U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

ASI Advanced Sensors and Instrumentation

Passive Monitors
Silicon Carbide

Advanced Sensors and Instrumentation (ASI)
Annual Program Webinar
November 4, 6-7, 2024

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Oak Ridge National Laboratory, Nuclear Energy & Fuel Cycle Division

Project Overview: SiC Passive Monitors

Purpose

- SiC frequently used as passive temperature monitor (TM) in irradiation experiments
- ASTM standard does not exist for SiC temperature monitors
- Historical SiC TM analysis methods better suited for high fluence experiments which achieve saturation swelling

Scope

- Plan for development of SiC thermometry ASTM standard & round robin testing
- Apply deep level transient spectroscopy (DLTS) as irradiated SiC analysis technique

Schedule

- Project completion **delayed** to FY25 – waiting to receive irradiated SiC
  - M3CT-24OR0703022: Research plan for the development of SiC thermometry ASTM standard and round robin testing
  - M3CT-24OR0703021: Assessment of the use of deep level transient spectroscopy (DLTS) for low fluence SiC temperature measurement

Participants

- INL: Malwina Wilding
- ORNL: Anne Campbell, Hsin Wang, David Glasgow

Technology Impact: SiC Passive Monitors

Benefits

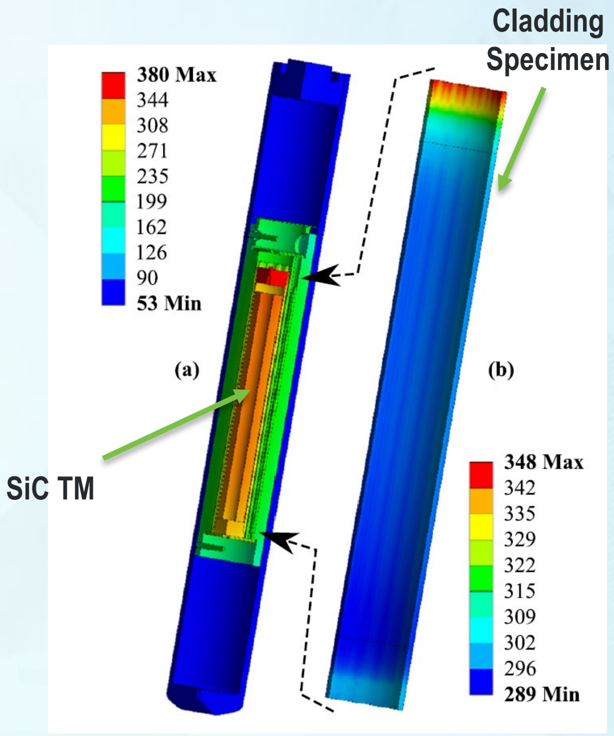
- Does not require instrument leads
- Continuous temperature indication ~200 – 1,000° C
- Compact geometry

Mechanism

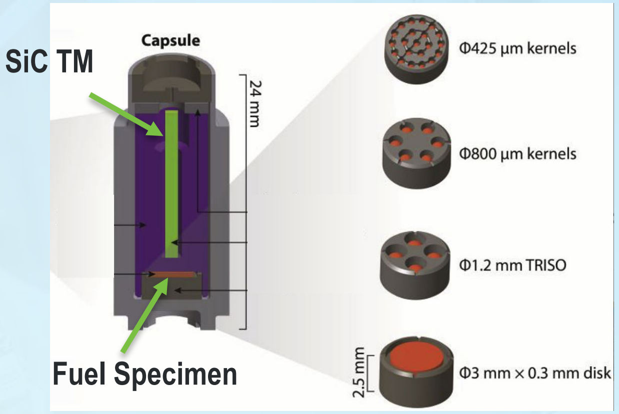
- Neutron irradiation damage causes point defects and lattice dilations in SiC
- Defects can be annealed post-irradiation when the annealing temperature exceeds irradiation temperature

Role in Nuclear Energy Industry

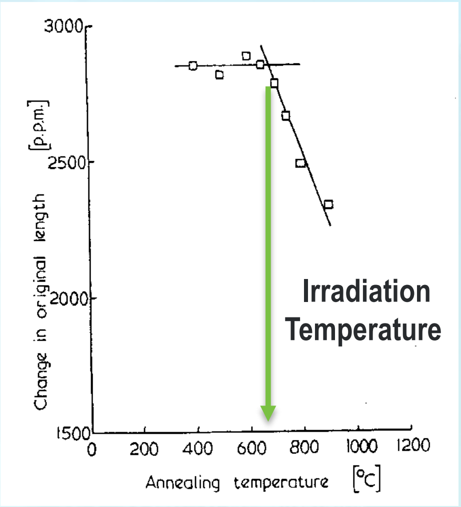
- Provides indication of temperature conditions during materials irradiations
  - Cladding materials
  - Moderators (graphite)
  - Fuels
- Data to support license/regulatory applications
- ***No standardized method for use***



Mulligan, et. al. JNM 588 (2024) 154808.



