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Status of the Optical Dilatometer Method of Evaluating the Peak Irradiation Temperatures of SiC Passive Monitors

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SUMMARY

The main objective of this project was to conduct a comparative assessment between the optical dilatometer method and resistivity method, using all 10 SiC temperature monitors provided by two Nuclear Science User Facility experiments: BSU-8242 and General Electric Hitachi. Unfortunately, due to multiple delays in acquiring, shipping, and cleaning the SiC temperature monitors, the project was only able to process one (1) SiC temperature monitor during this period. SiC temperature monitor KGT-3357 was evaluated via the optical dilatometer method to determine its peak irradiation temperature. The KGT-3357 sample was from the BSU-8242 experiment and designed for a temperature of 300°C and an exposure of 1 dpa. The optical dilatometer measurements indicated that the KGT-3357 SiC temperature monitor's peak irradiation temperature range was 240–267°C, with sensitivity of approximately ±20°C. Additionally, this temperature range falls within the evaluated melt wire temperature range of 238.6–271.5°C. The remaining six (6) SiC temperature monitors from the BSU-8242 experiment and three (3) from the General Electric Hitachi experiment will be used in the future work to further validate the optical dilatometer method for measuring SiC peak irradiation temperatures.

ACKNOWLEDGEMENTS

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TABLE OF CONTENTS

FIGURES

TABLES

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ACRONYMS

BSU	Boise State University
SiC	Silicon carbide

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1. INTRODUCTION

A silicon carbide (SiC) temperature monitor irradiated in the Advanced Test Reactor was evaluated via the optical dilatometer method to determine its peak temperature during irradiation. This temperature monitor was irradiated as part of the Nuclear Science User Facility's Boise State University (BSU) 8242 experiment[1], with a target dose of 1 dpa and a target temperature of 300°C. The BSU-8242 experiment featured a drop-in design and was primarily intended to assess the viability of using alloys manufactured via powder metallurgy and hot isostatic pressing as materials in nuclear reactor components [1].

