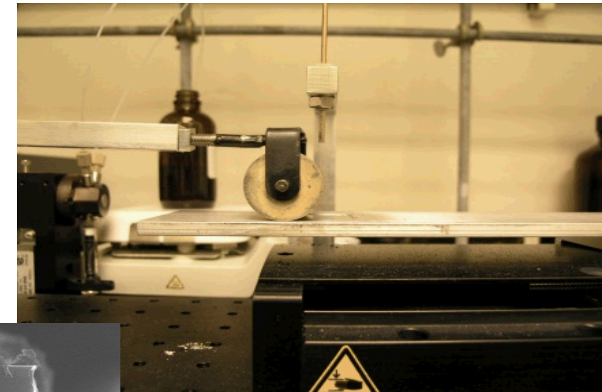
- A silicon template was developed to be filled using the material.
- Utilized a running bond design.
- Each bond line is 5 μm wide.
- Pattern is offset for support.
- Balanced stress for release etch.
- Etch depth ~ 5 μm.
- Period of ~1.2 μm (space)

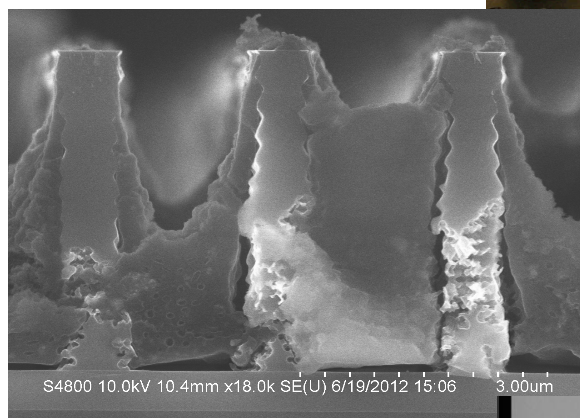


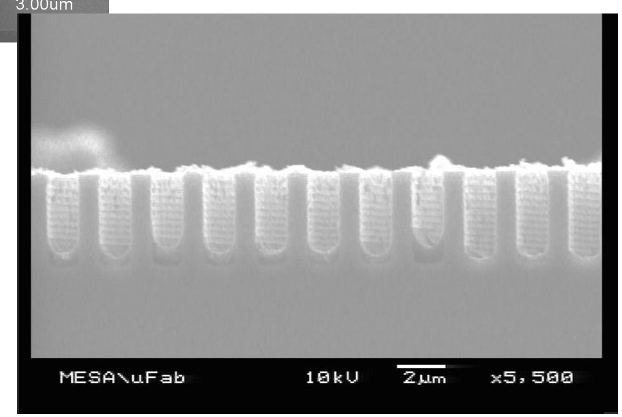


Engineering the flexible polarizer

- Large are coverage.
  - 1” X 1” size in the future.
  - Uniformity
  - Thickness
- Coating options:
  - Brush coat
  - Doctor blade
  - Roll coat
  - Surface preparation.

