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ASI | Advanced Sensors and Instrumentation

Benchmark Analysis for the Optical Dilatometry using Silicon Carbide Temperature Monitors

Advanced Sensors and Instrumentation (ASI)
Annual Program Webinar
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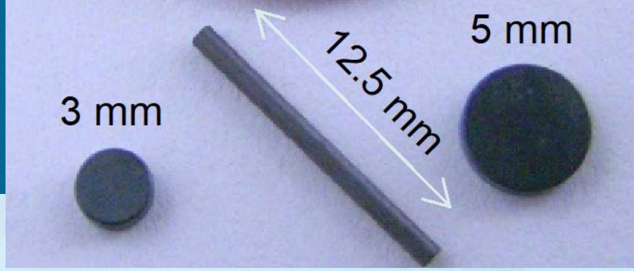
Project Overview



- **Brief summary of research scope:**

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. Passive temperature monitors are needed for when real-time sensors are not practical or economical to install in an irradiation test. The main purpose is to provide a practical and reliable approach to estimate peak irradiation temperature during post-irradiation examination (PIE) for direct integration in irradiation test designs.

- **Project Schedule:**

October 2022: Complete level 3 milestone (M3CT-22IN0702074) titled "Benchmark Analysis for the Optical Dilatometry using Silicon Carbide Temperature Monitors"

- **Participants:**

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Austin Fleming (TPOC)

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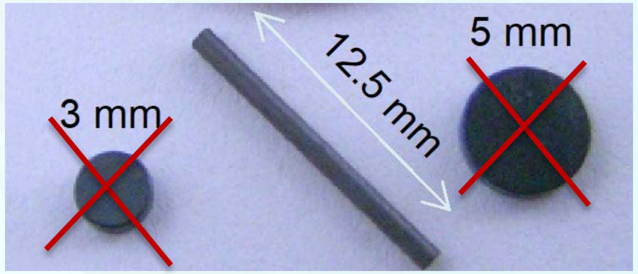
Technology Impact

- Passive monitors provide a practical, reliable, and robust approach to measure irradiation temperature during post-irradiation examination while requiring no feedthroughs/leads comparable to current more-complex real-time temperature sensors
- They have been chosen because they have a proven history for use by stakeholders for deployment and require continued development and characterization to assure successful integration with program schedules and objectives
- Further develop the temperature passive monitor capability for wider rage of temperatures, geometries and neutron damage
- Facilitates the development of advanced sensors and instrumentation with cross-cutting technology development to support the existing fleet, advanced reactor technology and advancing fuel cycle technology development

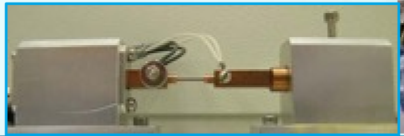
Resistivity Method

- Electrical resistivity is accepted as a robust measurement technique resulting in accuracies within 20ºC
- Very time and labor-intensive process with near-constant attention from trained staff (1 week to 3 week per sample)
- Labor time for Technician, Engineer, Radiological Control, and Administrative Assistant
- Adds many potential sources of measurement error:
  - Potentially result in oxidizing the SiC temperature monitor
  - Measurement error due to repeatedly transferring back and forth between the furnace and the test fixture
- Currently can only process rod-shaped SiC temperature monitors

SiC temperature monitors



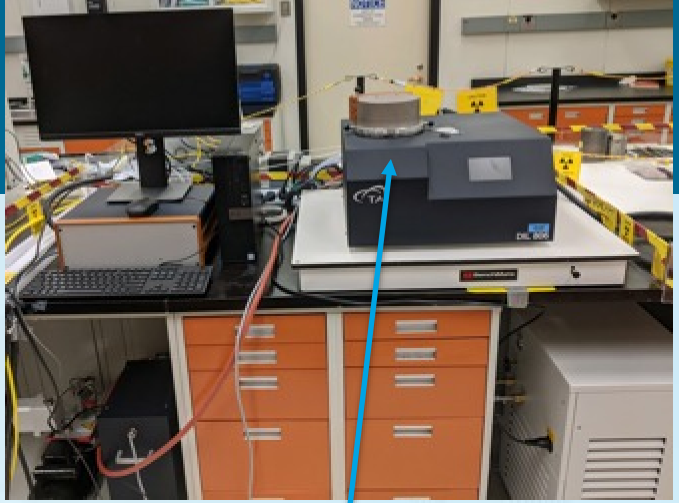
Spring loaded sample fixture.