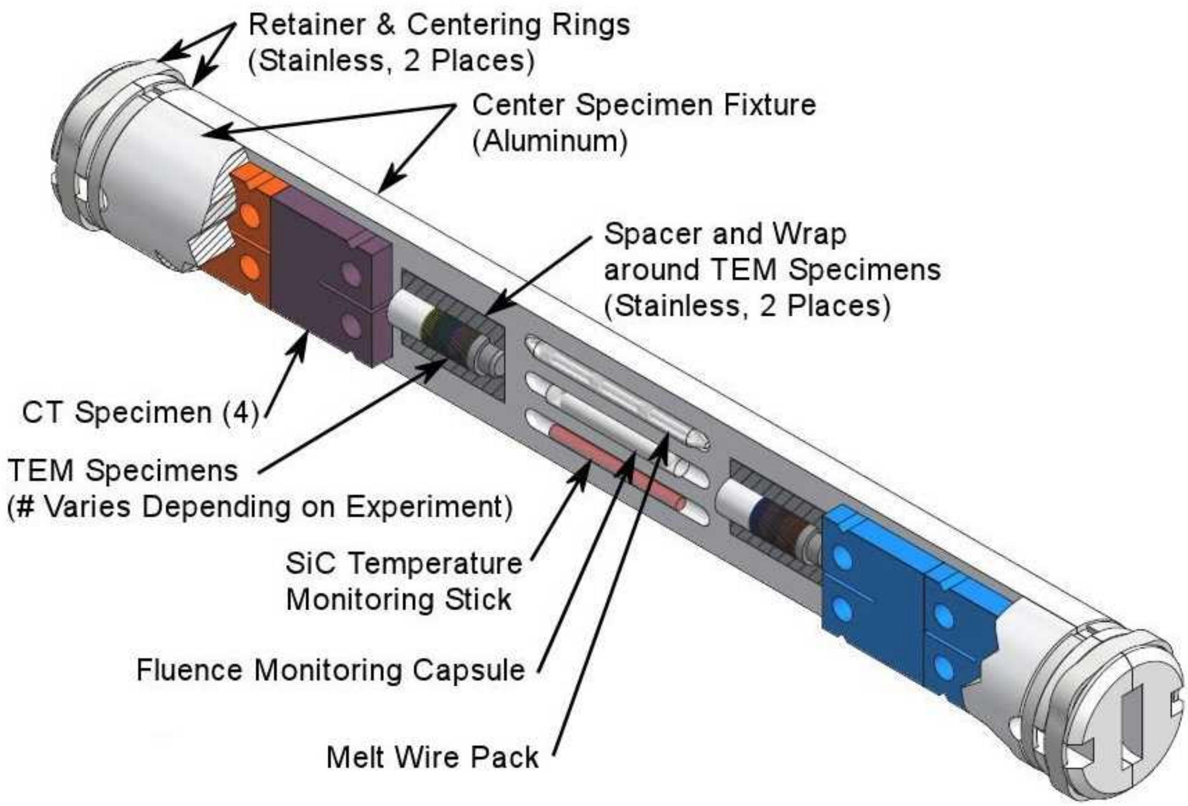

Figure 1. Typical BSU center specimen fixture, including the SiC temperature monitor location.[1]

Although, in instrumented lead tests, thermocouples typically provide the real-time temperature indications, other indicators (e.g., melt wires and SiC temperature monitors) are often included as secondary method for indicating peak temperatures experienced during irradiation. Less expensive static capsule tests, which have no leads attached for real-time data transmission, often rely only on melt wires and SiC temperature monitors as a post-irradiation technique for peak temperatures indication. A standard package of traditional melt wires typically contains 3 bulk materials. Each experiment has a projected (target) peak temperature, and all three-material selections are focused on that target temperature. The first material chosen has a melting point approximately 20 to 30˚C below the target temperature of an experiment. The second material selected melts at or near the target temperature, and the last material has a melting point that exceeds the experiment's target temperature by about 20 to 30˚C. However, melt wire package can only detect whether or not each material's melting temperature has been exceeded. Additionally, each melt wire package requires fabrication prior to any irradiation. SiC temperature monitors are advantageous because a single temperature monitor can be used to indicate the entire range of temperatures experienced during irradiation [2]. The SiC temperature monitors are ready to be inserted into any experiment from an INL's inventory of high-density chemical vapor deposition β-phase SiC material. The size and shape of the temperature monitors varies, whether rod- or disc-shaped, depending on the available internal dimensions of each experimental capsule. The BSU-8242 experiment used a rod-shaped SiC temperature monitor with a nominal size of 12.5 mm length x 1 mm diameter. Table 1 identifies (both pre- and post-irradiation) the SiC temperature monitor and gives the target temperature and evaluated melt wire temperature range.

Table 1. SiC temperature monitor identification, target temperature, and melt wire temperature range.

[redacted]

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 peak irradiation temperature [3]. Idaho National Laboratory has traditionally used resistivity measurements to gather peak irradiation temperature from SiC temperature monitors; however, significant efforts have been made to implement continuous optical dilatometry as the new automated evaluation method. The standard resistivity process involves repeated annealing of the SiC temperature monitors at incrementally increasing temperatures, with resistivity measurements taken after each annealing step. This process is very time consuming, could potentially result in oxidizing the SiC temperature monitor, and requires near-constant attention from trained staff. Besides the expensive, lengthy post-irradiation analysis required, the current process adds many potential sources of measurement error, as the sensor must be repeatedly transferred back and forth between the furnace and the test fixture. The aforementioned post-irradiation analysis largely accounts for the total cost of using these otherwise inexpensive sensors. An additional consideration made in this research is that, if the SiC post-processing was automated, such as it is when using the optical dilatometer, it could significantly reduce the time expense related to processing each SiC post-irradiation [4].

