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Evaluations of University of Wisconsin Silicon Carbide Temperature Monitors 300 LO and 400 LO B

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SUMMARY

Silicon carbide (SiC) temperature monitors 05R4-02-A KG1403 (300 LO) and 05R4-01-A KG1415 (400 LO B) were evaluated at the High Temperature Test Lab (HTTL) to determine their peak irradiation temperatures. HTTL measurements indicate that the peak irradiation temperature for the 300 LO monitor was 295 ± 20°C and the peak irradiation temperature for the 400 LO B monitor was 294 ± 25°C.

1. INTRODUCTION

Two silicon carbide (SiC) temperature monitors irradiated in the Advanced Test Reactor (ATR) were evaluated at the High Temperature Test Lab (HTTL) to determine their peak temperature during irradiation. These monitors were irradiated as part of the University of Wisconsin Pilot Project[1] with a target dose of 3 dpa. Temperature monitors were fabricated from high density (3.203 g/cm^3) SiC manufactured by Rohm Haas with a nominal size of 12.5 mm x 1.0 mm x 0.75 mm (see Attachment A). Table 1 provides identification for each monitor with an expected peak irradiation temperature range based on preliminary thermal analysis (see Attachment B). Post irradiation calculations are planned to reduce uncertainties in these calculated temperatures.

Table 1. SiC temperature monitors tested.

Monitor Identification	Expected Peak Irradiation Temperature Range (°C)
05R4-02-A KG1403 (300 LO)	287 - 343
05R4-01-A KG1415 (400 LO B)	411 - 442

Since the early 1960s, SiC has been used as a post-irradiation temperature monitor. As noted in Reference 2, several researchers have observed that neutron irradiation induced lattice expansion of SiC annealed out when the post-irradiation annealing temperature exceeds the peak irradiation temperature. As noted in Reference 3, INL uses resistivity measurements to infer peak irradiation temperature from SiC monitors. Figure 1 depicts the equipment at the HTTL used to evaluate the SiC monitors. The SiC monitors are heated in the annealing furnace using isochronal temperature steps that, depending on customer needs, can range from 50 to 800ºC. This furnace is located under a ventilation hood within the stainless steel enclosure. The ventilation system is activated during heating so that any released vapors are vented through this system. Annealing temperatures are recorded using a National Institute of Standards and Technology (NIST) traceable thermocouple inserted into an alumina tube in the furnace. After each isochronal annealing, the specimens are placed in a specialized fixture located in the constant temperature chamber (maintained at 30°C) for a minimum of 30 minutes. After the 30 minute wait time, each specimen's resistance is measured using the specialized fixture and a calibrated DC power analyzer.

This report discusses the evaluation of the SiC monitors and presents the results. Testing was conducted in accordance with Reference 3. Sections 2 and 3 present the data collected for each monitor and provide interpretation of the data. Section 4 presents the evaluated temperature results.

2. SiC Temperature Monitor 300 LO

An ohmic response curve was generated prior to heating for the 300 LO monitor, see Figure 2. These data were used to check for linearity and to select a target current that would result in minimal heating of the SiC monitor during resistance testing and remain within the range of the test instrumentation. For this testing, a target current range of 20 to 30 μA was selected. Using the data in Figure 2, an applied voltage of 2V was selected to provide the target current.

