INL/MIS-24-78380-Revision-0

Silicon Carbide Passive Temperature Sensors at INL

May 2024

Changing the World's Energy Future

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Idaho National Laboratory

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Silicon Carbide Passive Temperature Sensors at INL

Introduction

- Provide a practical and robust approach to estimate peak irradiation temperature during PIE for direct integration in irradiation test designs:

  - Less expensive static (drop-in) capsule tests with no leads will essentially be the only possibility for peak temperature indication
  - Determine a peak temperature reached within a relatively broad range (200 – 1200°C)
  - Neutron irradiation induced lattice expansion of SiC annealed out when the post-irradiation annealing temperature exceeds the peak irradiation temperature (0.5 dpa – 8 dpa)
  - Property change (lattice spacing, **dimensions, electrical resistivity**, thermal diffusivity, or bulk density)

ATR Overview

Figure 2. ATR Core Cross Section.

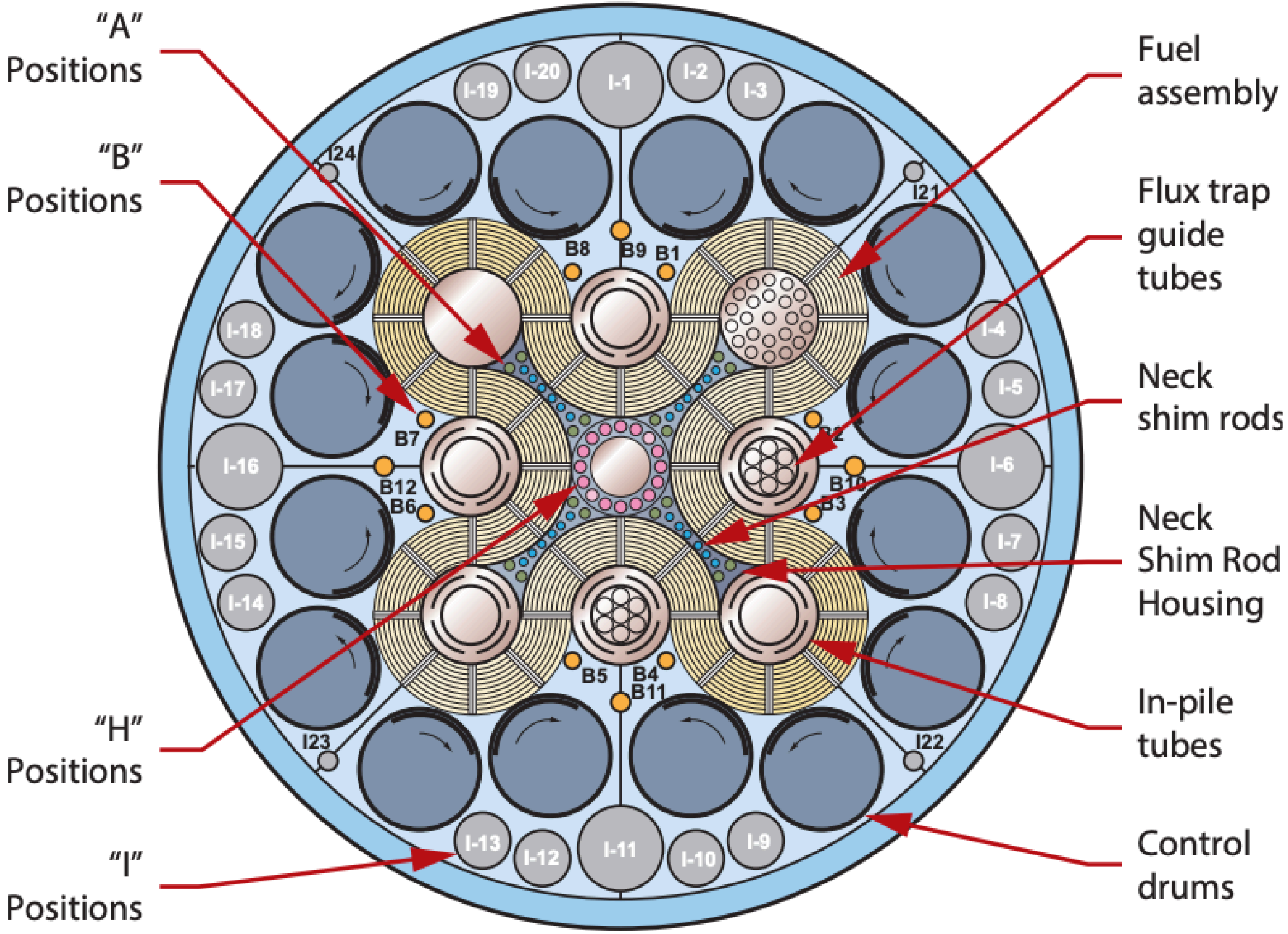
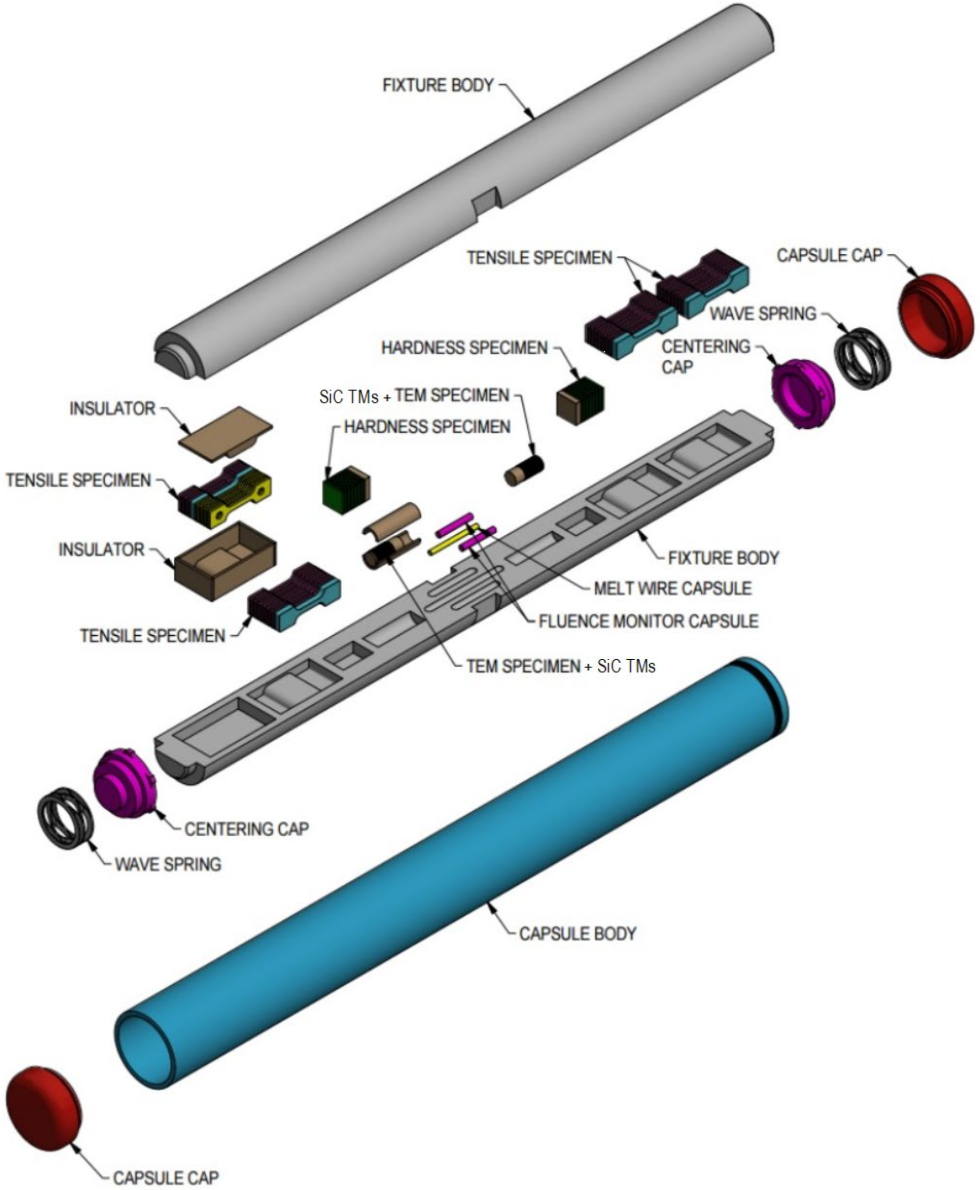
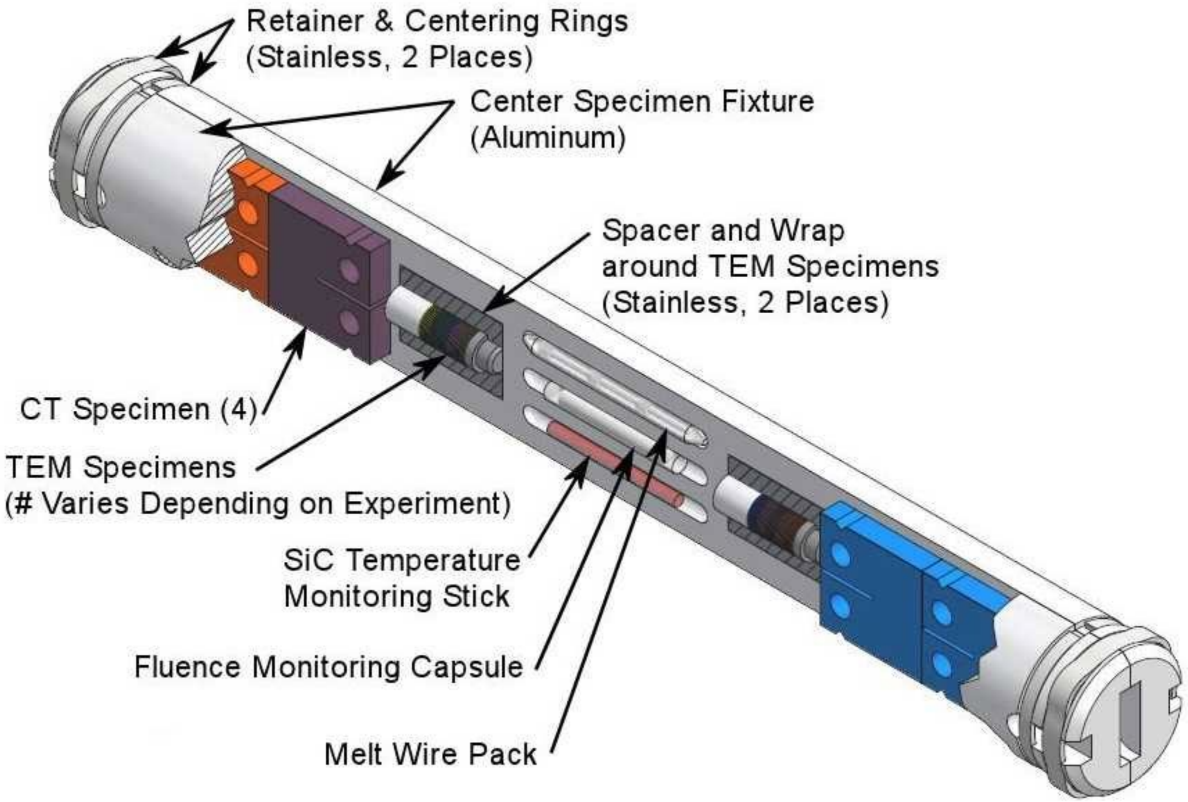