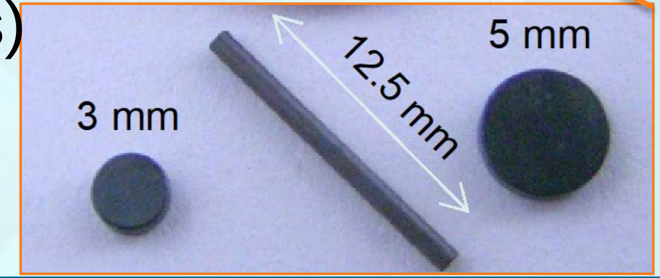


Resistivity Method Set-Up

Dilatometry Method

- Automated process requiring minimal setup time
- Dilatometer runs under vacuum or inert gas
  - key requirement for avoiding any oxidization issues
- Max. operating temperatures of 1400ºC (resolution of 0.1ºC)
  - SiC passive monitor temperatures are 200 – 1200ºC
- Reduced time expense
  - Time to process each sample 2 to 3 days (target temperature dependent)
- Contactless dilatometric measurement system
  - Allows samples to freely expand/shrink without any interference from mechanical contact
- Can process all SiC temperature monitors (rod and both discs)
  - 0.3–30 mm in length with a maximum height of 10 mm





Issues (Schedule/Cost/Technical)

- NSUF planned to provide 10 SiC temperature monitors:
  - BSU-8242 with 7 SiC rods: 1-SiC rod per capsule for 7 capsules (located in the middle of the capsule)
  - GE-Hitachi with 3 SiC rods: 3-SiC rods per 1 capsule (located in the bottom, middle, and top of the capsule)
- Multiple Delays:
  - Acquiring
  - Shipping
  - Cleaning (HF wash) the SiC temperature monitors
  - Oxidization of one SiC monitor for resistivity method
  - One of the GE-Hitachi sample broke (two pieces) during cleaning
- Highlight:
  - All 10 NSUF SiC temperature monitors successfully delivered in time to process them all

Benchmark Sample Selection

NSUF provided all 10 SiC temperature monitors:

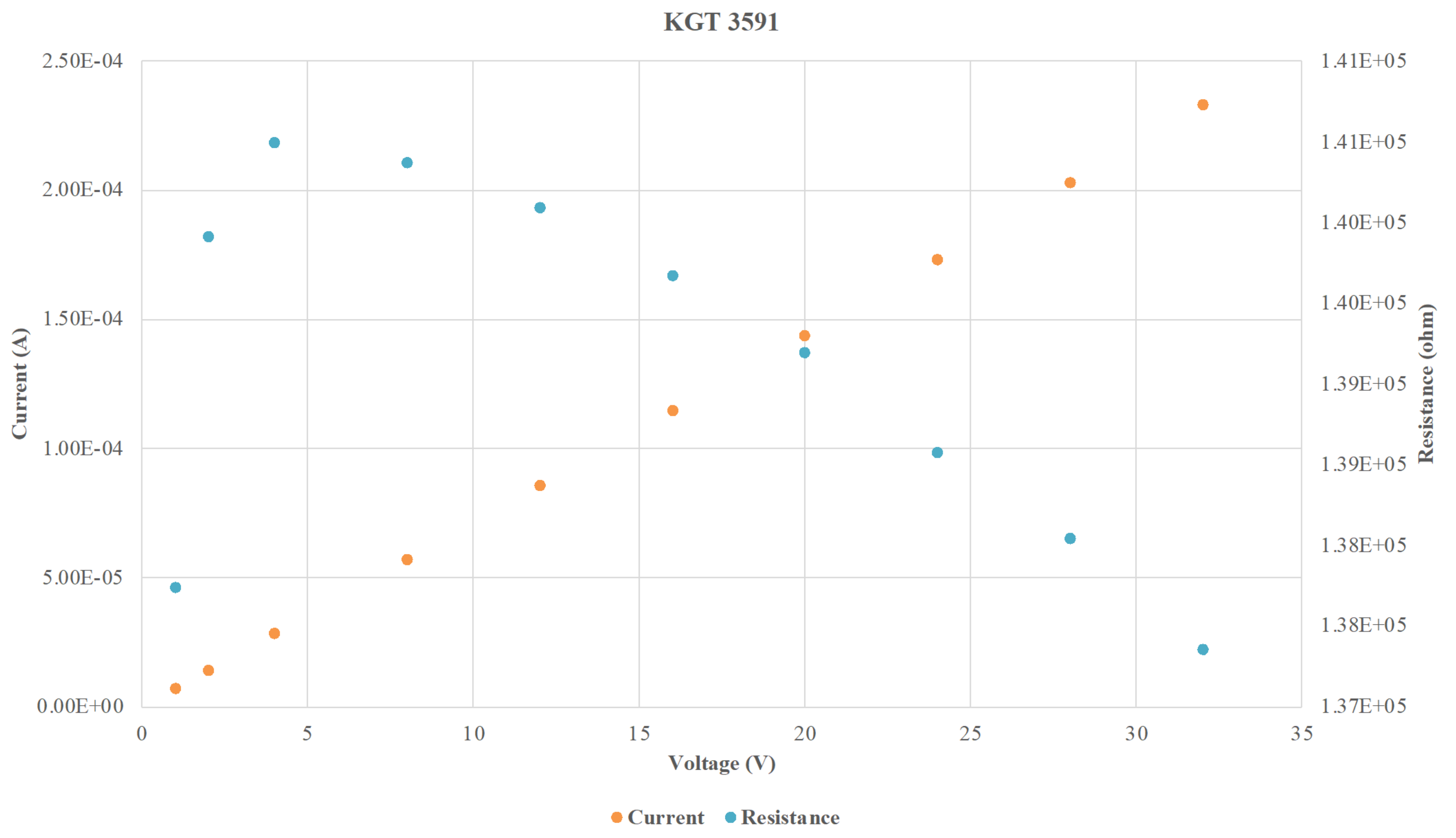
- Pair one for BSU-8242 are KGT-3591 and KGT-3597 with design temperature of 400℃ and exposure of 3 dpa