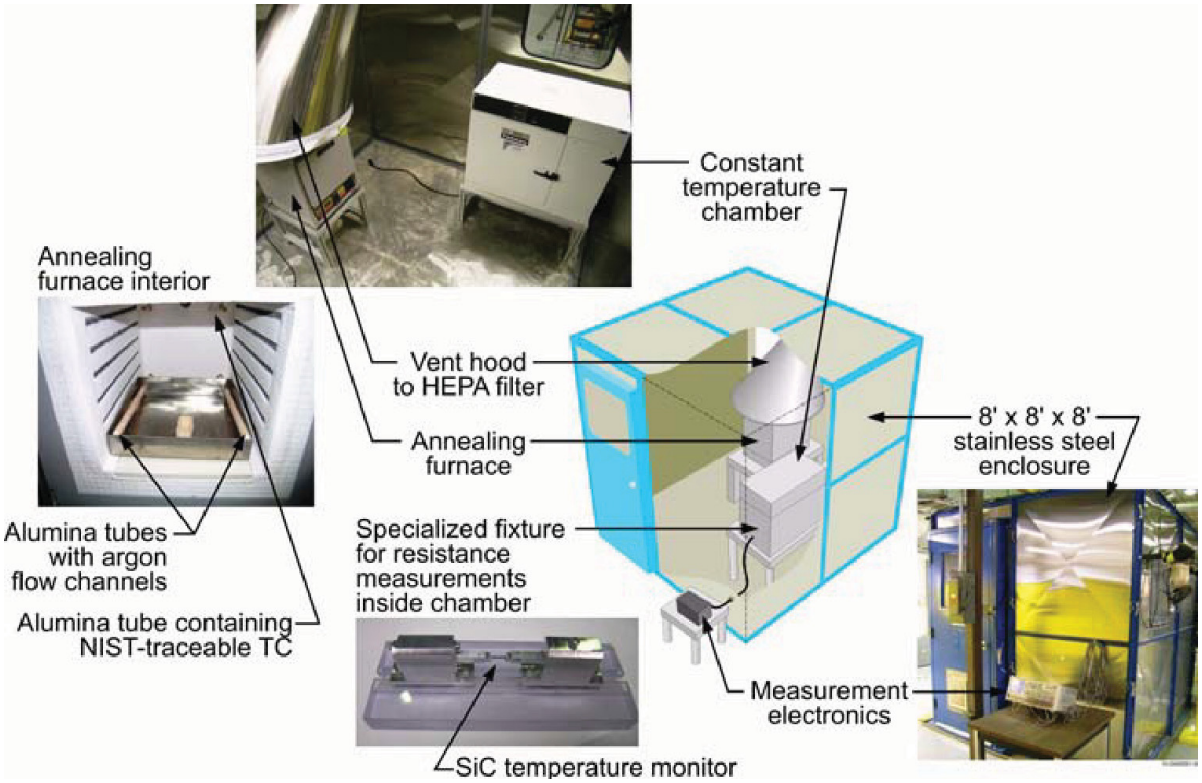

Figure 1. Setup for annealing and measuring SiC temperature monitors.

As documented in Ref. 3, electrical resistivity is used by INL to infer the peak irradiation temperature. Figure 3 presents the resistivity data taken at each isochronal annealing temperature. Reference 3 reports that the peak irradiation temperature using an electrical resistivity technique can be taken as the point where the resistivity begins, and consistently remains, above the error band. The error band bounds the data and is represented by the red dotted lines shown in Figure 3. Using this method, the first data point to consistently remain above the error band was taken at 6.073 ohm-m and 294.5°C. Using three significant digits to report this temperature, the inferred irradiation temperature was 295°C. This temperature falls within the planned irradiation temperature range of 287 to 343°C. As documented in Reference 3, the error using this technique is generally accepted to be 20°C.

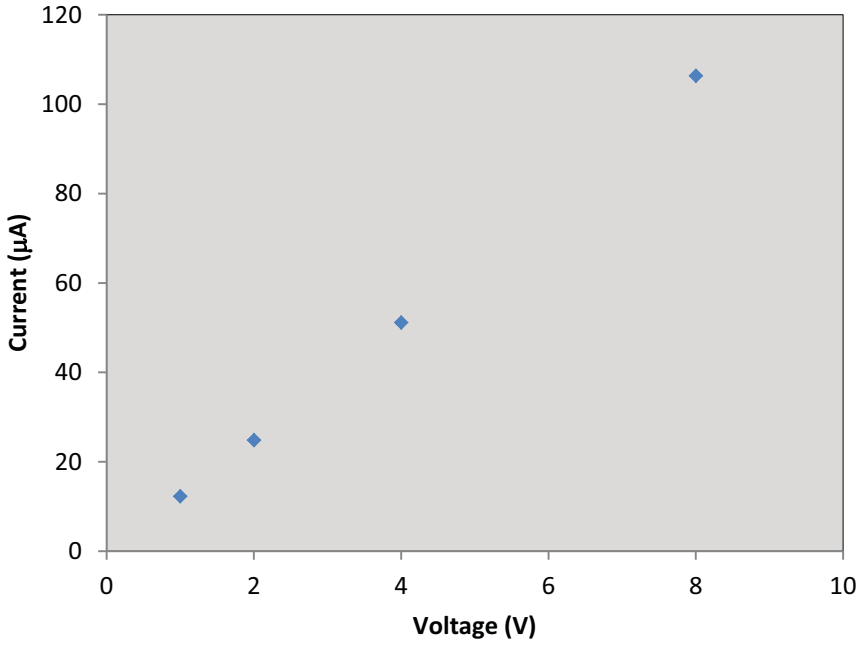

Figure 2. Ohmic response for the 300 LO monitor.