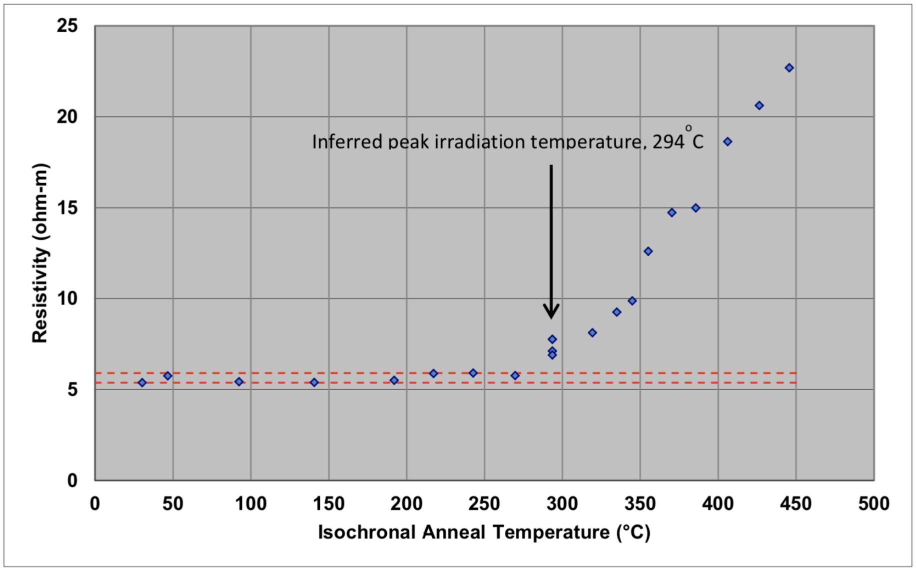

Figure 2. Typical electrical resistivity response for post-irradiation SiC monitor temperatures [4].

The curve in Figure 2 demonstrates resistivity-vs.-temperature behavior typical of the SiC resistivity method. The results in Figure 2 show that the peak irradiation temperature (294°C) is once the resistivity extend out beyond the band that corresponds to the maximum and minimum values measured at low temperatures and it continues to exponentially increase (or decrease) with increasing temperature. Comparisons between the temperatures gathered from SiC measurements and those inferred from the thermocouple data indicate that accuracies of approximately 20°C are possible for dose ranges of 1–8 dpa at temperatures ranging from 200°C to at least 800°C. The absolute temperature limits for resistivity techniques are typically considered to be 150°C (an amorphous threshold) and 875°C (due to recrystallization) [4].

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 presents how to use the thermal expansion from continuous optical dilatometry method to calculate the SiC peak irradiation temperature. This is an automated process that requires minimal setup time. The results show continuous optical dilatometry to be a reliable and less time-intensive process for determining irradiation temperature from passive SiC thermometry [3].

3.1 Optical Dilatometer

A TA Instruments DIL 806 optical dilatometer (Figure 3) was fully installed at the Measurements Science Lab to conduct post-irradiation processing of SiC temperature monitors to determine their peak irradiation temperatures. This instrument is a contactless dilatometric measurement system that allows the SiC temperature monitor to freely expand/shrink without any interference from mechanical contact. This allows a more precise determination of the passive monitor’s dimensional changes and the temperature at which these changes are detected. Furthermore, avoidance of any load resulting from 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 a test, 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. The optical dilatometer can process passive monitors 0.3–30 mm in length, with a maximum height of 10 mm. The maximum temperature for this model is 1400°C, with a temperature resolution of 0.1°C [5].