Pair two for GE-Hitachi are KGT-3341 and KGT-3336 (two pieces) with design temperature of 290℃ +/- 50℃ and exposure anywhere between 0.5-1 dpa

Sample Number	MSL Identification	HFEF Identification	NSUF Project Name	T Design [℃]	Damage [dpa]	Melt Wire Temp. Range [℃]		Evaluation Method
1	HTTL-605766-10	KGT-3347	BSU-8242	300	1	Less than	238.6	Dilatometry
2	HTTL-605767-10	KGT-3357	BSU-8242	300	1	238.6	271.5	Dilatometry
3	HTTL-605768-10	KGT-3573	BSU-8242	300	3	-	-	Dilatometry
4	HTTL-605769-10	KGT-3360	BSU-8242	400	1	327.5	399.4	Dilatometry
5	HTTL-605770-10	KGT-3373	BSU-8242	400	1	399.4	499.5	Dilatometry
6	HTTL-605772-10	KGT-3591	BSU-8242	400	3	-	-	Resistivity
7	HTTL-605771-10	KGT-3597	BSU-8242	400	3	-	-	Dilatometry
8	HTTL-1-GEH-BOT	KGT-3341	Ge-Hitachi-393	290	0.5-1	327.5	399.4	Resistivity
9	HTTL-1-GEH-MID	KGT-3336	GE-Hitachi 393	290	0.5-1	327.5	399.4	Dilatometry
9'	HTTL-1-GEH-MID	KGT-3336'	GE-Hitachi 393	290	0.5-1	327.5	399.4	Dilatometry
10	HTTL-1-GEH-TOP	KGT-3339	GE-Hitachi 393	290	0.5-1	271.5	279.5	Dilatometry

