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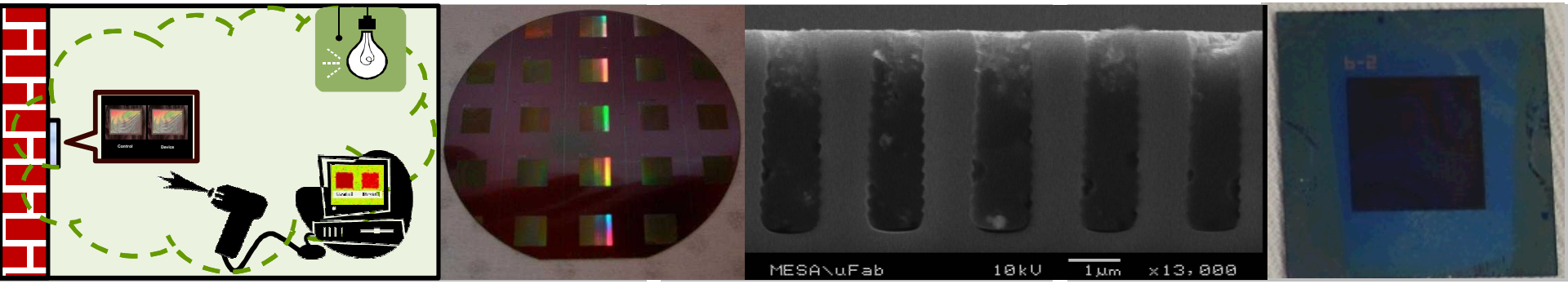

Polarization for Remote Chemical Detection

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Polarization Techniques & Technology – 24 Mar 2014

U.S. DEPARTMENT OF ENERGY | NNSA

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- *Collaborators:* Dennis Goldstein, Polaris Sensor Technologies

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Outline

- Motivation
- Approach
  - Dosimeter Tag Development
  - Dosimeter Tag Testing and Characterization
- Results
  - Exposure Results
  - Tag Sensitivity, Selectivity, and Optimization
- Conclusions and Future Work

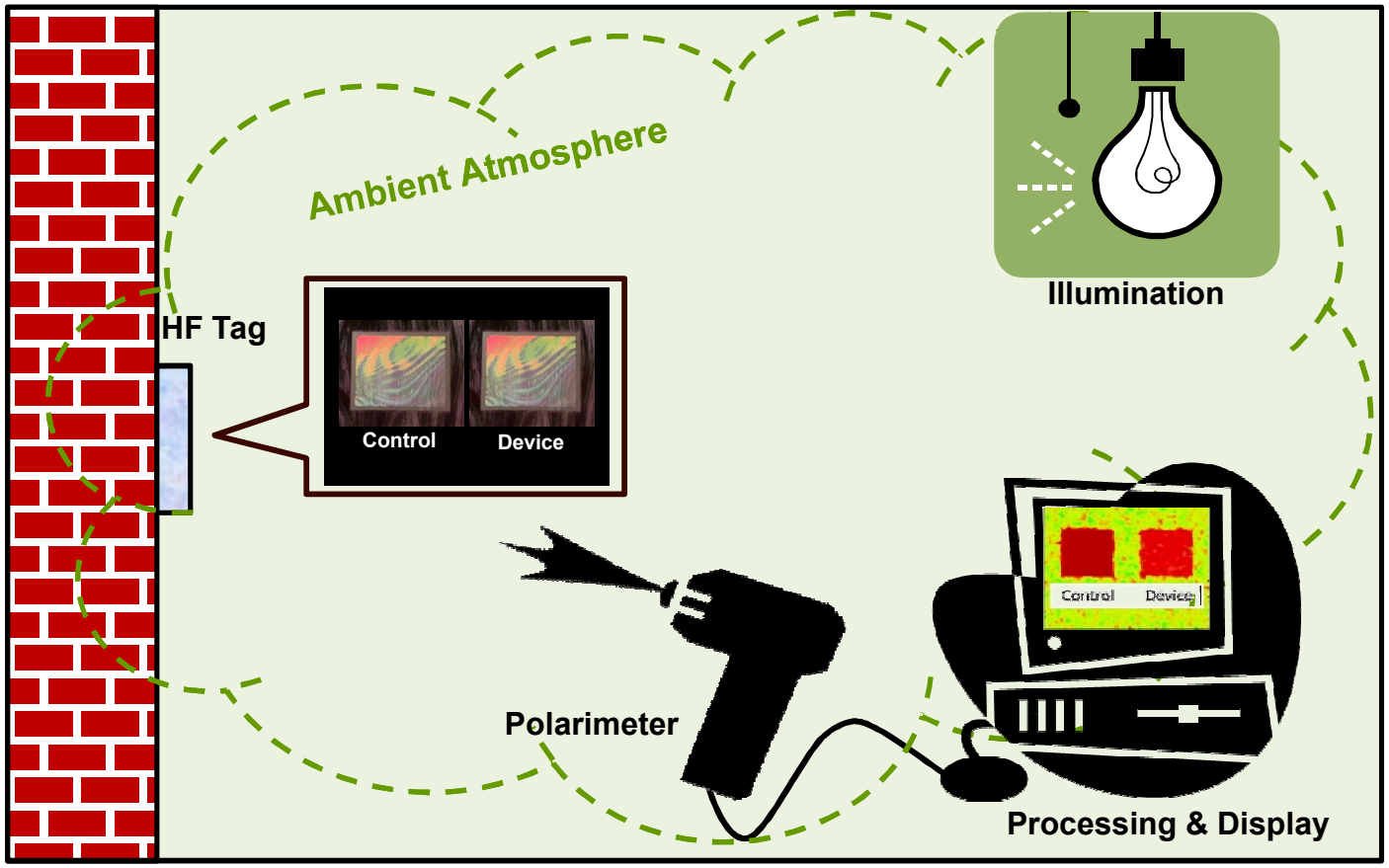
Motivation

- There is a need for persistent, passive, and non-invasive monitoring systems for detecting hazardous chemical vapors.

