INL/MIS-19-54412-Revision-0

# Evaluation of Low Dose Silicon Carbide Temperature Monitors

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July 2019


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**Prepared for the**
**U.S. Department of Energy**

**Under DOE Idaho Operations Office**
**Contract DE-AC07-05ID14517**

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# Evaluation of Low Dose Silicon Carbide Temperature Monitors

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**June 2019**

# Introduction


Since the early 1960s, SiC has been used as a post-irradiation temperature monitor.

- Neutron irradiation induced lattice expansion of SiC anneals out when the post-irradiation annealing temperature exceeds the peak irradiation temperature
- Irradiation temperature is determined by measuring a property change after isochronal annealing or during a continuously monitored annealing process
- There are many properties that may be measured including, electrical resistivity (method used), density, thermal diffusivity, lattice spacing, and thermal expansion.
- Peak irradiation temperatures 150 – 800°C
- Recommended dose 1 – 8 dpa
- Irradiation testing completed March 2017 electrical resistivity evaluations started March 2018 completed July 2018.

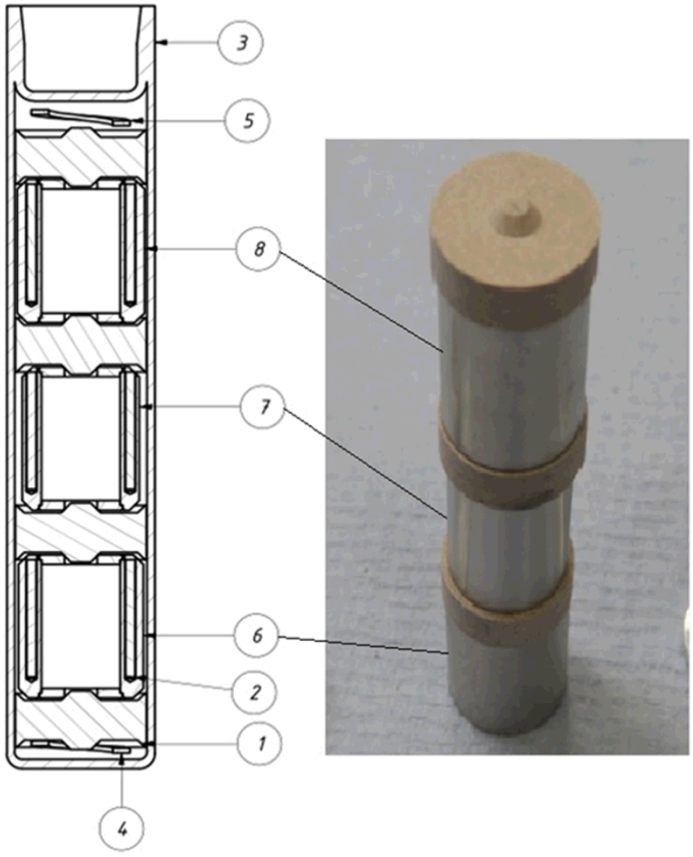
# Introduction

| | Measurement Accuracy | Calculated | Property Change @ 500°C Irradiation | Property Recovery above Irradiation Temperature (%/10°C anneal) | Recommended Sample Type | Technique Accuracy and Comments |
|---|---|---|---|---|---|---|
| **Dimensional Change** | 0.0003 mm | 0.5% | ~0.007 | ~3 | 12.7 mm L, 3.17 mm Diam Extreme \|\| Extreme Flatness | Measurement accuracy 12°C at 450°C 70°C at 700°C |
| **Electrical Resistivity (method used)** | 0.01-1 % | N?A | ~0.3 | ~0.03 | Bar 0.75 x 1 x Length | Within 20°C |
| **Thermal Diffusivity** | 1 – 5% | 90% | ~0.08 | ~13 | 2-4 mm L, 6-10 mm dia. | Within 40°C Good when length is limitation |
| **Density Gradient Column** | ~0.1% | 0.5% | ~0.021% | ~5 | Random, small | Within 30°C, Time consuming unless multiple samples irradiation. Good for small samples |
| **Lattice Spacing** | | | | | | Accuracy uncertain, Time consuming |

# Method


- Equipment used to evaluate the SiC temperature monitors.
  - Annealing furnace using isochronal temperature steps.
  - Resistivity measurement fixture located in the constant temperature chamber (maintained at 40°C) for a minimum of 30 minutes.
  - After the 30 minute wait time, each specimen's resistance is measured.

