## READ-OUT OF COLOR CENTERS WITH LIGHT-SHEET FLUORESCENCE MICROSCOPY

MINERAL DETECTION OF NEUTRINOS AND DARK MATTER WORKSHOP VIRGINIA TECH EXECUTIVE BRIEFING CENTER - JAN 11 2023

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#### THE PALEOCCENE CONCEPT READ-OUT OF COLOR CENTERS IN PASSIVE DETECTORS USING LIGHT-SHEET FLUORESCENCE MICROSCOPY



## NUMBER OF CENTERS CAN BE USED TO ESTIMATE PINDUCED CENTER PINDUCED CENTER PRIOR TO EXPOSURE AFTER EXPOSURE

TO REACTOR VS

**COLOR CENTERS** ABSORB AND RE-EMIT LIGHT IN <u>OPTICAL</u> WAVELENGTHS, ENABLING A <u>FAST READ-OUT</u>.

#### TESTING THE PALEOCCENE CONCEPT

### READOUT OF COLOR CENTERS WITH THE MESOSPIM



**SPIM:** Selective Plane Imaging Microscopy Scan speed at ~  $4 \mu m$  XYZ resolution: <10min/cm<sup>3</sup> STATE-OF-THE-ART LIGHT-SHEET MICROSCOPE THAT IMAGES CENTIMETER -SIZED SAMPLES WITHIN MINUTES. NAT. METH. (2019)

Benchtop meso-scale SPIM



#### THE MESOSPIM

### A "LARGE SCALE" LIGHT-SHEET FLUORESCENCE MICROSCOPY

FROM WHOLE-BODY RAT SCANS TO COLOR CENTER IMAGING IN CUBIC-CM SIZED CRYSTALS: New (2023) benchtop version was optimized for color center imaging\*, with pixel sizes down to 0.21  $\mu$ m compared to ~1  $\mu$ m in previous version.

This new version has: sample holder for crystal imaging, <u>larger sCMOS</u> <u>camera</u> (5056x2960 pixels, 4.25  $\mu$ m pixel size), <u>magnification up to 20x</u> (1.5  $\mu$ m x-y resolution), <u>smaller footprint and cost</u>. File sizes for a full crystal scan and ~4  $\mu$ m isotropic resolution: ~30 Gb. b, Mouse P14, vDISCO Obj: PlatinumTL™ 0.9x Staining: PI



i, CaF<sub>2</sub> crystal, γ-irradiated Obj: Mitutoyo 20x/0.28 Color centers induced by γ-rays



### IMAGING TRANSPARENT CRYSTALS WITH THE MESOSPIM SAMPLES: BLANK, IRRADIATED WITH γ-RAYS, OR NEUTRONS

1 cm<sup>3</sup> CaF<sub>2</sub> transparent crystals (all sides polished) were irradiated with  $\gamma$ -rays or neutrons and imaged in comparison to a **blank**.



 $\gamma$ -<u>RAY DOSES</u>: 100 kRad, 5 MRad (~10<sup>13</sup>-10<sup>14</sup> ph/cm<sup>2</sup> from a ~1 MeV source <sup>60</sup>Co source)

<u>NEUTRON DOSES</u>: neutron flux of ~10<sup>8</sup> n/cm<sup>2</sup>

CRYSTAL VENDORS: Crystran, or United Crystals

#### IMAGING TRANSPARENT CRYSTALS WITH THE MESOSPIM METHODS & ANALYSIS

**CaF<sub>2</sub> transparent crystals** (all sides polished, 1 cm<sup>3</sup>) were **irradiated** with  $\gamma$ -rays or neutrons and imaged in comparison to a **blank** crystal.



- 1. ESTIMATING BULK FLUORESCENCE: average fluorescence intensity within fiducialized volume.
- 2. FLUORESCENT TRACK-LIKE & POINT-LIKE STRUCTURES: Find pixel clusters & match repeated scans
- 3. ESTIMATING SINGLE COLOR CENTER FLUORESCENCE: matching of high-intensity pixels at 1-pixel level and comparison to random matches.