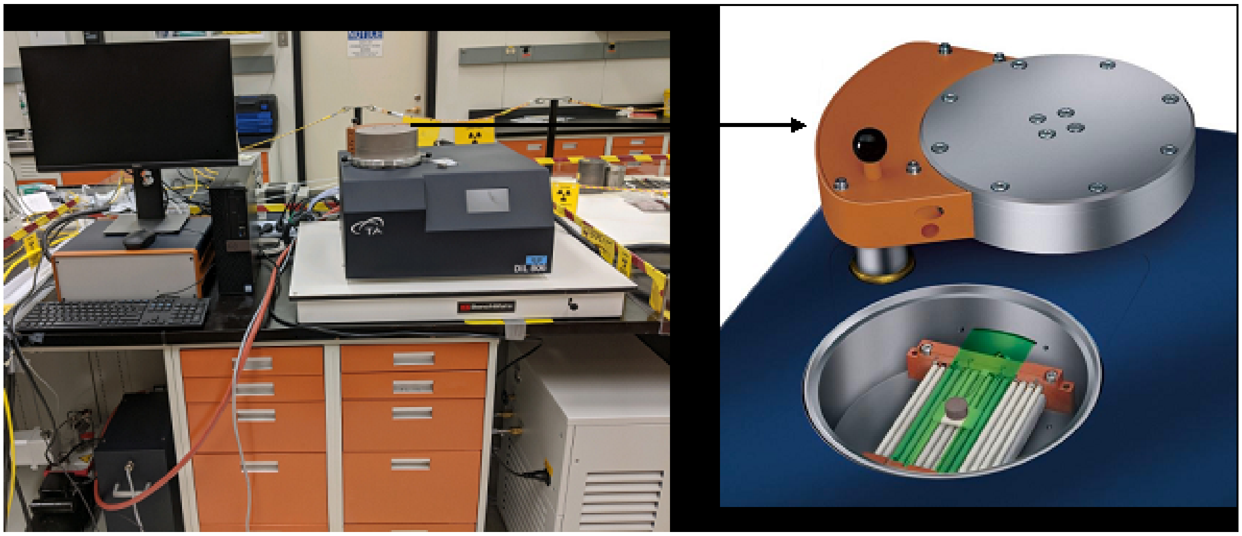

Figure 3. TA Instruments DIL 806 optical dilatometer, fully installed at the Measurements Science Lab.

Before each run, the optical dilatometer is pumped down with vacuum, back-filled with argon, pumped down, then back-filled with argon again. Finally, it is pumped down a third time to keep oxygen levels to a minimum. Additionally, during the run, the vacuum is maintained to prevent silica formation. One SiC temperature monitor is analyzed per dilatometer run. Each 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. This present analysis is not concerned with the quantitative expansion behavior, but rather how the SiC temperature monitor's delta change in length differs in response to heating and cooling. Therefore, a settling run is unnecessary, since the software that calculates the measurements essentially removes any noise caused by SiC temperature monitor settling during the analysis run [3].

4. RESULTS

4.1 Temperature Profile

As described in the previous paragraph, 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. Therefore, the optical dilatometer ran the KGT-3357 SiC temperature monitor from room temperature to 600°C (based on its target temperature of 300°C), held it there for 5 minutes, and then it cooled back down to room temperature, as shown in Figure 4. The results in Figure 4 reveal the furnace and sample temperature controls to be almost identical, with the furnace temperature as the chosen temperature control parameter for the optical dilatometer run. It was also found that continuous dilatometry method produces irradiation temperatures with a ± 20°C sensitivity[6].