Table 1. ATR General Characteristics

Reactor:	
Maximum thermal power	250 MW(Th)[a]
Maximum power density	1.0 MW/L
Maximum thermal neutron flux	1.0×10^{15} n/cm^2-sec[b]
Maximum fast flux	5.0×10^{14} n/cm^2-sec[b]
Number of flux traps	9
Number of experiment positions	77[c]
Core:	
Number of fuel assemblies	40
Active length of assemblies	1.2 m (4 feet)
Number of fuel plates per assembly	19
Reactor Coolant:	
Operating pressure	2.5 Mpa (360 psig)
Design temperature	115°C (240°F)
Light water maximum coolant flow	3.09 m^3/s (49,000 gpm)
Coolant temperature (operating)	<52°C (125°F) inlet, 71°C (160F) outlet

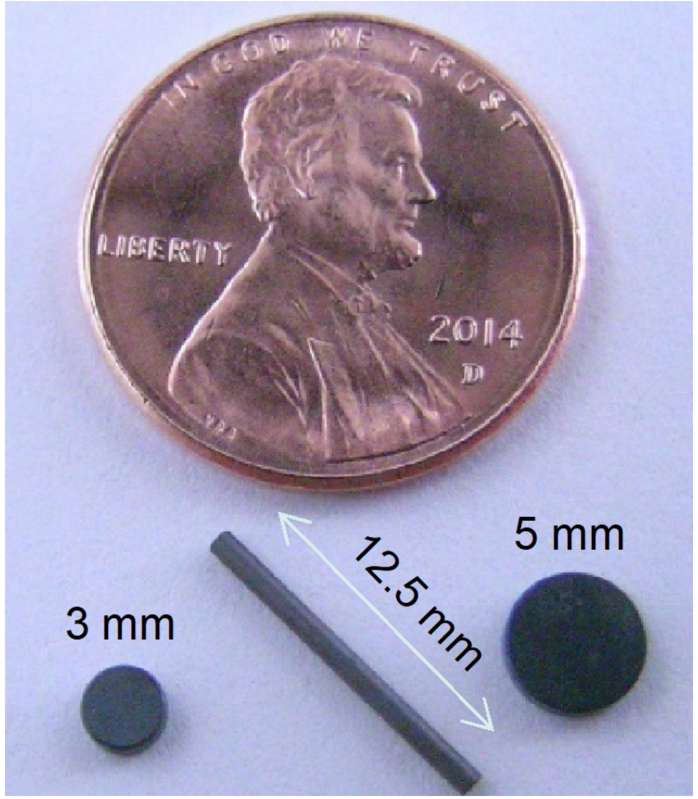
Drop In capsule and placement of SiC



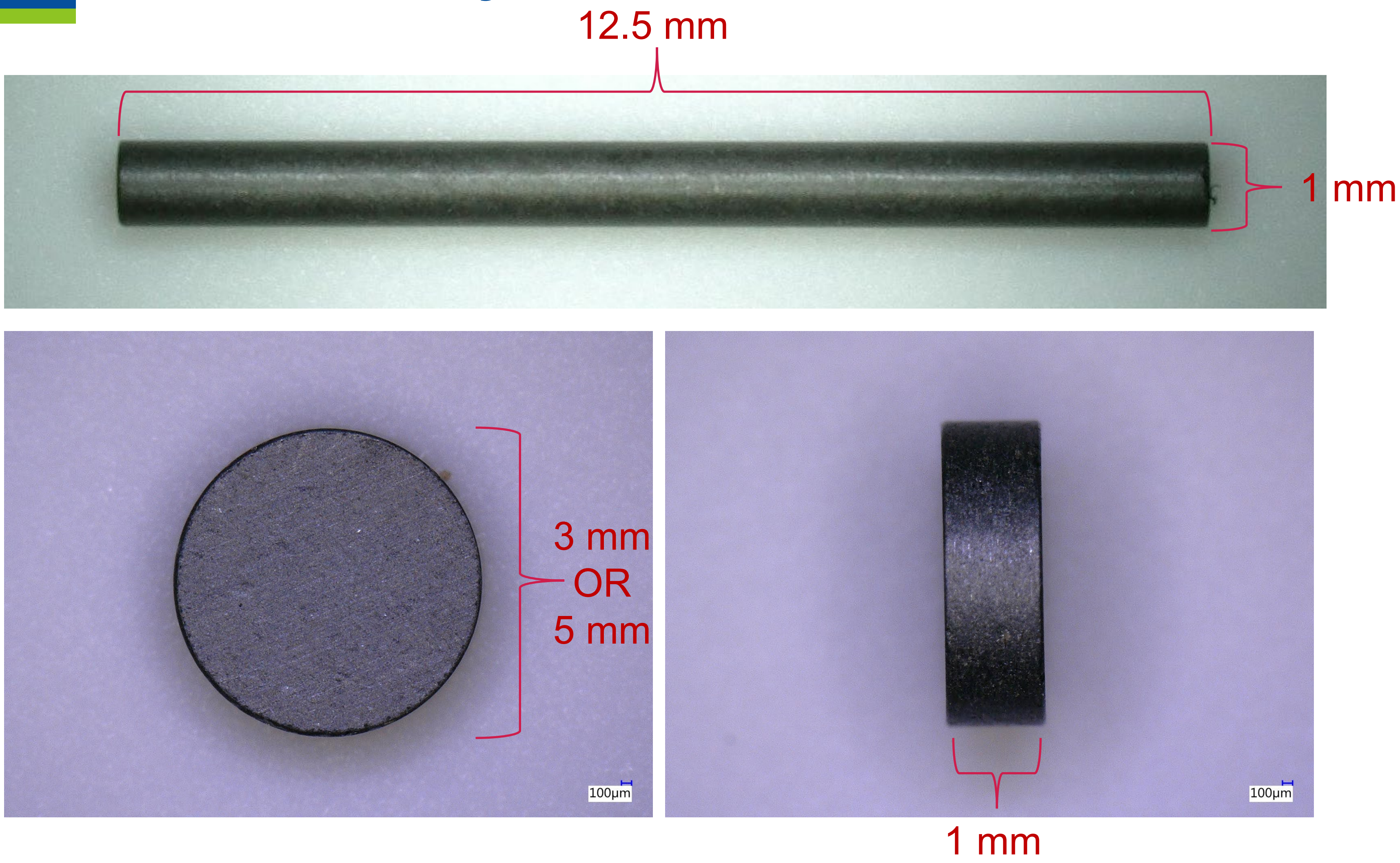


SiC Material

- SiC rod (1 mm OD by 12.5 mm) and disks (OD 3 mm or 5 mm by 1 mm)
  - Procured from PremaTech Advanced Ceramics
  - Follow Rohm and Haas specification sheet:
    - chemical vapor deposition (CVD) SiC
    - high resistivity grade (>1000 Ω cm)
    - highly pure (99.9995%) with theoretically density and with no voids or micro cracks
    - face-centered cubic ß crystal structure (isotropic characteristics)
    - performance at high temperatures (up to 1700°C)
    - resistance to corrosion, oxidation, erosion, wear, and abrasion
    - hardness (second only to diamond) with high strength



SiC Geometry



- **SiC Rods:**
  - 12.5 mm long by 1 mm OD
  - Quantity: 250
- **SiC Discs**
  - 3 mm OD by 1 mm thickness
  - Quantity: 40
  - 5 mm OD by 1 mm thickness
  - Quantity: 100

Pre-irradiation characterization

- Perform Supplemental Work Instruction for each SiC TMs that include:
  - Microscope images and their dimensions to make sure there are no issues with the surface
  - Measure dimensions with caliper and optical dilatometer
  - Ethanol and Acetone clean
  - Package, label and deliver to experiment

MSL
(Measurement Science Lab)
SILICON CARBIDE
Passive Temperature Monitors

for

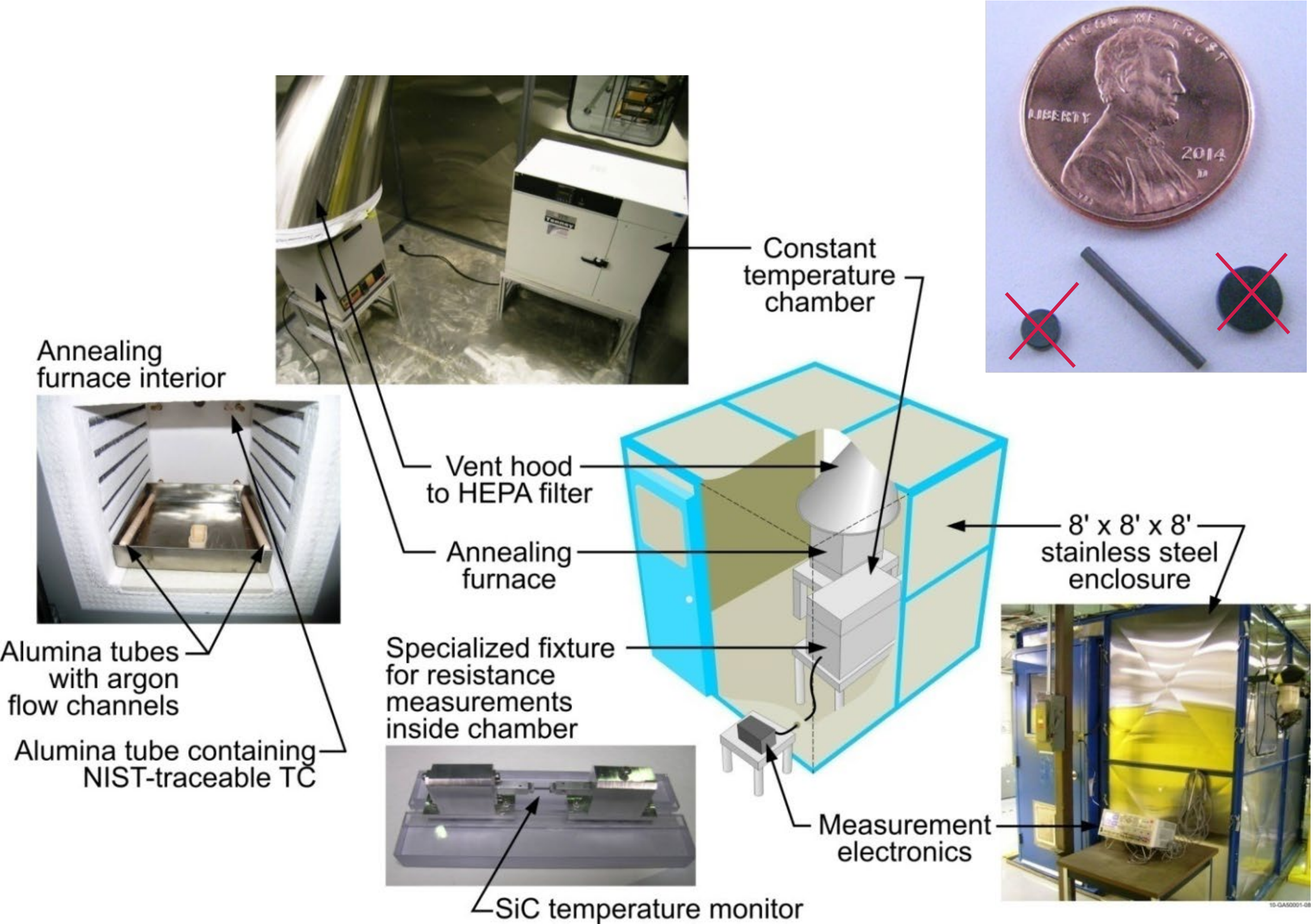
XXX
Experiment in **ATR**

SiC ID#: MSL- Experiment – SiC rod or disc
Temperatures: XXX °C
Neutron Damage: X dpa