# ESTIMATING THE FLUORESCENCE SIGNAL FROM COLOR CENTERS



CAMERA DARK COUNTS NOISE IS ESTIMATED IN DARK

SURFACE OILS, DUST, & POLISH ARE FLUORESCENT BACKGROUND

# ESTIMATING THE FLUORESCENCE SIGNAL FROM IRRADIATED CRYSTALS



- CAMERA DARK COUNTS NOISE IS ESTIMATED IN DARK
- BLANK VS IRRADIATED CRYSTALS ARE COMPARED



BLANK CaF<sub>2</sub> CRYSTALS YIELDED NO BULK SIGNAL

### ESTIMATING COLOR CENTER FLUORESCENCE SIGNAL





BLANK CaF<sub>2</sub> CRYSTALS YIELDED NO BULK SIGNAL

(\*)MEASUREMENT WITH LONG-PASS FILTER

### COLOR CENTER FLUORESCENCE SIGNAL BLANK VS IRRADIATED





CRYSTAL IS FLUORESCENT AFTER IRRADIATION\*

### **MEASUREMENTS OF ABSORPTION**

## CRYSTAL BECOMES LESS TRANSPARENT AFTER IRRADIATION & RE-EMITS LIGHT IN BLUE.



Absorption and emission spectra seemed to match literature values from fluorescing crystals containing rare-earth elements.



### **& EMISSION SPECTRA**

Fluorescence spectra from the irradiated crystal measured in response to  $400 \pm 10$  nm light



### BULK FLUORESCENCE FROM γ-IRRADIATED CRYSTALS COLOR CENTERS INDUCED BY IONIZATION

THE IONIZATION OF TRACE AMOUNTS OF RARE-EARTH ELEMENTS (REE)\* BY  $\gamma$ -RAYS IS LIKELY THE CAUSE OF THE STRONG FLUORESCENCE OBSERVED IN THE  $\gamma$ -RAY IRRADIATED SAMPLES.

Element	Crystals	
	Unit (ppb)	Unit (ppb)
	VT14	100K
La	80	1400
Ce	6875	3020
Pr	45	330
Nd	530	1430
Sm	205	315
Eu	85	85

Table: concentration of REEs measured from two  $CaF_2$  crystals that displayed high intensity of fluorescence after irradiation with neutrons (VT14) and  $\gamma$ -rays. (100kRad).

\*Normally found in their stable trivalent state (such as Sm3+, Eu3+ occupying the place of Ca ions, with charge compensated by interstitial F– ions), these ions can transition to fluorescing divalent states (Sm2+, Eu2+) upon acquiring an electron.



300 ppb of Eu is enough to yield >1 impurity /  $\mu$ m<sup>3</sup> and explain the homogeneous blue fluorescence observed in the 100 kRad irradiated crystal. <sup>12</sup>

### IMAGING COLOR CENTER STRUCTURES OR SINGLE COLOR CENTERS **DATA ANALYSIS METHODS**



Example of track-like structure found by a clustering algorithm and also observed in a repeated scan. (10x magnific., 0.4 µm pixel size).



These structures may span a few z-planes but hot pixels span many more z-planes.

- 1. **BULK FLUORESCENCE:** average fluorescence intensity within fiducialized volume.
- 2. FLUORESCENT TRACK-LIKE & POINT-LIKE STRUCTURES: Find pixel clusters & match repeated scans
- 3. ESTIMATING SINGLE COLOR CENTER FLUORESCENCE: matching of high-intensity pixels at 1-pixel level and comparison to random matches.

### IMAGING COLOR CENTER STRUCTURES OR SINGLE COLOR CENTERS **DATA ANALYSIS METHODS**

TO CONFIDENTLY IMAGE SMALL STRUCTURES/SINGLE COLOR CENTERS SEVERAL BACKGROUND SOURCES NEED TO BE ESTIMATED &/OR EXCLUDED FROM THE DATA.



