Silicon Carbide (SiC) Temperature Measurements in NSUF Experiments

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Introduction

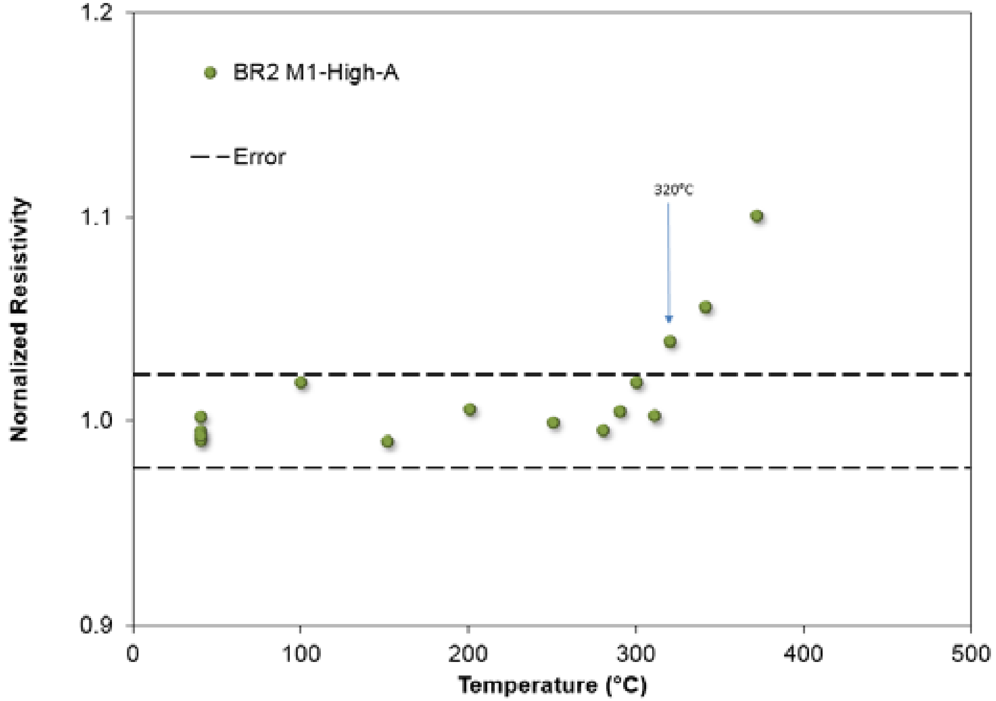
- Provide a practical and robust approach to estimate peak irradiation temperature during PIE for direct integration in irradiation test designs:
  - Less expensive static (drop-in) capsule tests with no leads will essentially be the only possibility for peak temperature indication
  - Determine a peak temperature reached within a relatively broad range (200 – 1200°C) resulting in accuracies within ±20°C
  - Neutron irradiation induced lattice expansion of SiC annealed out when the post-irradiation annealing temperature exceeds the peak irradiation temperature
  - Property change (lattice spacing, **dimensions, electrical resistivity**, thermal diffusivity, or bulk density)
- Joined collaboration between NSUF and Advanced Sensors and Instrumentation (ASI) Program

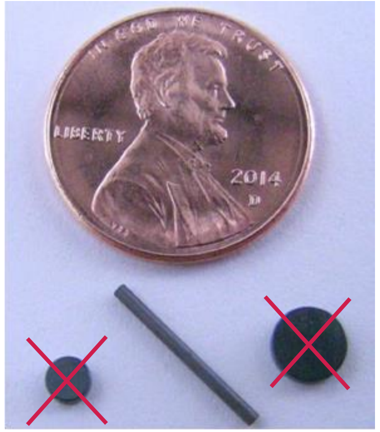
SiC Material

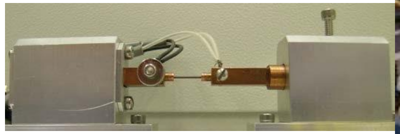
- SiC rod (1 mm OD by 12.5 mm) and disks (OD 3 mm or 5 mm by 1 mm)
  - Procured from PremaTech Advanced Ceramics
  - Follow Rohm and Haas specification sheet:
    - chemical vapor deposition (CVD) SiC
    - high resistivity grade (>1000 Ω cm)
    - highly pure (99.9995%) with theoretically density and 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