# Irradiation Capsule




(A)



(B)

- Basket for Material Irradiation (BAMI) Rig
  - (A) Cross-section of the aluminum irradiation capsule (3), stainless steel holders (6, 7 and 8) for SiC monitors (2). The holders are separated with ceramic discs (1) which are held in place with springs (4 and 5).
  - (B) Three holders and the three ceramic spacers stacked before irradiation.

# Calculated Dose and Peak Irradiation Temperature

## Dose Calculation

- Flux MCNP and cross sections SPECTER (based on ATR flux)

$$DPA_{rate} = \int_{E_m}^{E_M} \sigma_d(E)\phi(E)dE$$

- Second calculation MCNPX using BR2 F4 tally cards
- Both calculations produced comparable dose.

## Thermal Analysis (2D)

- Analytic Solution

$$\frac{1}{r}\frac{d}{dr}\left(k_i r \frac{dT_i}{dr}\right) + q_i = 0, i = 1 \ldots n$$

where $i$ is the layer number, n = 4. The following boundary conditions were applied:

$$T_1(0) < \infty$$

$$T_i(r = r_i) = T_{i+1}(r = r_i)$$

$$k_i \frac{dT_i}{dr}(r = r_i) = k_{i+1}\frac{dT_{i+1}}{dr}(r = r_i)$$

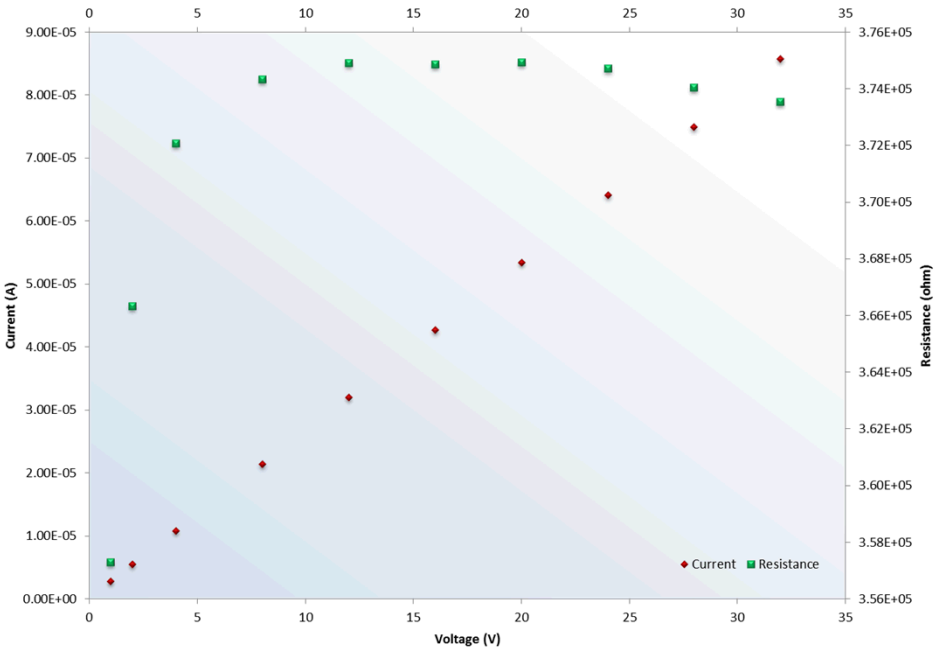
$$-k_n \frac{dT_n}{dr}(r = r_n) = h[T_n(r_n) - T_\infty]$$

where $T_\infty$ is the coolant temperature.