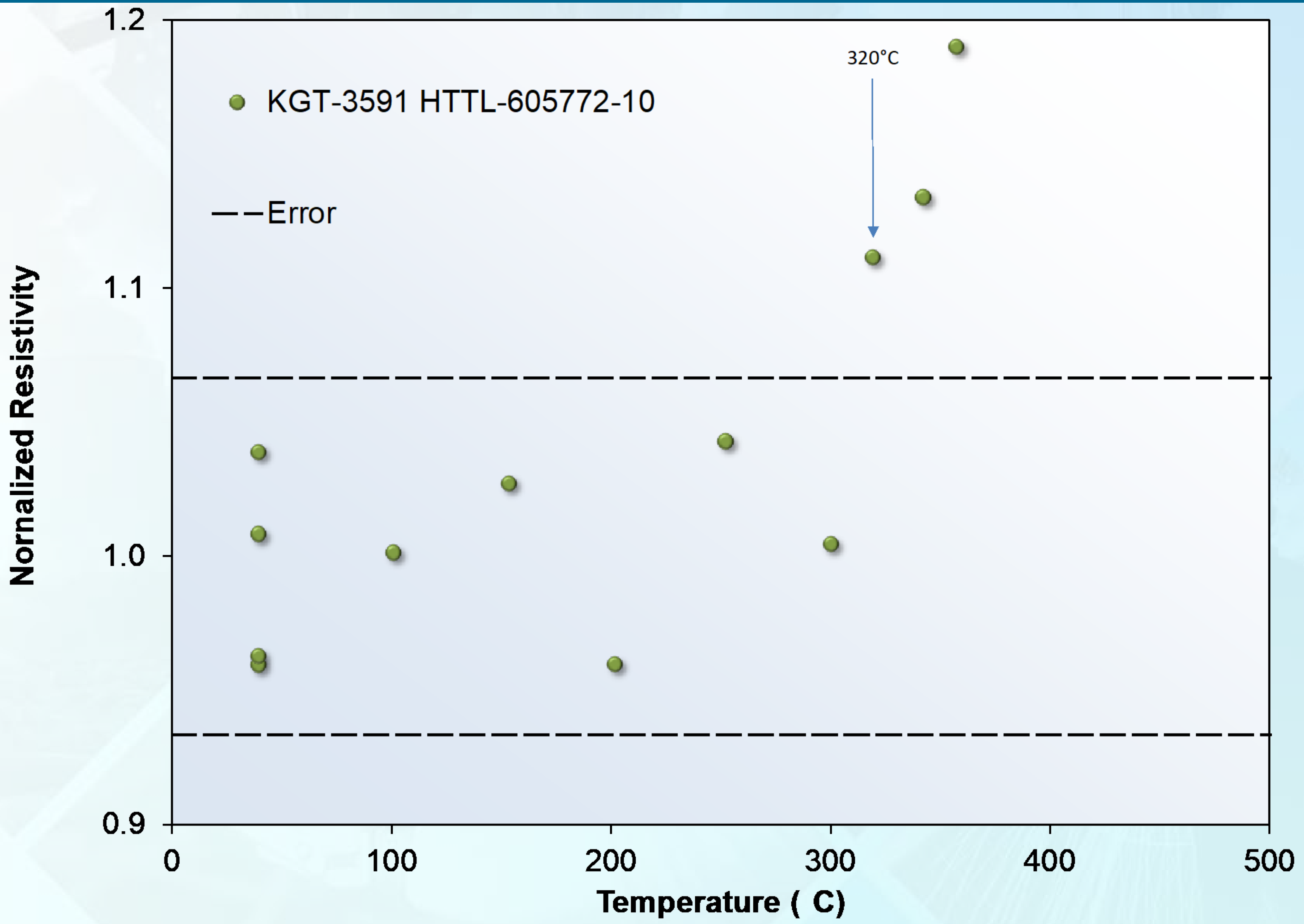
Resistivity Results for BSU-8242

- An ohmic response curve was generated for each monitor prior to heating
- BSU-8242 SiC temperature monitor KGT-3591 exhibited a typical ohmic response
- For this evaluation, voltage ranged between 4-8 V for both temperature monitors



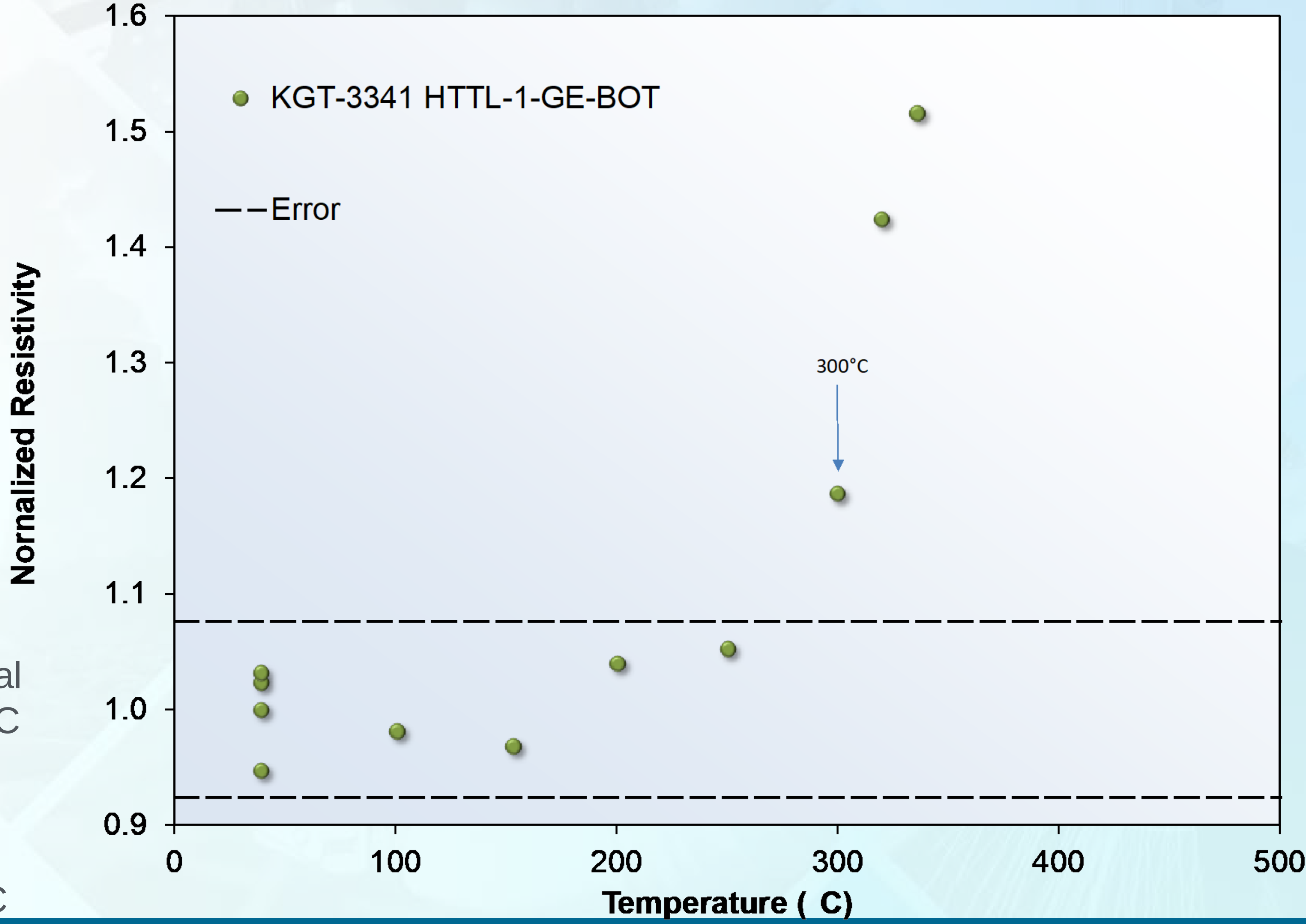
Resistivity Results for BSU-8242 Cont.

