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Benchmark Analysis for the Optical Dilatometry Method Using Silicon Carbide Temperature Monitors

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SUMMARY

The main objective of this project was to conduct a benchmark analysis for the optical dilatometry method by using NSUF's SiC temperature monitors: two (2) SiC temperature monitors provided by NSUF's BSU-8242 experiment and two (2) SiC temperature monitors provided by NSUF's GE-Hitachi experiment. Per the BSU-8242 experiment, KGT-3597 and KGT-3591 had a design temperature of 400°C and an exposure of 3 dpa. Per the GE-Hitachi experiment, KGT-3341 and KGT-3336 had a design temperature of 290°C +/- 50°C and an exposure of 0.5–1 dpa. The KGT-3336 SiC monitor was split into two pieces during the decontamination process, making the dilatometry method the only way to analyze both those pieces. The BSU-8242 monitors revealed peak irradiation temperatures under the design temperature, and the GE-Hitachi monitors revealed peak irradiation temperatures within the design temperature range. The optical dilatometry method measured the peak irradiation temperature of BSU-8242 KGT-3597 to be 330°C, while the resistivity method measured the peak irradiation temperature of BSU-8242 KGT-3591 to be 320°C +/- 20°C. The optical dilatometry method measured the peak irradiation temperatures of the pieces of GE-Hitachi KGT-3336 to be 260°C (for the larger piece) and 220°C (for the smaller piece), while the resistivity method measured the peak irradiation temperature of GE-Hitachi KGT-3341 to be 300°C, with an accuracy range of -50°C to +20°C. Both methods of SiC temperature monitor analysis produced very similar peak irradiation temperatures for each pair of SiC passive monitors from the two experiments, BSU-8242 and GE-Hitachi. The results show dilatometry method to be a reliable and less time-intensive process for determining irradiation temperatures from passive SiC thermometry.

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ACRONYMS

AM	additive manufactured
ATR	Advanced Test Reactor
BSU	Boise State University
GE	General Electric
MSL	Measurement Science Laboratory
NSUF	Nuclear Science User Facilities
PIE	post-irradiation examination
SiC	silicon carbide

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Benchmark Analysis for the Optical Dilatometry Method Using Silicon Carbide Temperature Monitors

1. INTRODUCTION

The Nuclear Science User Facilities (NSUF) is the U.S. Department of Energy, Office of Nuclear Energy's nuclear energy user facility. Its mission is to provide nuclear energy researchers with access to world-class capabilities, and to facilitate the advancement of nuclear science and technology. Boise State University (BSU)-8242 was a drop-in experiment irradiated in the Advanced Test Reactor (ATR) under the direction of the NSUF program. Weldability and inspectability issues continue to present challenges in the manufacture of nuclear reactor internal components. Components manufactured through powder metallurgy and hot isostatic pressing exhibit favorable structural uniformity, no chemical segregation, superior mechanical properties, and enhanced weldability. The BSU-8242 experiment was primarily aimed at building on existing research to demonstrate that alloys produced via powder metallurgy and hot isostatic pressing are suitable candidate structural materials for nuclear reactor technologies [1]. One way to measure irradiation temperature is via passive temperature monitors such as melt wires and silicon carbide (SiC) monitors. Figure 1 shows the placement of each passive temperature monitor inside the BSU-8242 capsule.

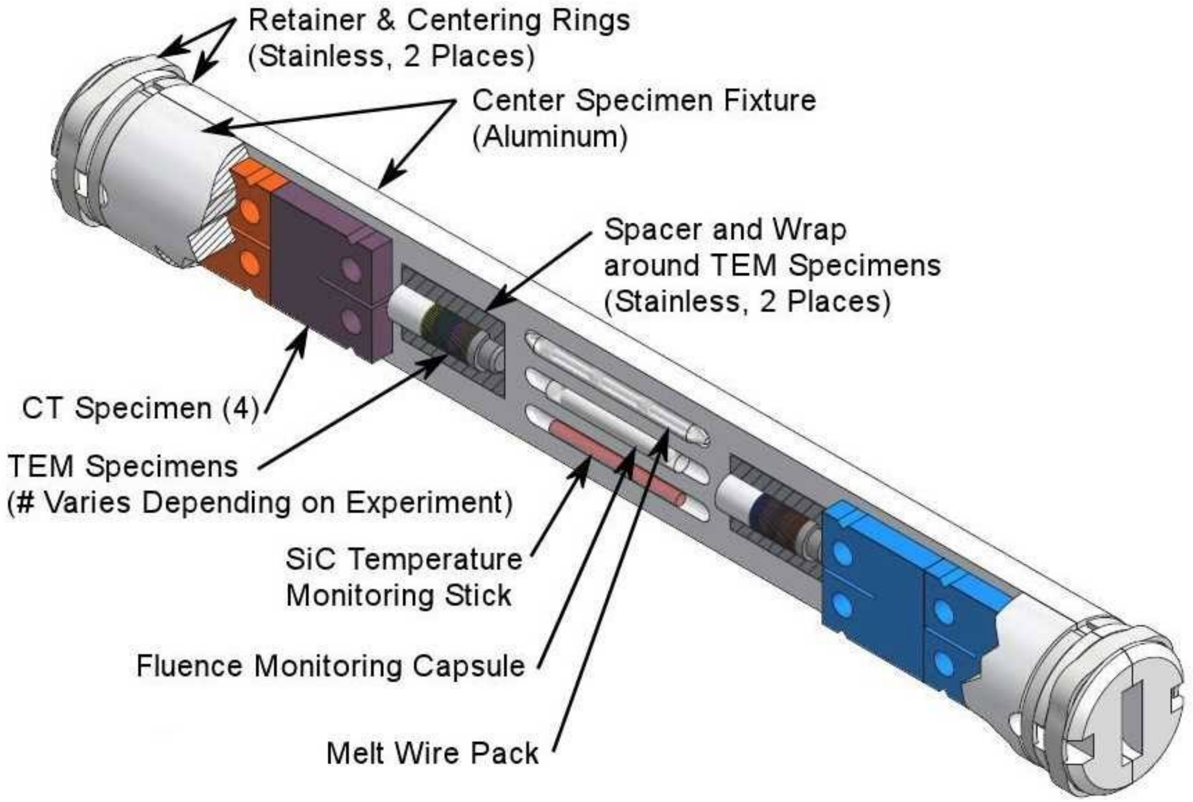

Figure 1. Typical BSU-8242 center specimen fixture, including the location of the SiC and melt-wire temperature monitors [1].