Example of track-like structure found by a clustering algorithm and also observed in a repeated scan. (10x magnific., 0.4 µm pixel size).



These structures may span a few z-planes but hot pixels span many more z-planes.

**Datasets are very large** (several Gb). **Analysis is computationally intensive** (exclusion of background sources at pixel level while finding track-like or point-like structures and matching them in repeated scans). -> **Development of Analysis Methods** (V. Aerne, MSc Thesis, UZH 2023)

### **& BACKGROUND SOURCES**

#### **EXTERNAL SOURCES:** <u>STRAY LIGHT & FILTER LEAKAGE:</u> Estimated with blank measurement.

#### CAMERA SHOT NOISE:

estimated by data taking with laser off.

### HOT PIXELS & LENS FLUORESCENCE:

Exclusion of fluorescent spots that appear in the same xy position across several z-planes.

#### SAMPLE SOURCES:

TRACE IMPURITIES FLUORESCENT UPON IONIZATION & FLUORESCENT SURFACE OILS, DUST, POLISH:

Avoided/excluded by fiducialization, comparison between neutron and  $\gamma$ -irradiated samples.

Further checks: Repeat scan and search for xyz matches. Estimate random matching.

### ANALYSIS METHODS FOR IMAGING COLOR CENTERS WITH THE MESOSPIM FINDING TRACK-LIKE (LARGE) FLUORESCENT STRUCTURES



Example of track-like structure found by a clustering algorithm and also observed in a repeated scan. 10x magnific., 0.4 µm pixel size (ps)





These structures may span a few z-planes but hot pixels span many more z-planes.

Only the same spot shows high correlation in the repeated scan. -> The structure is a real fluorescent feature in the crystal.

Work of MSc. Student V. Aerne

### **ANALYSIS METHODS:**

- 1) Automatic fiducialization of the data
- 2) Algorithm to find dense color center structures
  - Connects high-intensity pixels
  - Selects structures that exceed a certain threshold.
- 3) Structure matching
  - Compare structures to repeated scans
  - Quantify similarity

Origin of the structure is unknown. They were first identified by eye, but a couple more were found by this analysis method.

### ANALYSIS METHODS FOR IMAGING COLOR CENTERS WITH THE MESOSPIM SIGNAL INDUCED BY NEUTRONS: FINDING POINT-LIKE STRUCTURES

#### Work of MSc. Student V. Aerne



Example of bright pixels appearing at repeated scans imaged at 6x, 1µm ps. Only a few structures are observed in the data (but this data was taken at lower resolution and the structures are expected to be small).

-> use a different analysis method



# **Excess of matches in data of neutron irradiated crystal** is observed in comparison to random matching estimation. **Bulk fluorescence** measurements **also show excess** in comparison to blank.

### ANALYSIS METHOD:

- Automatic fiducialization of the data
- 2) Algorithm to find dense color center structures Find high intensity pixels.
- 3) Pixel matching

1)

- Find number of matching pixels
- Exclude defective pixels
- Compare to random matching

Still to investigate: Sample variability, intensity of neighboring pixels using high magnification scans (20x), level of signal excess for  $\gamma$ -irradiated crystals.

#### SUMMARY & OUTLOOK

Demonstrated imaging of radiation-induced color centers with light-sheet microscopy;

Current tests focus on the production of neutron induced color centers and identification of single color centers in this data;

Future tests will focus on:

- different materials (LiF, Al<sub>2</sub>O<sub>3</sub>, ...);
- relation between fluorescence istensity and neutron dose;
- imaging large ion-induced (or Li  $(n,\alpha)^{3}$ H) tracks. \_

Applications of imaging color centers with the mesoSPIM beyond PALEOCCENE ( $CE_vNS \& DM$  detection): Paleo detectors neutron detectors, Rock dating (geology)









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PALEOCCENE: 2104.13926, 2203.05525 mesoSPIM: bioRxiv 2023.06.16.545256

### **BACK UP SLIDES**