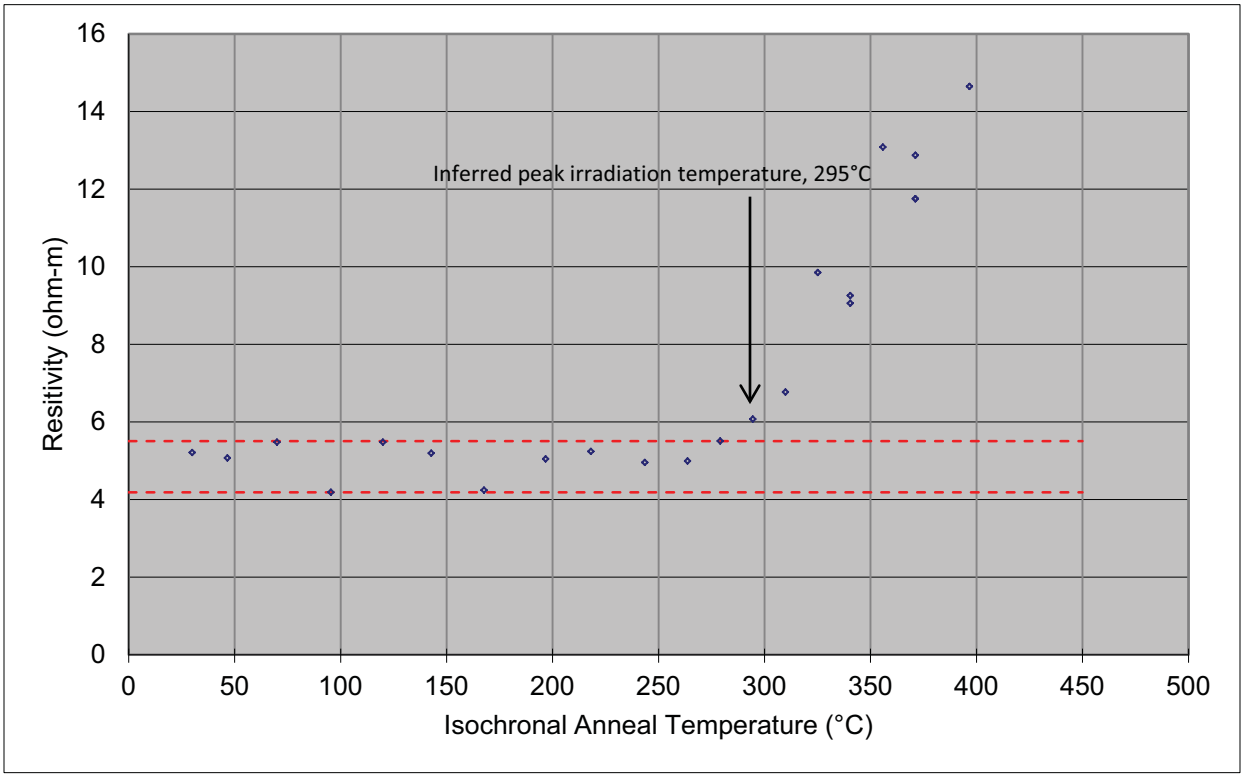

Figure 3. Resistivity data for the 300 LO monitor.

3. SiC Temperature Monitor 400 LO B

As a check for oxidation (temperatures >400°C) during the annealing process, the INL procedure for evaluating SiC temperature monitors[3] required the use of a reference unirradiated SiC monitor to be evaluated with the 400 LO B monitor. This procedure also required the use of an argon atmosphere during annealing. Figure 4 shows the ohmic response curve generated prior to heating the reference monitor and the 400 LO B monitor. For these samples, a target current range of 20 to 30 uA was selected. Based upon the data in Figure 4, an applied voltage of 2V was selected for the 400 LO B monitor, and an applied voltage 20V was selected for the reference monitor.