Resistivity Method

- Environment Temperatures in Air: 200 – 800°C
- Recommended dose ranging from 0.5 – 8 dpa
- Electrical resistivity is accepted as a robust measurement technique resulting in accuracies within 20°C
- Very time and labor intensive (1 week to 3 week per sample)
- Can only process SiC sticks



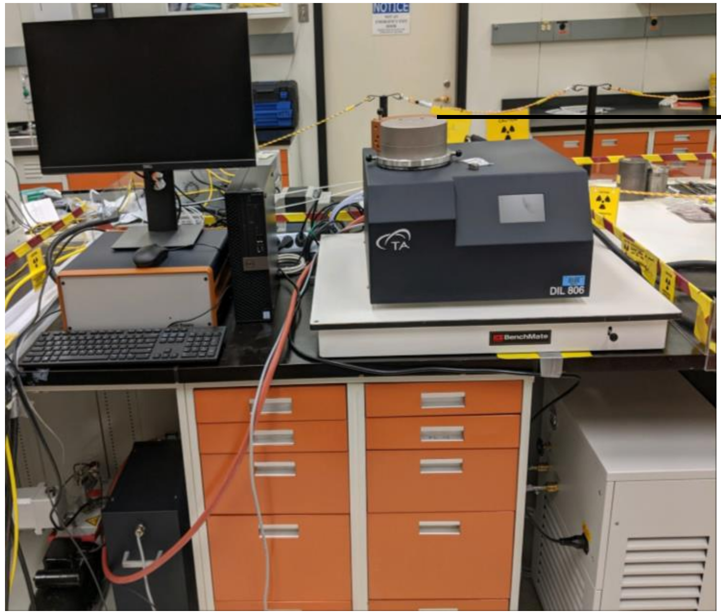


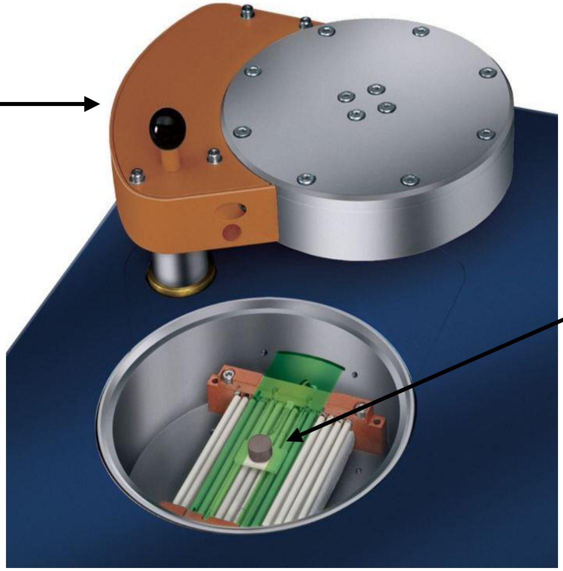


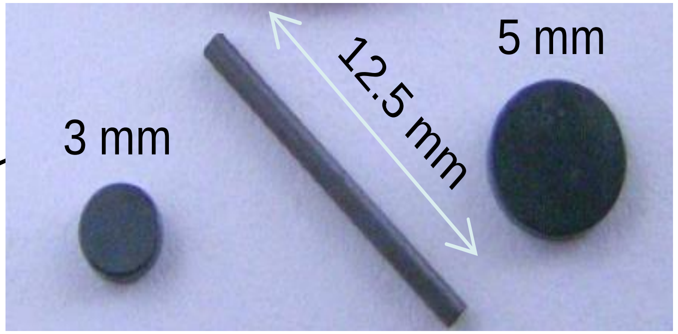
Spring loaded resistance measurement fixture. The SiC monitor is held in place between the two copper electrodes.