On the other hand, the General Electric (GE)-Hitachi experiment involved performing full irradiation and post-irradiation examination (PIE) on additive manufactured (AM) materials produced via direct metal laser melting. This experiment was a drop-in capsule irradiated in position B-11 of ATR and aimed to compare the properties of irradiated and unirradiated AM specimens [2]. The placement of each passive temperature monitor (melt wires and SiC) is shown in Figure 2.

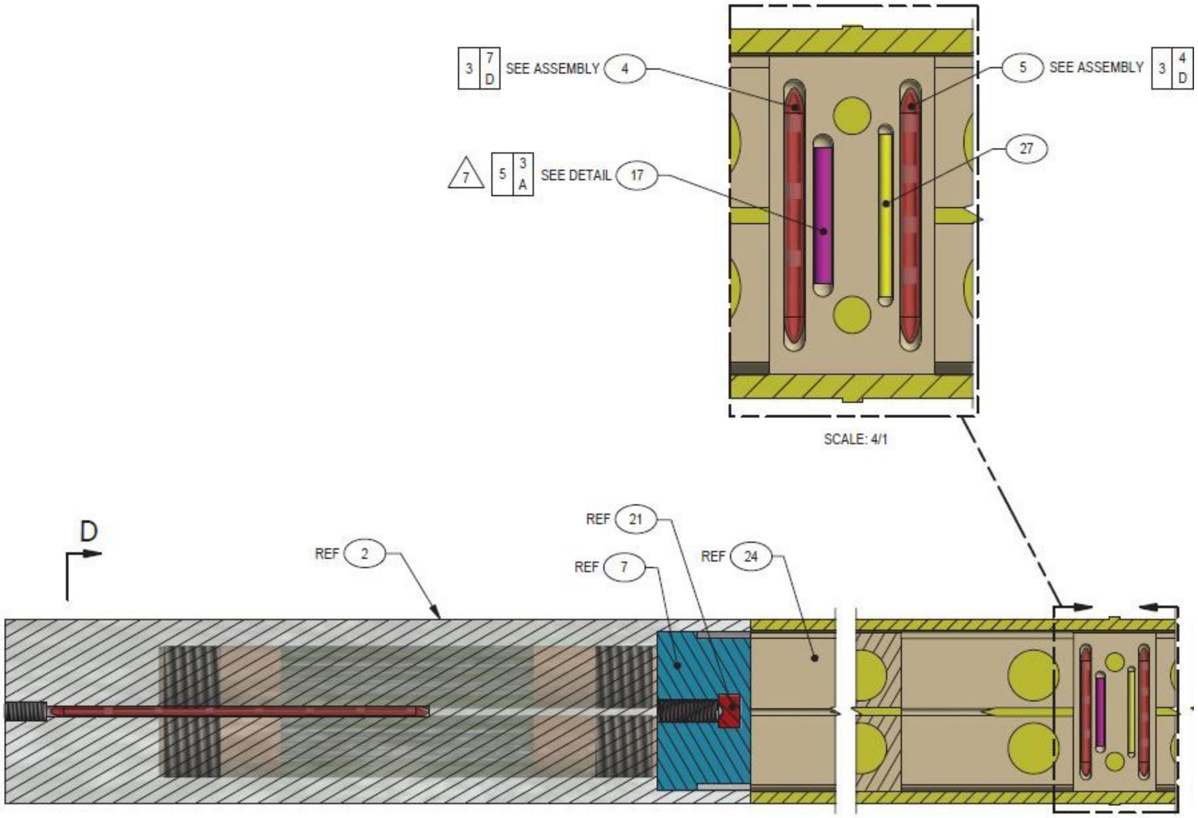

Figure 2. Graphical snip (the bottom end of the GE-Hitachi capsule to its middle) taken from Idaho National Laboratory drawing number 605754. The placement of each melt wire is shown in red (items 4 and 5), whereas that of the SiC monitor is shown in yellow (item 27) [3].

The SiC temperature monitors irradiated in the ATR were evaluated using both optical dilatometry and resistivity methods to determine peak temperatures during irradiation and to benchmark SiC monitors in terms of the dilatometry method. Benchmark analysis of the optical dilatometry method required a pair of SiC monitors, both irradiated under similar conditions. Thus, two pairs of SiC monitors (one from the BSU-8242 experiment and one from the GE-Hitachi experiment) were chosen for this project. The two SiC temperature monitors (i.e., one pair) had been irradiated as part of NSUF’s BSU-8242 experiment [1], at a target dose of 3 dpa and a target temperature of 400°C. Furthermore, two additional SiC temperature monitors had been irradiated as part of NSUF’s GE-Hitachi experiment [2], at a target dose of 0.5–1 dpa and a target temperature of 290°C +/- 50°C. These SiC temperature monitors are listed in Table 1.

Table 1. SiC temperature monitor MSL/HFEF identification names, experiment names, target temperatures, exposure levels, and evaluation methods.

Pair Number	Experiment	MSL Identification	HFEF Identification	Design Temp. (°C)	Exposure (dpa)	Evaluation Method
1	BSU-8242	HTTL-605771-10	KGT-3597	400	3	Dilatometry
		HTTL-605772-10	KGT-3591	400	3	Resistivity
2	GE-Hitachi	HTTL-GEH-BOT	KGT-3341	290	0.5-1	Resistivity
		HTTL-GEH-MID	KGT-3336	290	0.5-1	Dilatometry

2. BACKGROUND

Since the early 1960s, SiC has been used as a post-irradiation temperature monitor. Researchers observed that SiC’s neutron-irradiation-induced lattice expansion annealed out when the post-irradiation annealing temperature exceeded the SiC’s peak irradiation temperature [4]. Idaho National Laboratory has traditionally used resistivity measurements to obtain peak irradiation temperatures from SiC temperature monitors; however, significant efforts have been made to implement continuous optical dilatometry as a new automated evaluation method. The standard resistivity method involves repeated annealing of the SiC temperature monitors at incrementally increasing temperatures, with the resistivity measurements being taken after each annealing step. This process is very time consuming, potentially causes oxidization of the SiC temperature monitor, and requires near-constant attention from trained staff. Besides the expensive and lengthy post-irradiation analysis that is required, and which largely accounts for the total cost of using these otherwise inexpensive sensors, the current process adds many potential sources of measurement error, as the sensors must be repeatedly transferred back and forth between the furnace and the test fixture. Another consideration in this research is that automated post-processing of the SiC—such as is achieved by using the optical dilatometer—could significantly reduce the time needed to process each SiC post-irradiation [5].