- Confirmed with ANSYS numerical simulations

| Monitor Identification | Dose (dpa) | Calculated Peak Irradiation Temperature (°C) |
|---|---|---|
| BR2 M1-Low-A | 0.5 | 255°C |
| BR2 M2-Low-A | 1.1 | 255°C |
| BR2 M1-High-A | 0.5 | 310°C |
| BR2 M2-High-A | 1.1 | 310°C |
| BR2 M1-Med-A | 0.5 | 410°C |
| BR2 M2-Med-A | 1.1 | 410°C |

# SiC Temperature Monitor Evaluations

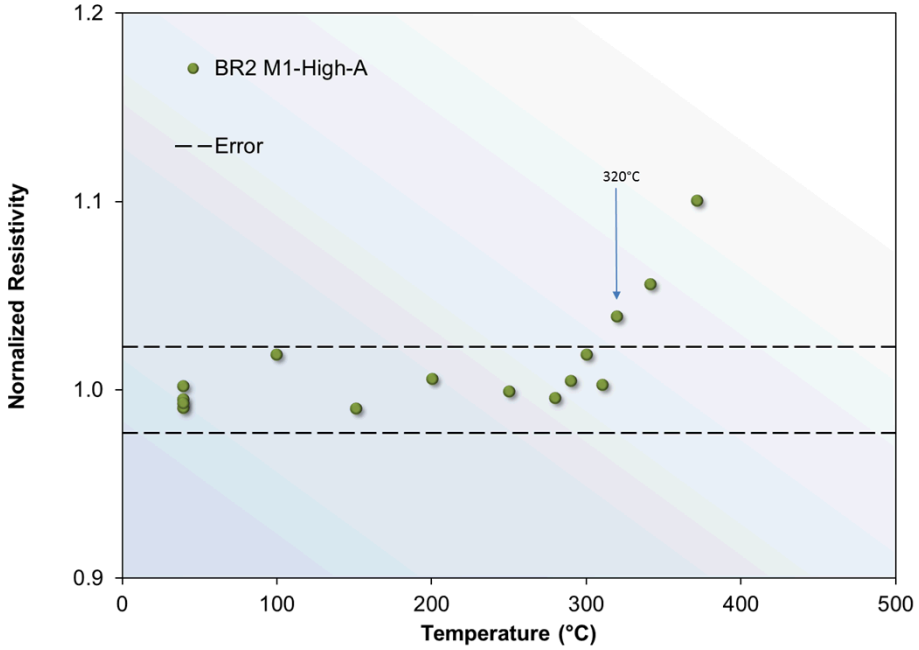



Typical ohmic response as demonstrated by SiC temperature monitor BR2 M1-High-A

## Ohmic response curve

- Generated for each monitor prior to heating
- Check for linearity.
- Select target voltage (with corresponding current) to minimize heating.
- Voltage ranged from 16 – 20V.

# SiC Temperature Monitor Evaluations




Typical peak irradiation temperature evaluation as demonstrated by SiC temperature monitor BR2 M1-High-A

## Peak irradiation temperature

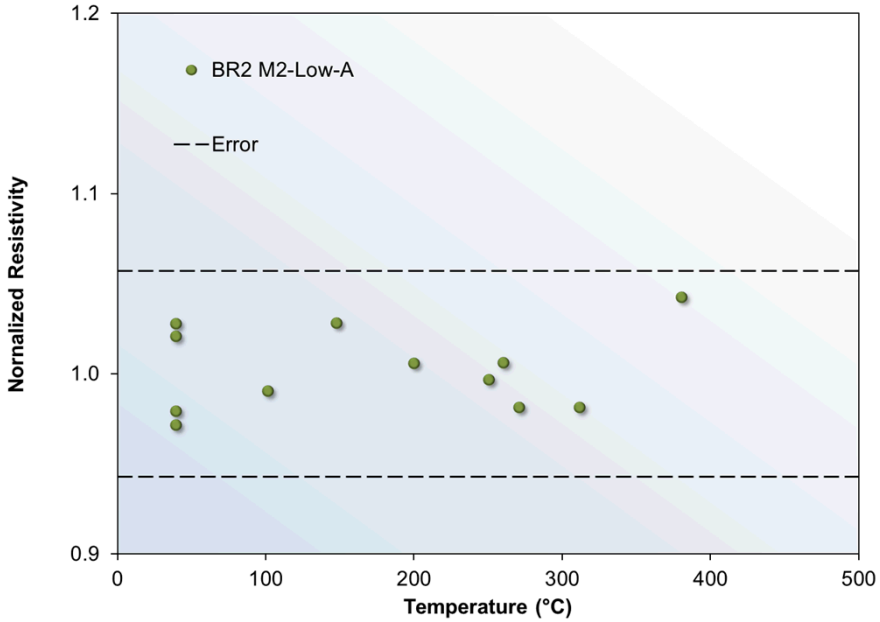
- Electrical resistivity technique
- Evaluation point where the resistivity begins, and consistently remains, above the error band (dotted lines)
- Error band established as ±2σ (based on first five data points taken below 150°C).