SWI - Date – Performer Initials

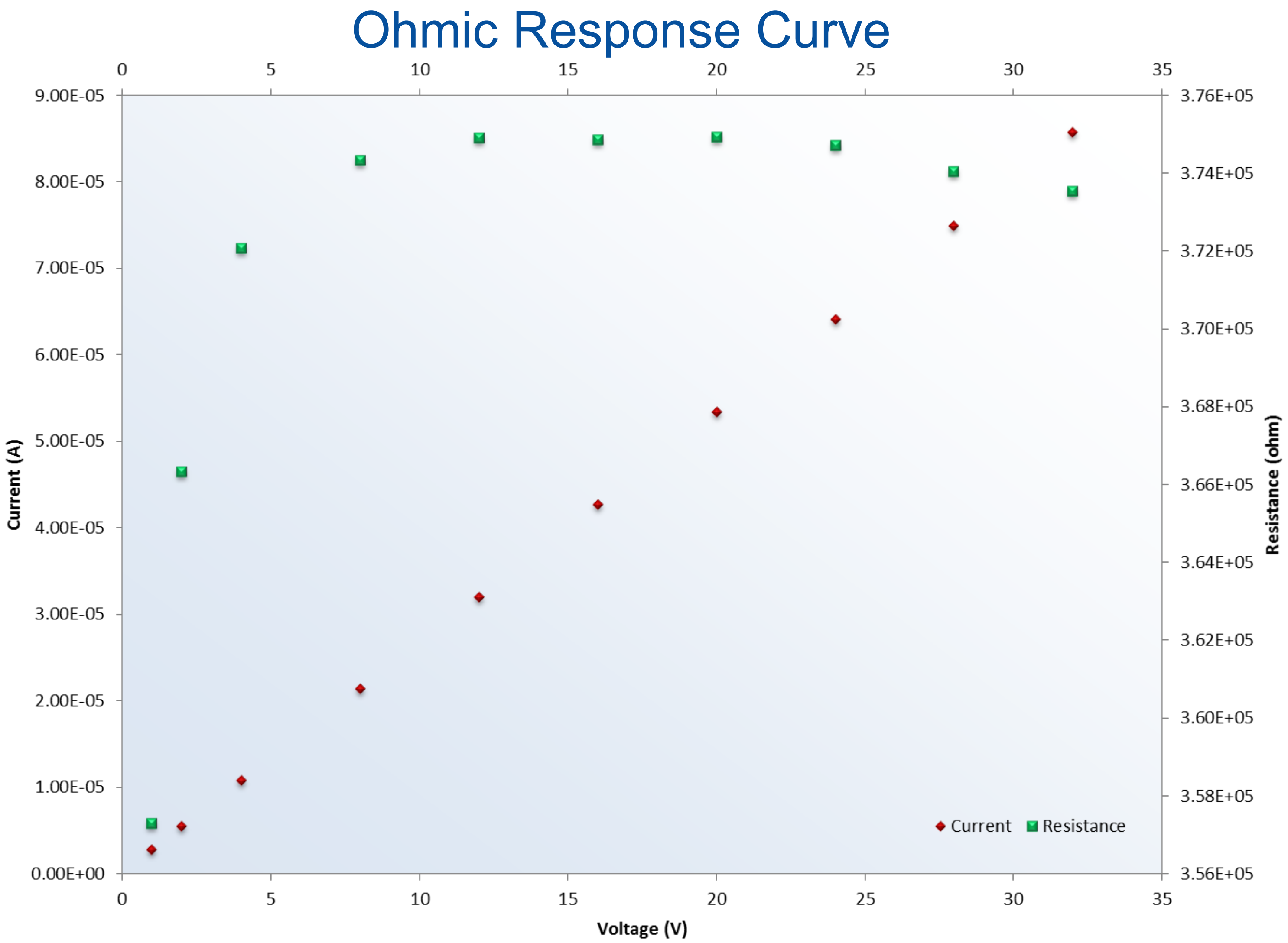
1

SiC Temperature Monitor Resistance Method



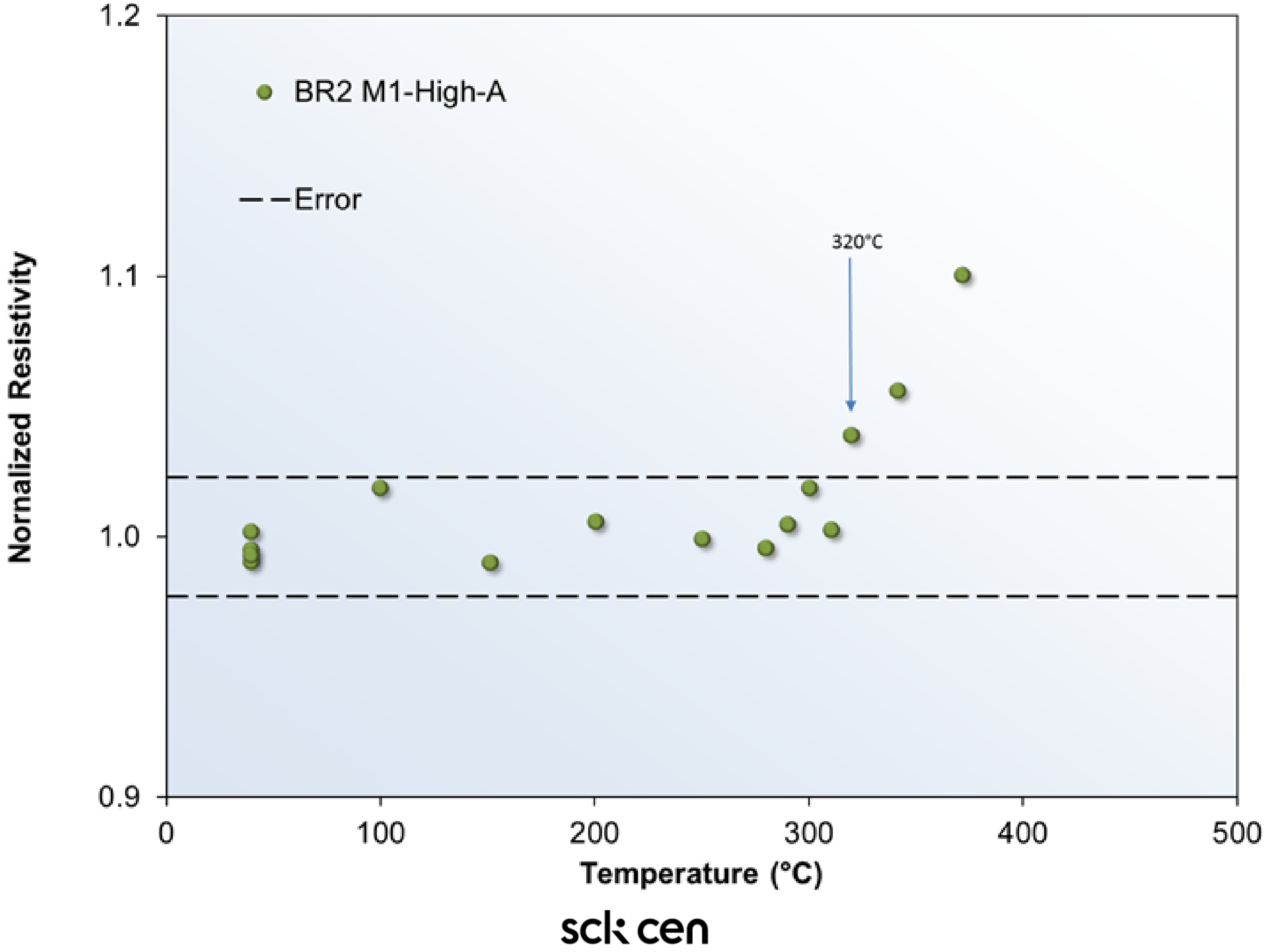
- Wide range of temperatures (200 – 800 °C) and doses (0.5 to 8 dpa)
- Only Small (1 mm x 1 mm x 10 mm pillar or 1 mm x 12.5 mm rod) SiC monitors
- Isochronal heating (30 minutes) in furnace with NIST-traceable thermocouples
- Specialized fixturing developed for resistance measurements
- Measurements made in constant temperature chamber at 40 °C
- High accuracy (9 digit) electronics