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 (accuracies +/-20℃)
- 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 (Resistance Method cannot process disks)







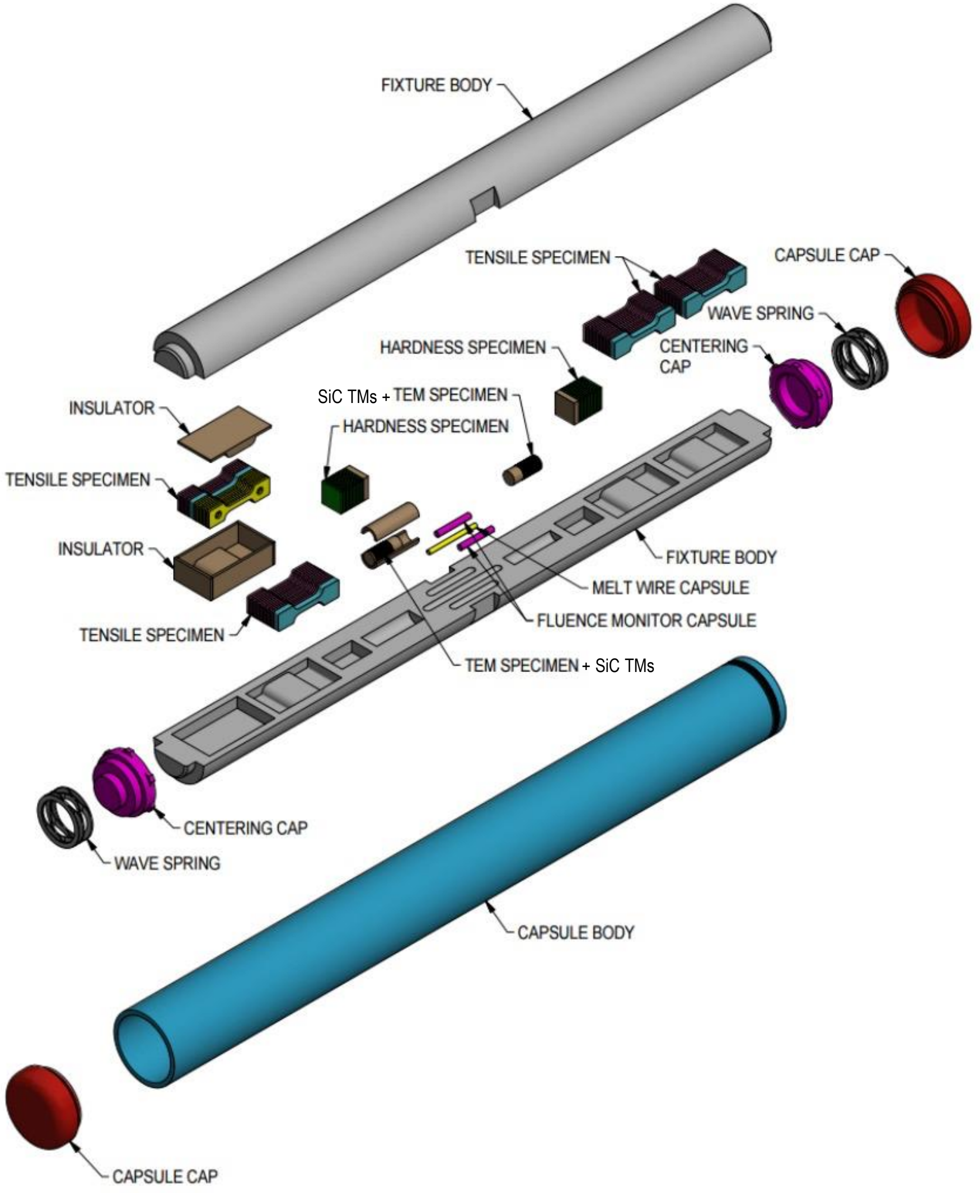
Dilatometer Advantages

- 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

NSUF ISU N-SERT Experiment

- Awarded under NSUF number ISU-10537
- Collaborated with ISU - Dr. Haiming Wen as the principal investigator
- Objective: Irradiation performance of ultrafine-grained and nanocrystalline variations of reactor structural and cladding steels produced using severe plastic deformation manufacturing techniques

Capsule Number	Design Temperature [℃]	Exposure [dpa]	Capsule Position in ATR
1	300 +/−50	2 +/−10%	Top
2	300 +/−50	6 +/−10%	Middle Top
3	500 +/−50	6 +/−10%	Middle Bottom
4	500 +/−50	2 +/−10%	Bottom