- Evaluation data for BSU-8242 SiC temperature monitor KGT-3591:
  - T design is 400 °C
- The error band was based on a 2-sigma value calculated from the data points collected up to 100°C
- The peak irradiation temperature for this monitor is evaluated at **320℃ with +/- 20°C accuracy**
  - 80 °C below T design
- Melt wire range was undetermined for this position



Resistivity Results for GE-Hitachi

- Evaluation data for GE-Hitachi SiC temperature monitor KGT-3341:
    - 290 °C with +/- 50 °C
- The error band was based on a 2-sigma value calculated from the data points collected up to 100°C
- The peak irradiation temperature for this monitor is evaluated at **300°C with accuracies between +20°C and -50°C**
    - 10°C above T design
- The lower evaluation temperature was unexpected, thus larger isochronal heating steps were taken when the SiC temperature monitor responded ->lower error bound of -50°C
- Melt wire range was 327.5 to 399.4 °C



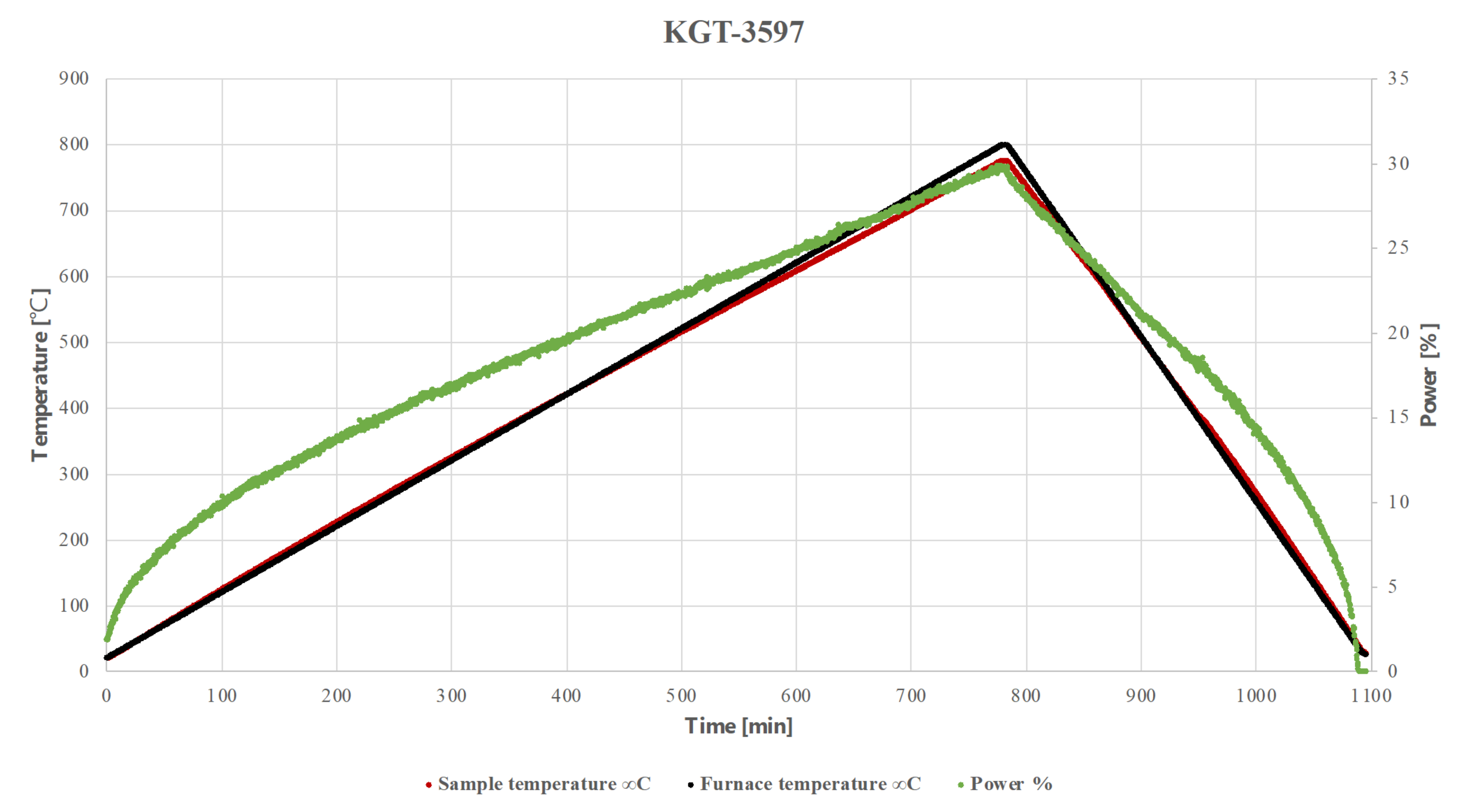
Dilatometry Results for BSU-8242

Dilatometer Program:

- 800℃ max. temp
- 1℃/min heating rate
- Hold 5 min at 800℃
- -2.5℃/min cooling rate

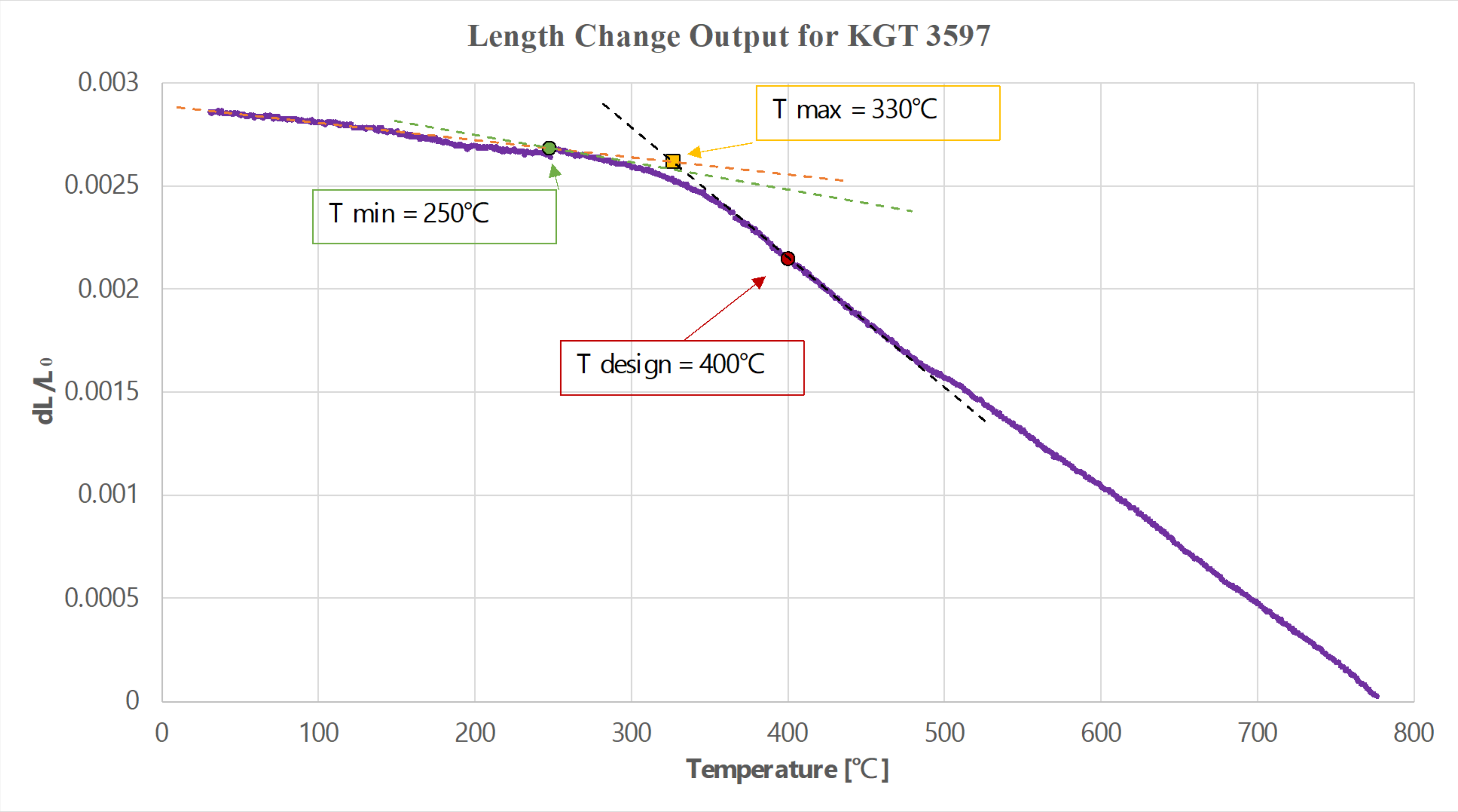
Both monitors showed similar results.

Furnace and sample temperature controls are almost identical, with the furnace temperature as the chosen temperature control parameter for the optical dilatometer run.



Dilatometry Results for BSU-8242 Cont.

- Evaluation data for BSU-8242 SiC temperature monitor KGT-3597
- The irradiation started at ~250°C
- The peak irradiation temperature for this monitor is evaluated at **330°C**
  - 70°C below T design
- Melt wire range was undetermined for this position
- Compared to resistivity method (320°C) for similar positioned -> very close results