The quality of the material used to manufacture the SiC temperature monitor has a major impact on its radiation-induced swelling and, thus, on the ensuing peak irradiation temperature evaluation. Temperature monitors were fabricated from material that meets the Rohm Haas specifications for grade SC003. This material was produced via a chemical vapor deposition process that enabled high purity (99.9995%) and an actual density equal to the theoretical density. Due to its polycrystalline β-cubic structure, the SiC material possesses isotropic characteristics. By taking advantage of these characteristics, the SiC monitors were manufactured to exceed a resistivity of 10 Ωm. The SiC monitors used in the experiment were manufactured as cylinders 1 mm in diameter and 12.5 mm in length (Figure 5) [6].

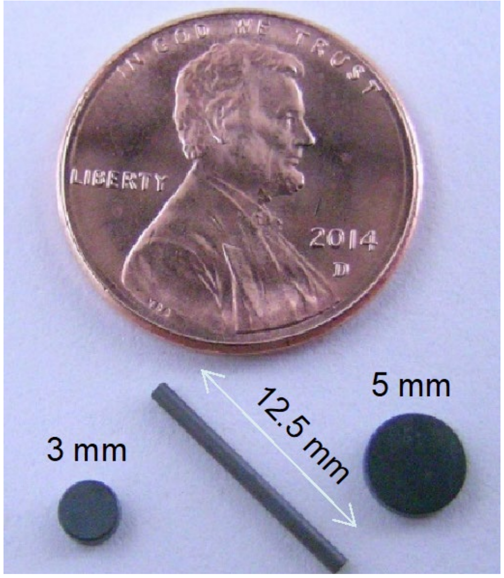

Figure 3. The SiC temperature monitors available for use in irradiation testing were comprised of small rods and discs. Only the rods were used for the purposes of this report [6].

3. METHODS

Irradiation temperature is determined by measuring a property change after isochronal annealing (i.e., lattice spacing, dimensions, electrical resistivity, thermal diffusivity, or bulk density). This work uses electrical resistivity and thermal expansion from dilatometry to calculate the SiC peak irradiation temperatures. The results show continuous optical dilatometry to be a reliable and less time-intensive process for determining irradiation temperature from passive SiC thermometry [5].

3.1 Resistivity

Electrical resistivity measurements were used to determine the peak irradiation temperatures of two of the four SiC temperature monitors. Figure 4 demonstrates the Measurements Science Laboratory (MSL) equipment used to evaluate the SiC temperature monitors, which were heated isochronally in the annealing furnace. The annealing temperatures were recorded using a National Institute Standards and Technology traceable calibrated thermocouple inserted into an alumina tube within the furnace. After each isochronal annealing, the specimens were placed in a specialized fixture designed for recording resistance measurements, then inserted into a constant temperature chamber (normally maintained at 40°C) for a minimum of 30 minutes. Afterward, the resistance was measured via a specialized fixture connected to a calibrated direct-current power analyzer (see Figure 4). Copper spring-loaded rods hold the SiC monitor in place. Each rod has a conical recess machined into one end to secure the SiC monitor in place. Electrical contacts are held in place with screws. A comprehensive description of the equipment and processes that were used can be found in [4] and [6].

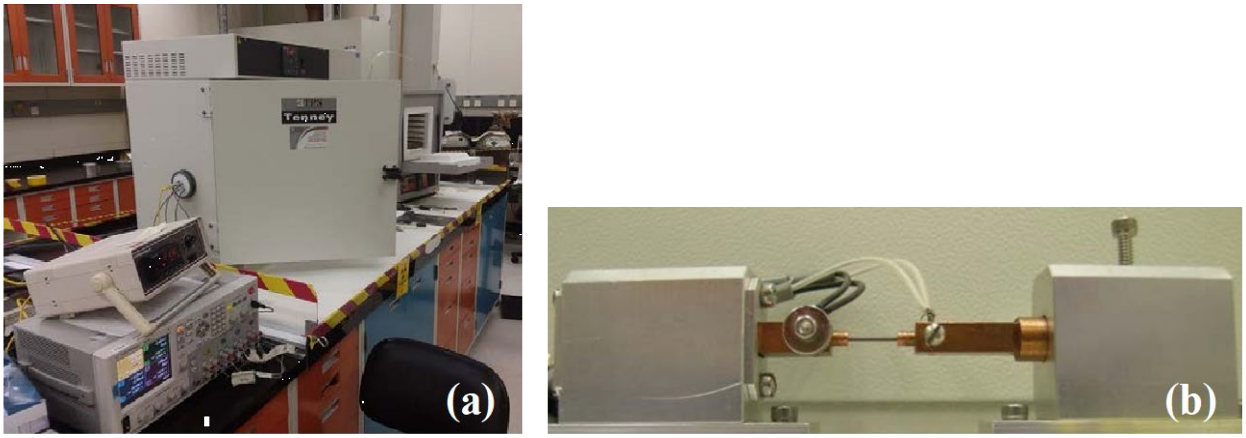

Figure 4. SiC resistivity measurement system: (a) annealing furnace and data acquisition system, and (b) spring-loaded resistance measurement fixture for SiC temperature monitors [6].

3.2 Optical Dilatometry

A TA Instruments DIL 806 optical dilatometer (Figure 5) was used for post-irradiation processing of the SiC temperature monitors to determine their peak irradiation temperatures. The DIL 806 is a contactless dilatometric measurement system that allows the SiC temperature monitor to freely expand/shrink without any interference from mechanical contact. This fosters a more precise determination of the passive monitor's dimensional changes and the temperature at which the changes are detected. Furthermore, avoidance of any load caused by contact with a measuring system enables the analysis to be extended well beyond the softening point into the melt. Additionally, the optical dilatometer offers effective environmental control during the testing, enabling users to analyze samples not only in air but also under a vacuum or in an inert atmosphere—a key requirement for avoiding any oxidization issues involving the SiC temperature monitors. A comprehensive description of the equipment and processes can be found in [4], [5], and [7].