- Hydrogen fluoride (HF) gas monitoring is the particular focus of this work.
  - HF is the principal industrial source of fluorine.

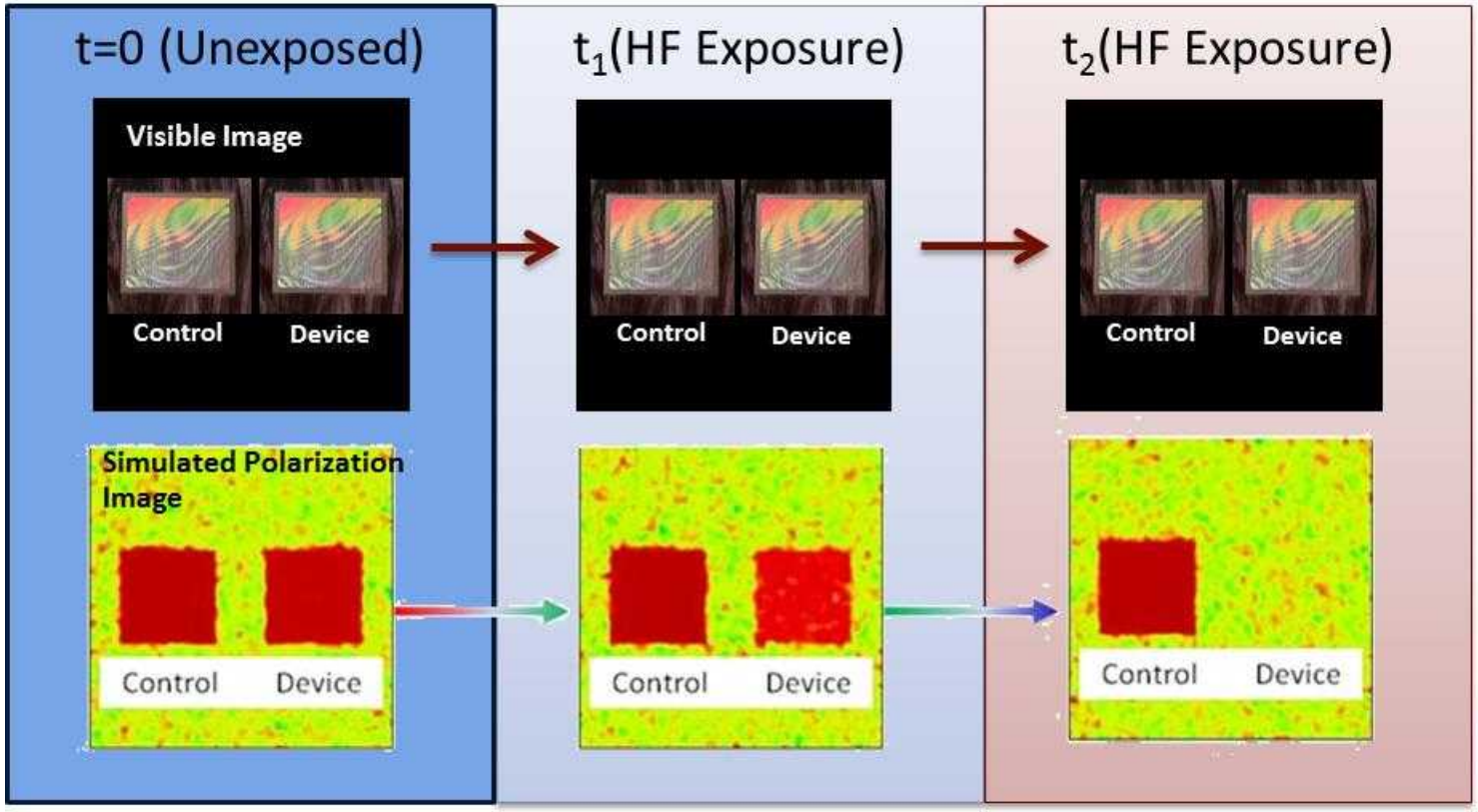
Approach

- Develop HF-sensitive dosimeter tags that can be used to monitor a facility for a period of weeks to months.



Approach

- Monitoring mechanism: optical polarization properties of the tags change upon exposure to HF (~1-3 ppm for 30+ days).