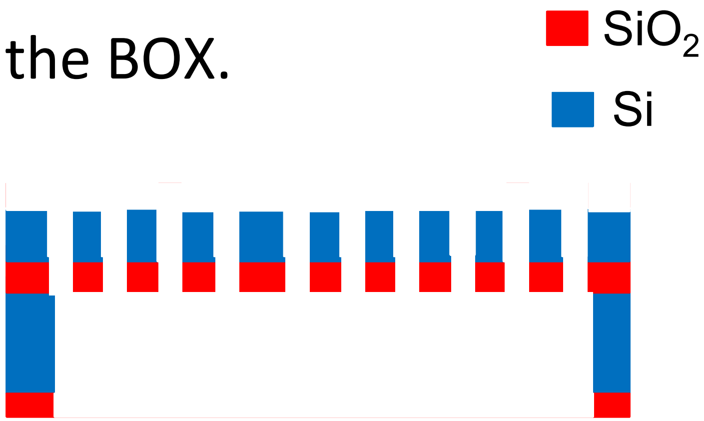




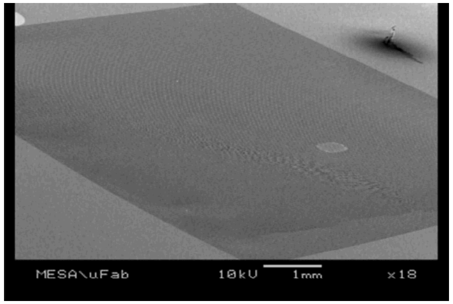


Engineering the flexible polarizer

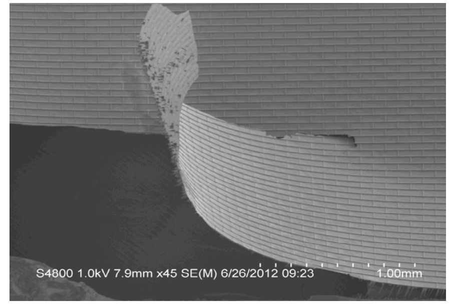
- Moved to an SOI wafer to leverage the oxide as a stopping layer.
- Performed a backside DRIE etch to the BOX.
- 5 μm device layer
- 300nm SiO_2 layer
- Lesson's learned.
  - Thin membrane handling.
    - Static, vacuum, cleaning, etc.
  - Finish front side DRIE last with SiO2 hard mask to maintain integrity.
  - Dry etch using RIE to remove hard mask.
  - Drop cast final material – do not roll coat.



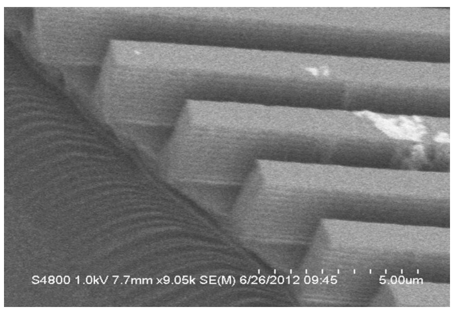
Engineering the flexible polarizer



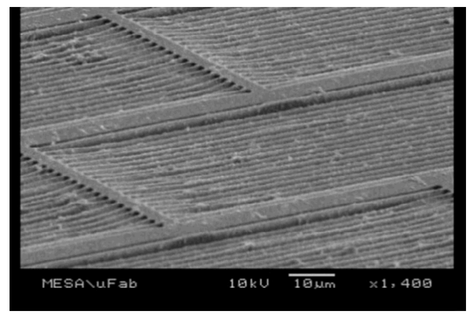
(a)



(b)

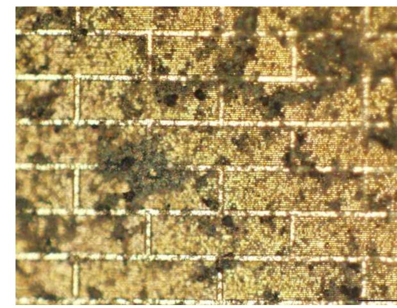


(c)



(d)