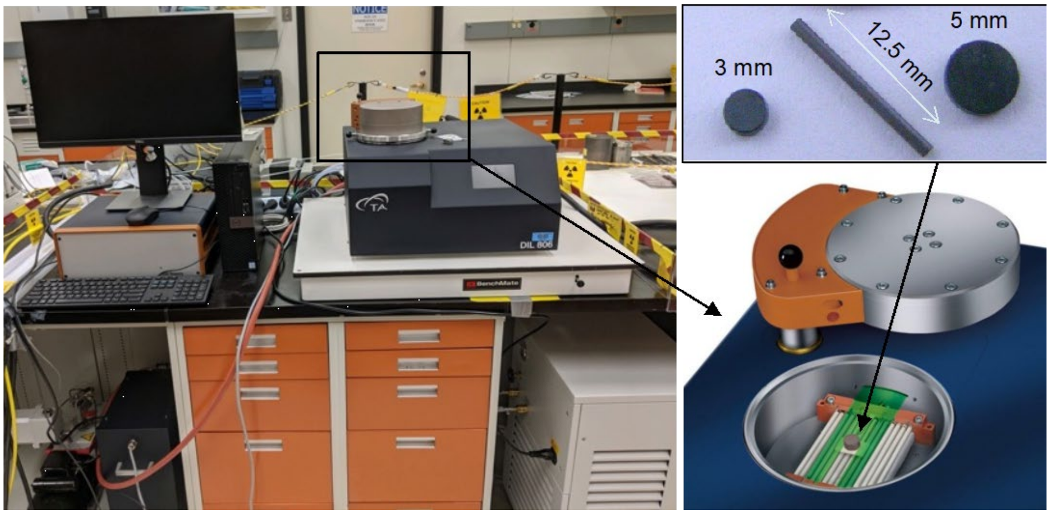

Figure 5. TA Instruments DIL 806 optical dilatometer, fully installed at the MSL [4].

4. RESISTIVITY EVALUATIONS

This section discusses and presents the results of the SiC monitor evaluation in which the resistivity measurement system was used. This work was conducted in accordance with an approved Evaluation Plan: 3473 [6]. An ohmic response curve was generated for each monitor prior to heating. Figure 6 shows a typical ohmic response. The data were used to check for linearity and to select a target voltage and corresponding current that would result in minimal heating of the SiC monitor during resistance testing but remain within the range of the test instrumentation.

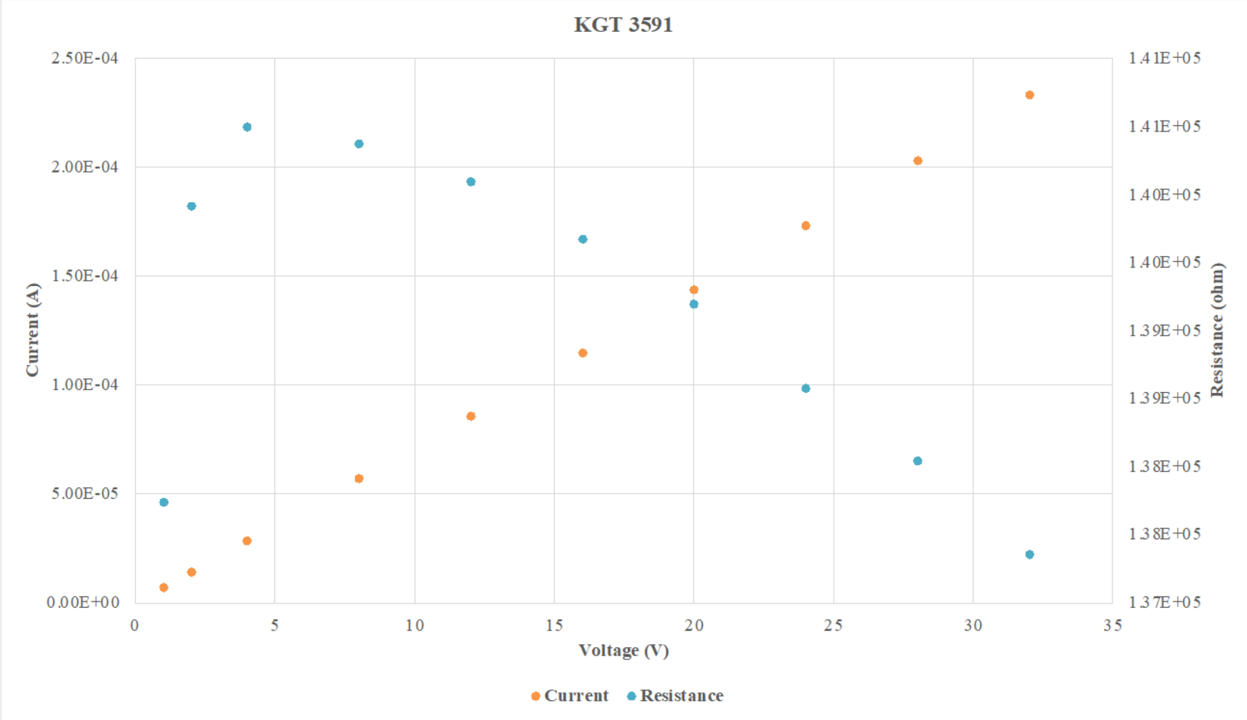

Figure 6. Typical ohmic response, demonstrated by BSU-8242 SiC temperature monitor KGT-3591.

Figure 7 shows the evaluation data for BSU-8242 SiC temperature monitor KGT-3591. The error band was based on a 2σ value calculated from the data points collected up to 100°C. The peak irradiation temperature for this monitor was evaluated to be 320° +/- 20°C.