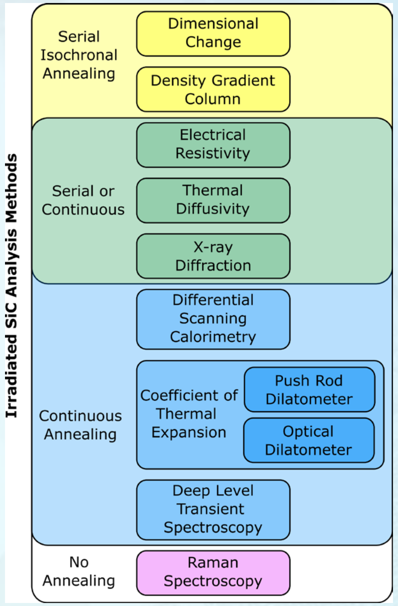
Petrie, et. al. JNM 526 (2019) 151783.

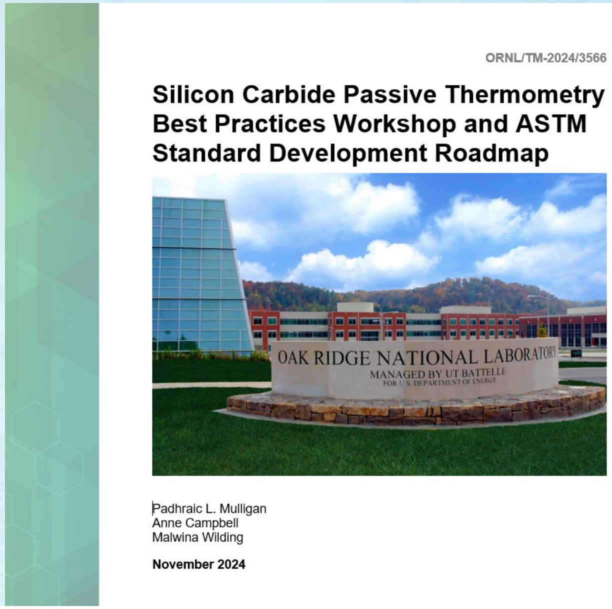


R.P. Thorne, V.C. Howard, B. Hope, Proc. Br. Ceram. Soc. 7 (1967) 449–459.

SiC Passive Monitors Workshop

- Hosted SiC passive thermometry workshop May 8-9, 2024
  - 27 attendees from US/international labs and industry
  - Discussed various SiC analysis methods/best practices
  - Several sessions on SiC TM technique, tours of HFIR, LAMDA, hot cells
  - Developed high-level plan for ASTM standard development
- Workshop outcomes, review of SiC analysis methods, and ASTM standard development plan included in upcoming ORNL Technical Memo

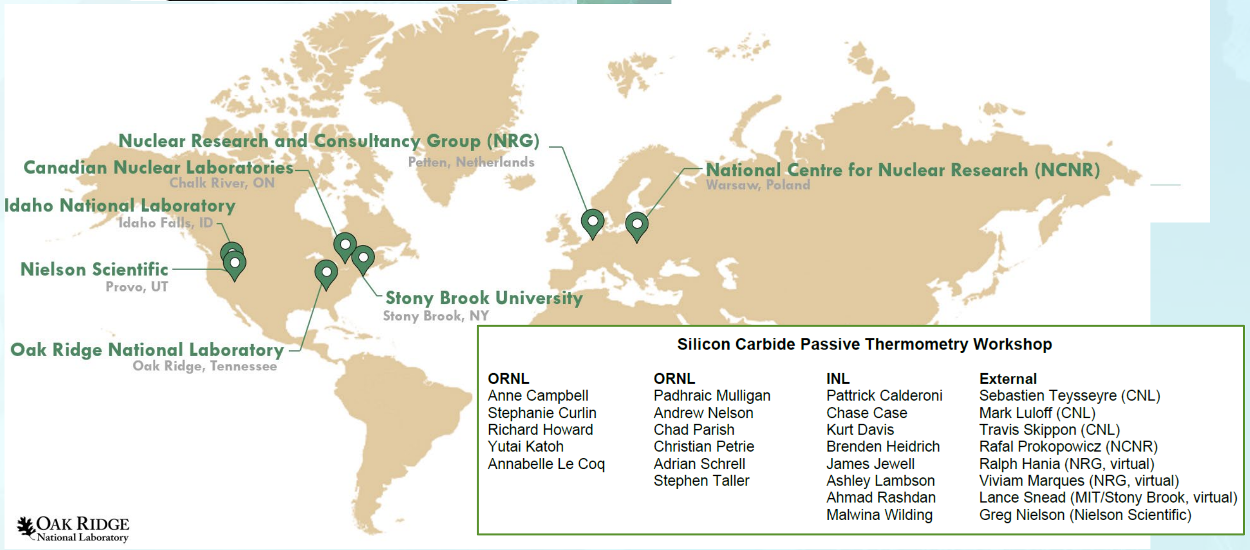



ORNL/TM-2024/3566

Silicon Carbide Passive Thermometry Best Practices Workshop and ASTM Standard Development Roadmap