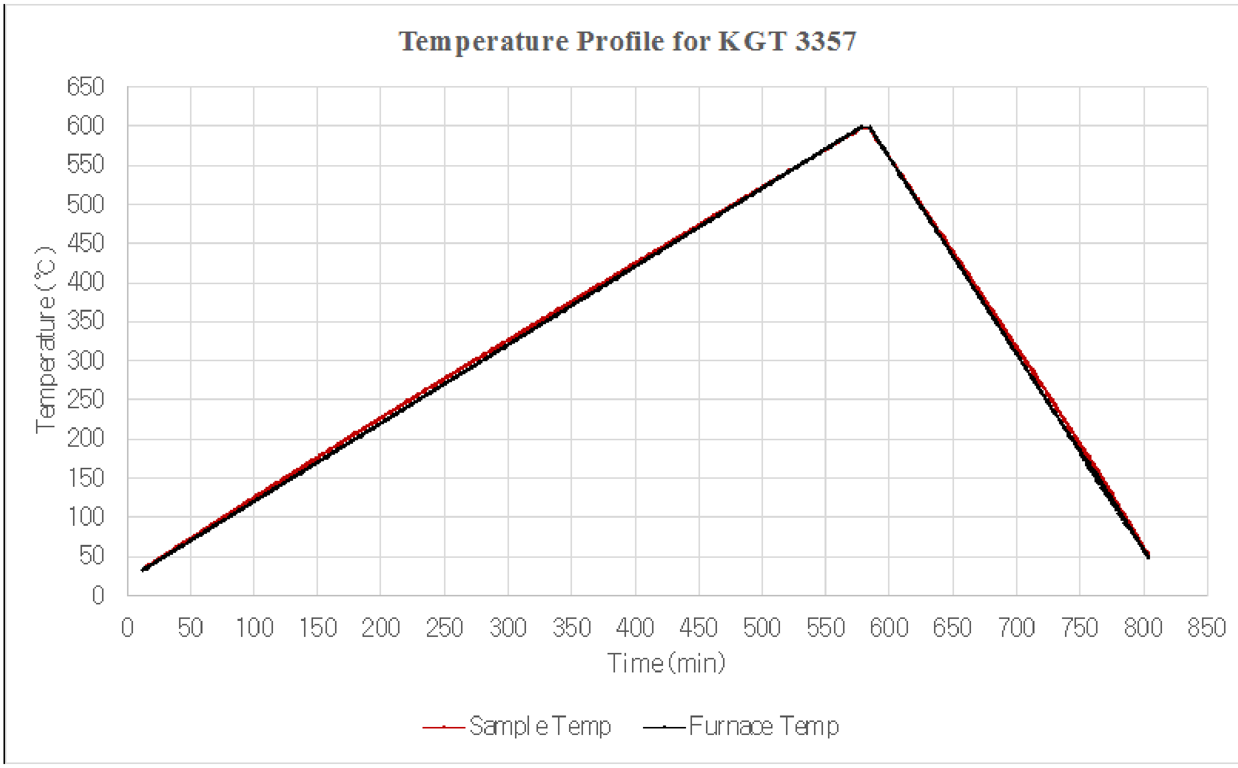

Figure 4. Temperature profiles of the furnace and sample controls for the KGT-3357 SiC temperature monitor.

SiC temperature monitors irradiated in the reactor will increase in volume due to radiation-induced swelling, as shown in Table 2. Furthermore, there was shown to be 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 monitor's length increasing post-irradiation by 0.108 mm, followed by a decrease of 0.027 mm from the post-irradiation examination annealing process in the dilatometer.

Table 2. KGT-3357's change in length pre-irradiation, and before and after annealing in the dilatometer.

[redacted]

4.2 Change in Length

Figure 5 plots the change in length (strain) of SiC temperature monitor KGT-3357 (design temperature: 300°C) during an optical dilatometer run. The heating segment is shown in red, the cooling segment in blue, and the difference of the two (delta) in black. The yellow dot represents the temperature of approximately 264°C at the maximum length change during heating segment. These same data are used for all subsequent analyses in this report.