Figure 5 presents the resistivity data taken at each isochronal annealing temperature for the reference monitor. Oxidation of the reference monitor during the annealing process would have resulted in a substantial increase in resistivity. No substantial increase was observed.

Figure 6 presents the resistivity data taken at each isochronal annealing temperature for the 400 LO B monitor. As shown in Figure 6, the first data point to consistently remain above the error band was obtained after an annealing temperature of 294°C. This temperature is considerably lower than the planned irradiation temperature range of 411 to 442°C indicated by the customer. Because of this unexpected result, the resistivity measurement was repeated several times. Each data point taken consistently remained above the error band. Hence, measurements indicated that the peak irradiation temperature is 294°C with an uncertainty estimated as ± 25°C. Note that the larger uncertainty is because larger temperature increments (25°C) were selected when significant increases in resistivity were observed. Smaller increments in annealing temperatures had not been planned until temperatures that were closer to the expected values of 411 to 442°C.

As noted in Reference 3, there are several limitations associated with the use of SiC temperature monitors that limit their accuracy. For example, if the irradiation temperature was lower for a time period after the peak irradiation temperature occurred, lower peak irradiation temperatures will be inferred from SiC resistivity measurements. Expected peak irradiation temperatures were preliminary values and calculations are planned to reduce uncertainties in these predictions.