Padhraic L. Mulligan
Anne Campbell
Malwina Wilding

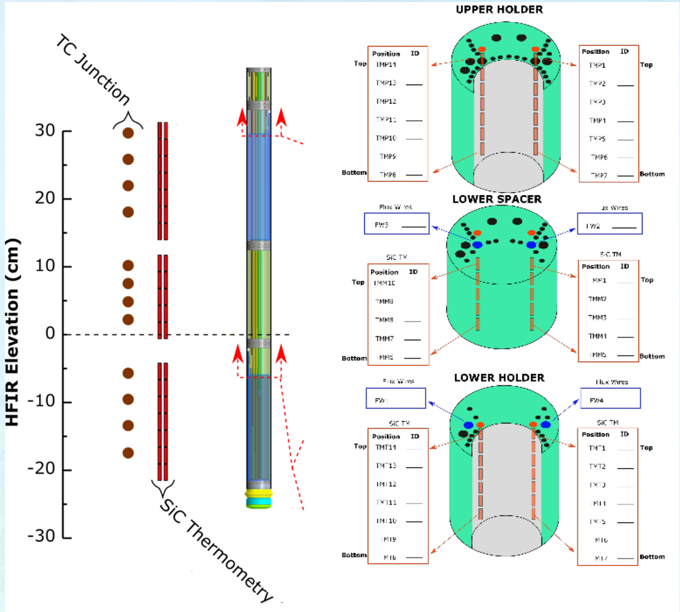
November 2024

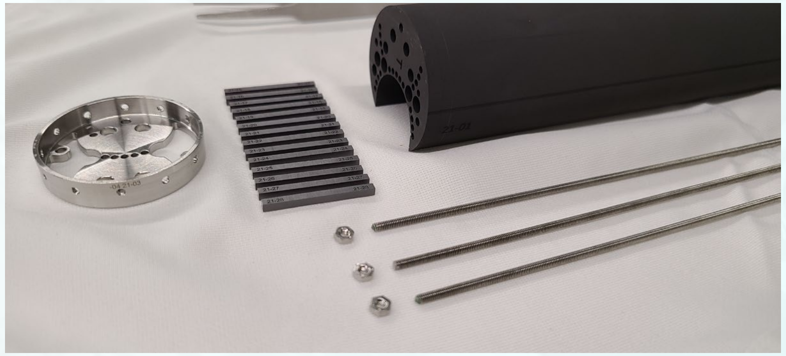


SiC Passive Monitors Workshop (Round Robin Testing)

- Developed plan for round robin SiC analysis
  - Pre-irradiated SiC from three reactors
    - HFIR, ATR, MITR
    - Range of temperatures & fluences
  - Six laboratories to perform analysis
    - INL, ORNL, MIT, CNL (Canada), NRG (Netherlands), NCNR (Poland)
- Discussed ASTM standard development with ASTM E10 Committee
  - Requested a "best practices" document to subcommittee chairs for discussion
- Awaiting shipment of HFIR irradiated experiment