SiC Temperature Monitor Resistance Method



- Generated for each monitor prior to heating
- Check for linearity
- Select target voltage (with corresponding current) to minimize heating

SiC Temperature Monitor Resistance Method



Peak Irradiation Temperature

- Evaluation point where the resistivity begins, and consistently remains, above the error band (dotted lines)
- Error band established as ±2σ (based on first five data points taken below 150°C)

SiC Temperature Monitor Resistance Method

NSUF
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sck cen

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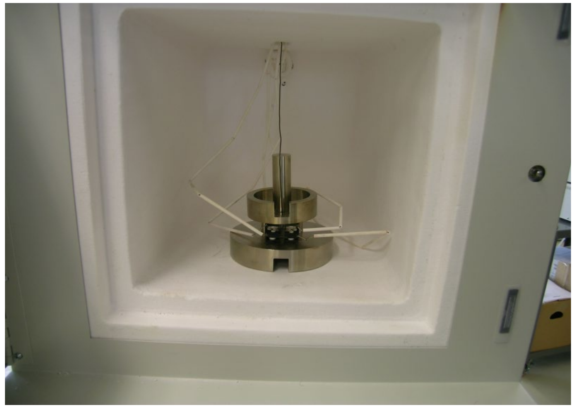
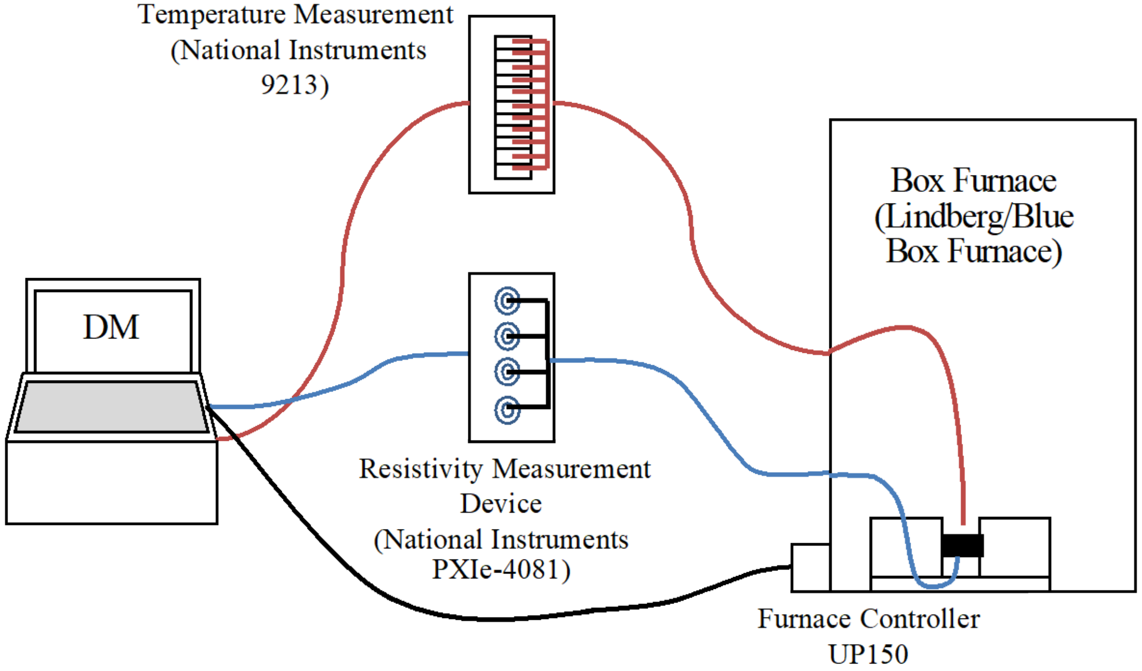
ELECTRIC POWER RESEARCH INSTITUTE