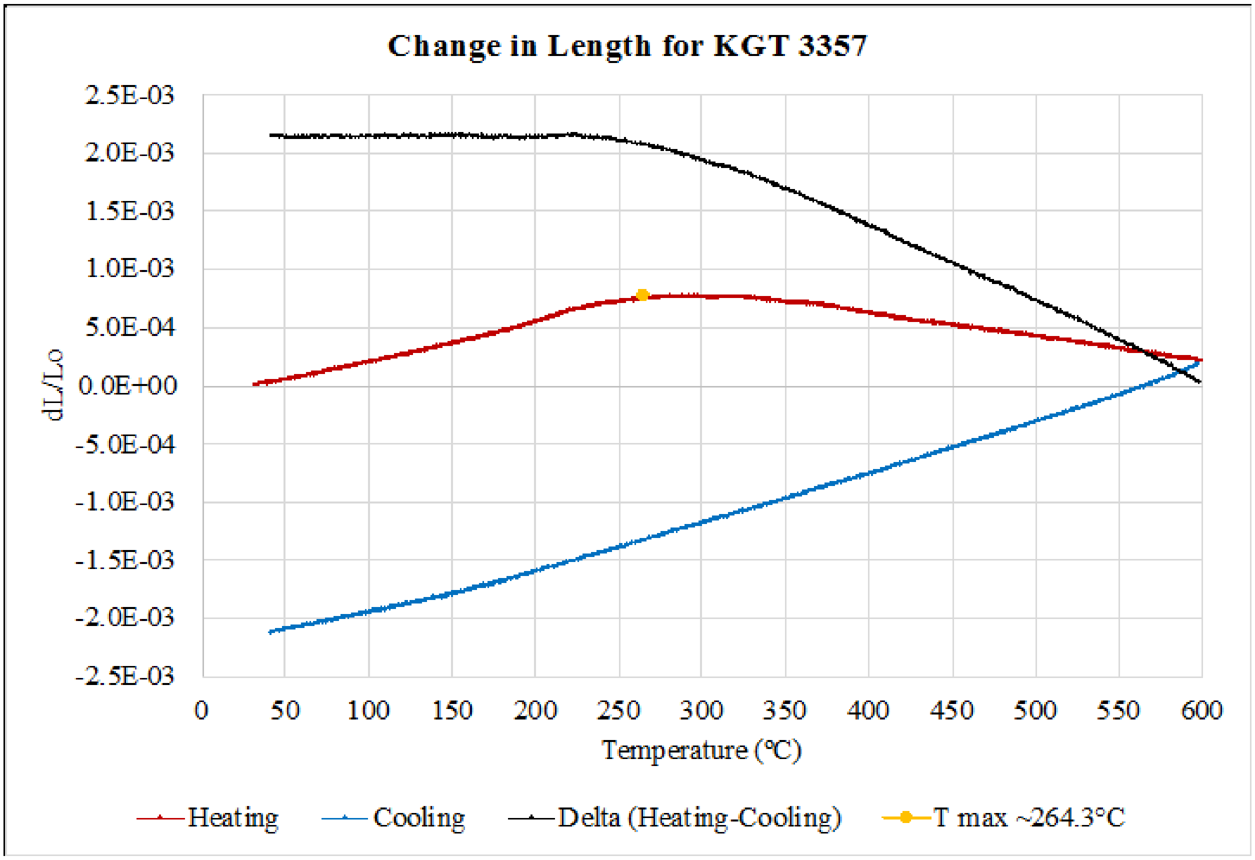

Figure 5. Change in length (strain) vs. temperature for SiC temperature monitor KGT-3357.

5. ANALYSIS AND DISCUSSION

5.1 Method 1

This method utilized the data collected from the length change of SiC temperature monitor KGT-3357, as discussed in Section 4.2 of this report. The first step of this method required fitting a straight line (shown in black) to the data below the temperature of 264°C (found in Section 4.2), as shown in Figure 6. The next step involved fitting a line (orange line) to the relatively straight section of the length change curve above the temperature of 264°C. The two fitted lines (black and orange) intersected at an irradiation temperature of 265.5°C indicated with a black circle. This irradiation temperature falls within the evaluated melt wire temperature range of 238.6–271.5°C [2].