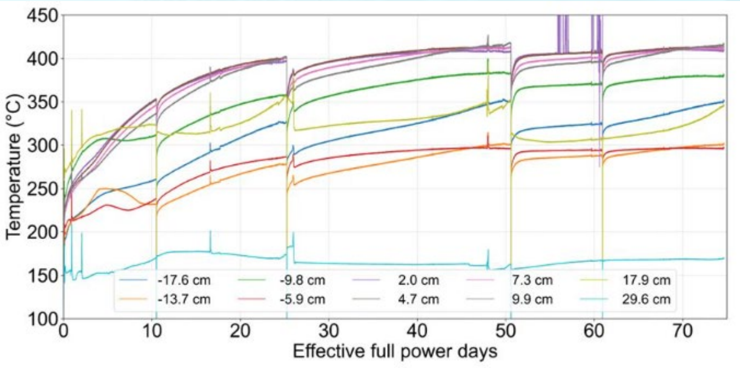
SiC TM and TC Locations





SiC Thermometry in Upper Holder

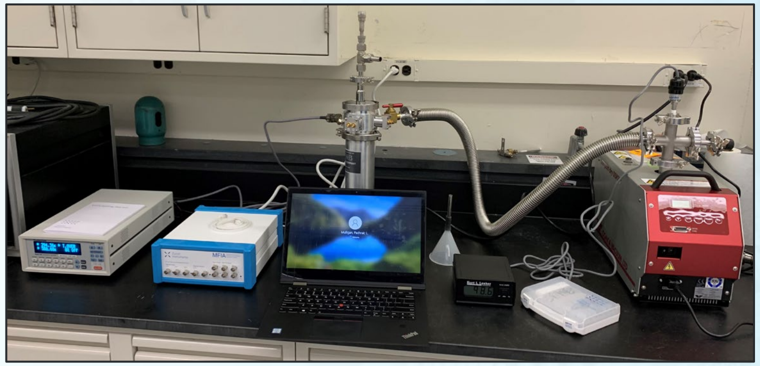
WIRE-21 Thermocouple Measurements

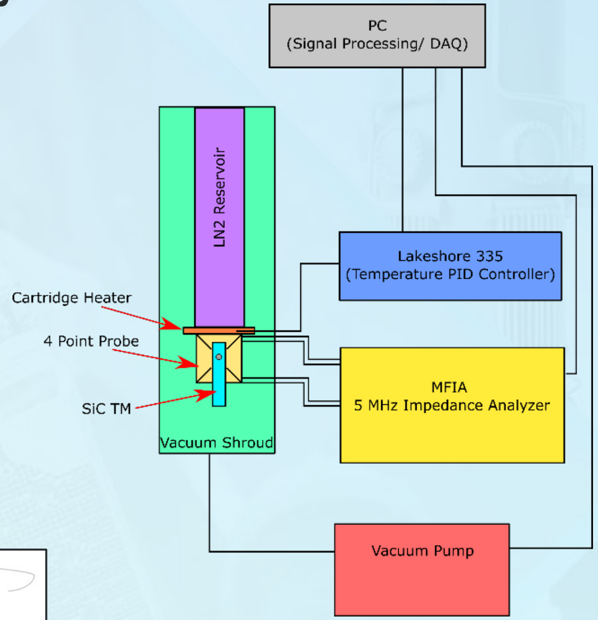


SiC Deep Level Transient Spectroscopy

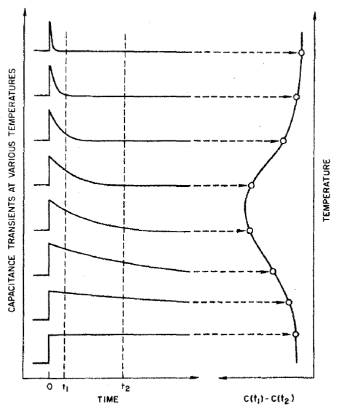
- DLTS can identify electrically active defects in semiconductor
  - Activation energy (ΔE)
  - Capture cross section (σ_n)
  - Defect concentration (N_T)
- Requires
  - Rectifying junction (Schottky or p-n)
  - Well-behaved depletion region
  - High defect concentration (10^{-5} x N_d)
- Setup and integrated DLTS system at ORNL
  - 77 – 500 K
  - DC – 5 MHz
  - -10 – 10 V bias
  - GUI controlled/automated
- Traditional lock-in amplifier/pulse generator/DAQ replaced with programable MFIA (Zurich Instr.)

DLTS System

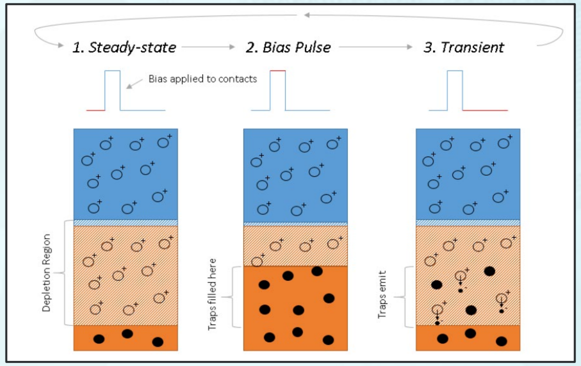




DLTS Process

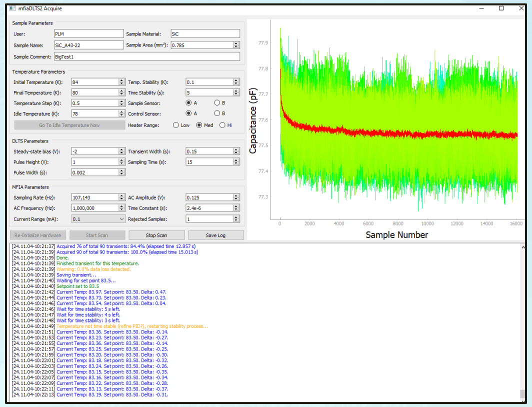


Lang, J. Appl. Phys. 45 (1974) 3023-3032



Nelson, "Native and Radiation-Induced Defects in III-V Solar Cells and Photodiodes", Thesis, 2019.

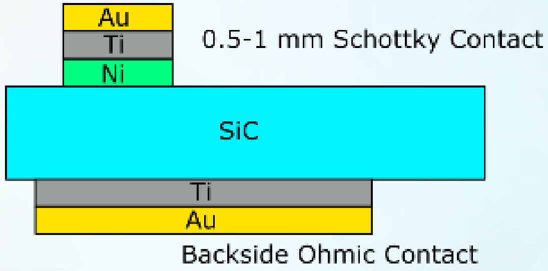
DLTS Signal: $S = C(t_1) - C(t_2) = \Delta C(0) \times (e_1^{-\frac{t_1}{\tau}} - e_1^{-\frac{t_2}{\tau}})$