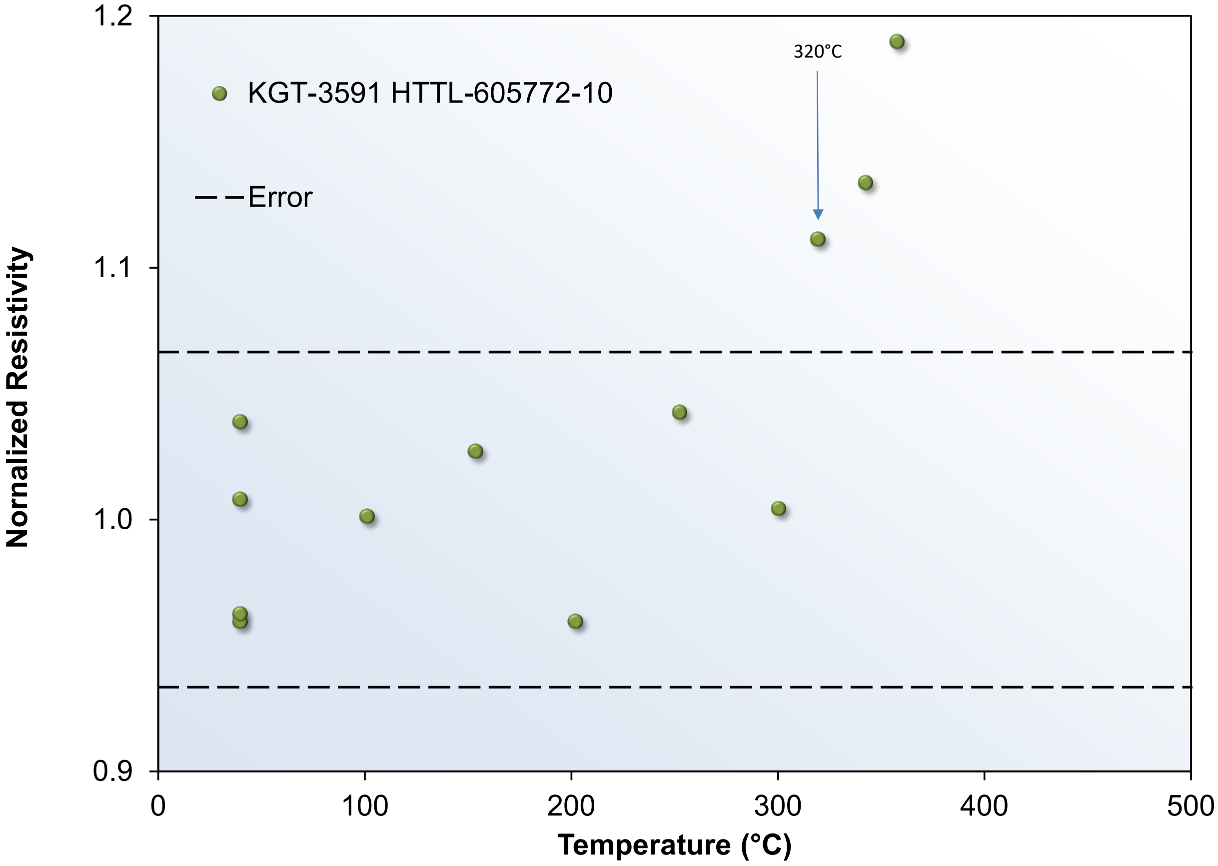

Figure 7. Evaluation of BSU-8242 SiC temperature monitor KGT-3591, using the resistivity method.

Figure 8 shows the evaluation data for GE-Hitachi SiC temperature monitor KGT-3341. The error band was based on a 2σ value calculated from the data points collected up to 100°C. The peak irradiation temperature for this monitor was evaluated to be 300°C, with an accuracy ranging between -50°C to +20°C. This peak temperature was lower than expected, therefore, larger isochronal heating steps were taken when the SiC temperature monitor responded—hence the lower error bound of -50°C.

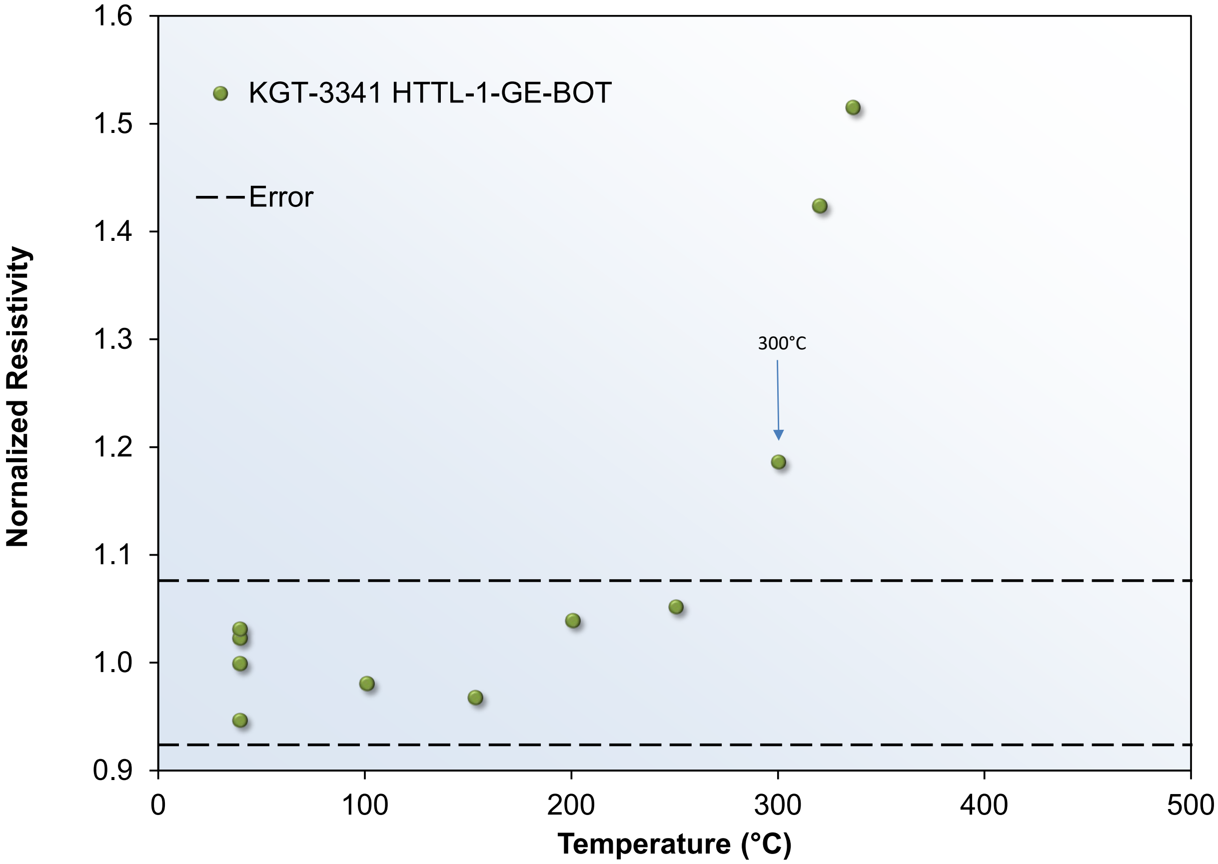

Figure 8. Evaluation of GE-Hitachi SiC temperature monitor KGT-3341, using the resistivity method.