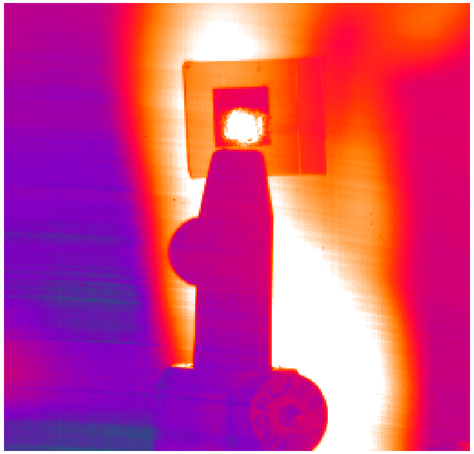
Optical Pic. Of coated device



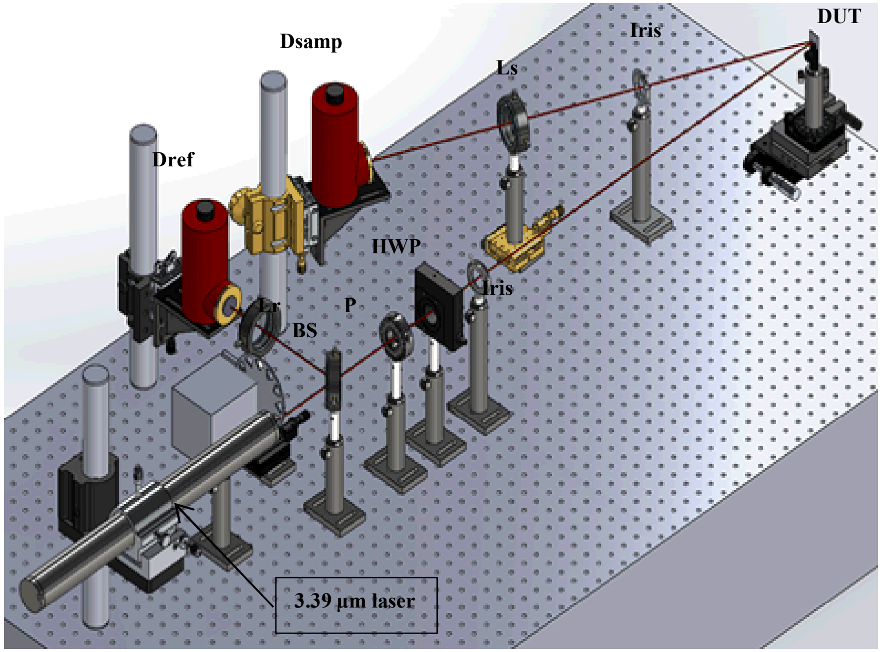
(a) shows an SEM of a 1 cm X 1cm release tag
(b) is an image of a torn membrane
(c) are the silicon fins remaining on the oxide membrane
(d) is a coated device after processing.

Optical Characterization

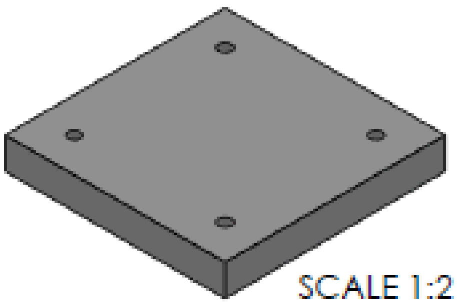
- 3.39 μm HeNe source in reflection mode.
- HgCdTe detector (LN2 cooled)
- Device remained in the window screen for handling.