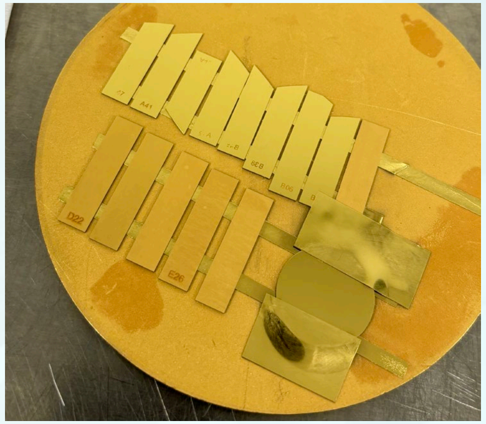


DLTS Software Credit: George Nelson, RIT
https://github.com/nelsongt/mfiaDLTS

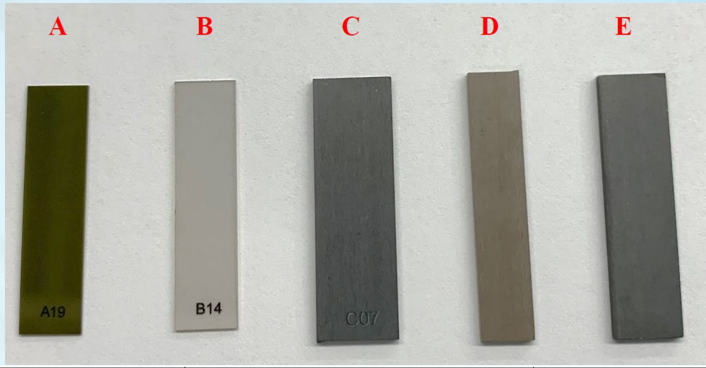
DLTS Specimen Fabrication

- Five different types of SiC substrates
- Metal contact deposition performed in Center for Nanophase Materials Science cleanroom
  - Electron-beam evaporation
  - Full backside ohmic contact
  - Shadow mask used for topside Schottky contacts
  - Avoided annealing of contacts
- Deposited on both Si- and C-face of wafer



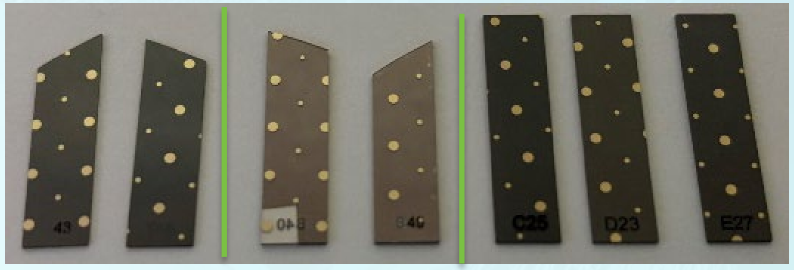


Five Types of Commercially Available SiC



ID Prefix	Vendor	SiC Type	Form Factor
A	MSE	4H, N-type, 4.0° off axis toward <11-20>	0.35×101 mm diameter wafer
B	MSE	4H, Semi Insulating (SI), on axis <0001>	0.5×101 mm diameter wafer
C	Dow / Rohm & Haas	3C, High Resistivity	6.35×152 mm square plate
D	CoorsTek	3C PureSiC®, High Resistivity CVD	5.0×200 mm square plate
E	ASCM	3C, High Resistivity	6.35×152 mm square plate