5. DILATOMTERY EVALUATIONS

Each dilatometer run starts at room temperature and climbs—at a rate of 1°C/min—to at least 300°C above the expected irradiation (target) temperature, is held there for 5 minutes, and is then cooled back down to room temperature at a rate of 2.5°C/min. Hence, the optical dilatometer ran the remaining two NSUF SiC temperature monitors from room temperature to 800°C, held them there for 5 minutes, then cooled them back down to room temperature, as shown in Figure 9. The results in Figure 9 pertain to BSU-8242 SiC temperature monitor KGT-3597, revealing the furnace and sample temperature controls to be almost identical, with the furnace temperature being the chosen temperature control parameter for the optical dilatometer run. Additionally, the power output was mostly smooth across the whole run, as expected. All furnace temperature profiles were similar for each SiC monitor processed.

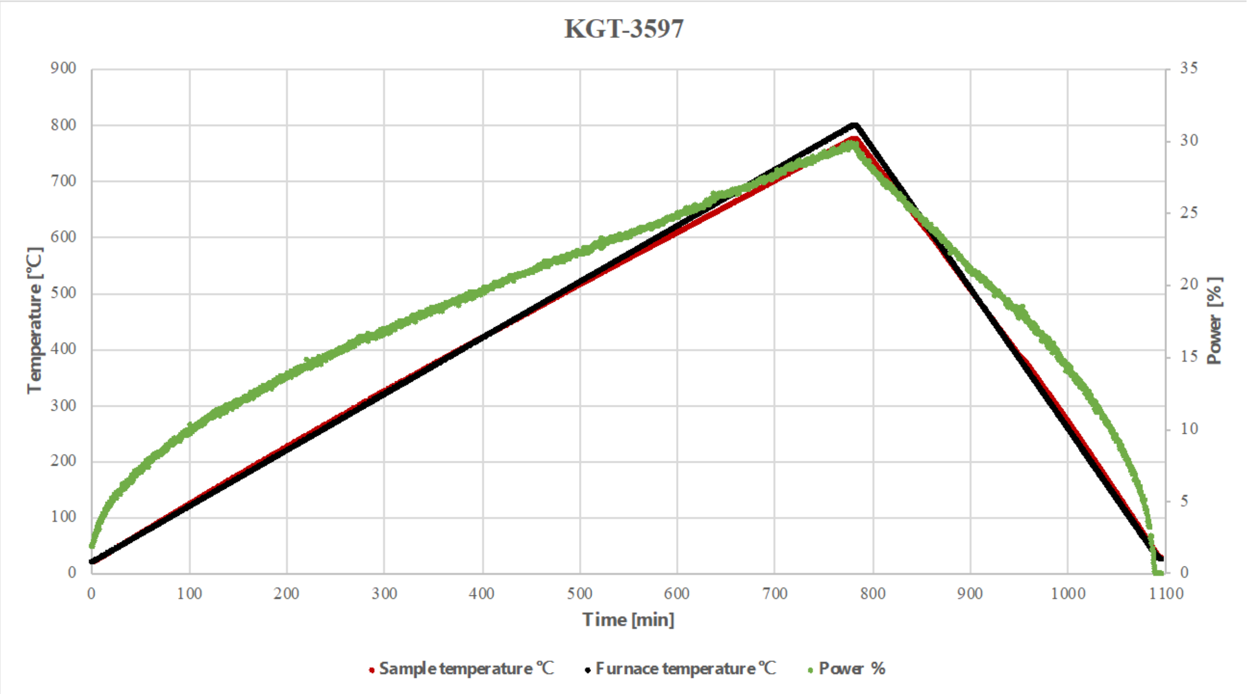

Figure 9. Temperature profiles of the furnace, sample controls, and power for BSU-8242 SiC temperature monitor KGT-3597.

SiC temperature monitors irradiated in the reactor will increase in volume due to radiation-induced swelling. Furthermore, there was little change in the crystal volume at annealing temperatures lower than the irradiation temperature, but a dramatic decrease in volume when the annealing temperature surpassed the irradiation temperature. This is well demonstrated in Table 2, with the SiC temperature monitors' lengths increasing post-irradiation by 92 µm for the BSU-8242 experiment and by 26 µm for the GE-Hitachi experiment, followed by a length decrease of 36 µm for the BSU-8242 experiment and of 52 µm for the GE-Hitachi experiment following the annealing process in the dilatometer.

Table 2. SiC temperature monitors' changes in length pre- and post-irradiation, and after annealing in the dilatometer (post-PIE).

Experiment	MSL Identification	HFEF Identification	Length (µm)				
			Pre-Irradiation	Post-Irradiation	Δ Length	Post-PIE	Δ Length
BSU-8242	HTTL-605771-10	KGT-3597	12500	12592.4	92.4	12556.3	-36.1
GE-Hitachi	HTTL-1-GEH-MID	KGT-3336	12556	12581.9	25.9	12529.7	-52.2

Figure 10 plots the difference between the heating and the cooling changes in length for BSU-8242 SiC temperature monitor KGT-3597, based on data collected from an optical dilatometer run. The design temperature for KGT-3597 was 400°C, with an exposure of 3 dpa. Figure 10 revealed the maximum peak irradiation temperature to be 330°C, and the minimum irradiation temperature to be 250°C. Furthermore, the peak irradiation temperature is 70°C below the design temperature, but only 10°C away from what the resistivity method revealed. However, visual inspection of the melt wire was inconclusive [3]. Thus, the resistivity and dilatometry methods revealed very similar irradiation temperatures for the two BSU-8242 SiC temperature monitors chosen, due to their having the same target temperature and neutron damage.