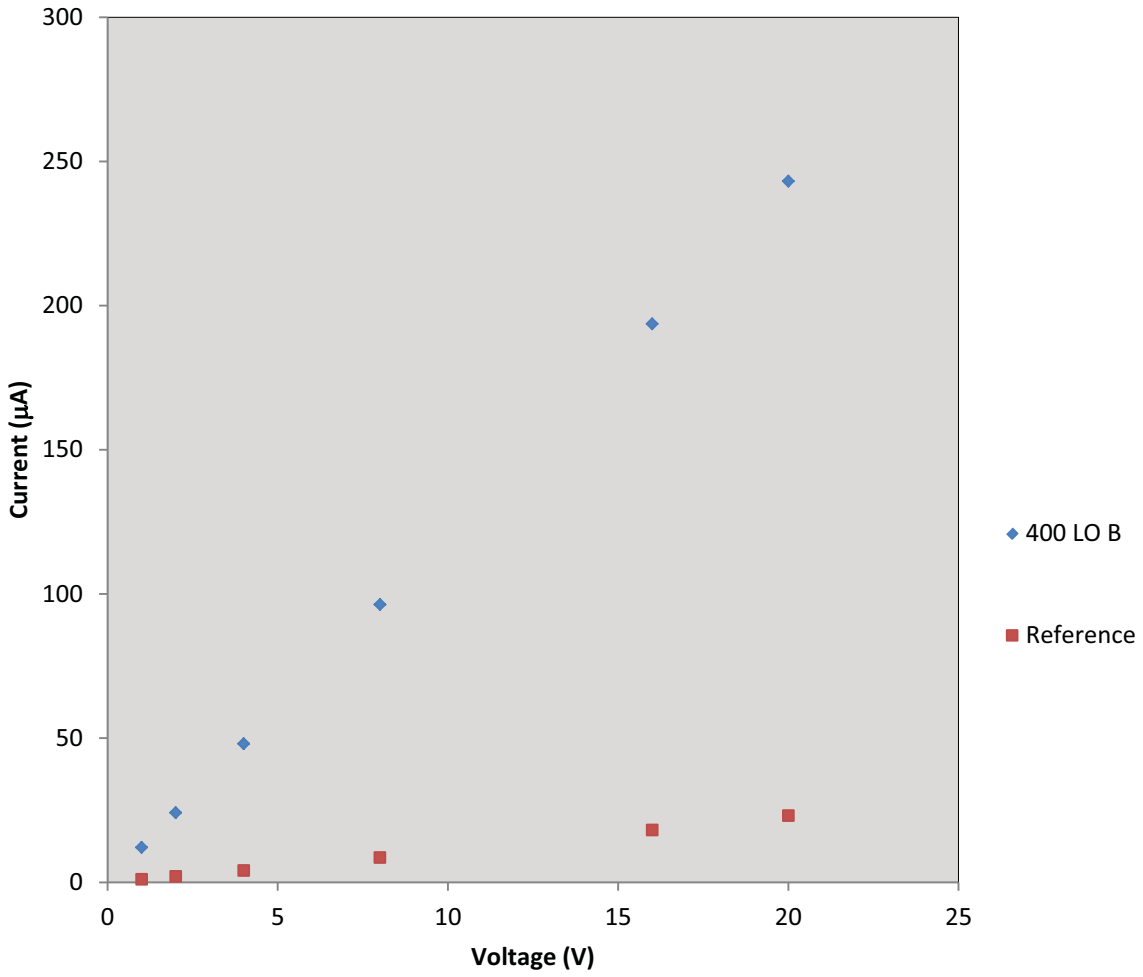

Figure 4. Ohmic response for the 400 LO B monitor.

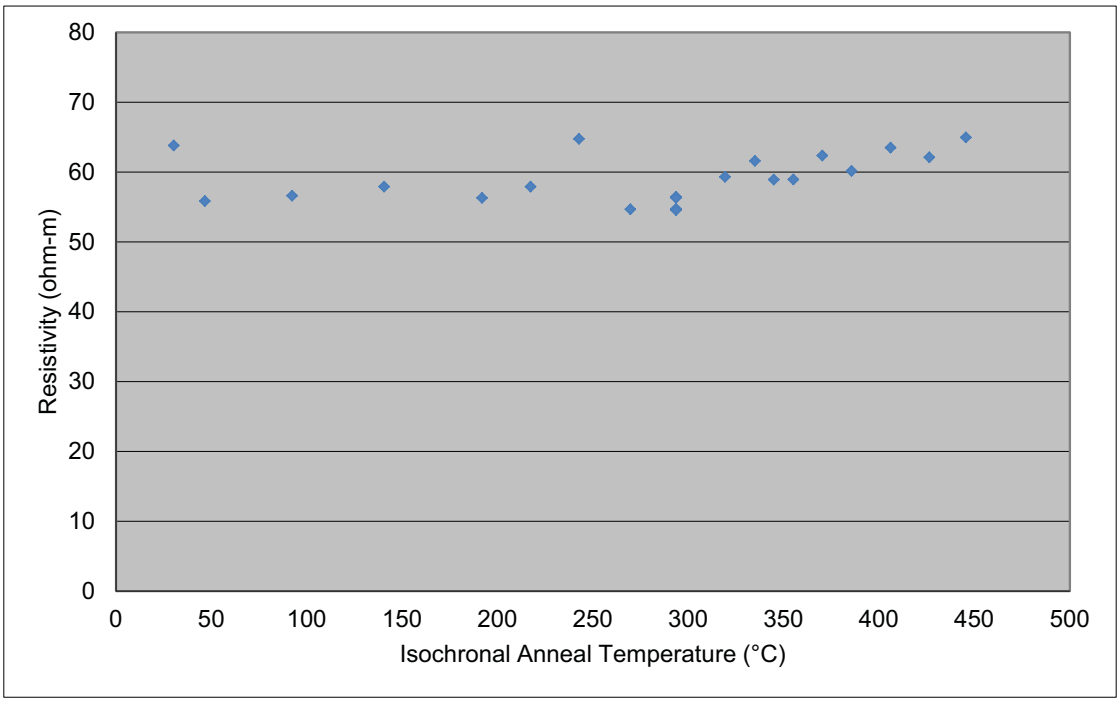

Figure 5. Resistivity data for the reference monitor.