4H N-type SiC 3C SiC

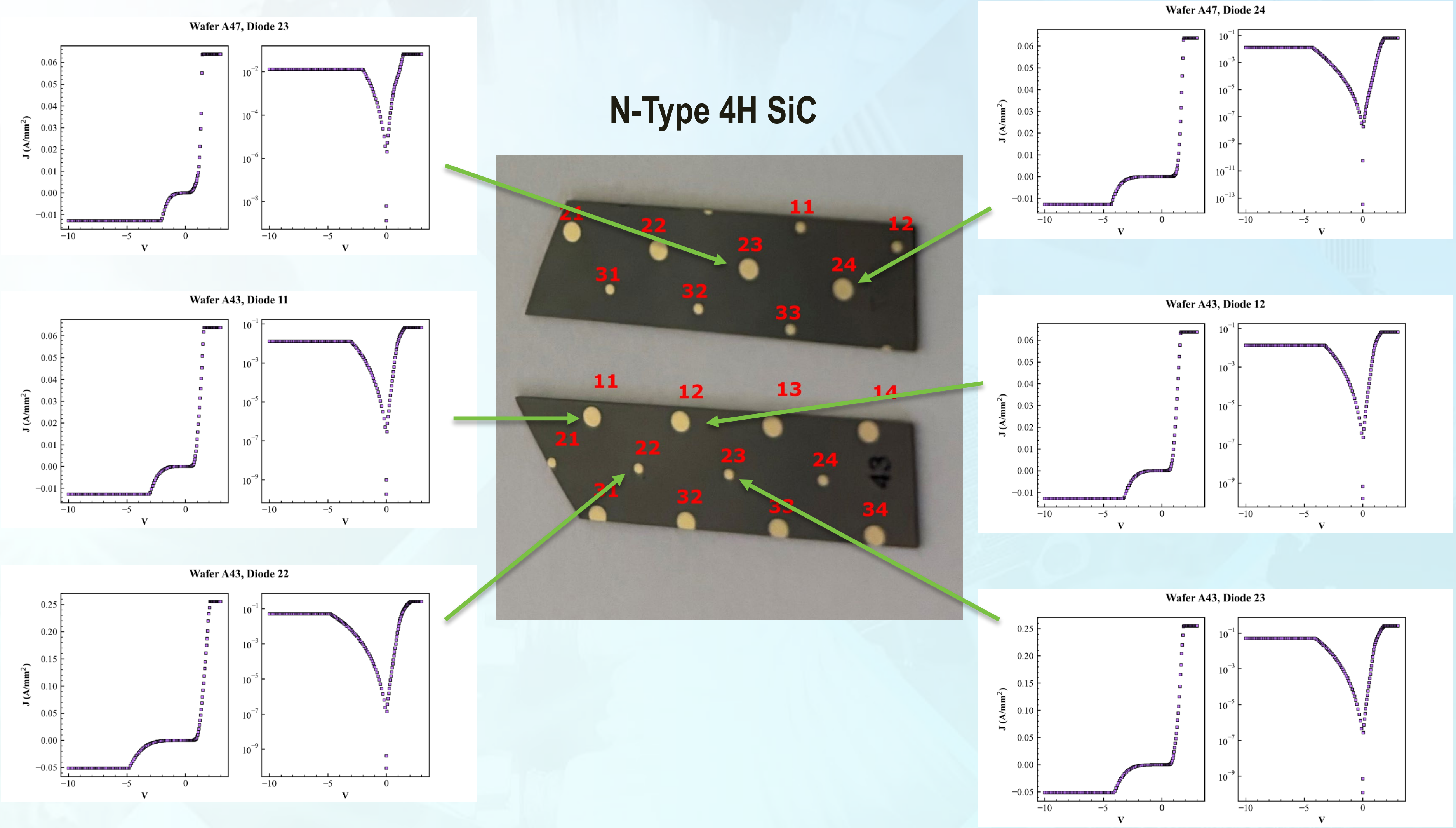


4H Semi Insulating SiC

DLTS Specimen Characterization

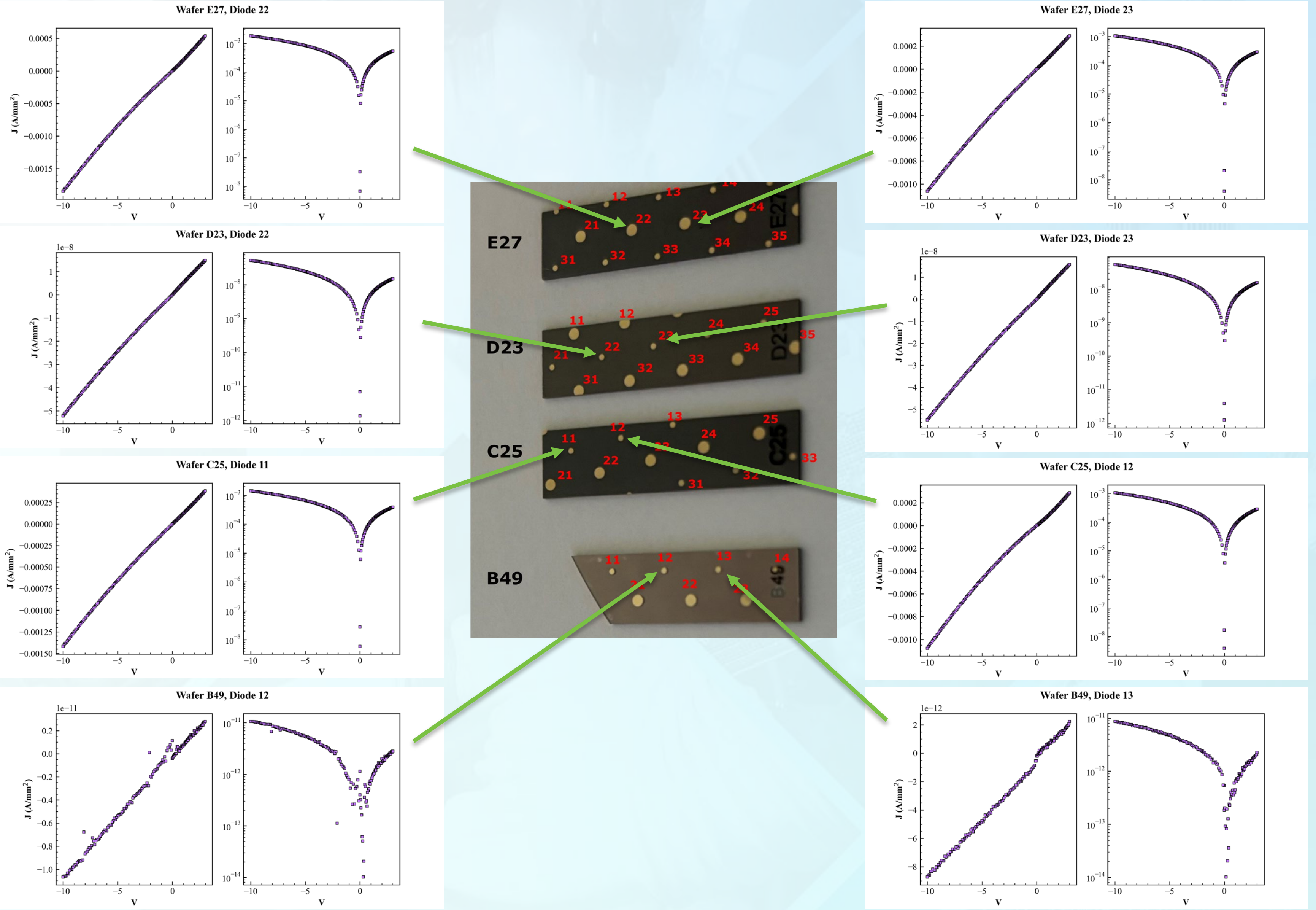
I-V measurements showed N-type 4H material produced good rectify contacts