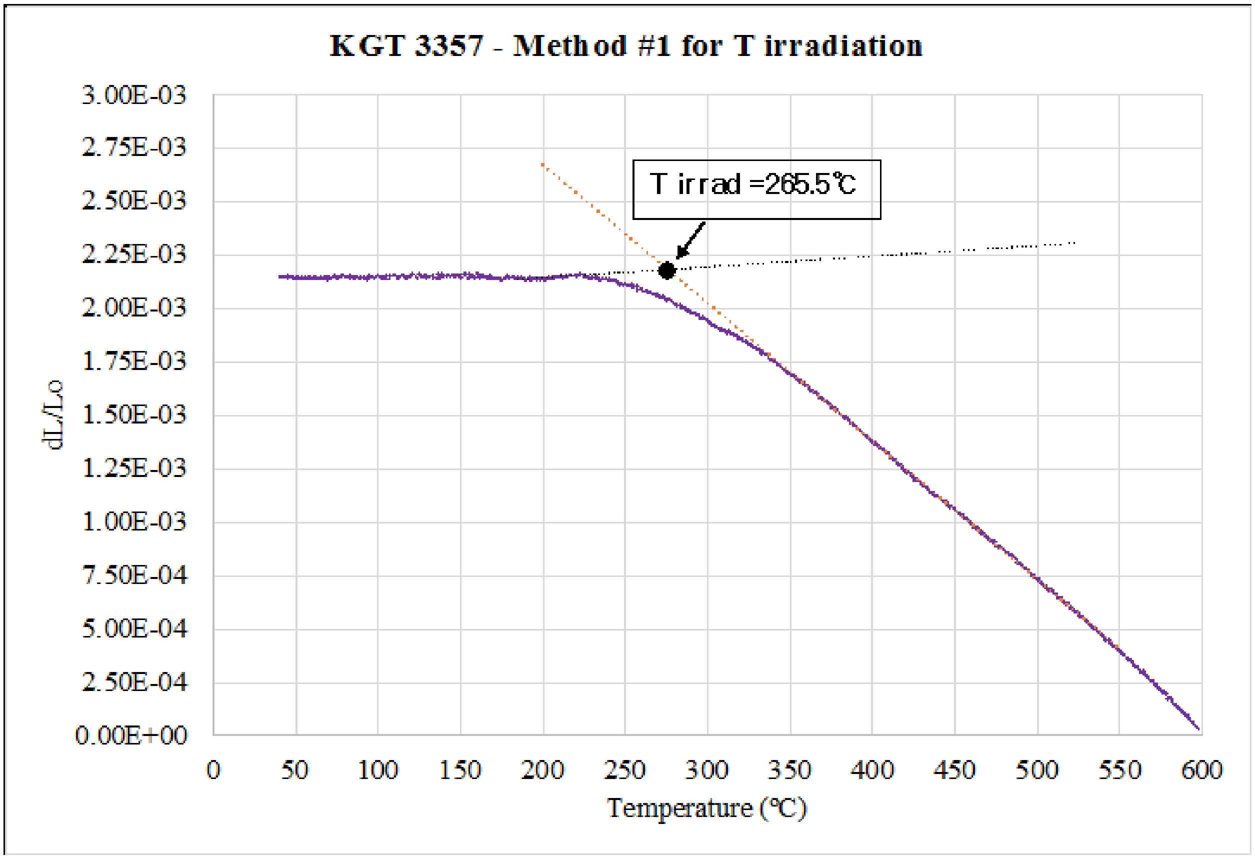

Figure 6. Method of measuring irradiation temperature via the delta of change in length of KGT-3357 SiC temperature monitor.

5.2 Method 2

This method also utilized the data collected from the length change of SiC temperature monitor KGT-3357,where it (Figure 7) required fitting two additional lines to the length change curve (Figure 5) compared to the first method. The first step of the second method required fitting again a straight line (orange line) to the data below the temperature of 264°C, as shown in Figure 7. The next fitted line (black line) was centered at the design irradiation temperature (target temperature of 300°C) shown as a red circle, and the third (green line) fitted line was centered around where the data began curving downward subsequently the flat region below the temperature of 264°C. The intersection of the orange and black lines was specified as the maximum temperature (yellow square) of 266.7℃, whereas the minimum temperature (i.e., 240°C) was specified as the intersection of the green and orange lines (green circle). This second method demonstrated better results by measuring the full irradiation range of the SiC temperature monitor. Furthermore, both the minimum and maximum temperatures fell within the melt wire evaluation temperature range of 238.6–271.5°C [2].