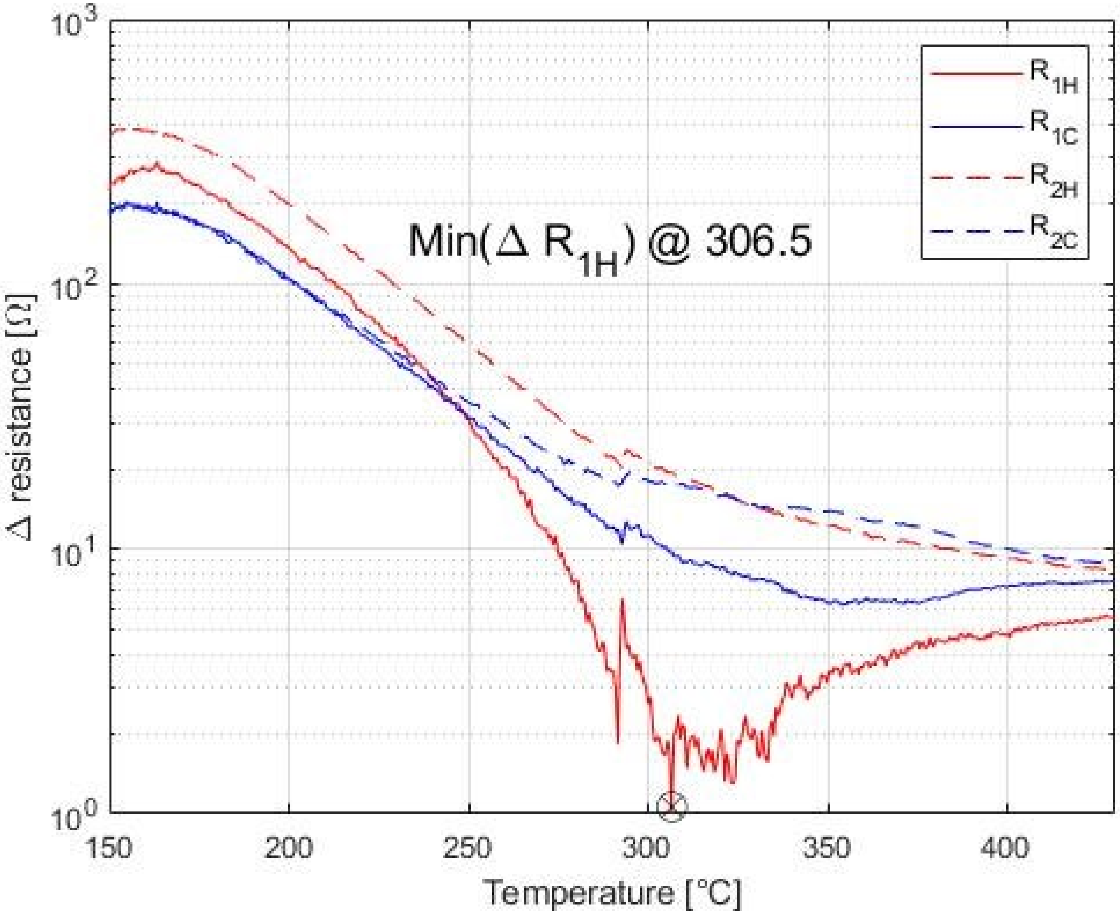
GE HITACHI

UNIVERSITY OF ILLINOIS URBANA-CHAMPAIGN

Automated Resistivity Method

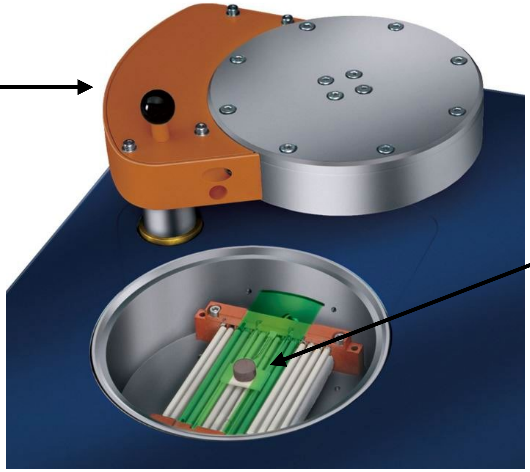


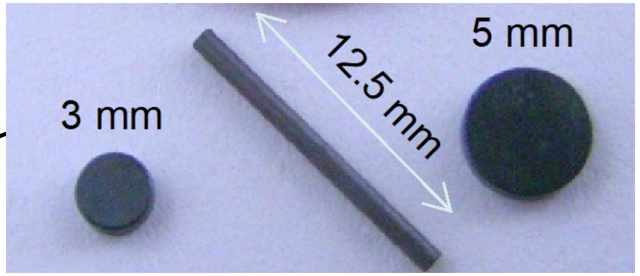




SiC Temperature Monitor
Optical Dilatometry

- Thermal expansion from continuous dilatometry is an automated process requiring minimal setup time (simple process)
- Dilatometer runs under vacuum (no oxidation issues as can happen with Resistance Method)
- Temperatures: 200 – 1200°C
- Recommended dose ranging from 0.5 – 8 dpa
- Time to process each sample 2 to 3 days (Resistance Method can be 1 to 3 weeks)
- Rods, square shaped pillars and disks may be used (any type of geometry works)