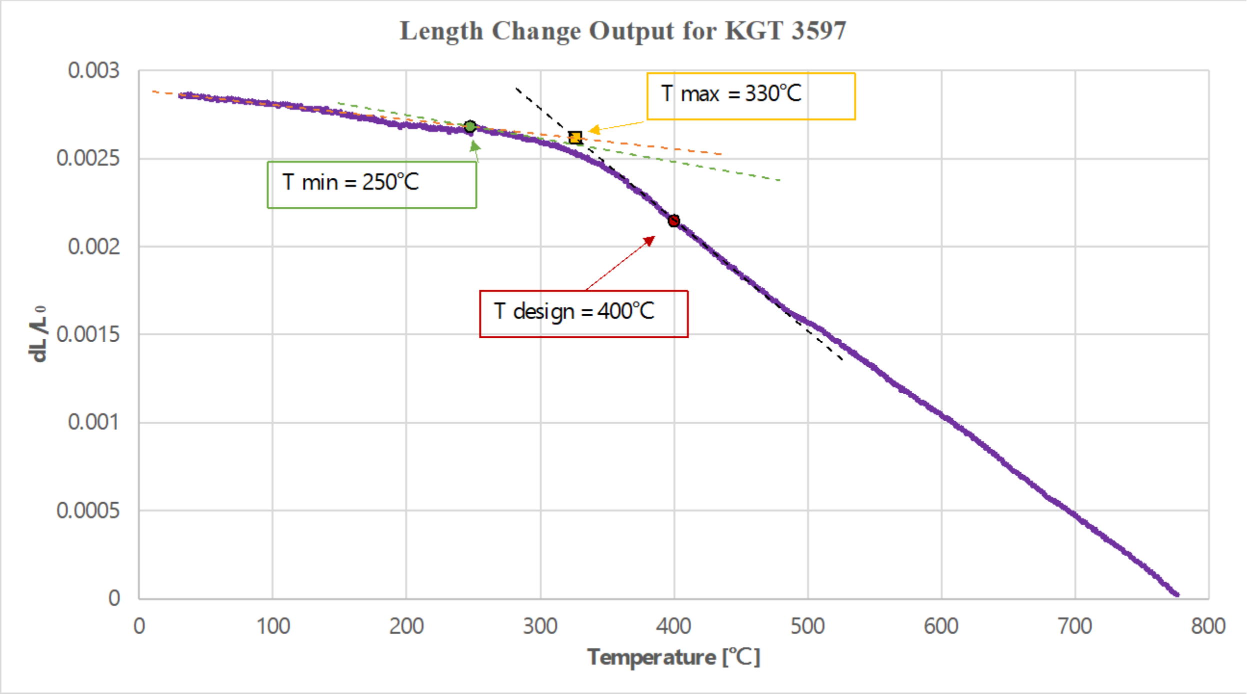

Figure 10. Irradiation temperatures determined based on the delta change in length and the design temperature of BSU-8242 SiC temperature monitor KGT-3597.

Figure 11 plots the difference between the heating and the cooling changes in length for the first (and longer) of the two pieces of GE-Hitachi SiC temperature monitor KGT-3336, based on data collected from an optical dilatometer run. The design temperature for KGT-3336 was 290°C +/- 50°C, with an exposure of 0.5–1 dpa. Figure 11 revealed the maximum peak irradiation temperature to be 260°C, and the minimum temperature to be 230°C. Furthermore, the peak irradiation temperature was 30°C below the design temperature, but still within its accuracies. Also, it is only 40°C away from what the resistivity method revealed, but still withing its accuracies. However, visual inspection of the melt wire revealed a temperature range of 327.5–399.4°C [3], which unfortunately are much higher to what the resistivity and dilatometry methods revealed for the first piece.

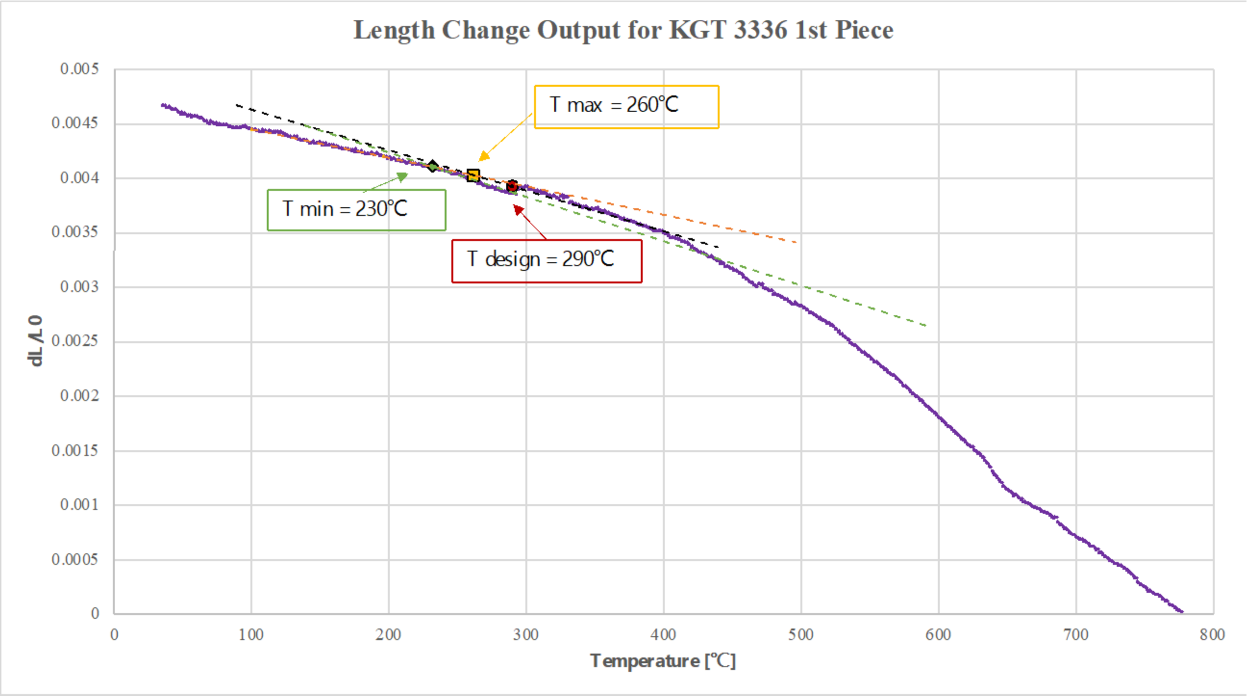

Figure 11. Irradiation temperatures based on the delta change in length and the design temperature of the first (and larger [8.13 mm in length]) piece of GE-Hitachi SiC temperature monitor KGT-3336.