- $V_{bi} \approx 1.45 – 1.66$ V
- Ideality factor ≈ 1.22 – 1.63
- $\Phi_B \approx 1.25 – 1.41$



DLTS Specimen Characterization

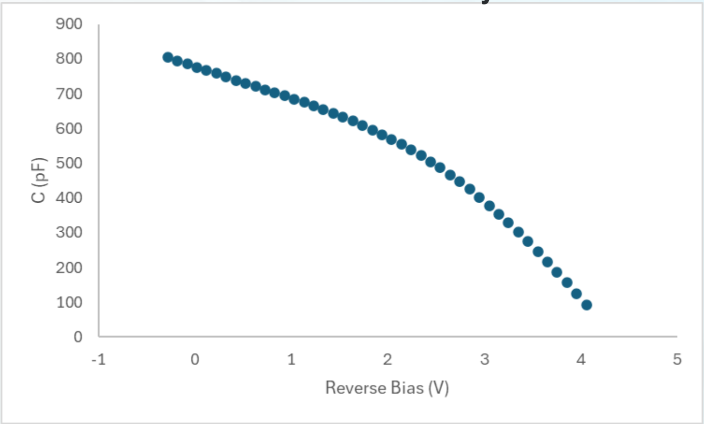
Semi-insulating 4H SiC and three types of 3C SiC all produced ohmic contacts



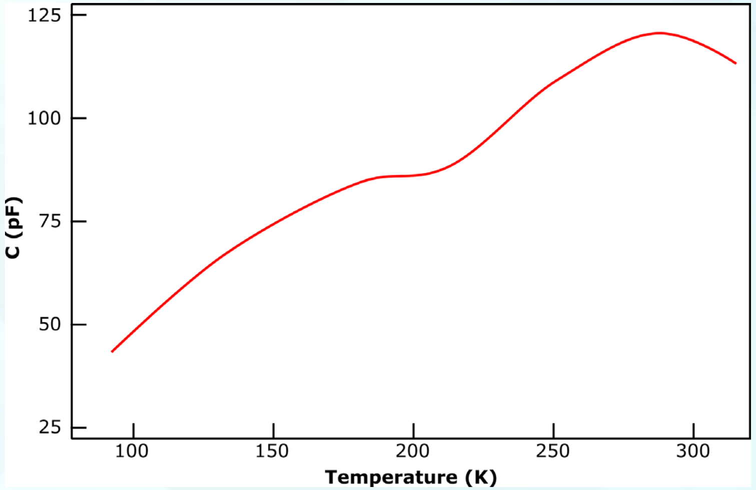
DLTS-Unirradiated SiC

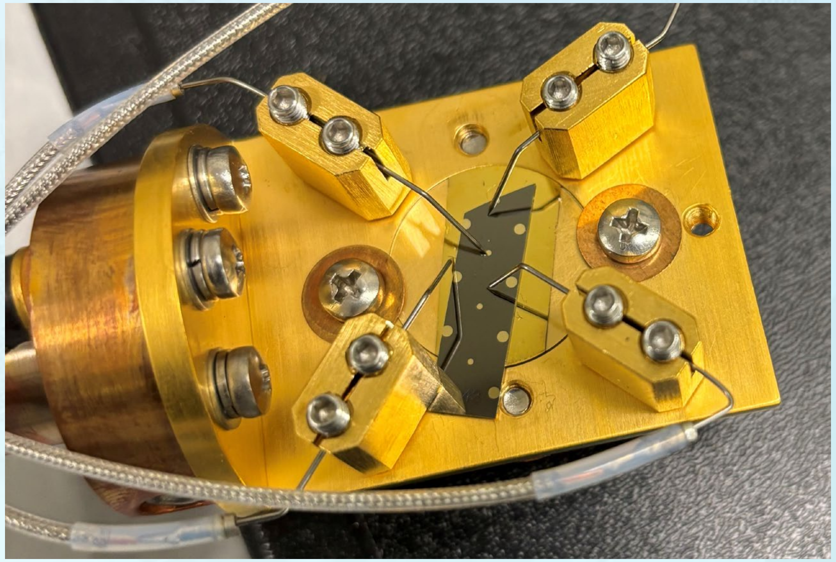
- Unirradiated SiC analyzed using DLTS system
  - 80 – 320 K
  - 1 MHz
  - 2V reverse bias
  - 1V pulse height, 2 ms pulse width
- Identified two defect levels
  - $E_C - 0.35$, $E_C - 0.66$ eV
- Doping concentration measured
  - $N_d = 6.1 \times 10^{14}\ cm^{-3}$
- Working to incorporate frequency and bias voltage sweep into measurements
- Defect concentrations will be compared to the irradiated SiC material