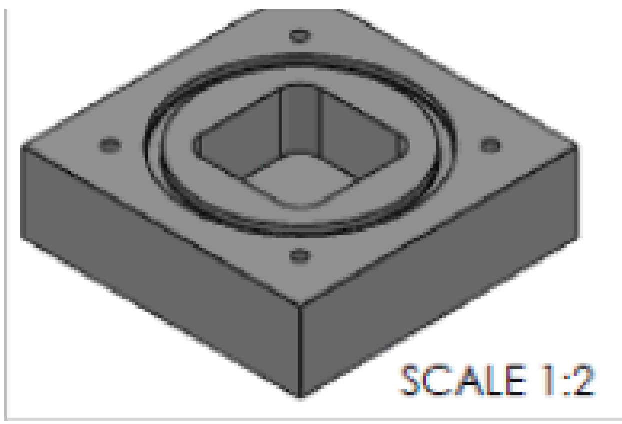


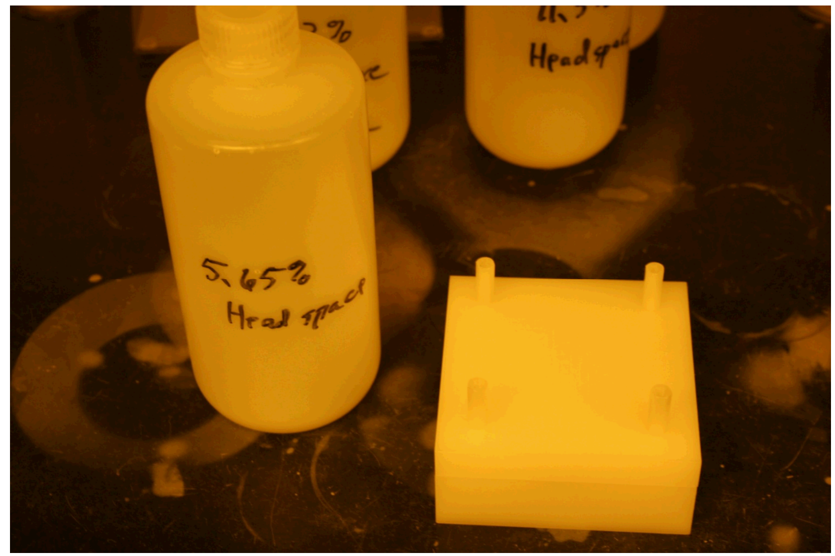
J. Hunker – IR camera



Vapor phase HF exposure



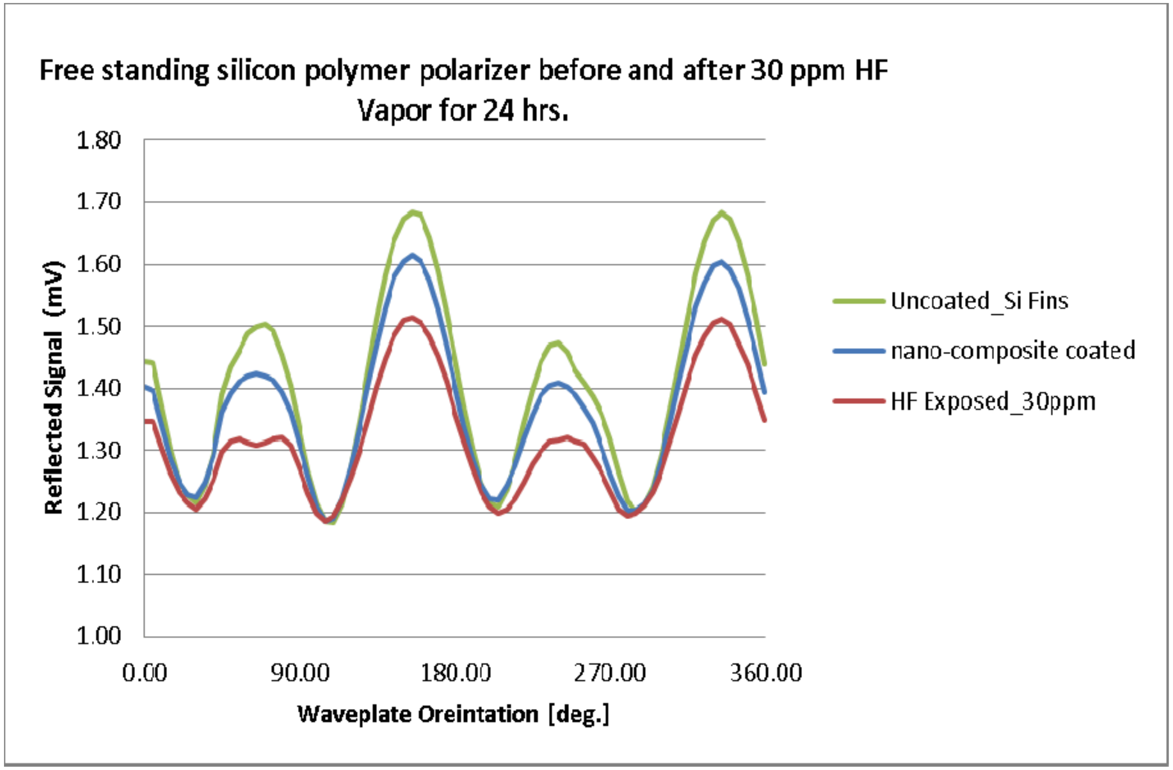




Viton O-ring for sealing. Small block teflon in bottom.

Results

- Produced 2-full wafers to date using this technique.
- Being the structure may not be perfectly planar the beam is walking some in reflection mode.



Conclusion

- Demonstrated a proof of concept device.
- DRIE etch development to minimize micro-masking.
- Showed a possible transfer technique to Kapton tape surfaces.
- Future work includes:
  - Moving to 1” X 1” area.
  - Investigate transfer options for optimal performance.
  - Optical performance as a function of HF response.
- Thanks for your time.

References/Acknowledgements

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