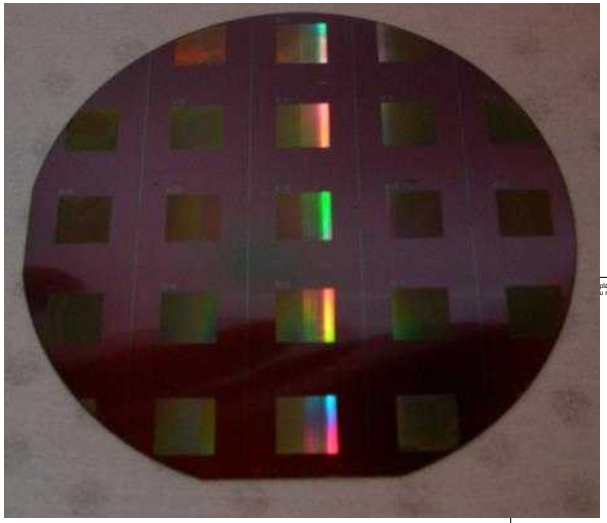
Low Impact & Passive Measurement

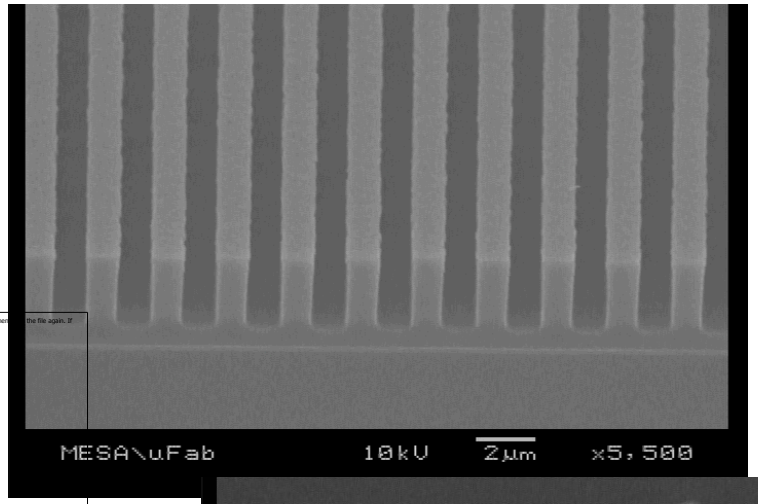
Persistent Monitoring Capability

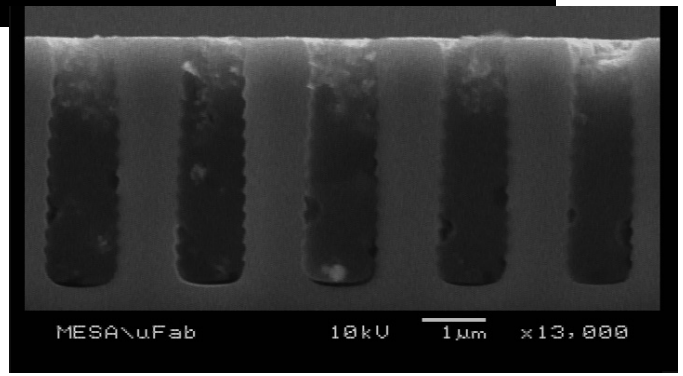
Simplified Data Products

Dosimeter Tag Production

- Standard lithographic approach used for dosimeter tag production.
  - 2.3 µm periodic structure etched in silicon forms tag substrate.
  - Substrate is coated with HF-sensitive material.

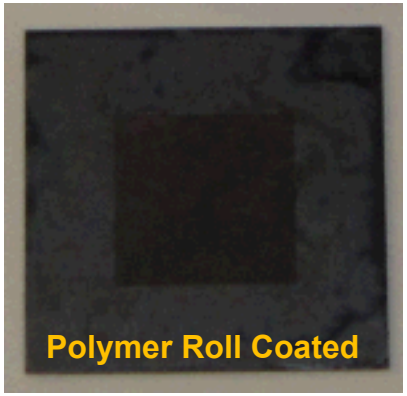


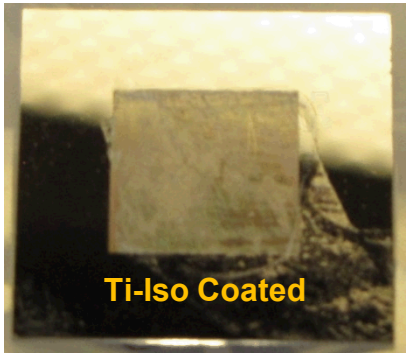


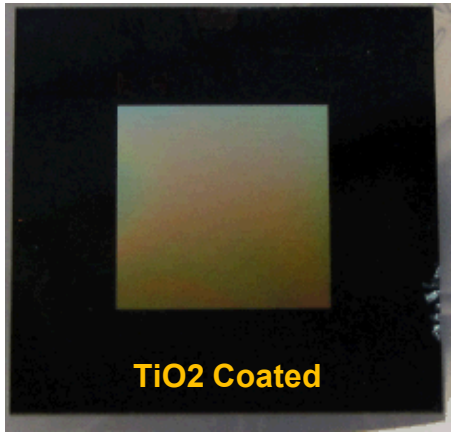


Tag Development

- Many tag fabrication approaches investigated:
  - Polymer roll coated
  - Polymer polished
  - Ti-Isoproproxide coated
  - Ti evaporative deposition
  - TiO_2 evaporative deposition
- Tested all fabrication approaches developed at 30 ppm for 24 hours and ~35% RH.
  - Tags coated with TiO_2 produced largest polarization change.