C-V measurement of Schottky diode



Diode capacitance as a function of temperature



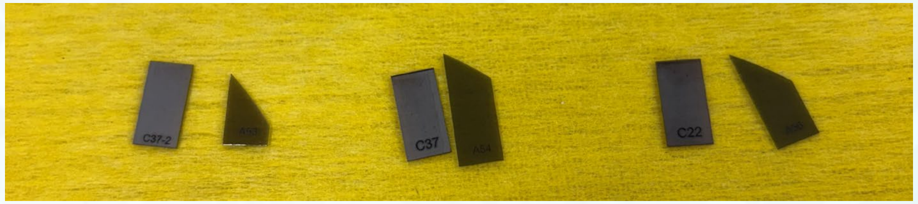


N-type 4H SiC in cryostat probe station

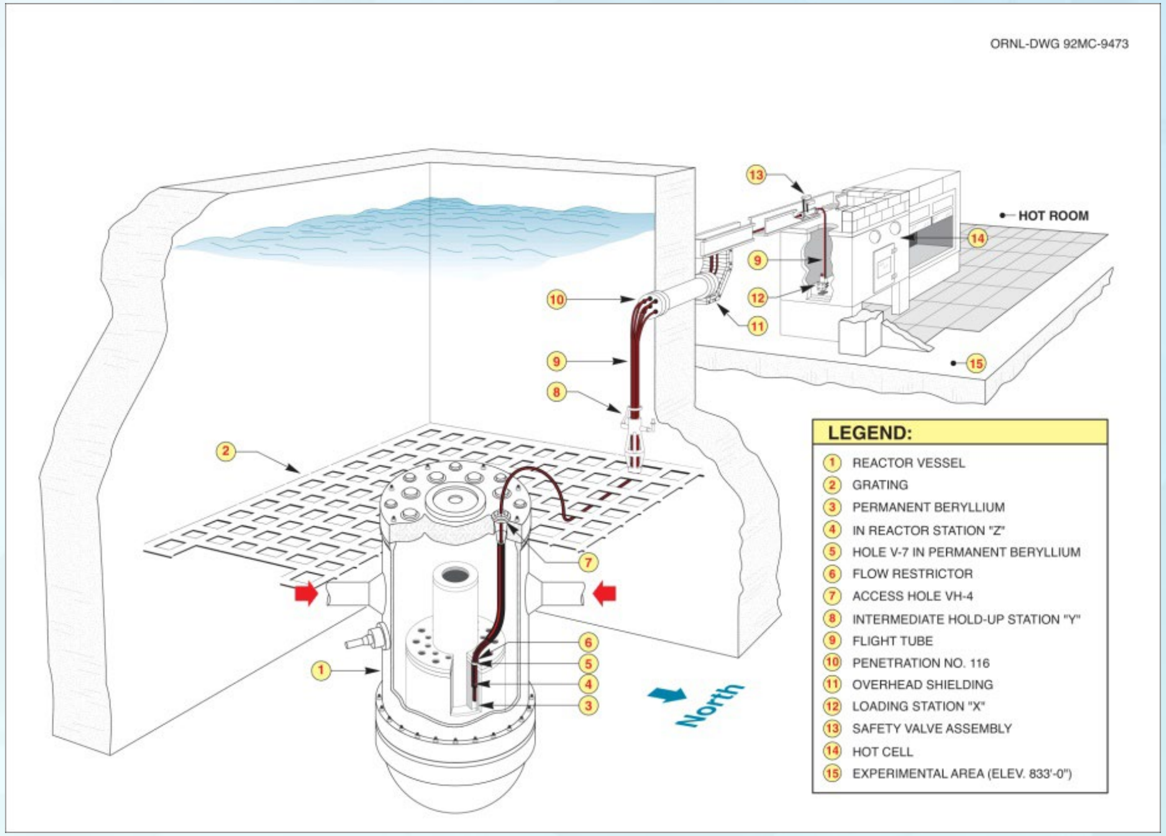
Irradiated SiC

- SiC specimens irradiated in graphite rabbits using the pneumatic tube of HFIR Neutron Activation Analysis Laboratory
- 4 different fluences
- 3C- and 4H- SiC sample for each fluence
  - 3C- for dilatometry measurement
  - 4H- for DLTS measurement
- Ready for sample preparation by end of year

Sample	Irradiation Time (min)	Total Fluence (n/cm^2)
A	5	1.92E+17
B	10	3.84E+17
C	20	7.68E+17
D	30	1.15E+18



Irradiated SiC Specimens



HFIR NAA Laboratory

Concluding Remarks

ASTM Development

- Hosted SiC Passive Thermometry Workshop
- Plan for round robin testing of pre-irradiated SiC

DLTS Analysis of SiC

- DLTS system is integrated and operational
- Fabricated specimens for DLTS measurements on five types of SiC
  - Semi insulating and polycrystalline SiC are not good candidates for DLTS measurements
- Irradiated four samples of 3C- and 4H- to 1.9E+17 – 1.2E+19 n/cm^2

Future Work (CO, no new BA)

- Issue report on SiC Workshop and round robin
- Perform DLTS on irradiated SiC specimens

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Thank You