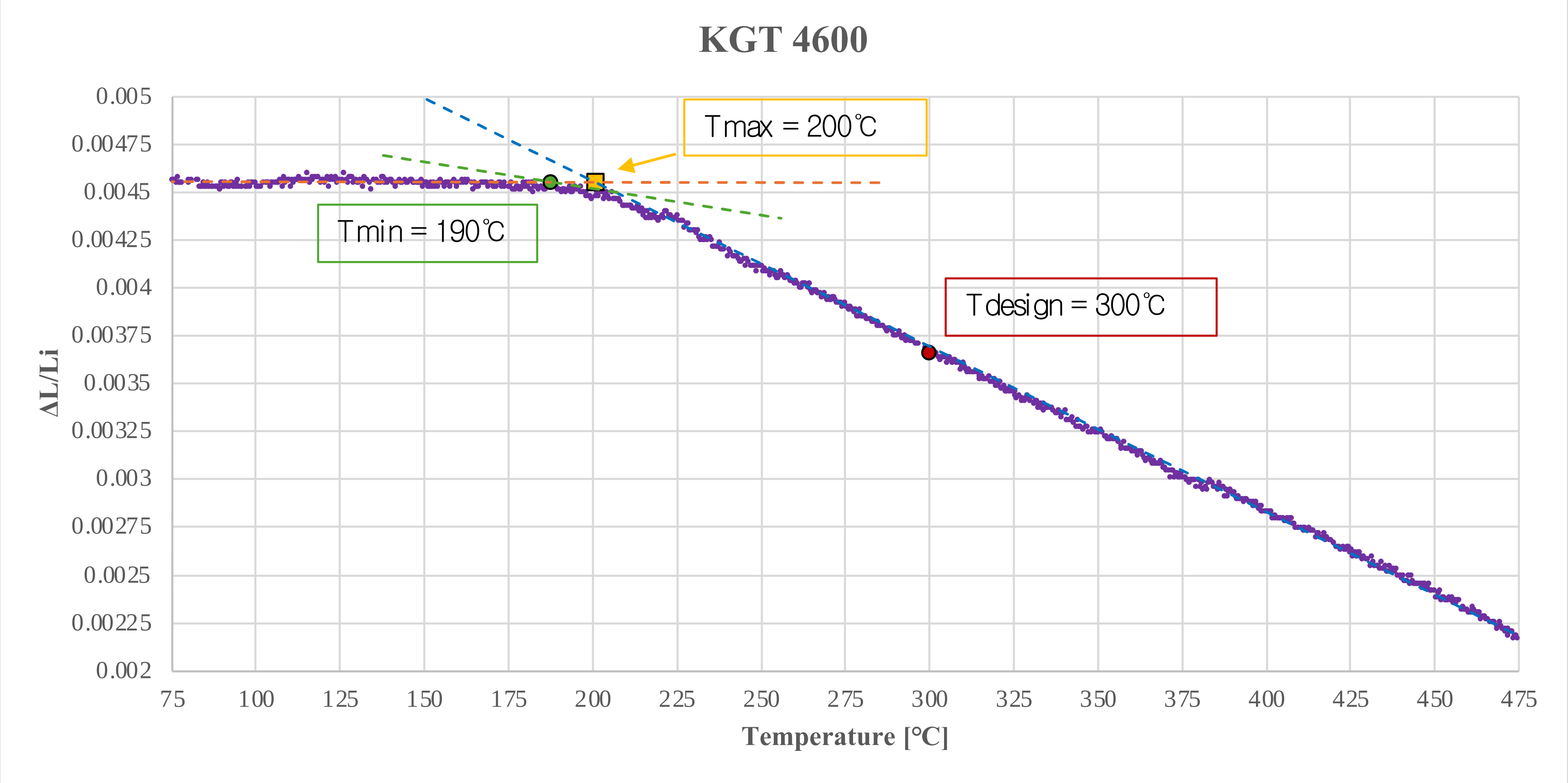


SiC temperature Monitors

- Total of 8 SiC TMs
  - Each capsule had two SiC TMs (placed between TEM specimens)
  - Provided thermal model estimates for each SiC location in the capsule