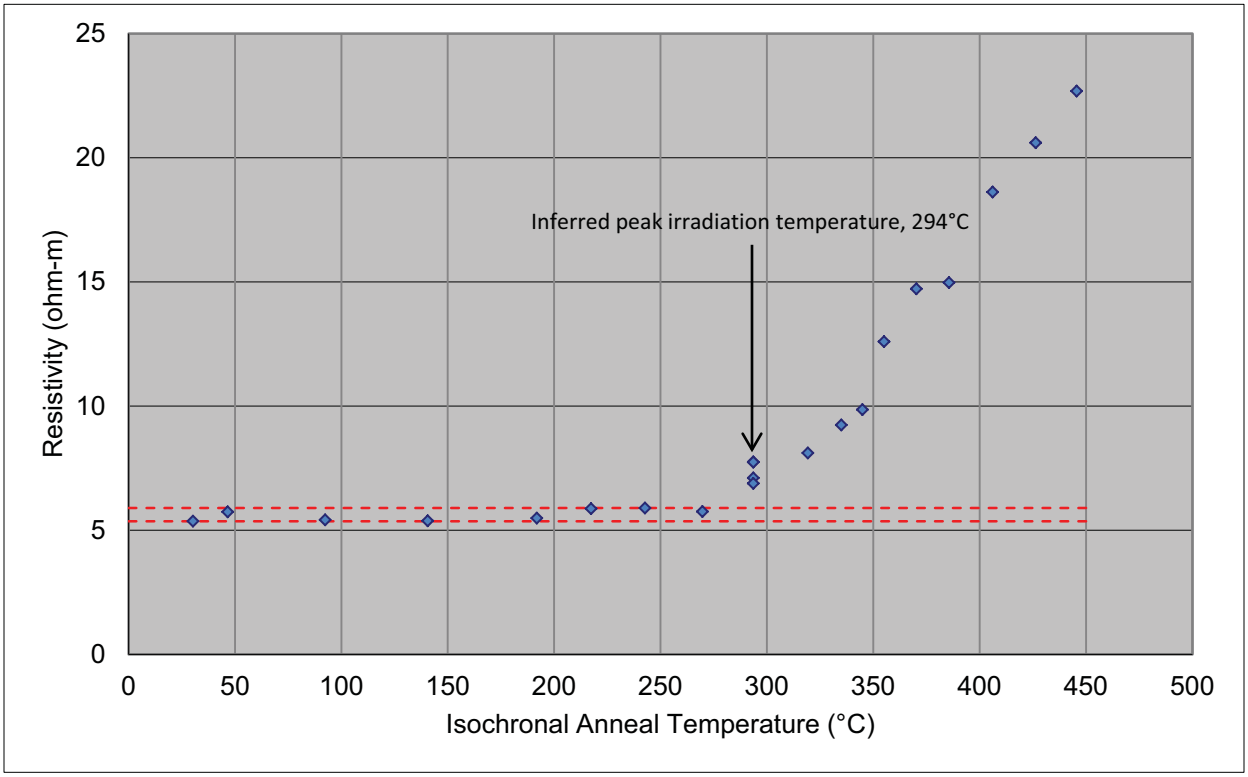

Figure 6. Resistivity data for the 400 LO B monitor.

4. Results

Table 2 summarizes peak irradiation temperatures inferred from resistivity measurements of the 300 LO and 400 LO B SiC monitors. The peak irradiation temperature inferred for the 300 LO monitor was 295 ± 20°C. This temperature was within the expected peak irradiation temperature range. The peak irradiation temperature inferred for the 400 LO B monitor was 294 ± 25°C. Post irradiation thermal analysis may prove helpful in explaining the disparity between the peak irradiation temperature (294°C) and the expected irradiation temperature range (411- 442°C). No anomalous data were obtained during the SiC resistivity measurements. Data obtained from an unirradiated reference SiC monitor did not indicate that any oxidation occurred during annealing.

Table 2. Test results for the SiC monitors tested.

Monitor Identification	Inferred Peak Irradiation Temperature (°C)	Error (°C)	Expected Peak Irradiation Temperature Range (°C)
05R4-02-A KG1403 (300 LO)	295	±20	278 - 343
05R4-01-A KG1415 (400 LO B)	294	±25	411- 442

5. References

1. MacLean, H.J, and K. Sridharan, T. A. Hyde, “Irradiation Test Plan for the ATR National Scientific User Facility - University of Wisconsin Pilot Project,” PNL-2784, Revision 1, Idaho National Laboratory, July 21, 2008.

2. J. L. Rempe, K. G. Condie, D. L. Knudson, and L.L. Snead, “Silicon Carbide Temperature Monitor Measurements at the High Temperature Test Laboratory,” INL/EXT-10-17608, January 2010.