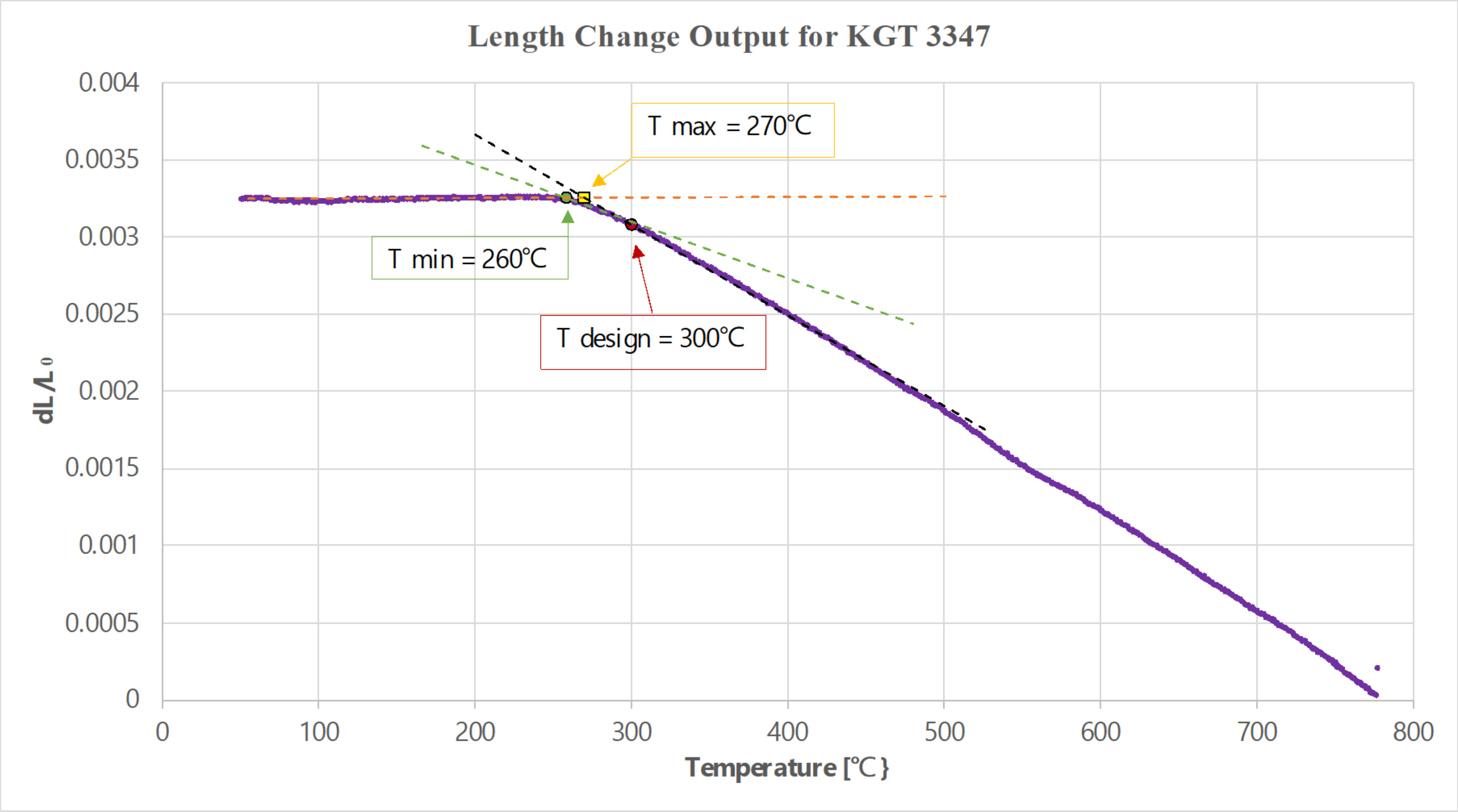


SiC Temperature Monitor Optical Dilatometry

Optical Dilatometer Advantages

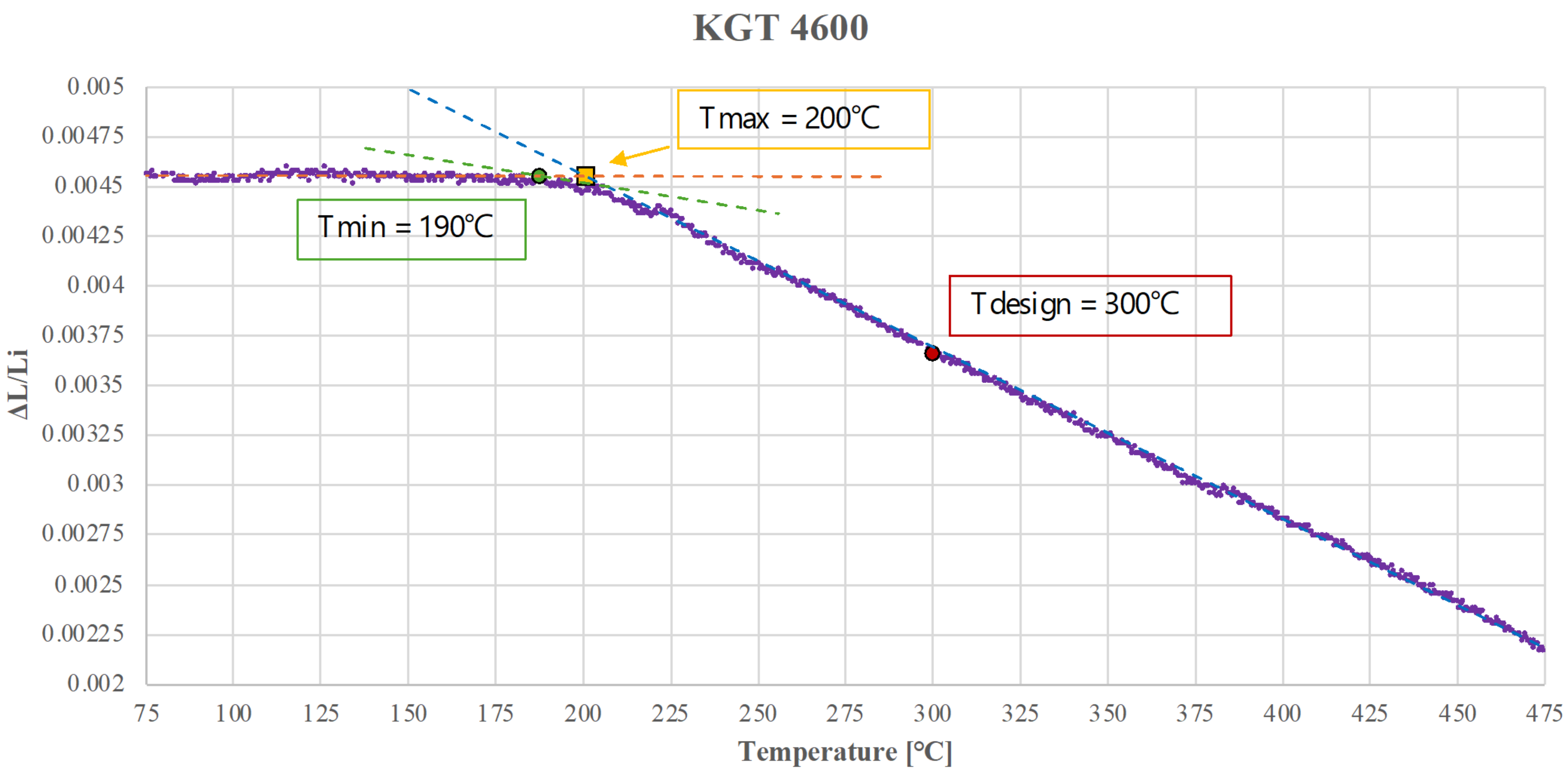
- Contactless dilatometric measurement system (no error due to sample placement with push-rod dilatometer)
  - freely expand/shrink without any interference from mechanical contact
  - more precise determination of the passive monitor’s dimensional changes and the temperature at which the changes are detected
- Effective environmental control during the testing