Dilatometry Results for GE-Hitachi 1st Piece

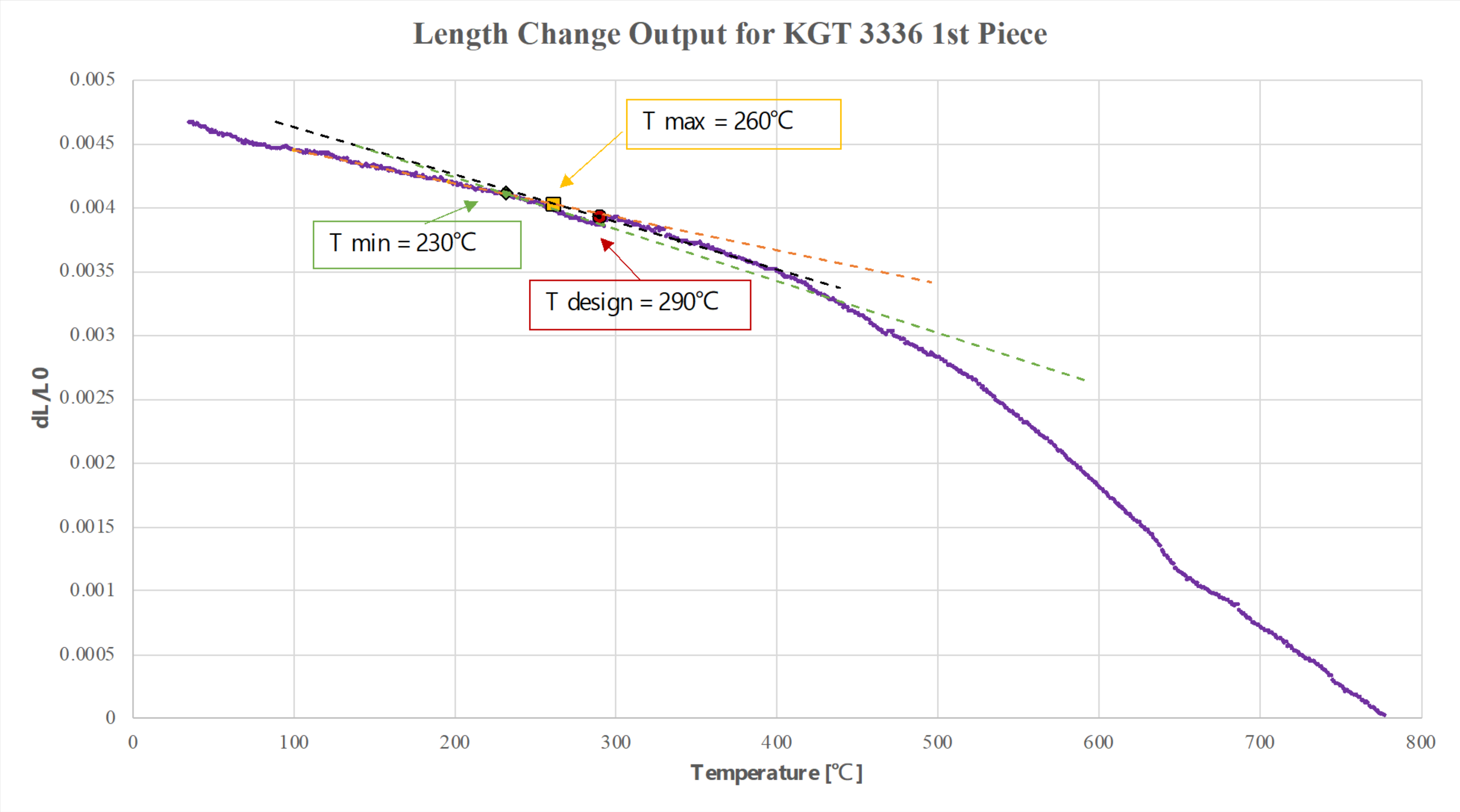
- Evaluation data for BSU-8242 SiC temperature monitor KGT-3336 1st piece (length ~8125.6 µm)

- The irradiation started at ~230℃

- The peak irradiation temperature for this monitor is evaluated at **260℃**
  - 30°C below. T design

- Compared to resistivity method (300℃) for similar positioned -> very close results due to the lower error bound