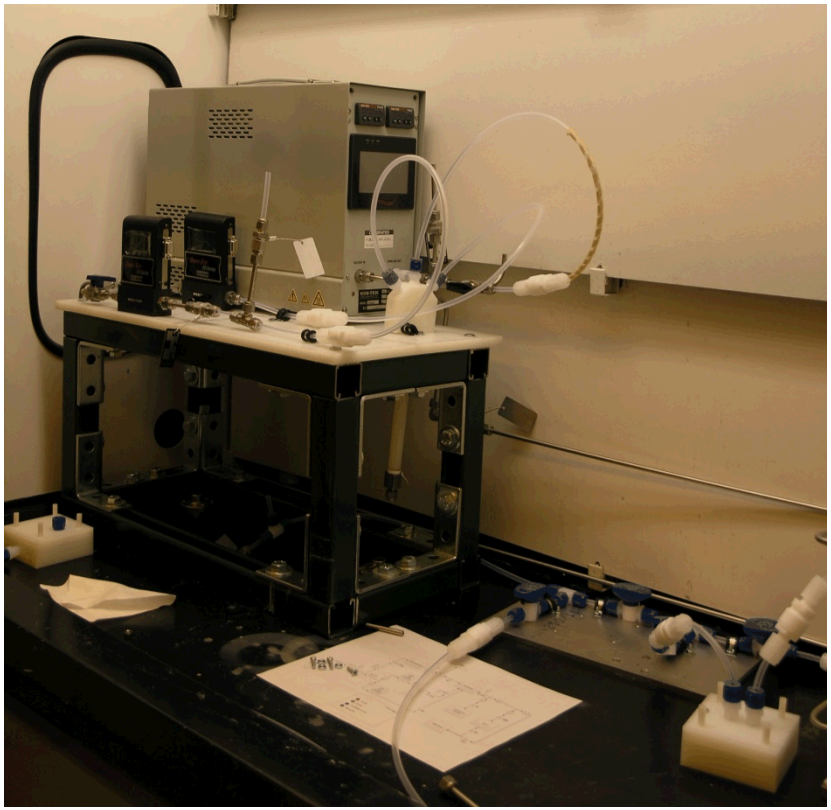


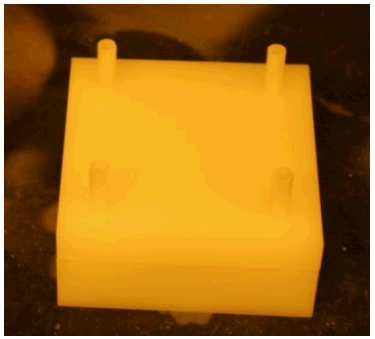




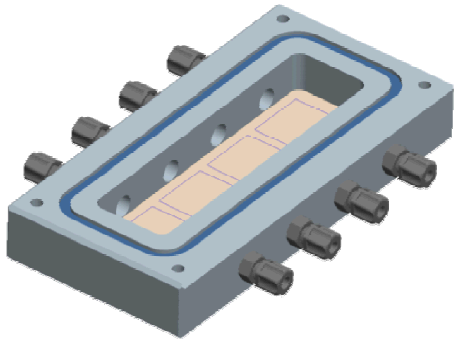
Simulating Tag Exposure

- Developed low concentration HF exposure system to mimic facility exposure conditions.
  - HF vapor exposure, 1-30 ppm.
  - Lab room temperature (~22 C).
  - Variable humidity (~30%)
- 1-8 samples per exposure





Single Channel DUT

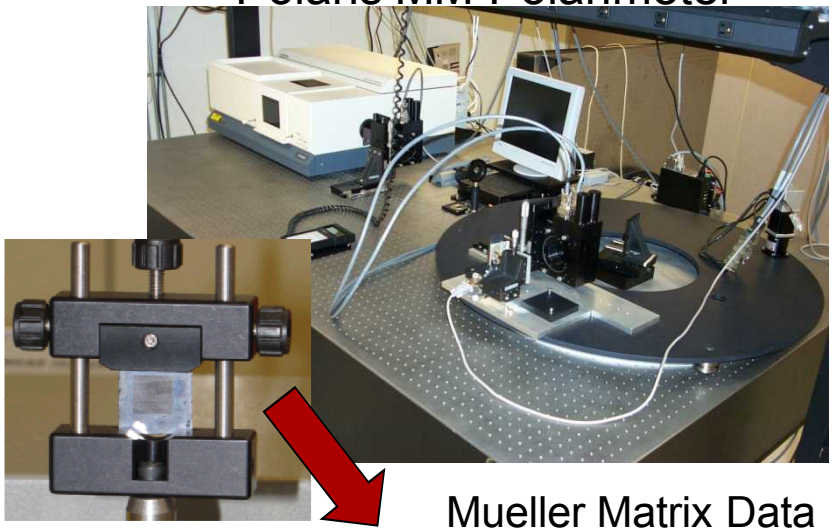


Multi- Channel DUT

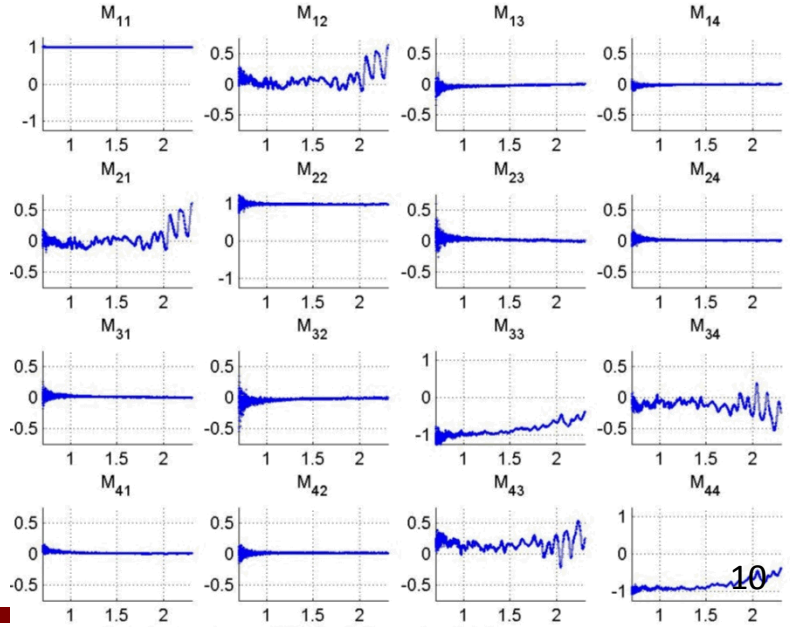
Mueller Matrix Tag Characterization

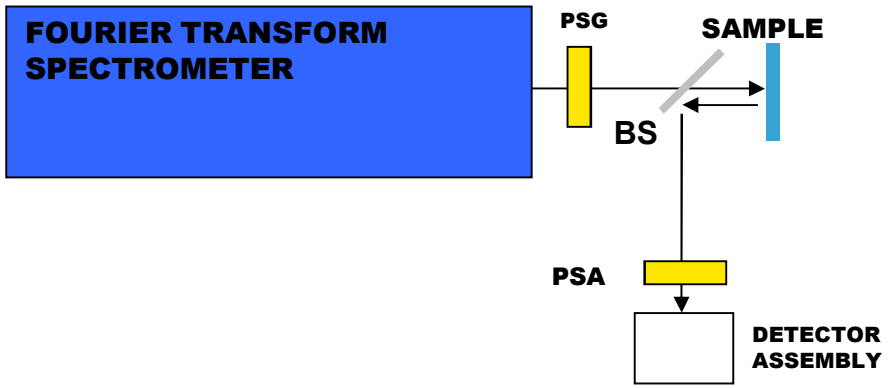
- Spectropolarimetric Mueller matrix (MM) measurements of the tags accomplished through a measurement partnership with Polaris Sensor Technologies.
  - Full polarimetric characterization over λ=0.7-2.3 µm and λ=4.0-12.0 µm.
  - NIR, SWIR and LWIR of primary interest due to the availability of COTS uncooled detectors.