# SiC Temperature Monitor Evaluations

| ID | Measured | Calculated | Deviation (measured - calculated) | % Deviation | Initial resistivity at 40°C |
|---|---|---|---|---|---|
| BR2 M1-Low-A | 240°C | 255°C | -15°C | -6% | 20 (Ω-m) |
| BR2 M2-Low-A | Indeterminate | 255°C | n/a | n/a | 16 (Ω-m) |
| BR2 M1-High-A | 320°C | 310°C | 10°C | 3% | 24 (Ω-m) |
| BR2 M2-High-A | 330°C | 310°C | 20°C | 6% | 14 (Ω-m) |
| BR2 M1-Med-A | 390°C | 410°C | -20°C | -5% | 43 (Ω-m) |
| BR2 M2-Med-A | 380°C | 410°C | -30°C | -8% | 25 (Ω-m) |

- All of the monitors responded with the exception of the BR2 M2-Low-A.
- BR2 M2-Med-A outside of the expected deviation of 20°C

# SiC Temperature Monitor Evaluations

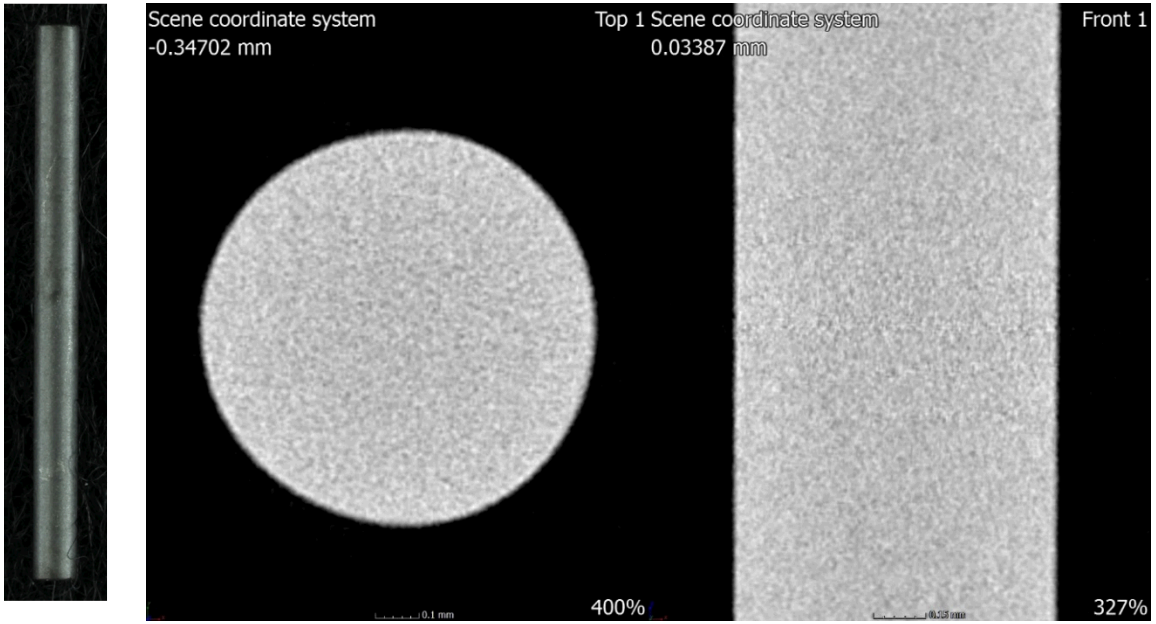



Evaluation data for BR2 M2-Low-A

BR2 M2-Low-A

- No response
- Lowest dose, 0.5 dpa
- Lowest temperature exposure (255°C).
- Very large error band

# 3D Micro-focus Computed Tomography




BR2 M2-Low-A

- Scan @50 kV, resolution of 6 µm
- Uniform density
- No voids or cracks
- Destructive examination next, including SEM

# Conclusion

- Successful evaluation of SiC temperature monitors irradiated in BR2 as part of an NSUF Project.
- Resistivity method proved robust.
- Low dose (0.5 dpa) SiC monitors may be evaluated within 240 – 380° C.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