- Melt wire range was 327.5 to 399.4 °C



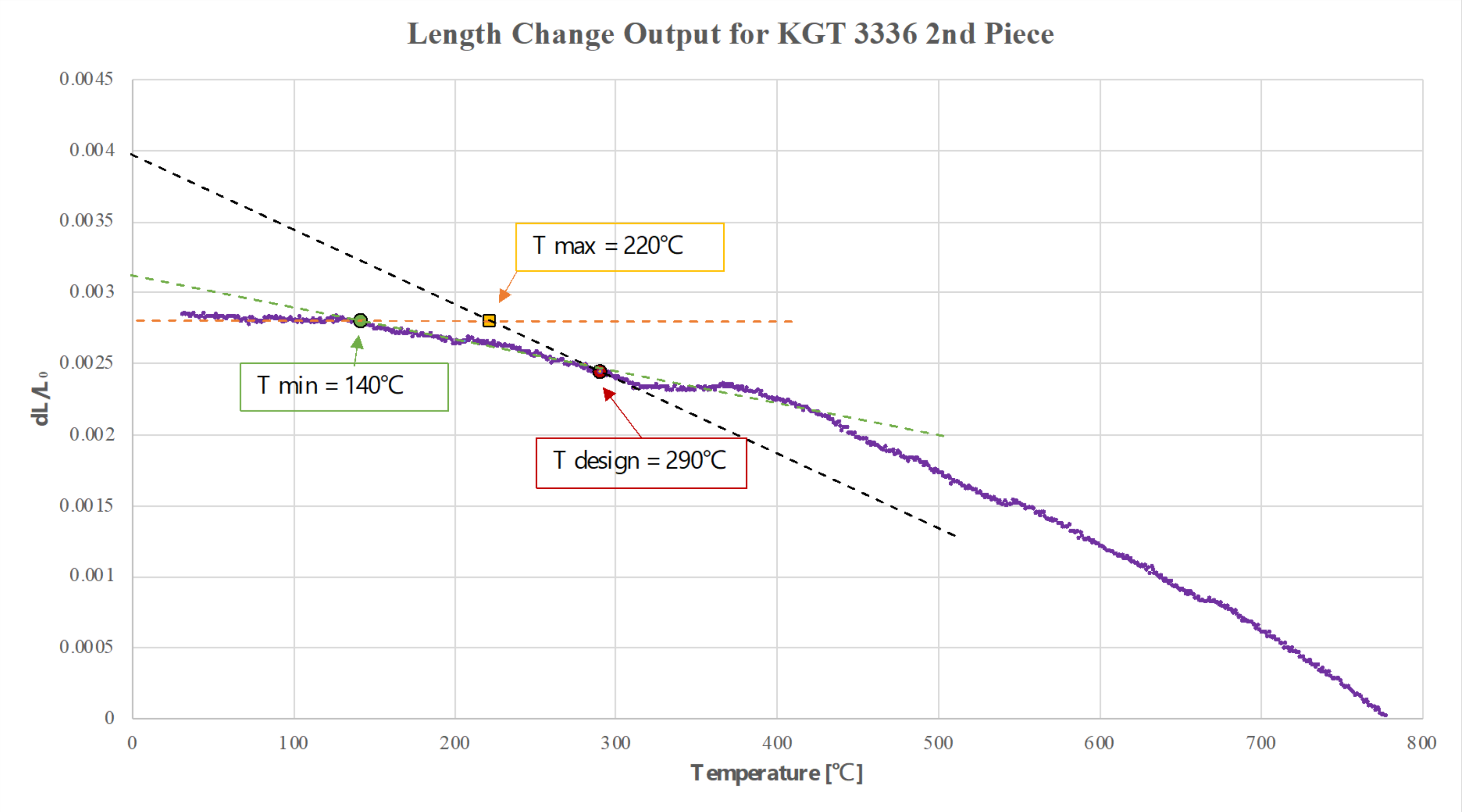
Dilatometry Results for GE-Hitachi 2nd Piece

- Evaluation data for BSU-8242 SiC temperature monitor KGT-3336 2nd piece (length ~4456.3 µm)

- The irradiation started at ~140℃ (90℃ lower than 1st piece)

- The peak irradiation temperature for this monitor is evaluated at **220℃**
  - 70°C below T. design and 40℃ below 1st piece

- Smaller pieces of SiC seem to have much harder results to read (almost flat line)



Summary of Results

Two pairs of SiC monitors were chosen:

- BSU-8242 -> KGT-3597 and KGT-3591 with T design of 400℃ and exposure of 3 dpa
- Ge-Hitachi -> KGT-3341 and KGT-3336 with T design of 290℃ +/- 50℃ and an exposure between 0.5 to 1 dpa

-> KGT-3336 SiC monitor was split into two pieces during decontamination process

Evaluation of peak Irradiation temperatures are as follow:

- BSU-8242: Resistivity of KGT-3591 at 320℃ +/- 20 ℃ and Dilatometry of KGT-3597 at 330℃
- GE-Hitachi: Resistivity of KGT-3341 at 300℃ -50℃ to 20℃ and Dilatometry of KGT-3336 larger piece at 260℃

Both methods of analyzing the SiC temperature monitors showed very similar peak irradiation temperatures for each pair of the SiC passive monitors.

Pair Number	MSL Identification	HFEF Identification	Design Temp.[℃]	Exposure [dpa]	Evaluation Method	Irrad. Temp. [℃]	Delta with T design
1	HTTL-605771-10	KGT-3597	400	3	Dilatometry	330	-70
	HTTL-605772-10	KGT-3591	400	3	Resistivity	320	-80
2	HTTL-GEH-BOT	KGT-3341	290	0.5-1	Resistivity	300	10
	HTTL-GEH-MID	KGT-3336	290	0.5-1	Dilatometry	260	-30

Conclusion and Future Work

- Conducted a benchmark analysis between the optical dilatometer and resistivity method using two pairs from each NSUF experiment's SiC temperature monitors
- Encounter multiple delays in acquiring, shipping, and cleaning the SiC temperature monitors
- The GE-Hitachi KGT-3336 was split into two pieces during cleaning process
- Results revealed continuous optical dilatometry to be a valid method of measuring the peak irradiation temperature of passive SiC temperature monitors
- 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
- Future work will focus on analyzing SiC discs (5-mm and 3-mm)

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Thank You