Polaris MM Polarimeter



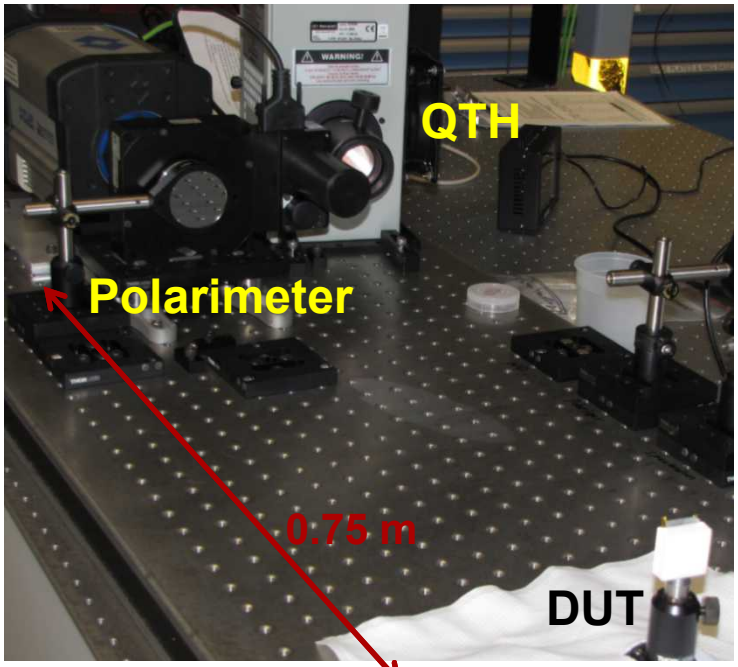
Mueller Matrix Data

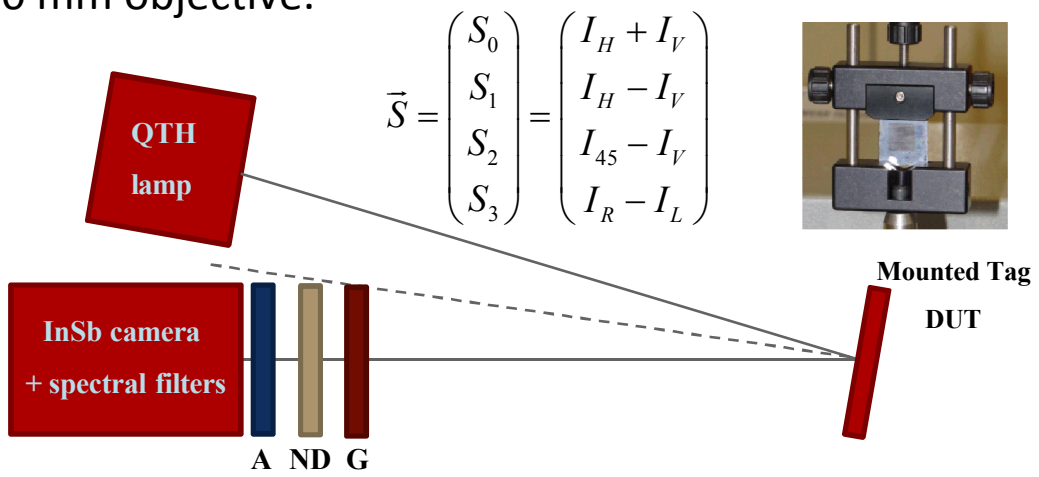




SWIR Linear Stokes Polarimeter

- Rotating polarizer polarimeter characterizes the degree of linear polarization (DOLP) reflected off the tags in the SWIR.
  - QTH 50 W collimated light source.
  - 100 nm spectral filters centered at λ = 2.14, 2.20, and 2.36 µm.
  - FLIR InSb imaging camera with 50 mm objective.





$$\vec{S} = \begin{pmatrix} S_0 \\ S_1 \\ S_2 \\ S_3 \end{pmatrix} = \begin{pmatrix} I_H + I_V \\ I_H - I_V \\ I_{45} - I_V \\ I_R - I_L \end{pmatrix}$$

A: Analyzer ND: OD2 filter

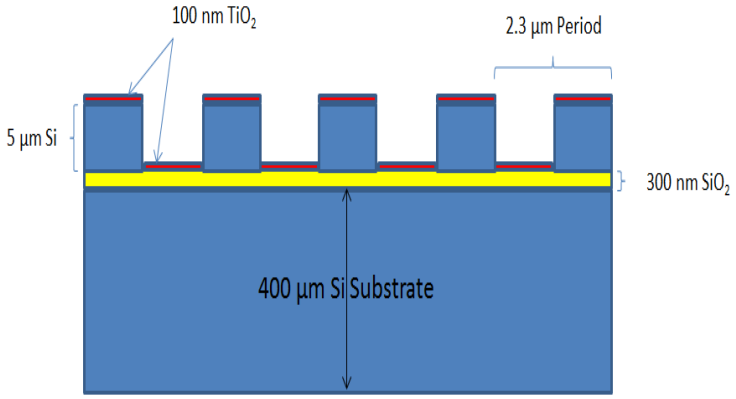
G: Generator (removed for tag and offset measurements)

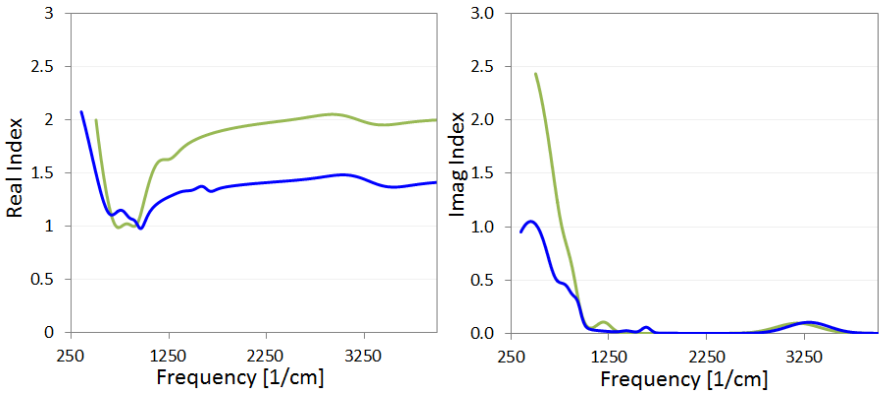
$$DOLP = \frac{\sqrt{S_1^2 + S_2^2}}{S_0}$$