  - vacuum or in an inert atmosphere—a key requirement for avoiding any oxidization issues, which prolongs the resistivity method
- Possible to detect multiple irradiation temperature ranges
  - If experiment has a wide variation of temperature variations, the thermal expansion will detect it compared to the resistivity method of detecting the lowest irradiation temperature

SiC Rod annealed in Optical Dilatometer

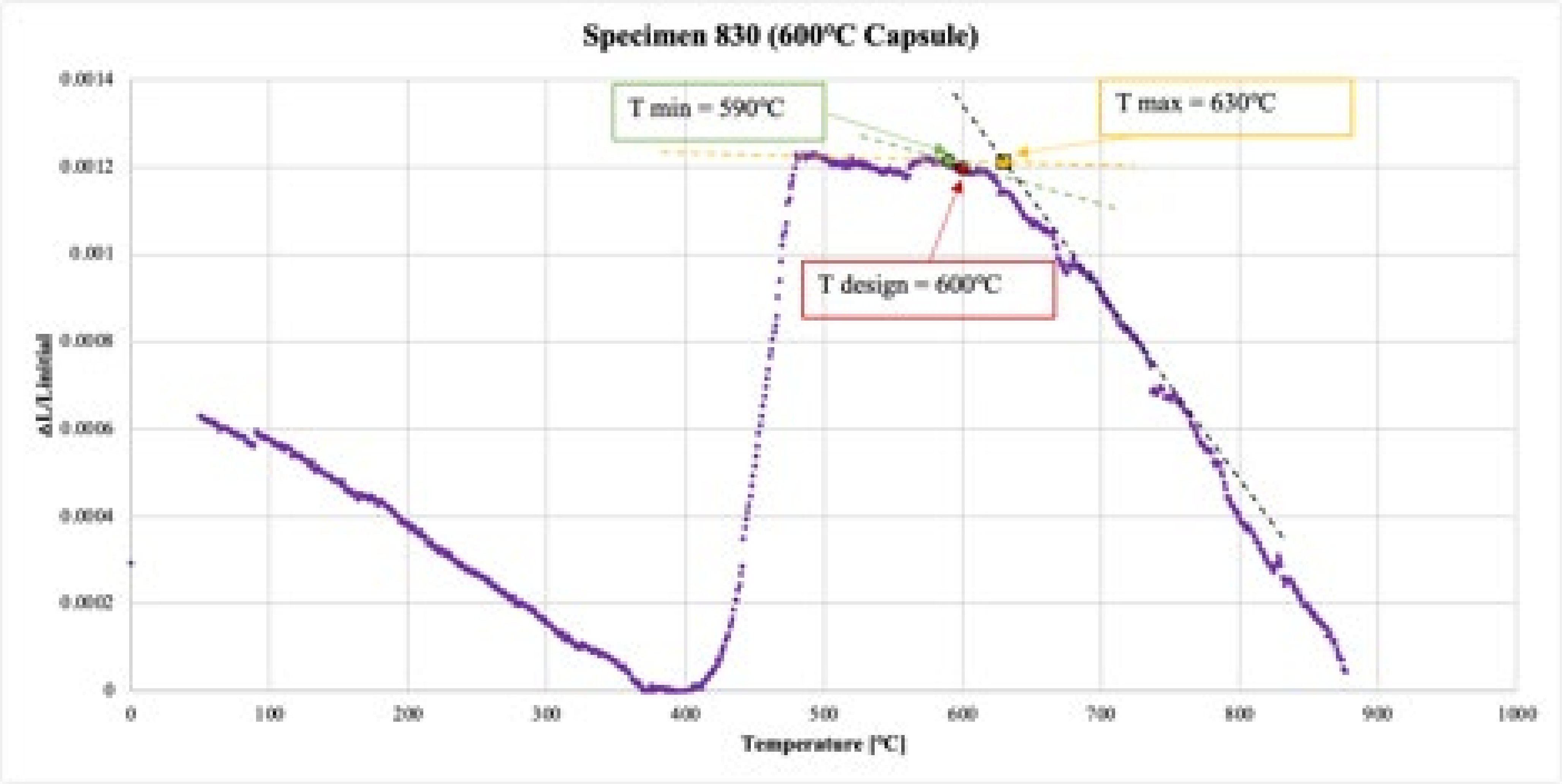


- ATR has many positions that vary throughout each cycle with temperature
- Not a single SiC will have same results - no program to analyze without introducing error or faulty results