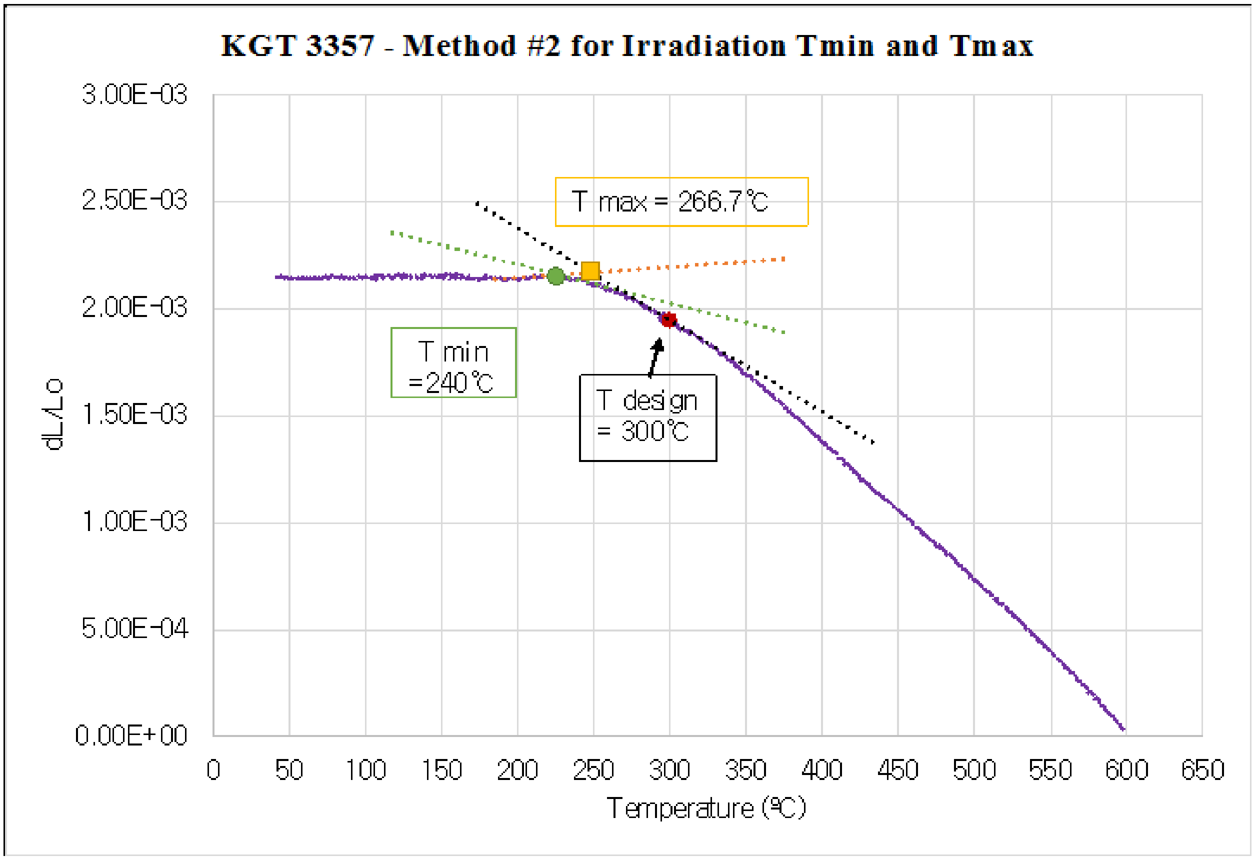

Figure 7. Method of measuring the irradiation temperature range from the delta change in length and the design temperature of the KGT-3357 SiC temperature monitor.

6. CONCLUSION

This summary report shows continuous optical dilatometry to be a valid method of measuring the irradiation temperature range of passive SiC temperature monitor. The optical dilatometer uses an automated process that only requires a small amount of time to run and is easy to use, thus saving on valuable labor time in comparison to the traditional resistivity method. The main objective of this project was to conduct a comparative assessment between the optical dilatometer method and resistivity method, using all 10 SiC temperature monitors provided by two Nuclear Science User Facility experiments: BSU-8242 and General Electric Hitachi. Unfortunately, due to multiple delays in acquiring, shipping, and cleaning the SiC temperature monitors, the project was only able to process one (1) SiC temperature monitor during this time. SiC temperature monitor KGT-3357 was evaluated via the optical dilatometer method to determine its peak irradiation temperature range. The KGT-3357 sample was from the BSU-8242 experiment and designed for a target temperature of 300°C and an exposure of 1 dpa. The optical dilatometer measurements indicated that the KGT-3357 SiC temperature monitor's peak irradiation temperature range was 240–267°C, with sensitives of approximately ±20°C. This temperature range falls within the evaluated melt wire temperature range of 238.6–271.5°C. However, more than one sample is required before finalizing and validating the benefits of the optical dilatometer method (e.g., reduced process times, lower costs, and enhanced accuracies).

7. FUTURE WORK

Future activities will focus on processing the remaining six (6) SiC temperature monitors from the BSU-8242 experiment as well as the three (3) from the General Electric Hitachi experiment in order to further validate the optical dilatometer method for effectively measuring SiC peak irradiation temperature.

8. REFERENCES

1. Guillen, Donna, et al. “Boise State University (BSU)-8242 Experiment Execution Plan,” PLN-5248, Rev. 1, Project 32349, April 2017.
2. Davis, Kurt L., and Hone, Lance A. “NSUF Melt Wire Evaluations for BSU 8242 and GE Hitachi-10393 Irradiation Experiments.” 2020, https://doi.org/10.2172/1633621.
3. Campbell, Anne A., et al. “Method for Analyzing Passive Silicon Carbide Thermometry with a Continuous Dilatometer to Determine Irradiation Temperature.” *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, vol. 370, 2016, pp. 49–58., https://doi.org/10.1016/j.nimb.2016.01.005.
4. Rempe, J. L., Condie, K. G., and Knudson, D. L. “Silicon Carbide Temperature Monitor Evaluation,” PLN-3473, Revision 0, Idaho National Laboratory, May14, 2010.
5. “Dil 806 Optical Dilatometer.” *TA Instruments*, https://www.tainstruments.com/dil-806/.
6. Field, Kevin G., et al. “Evaluation of the continuous dilatometer method of silicon carbide thermometry for passive irradiation temperature determination.” *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, vol. 445, 2019, pp. 46–56., https://doi.org/10.1016/j.nimb.2019.02.022.