Figure 12 plots the difference between the heating and the cooling changes in length for the second piece of GE-Hitachi SiC temperature monitor KGT-3336 (the second piece), based on data collected from an optical dilatometer run. The design temperature for KGT-3336 was 290°C +/- 50°C, with an exposure of 0.5–1 dpa. Figure 12 reveals the maximum peak irradiation temperature to be 220°C, and the minimum temperature to be 140°C. The smaller the SiC monitor piece, the higher the accuracy of the dilatometer measurements; therefore, this higher irradiation range is expected. Furthermore, the peak irradiation temperature is 70°C below the design temperature, and 80°C away from what the resistivity method revealed. Furthermore, visual inspection of the melt wire revealed a temperature range of 327.5–399.4°C [3], which unfortunately are much higher to what the resistivity and dilatometry methods revealed for the second piece.

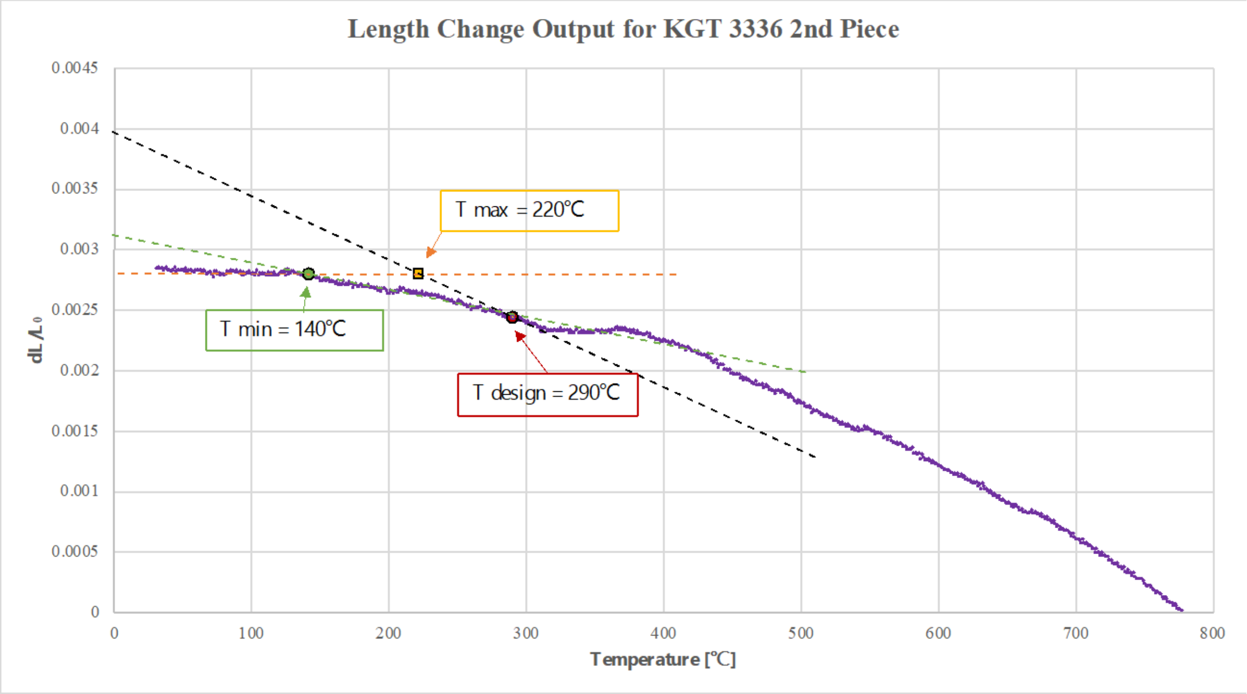

Figure 12. Irradiation temperatures based on the delta change in length, and the design temperature of the second (and smaller [4.46 mm in length]) piece of GE-Hitachi SiC temperature monitor KGT-3336.

6. CONCLUSION

This summary report reveals continuous optical dilatometry to be a valid method for measuring peak irradiation temperatures of passive SiC temperature monitors. The optical dilatometer utilizes an automated process, requires only a small amount of time to run, and is easy to use, thus saving valuable labor time in comparison to the traditional resistivity measurement method. The main objective of this project was to conduct a benchmark analysis for the optical dilatometry method by using NSUF's SiC temperature monitors: two (2) SiC temperature monitors provided by NSUF's BSU-8242 experiment and two (2) SiC temperature monitors provided by NSUF's GE-Hitachi experiment. Per the BSU-8242 experiment, KGT-3597 and KGT-3591 had a design temperature of 400°C and an exposure of 3 dpa. Per the GE-Hitachi experiment, KGT-3341 and KGT-3336 had a design temperature of 290°C +/- 50°C and an exposure of 0.5–1 dpa. The KGT-3336 SiC monitor was split into two pieces during the decontamination process, making the dilatometry method the only way to analyze both those pieces. The BSU-8242 monitors revealed peak irradiation temperatures under the design temperature, and the GE-Hitachi monitors revealed peak irradiation temperatures within the design temperature range. The optical dilatometry method measured the peak irradiation temperature of BSU-8242 KGT-3597 to be 330°C, while the resistivity method measured the peak irradiation temperature of BSU-8242 KGT-3591 to be 320°C +/- 20°C. The optical dilatometry method measured the peak irradiation temperatures of the pieces of GE-Hitachi KGT-3336 to be 260°C (for the larger piece) and 220°C (for the smaller piece), while the resistivity method measured the peak irradiation temperature of GE-Hitachi KGT-3341 to be 300°C, with an accuracy range of -50°C to +20°C. Both methods of SiC temperature monitor analysis produced very similar peak irradiation temperatures for each pair of SiC passive monitors from the two experiments, BSU-8242 and GE-Hitachi. Therefore, results show dilatometry method to be a reliable and less time-intensive process for determining irradiation temperatures from passive SiC thermometry.

7. REFERENCES

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