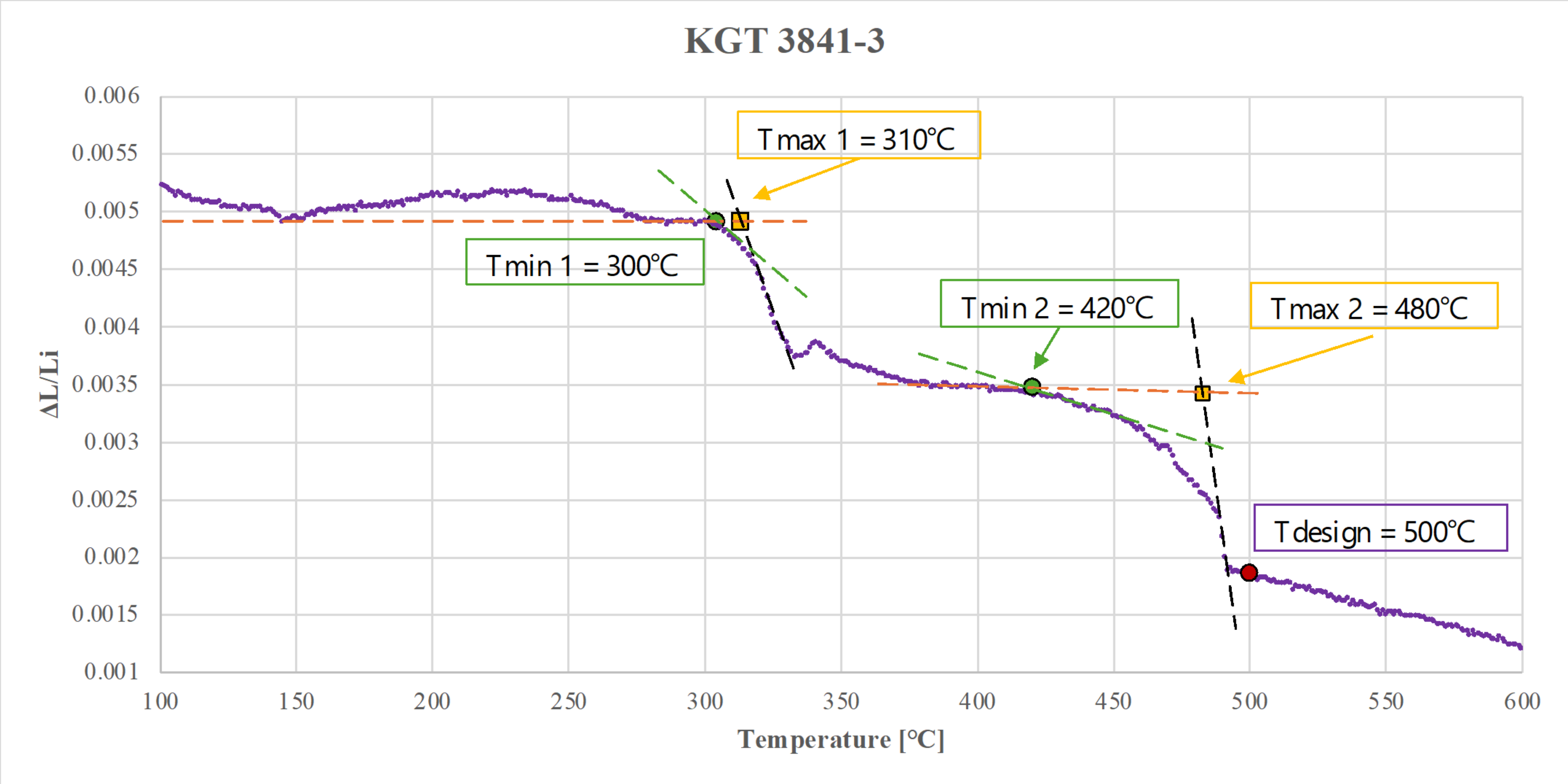
SiC 3-mm Disc annealed in optical dilatometer



Broken SiC rod



Wide Temperature Variations during Neutron Irradiation



- Were able to detect two temp. ranges, one closer to 300℃ and second closer to 500℃ design temp.
- This capsule was placed in the middle between two target temperatures (300℃ and 500℃)

SiC swelling

- Increase in volume due to radiation-induced swelling
- Little change in the crystal volume is expected at annealing temperatures lower than the irradiation temperature
- Dramatic decrease in volume is expected when the annealing temperature surpasses the irradiation temperature
- Averaged anywhere between 40 µm and 100 µm swelling in rods and averaged 30 µ for 3-mm discs
- Averaged shrinkage from PIE annealing as little as 30 µm to 50 µm for rods and 10 µm for 3-mm discs
- Critical to include enough space in capsule design to avoid sensor getting stuck

Limitations

- Temperatures are inferred by post-irradiation detection of changes in material properties:
  - If there is a power outage to the equipment during your annealing process – the results are lost (you can only anneal the point defects once)
  - Ideal sensor for steady-state power -> if more temp. fluctuations the more difficult to determine peak irradiation temp. and higher error
  - It will not detect irradiation temperatures that had lower than 0.1 dpa of neutron damage (i.e., spikes of power/temperature during irradiation)

ASTM Considerations

1. Materials and sourcing (Rohm-Haas material and PremaTech or other options?)
2. Physical dimensions (rods, pillars, or discs) – what's the smallest and largest volume allowed
3. Shipping and handling – how to properly decontanimate and process these in PIE
4. PIE measurement methods (lattice spacing, **dimensions, electrical resistivity**, thermal diffusivity, or bulk density)
5. Experimental conditions (max. and min. temperatures and neutron damage rates in dpa)
6. Disposal (how to properly dispose of the rad materials)

Conclusions

- Successfully deployed SiC temperature monitors for irradiation at ATR, MITR and HFIR
- Resistivity method was the most used processing method for SiC TMs generating results within +/- 20℃
  - It's not automated, so it can take anywhere between 1-3 weeks to process one SiC TM
  - Oxidation is an issue during processing as the environment is not inert, that can alter and induce error in the measurement
  - Can only process SiC pillars and rods (at least 10 mm long)
- Dilatometry is the second most used processing method that also produces results within +/- 20℃
  - Fully automated process that only requires 2-3 days of processing time
  - Optical dilatometer can process any type of SiC (rods, pillar or disks)
  - No oxidation issues due to inert or vacuum environment

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