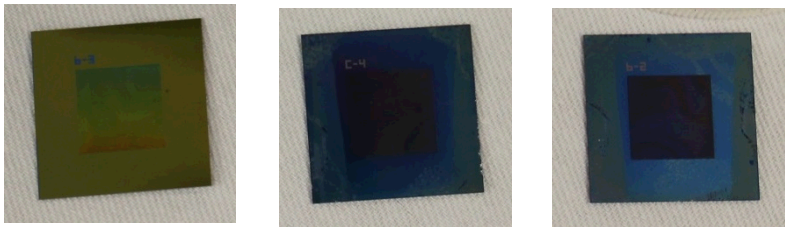
$$\Delta DOLP = DOLP_{\text{exposed}} - DOLP_{\text{unexposed}}$$

Optimizing Tag Production

- Material behavior before and after HF exposure is being characterized using several techniques.
  - Ellipsometry
  - X-Ray Diffraction, Raman Spectroscopy
  - Interferometry, AFM
- Amorphous TiO2 is most sensitive to HF
  - Thickness variation being modeled and tested.
  - Lower bound of sensitivity currently >1 ppm for 30 days.
- Selectivity experiments being conducted.
  - Tests with other acid vapors (HCl and HNO_3) indicate little to no change in polarization state.



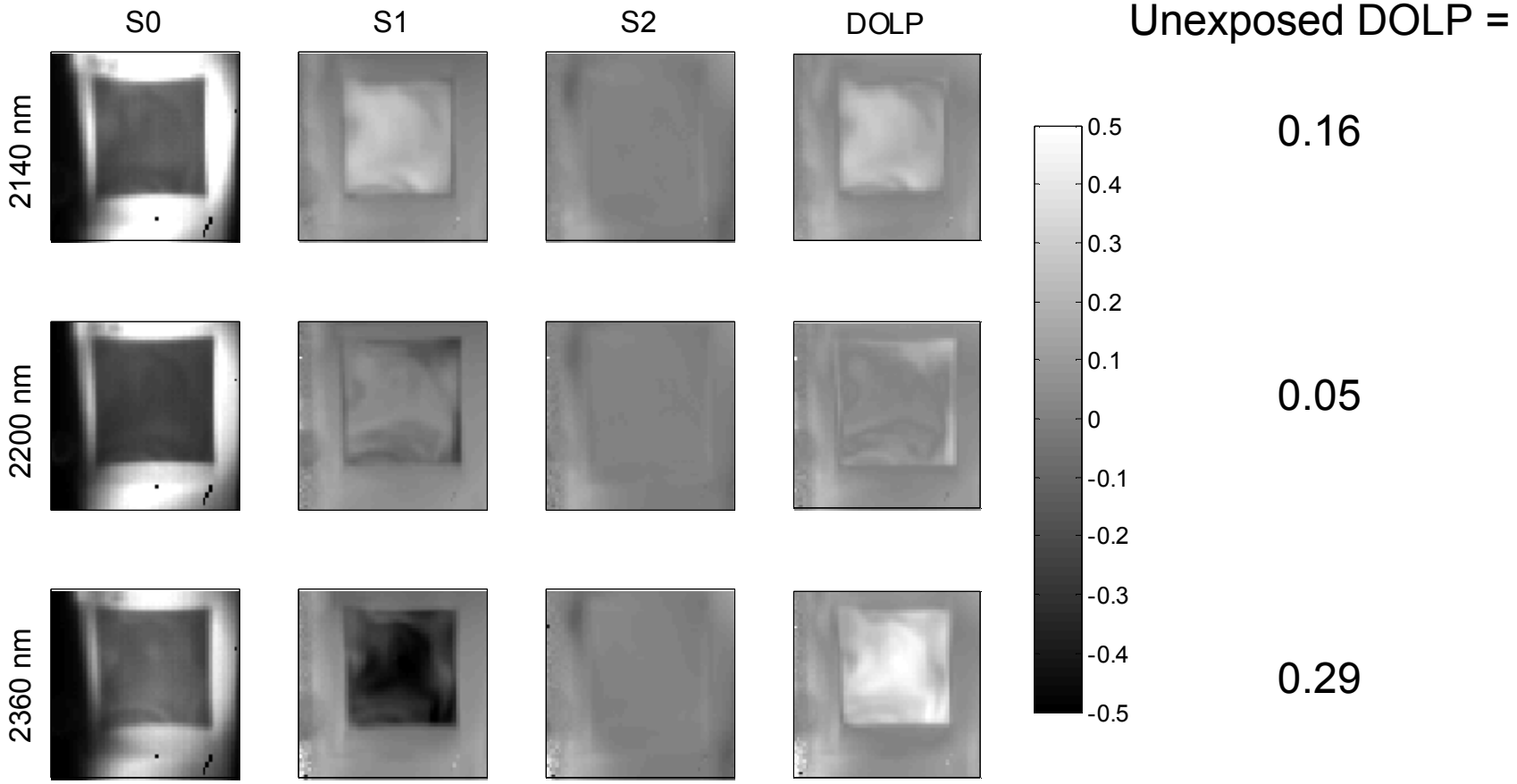




unexposed exposed exposed

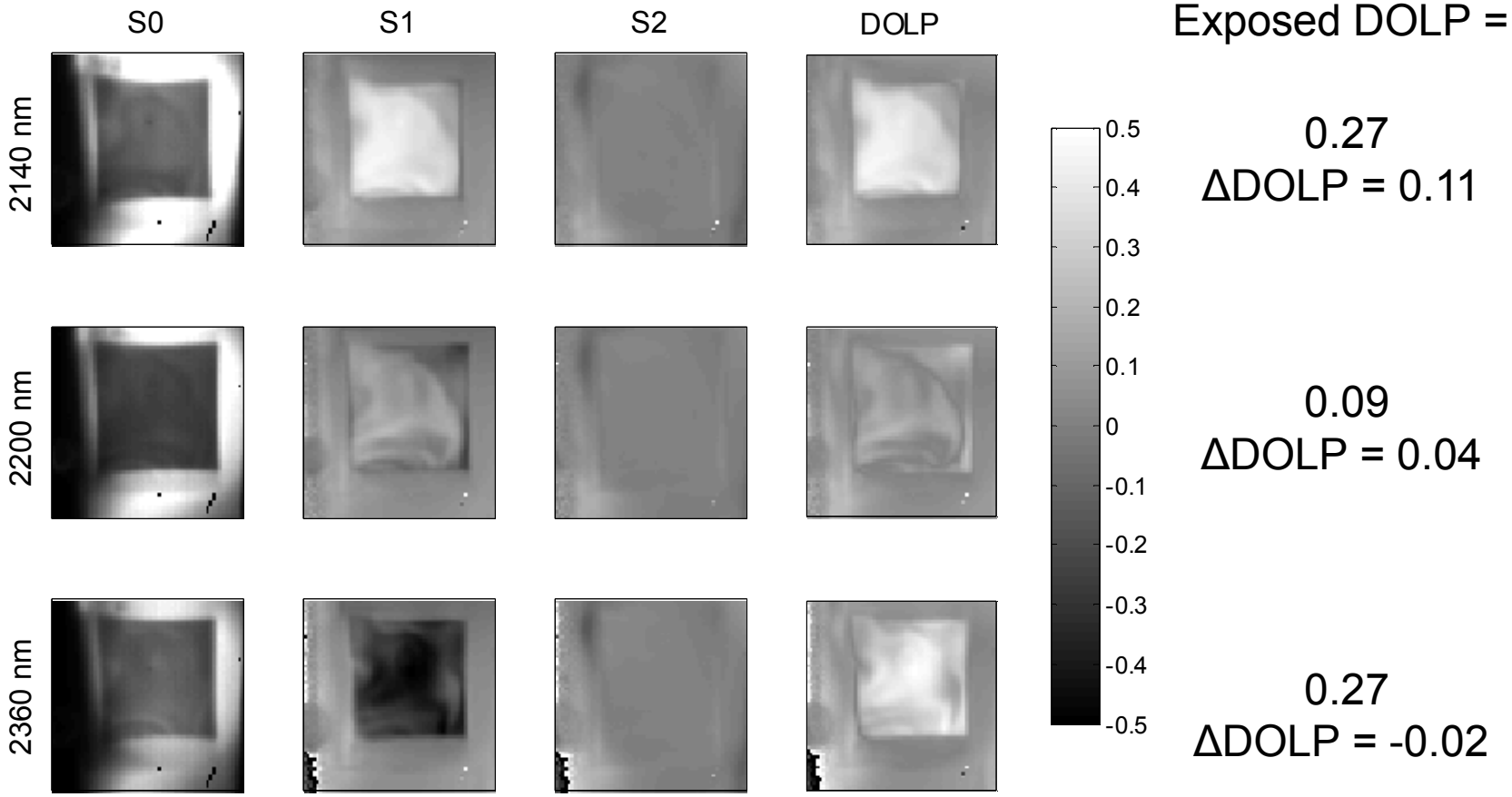
Tag Sensitivity Results

- 30 ppm exposure for 24 hours, ~25% RH
- Unexposed



Tag Sensitivity Results

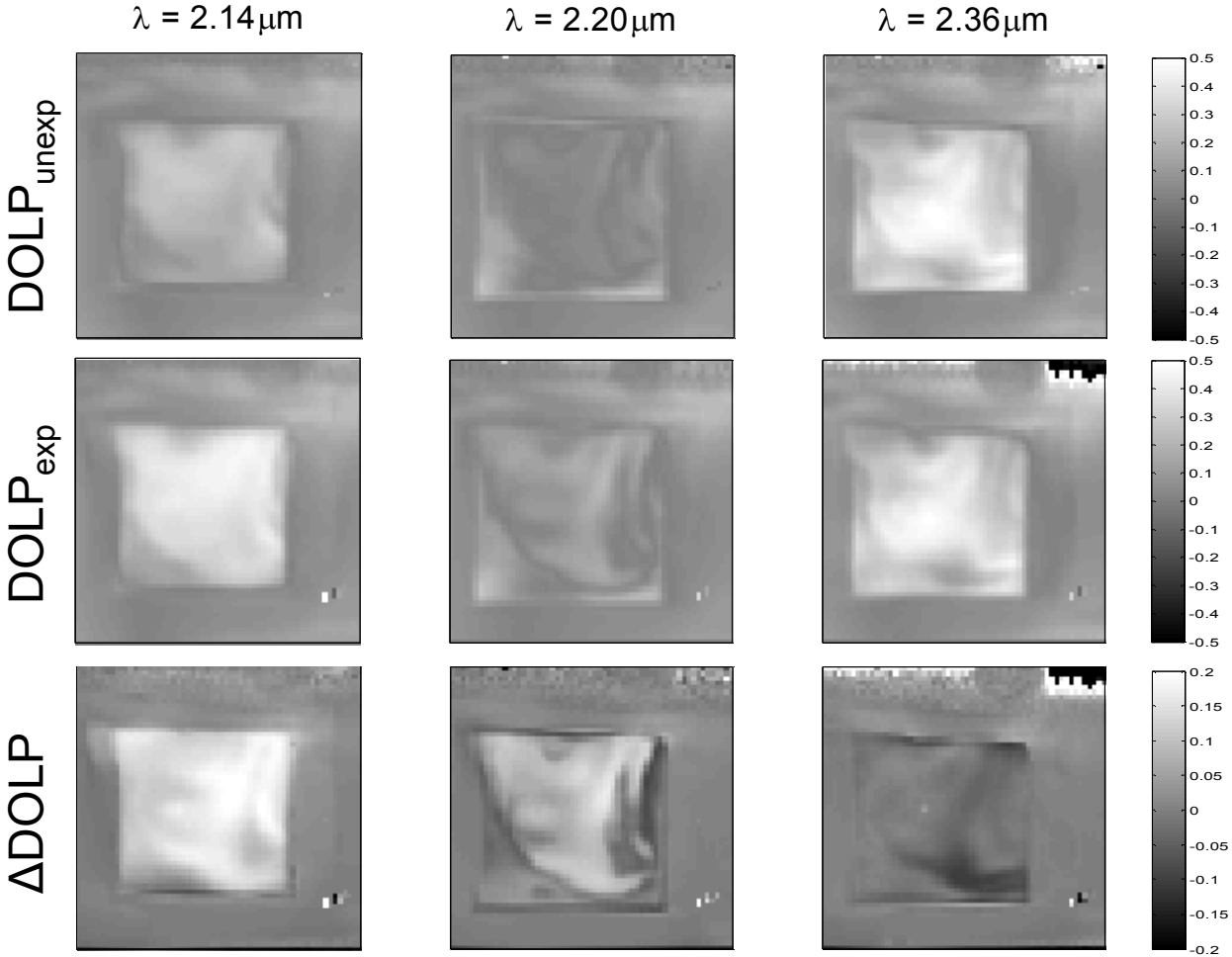
- 30 ppm exposure for 24 hours, ~25% RH
- Exposed