SiC Number	MSL Identification	HFEF Identification	Temperature Estimates for SiC [℃]		
			T min	T max	Avg
1	HTTL-244L2-R1	KGT 3828-1	336	337	336.5
2	HTTL-243U1	KGT 3828-2	333	335	334
3	HTTL-242L1-R1	KGT 4600	345	347	346
4	HTTL-244-U2	KGT 4609	341	343	342
5	HTTL-243L1-R1	KGT 4639-C	506	507	506.5
6	HTTL-243L2	KGT 4639-D	499	501	500
7	HTTL-241L2	KGT-3841-3	517	519	518
8	HTTL-242U2	KGT-3841-4	509	511	510

SiC TMs annealed in Optical Dilatometer



- Thermal expansion from continuous dilatometry used to calculate the SiC irradiation temperature
- Annealing Max. Temp: 800 C
- Design/Target Temp: 300℃
- Irradiation temperature measured using the difference of the SiC length change between heating and cooling
- Peak irradiation temp. was 200℃ +/- 20℃
- The steady-state irradiation temperature ranged between 190℃ and 200℃

SiC TM Results

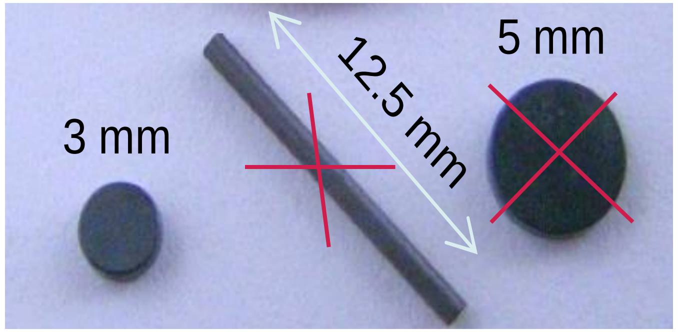
- All 8 SiC were successfully processed in the optical dilatometer
  - All but 3 SiC TMs met the target/design temperature ranges
  - All thermal temperature predictions were overestimated

HFEF Identification	Design Temp. [℃]	Thermal Model Avg. Temp. [℃]	Avg. Peak Irradiation Temp. [℃]	Met Design Temp.	Met Thermal Model Avg. Temp.
KGT 3828-1	300+/-50	336.5	270+/-20	YES	Below
KGT 3828-2	300+/-50	334	230+/-20	YES	Below
KGT 4600	300+/-50	346	200+/-20	Below	Below
KGT 4609	300+/-50	342	300+/-20	YES	Below
KGT 4639-C	500+/-50	506.5	440+/-20	YES	Below
KGT 4639-D	500+/-50	500	280+/-20	Below	Below
KGT-3841-3	500+/-50	518	480+/-20	YES	Below
KGT 3841-4	500+/-50	510	410+/-20	Below	Below

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 27.9 µm neutron-induced swelling & 11.2 µm post annealing shrinkage
  - Critical to include enough space in capsule design to avoid sensor getting stuck

SiC Number	Diameter [µm]				
	Pre-Irradiation	Post-Irradiation	ΔOD	Post-Annealing	ΔOD
1	3000	3030.8	30.8	3020.3	-10.5
2	3000	3031.4	31.4	3018.3	-13.1
3	3000	3031.4	31.4	3017.3	-14.1
4	3000	3023.2	23.2	3009.9	-13.3
5	3000	3021.7	21.7	3016.1	-5.6
6	3000	3027.5	27.5	3017.6	-9.9
7	3000	3030.8	30.8	3013.4	-17.4
8	3000	3026.6	26.6	3020.8	-5.8
Average	**3000.0**	**3027.9**	**27.9**	**3016.7**	**-11.2**



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)

Conclusions

- Optical dilatometer can successfully process SiC sticks and disks
- No oxidation issues (inert or vacuum chamber)
- All but 3 SiC TMs met the design/target temperature ranges
- All thermal temperature predictions were overestimated

Future Work

- Work with NSUF on creating a road map for using passive sensors in irradiation experiments
- Attend "Best Engineering Practices" event at ORNL to start the process of creating an ASTM standard for using and processing SiC TMs

- Technical details can be found in "Assessment of Readout Techniques for Passive Monitors" by M. Wilding, Summary Report, March 2024
- E-mail: Malwina Wilding at Malwina.wilding@inl.gov for details

Idaho National Laboratory

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