3. Rempe, J.L. and K.G. Condie, D. L. Knudson, “Silicon Carbide Temperature Monitor Evaluation,” PLN-3473, Revision 0, Idaho National Laboratory, May14, 2010.

ATTACHMENT A

ROHM AND HAAS
Advanced Materials, Woburn Ma.

Material Analysis

Part Number	12.5mm x 1mm x .75mm thick plate SC003
Customer	Tyler Gerczak
Customer P.O. Number	N/A
Material	CVD Silicon Carbide
Resistivity	> 1,000 Ohm-cm

Gas Discharge Mass Spectroscopy Measurement

Element	Concentration [ppm wt]	Element	Concentration [ppm wt]	Element	Concentration [ppm wt]	Element	Concentration [ppm wt]
Li	< 0.001	Mn	< 0.01	Ag	< 0.01	Tm	< 0.005
Be	< 0.005	Fe	< 0.01	Cd	< 0.05	Yb	< 0.005
B	0.47	Co	< 0.005	In	< 0.01	Lu	< 0.005
C	Matrix	Ni	< 0.01	Sn	< 0.05	Hf	< 0.005
N	=< 10	Cu	< 0.05	Sb	< 0.01	Ta	< 5
O	=< 70	Zn	< 0.05	Te	< 0.01	W	< 0.01
F	< 0.1	Ga	< 0.05	I	< 0.005	Re	< 0.01
Na	0.06	Ge	< 0.05	Cs	< 0.005	Os	< 0.01
Mg	< 0.01	As	< 0.01	Ba	< 0.005	Ir	< 0.005
Al	< 0.01	Se	< 0.05	La	< 0.005	Pt	< 0.01
Si	Matrix	Br	< 0.05	Ce	< 0.005	Au	< 0.05
P	< 0.01	Rb	< 0.01	Pr	< 0.005	Hg	< 0.05
S	0.05	Sr	< 0.005	Nd	< 0.005	Tl	< 0.005
Cl	0.19	Y	< 0.01	Sm	< 0.005	Pb	< 0.005
K	< 0.05	Zr	< 0.01	Eu	< 0.005	Bi	< 0.005
Ca	< 0.05	Nb	< 0.01	Gd	< 0.005	Th	< 0.001
Sc	< 0.001	Mo	< 0.05	Tb	< 0.005	U	< 0.001
Ti	0.02	Ru	< 0.01	Dy	< 0.005		
V	< 0.005	Pd	< 0.01	Ho	< 0.005		
Cr	< 0.1	Rh	< 0.005	Er	< 0.005		

ATTACHMENT B

Heather J MacLean Chichester/MACLHJ/CC01/INEEL/US
08/31/2011 12:45 PM

To: Joy L Rempe/YOJ/CC01/INEEL/US
cc: Collin J Knight/KNIGCJ/CC01/INEEL/US@INEL, darrell.knudson@inl.gov, Kurt L Davis/DAVIKL/NON/INEEL/US@INEL
bcc:
Subject: Re: Fw: HF Wash of SiC monitors at CFA-625

Hi Joy,

The calculated temperatures (based on planned rather than actual irradiation) were:

SiC from 300 LO capsule:
minimum T = 287 C
maximum T = 343 C

SiC from 400 LO B Capsule:
minimum T = 411 C
maximum T = 442 C

We had a possible shift in irradiation position during 1 cycle, so the temperature values could be slightly higher than originally calculated, but the range should give you a good idea.

Please let me know if you have other questions.
Thanks,
Heather

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Joy L Rempe/YOJ/CC01/INEEL/US

Joy L Rempe/YOJ/CC01/INEEL/US
08/24/2011 04:50 PM

To: Collin J Knight/KNIGCJ/CC01/INEEL/US@INEL
cc: Heather J MacLean Chichester/MACLHJ/CC01/INEEL/US@INEL, darrell.knudson@inl.gov, Kurt L Davis/DAVIKL/NON/INEEL/US@INEL
Subject: Re: Fw: HF Wash of SiC monitors at CFA-625

Thanks for the update. Heather, Could you let us know the approximate peak irradiation temperatures?

Joy