- Latest results: 4/5 tags tested had a ΔDOLP >= 0.04 at λ = 2.14 µm

Tag Sensitivity Results

- Change in DOLP map, exposed vs. unexposed



Exposed DOLP =

0.27
ΔDOLP = 0.11

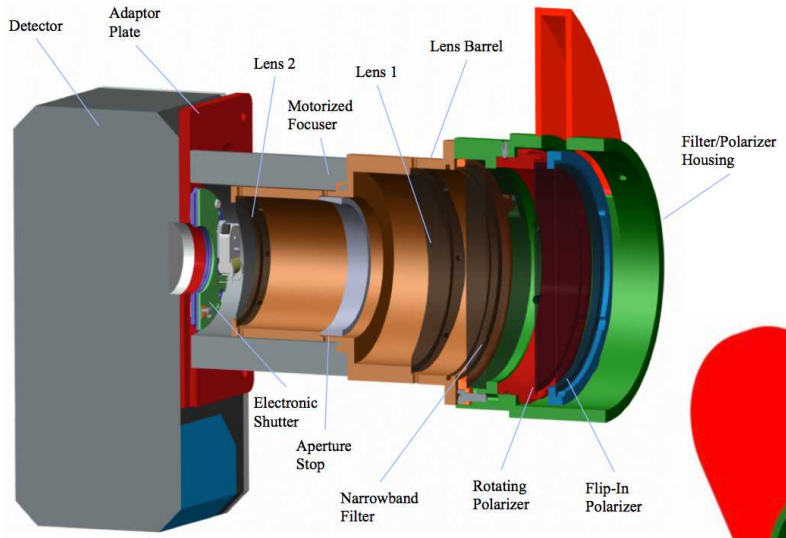
0.09
ΔDOLP = 0.04

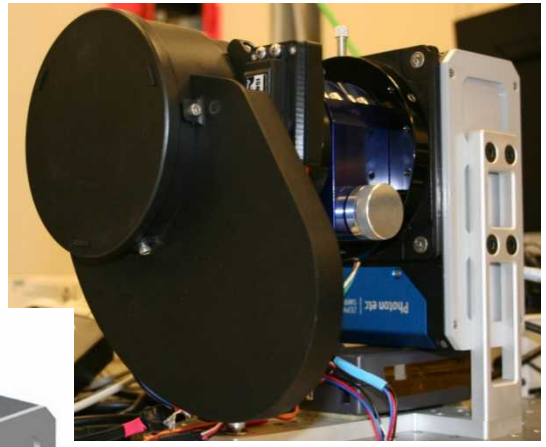
0.27
ΔDOLP = -0.02

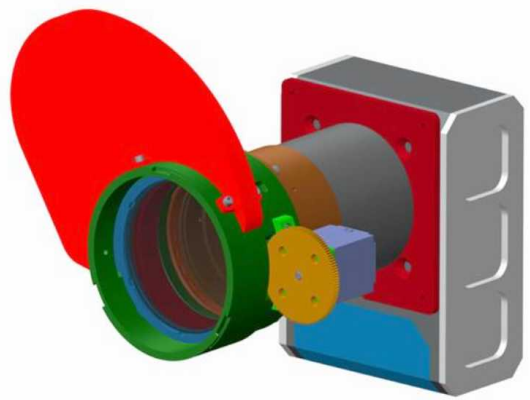
$$\Delta DOLP = DOLP_{\text{exposed}} - DOLP_{\text{unexposed}}$$

Polarimeter Prototype

- SWIR (λ=2.1 – 2.4 µm) imaging polarimeter can be used to interrogate the dosimeter tags.
  - Straightforward data products and processing, field portable, simple automated operation.
  - Polarimeter is assembled and is currently being tested at SNL.







Conclusions and Future Work

- This effort demonstrates a polarimetric approach to monitoring for gases.
  - Passive and persistent monitoring using an unconventional approach.
  - Tags are simple and small and could be installed nearly anywhere.
  - HF monitoring is the focus of this project, but could modify general approach for detecting other chemicals of interest.
- Future work will be focused on further optimization of the dosimeter tags and testing of the complete monitoring system.

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END

Thank you!