INL/EXT-15-36738
Revision: 1

Silicon Carbide Temperature Monitor Process Improvements

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The INL is a U.S. Department of Energy National Laboratory
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Prepared for the
U.S. Department of Energy
Office of Nuclear Energy, Science, and Technology
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

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1. Introduction

Silicon carbide (SiC) temperature monitors are used as temperature sensors in Advanced Test Reactor (ATR) irradiations at the Idaho National Laboratory (INL). Although thermocouples are typically used to provide real-time temperature indication in instrumented lead tests, other indicators, such as melt wires, are also often included in such tests as an independent technique for detecting peak temperatures incurred during irradiation. In addition, less expensive static capsule tests, which have no leads attached for real-time data transmission, often rely on melt wires as a post-irradiation technique for peak temperature indication. Melt wires are limited in that they can only detect whether a single temperature is or is not exceeded. SiC monitors are advantageous because a single monitor can be used to detect for a range of temperatures that occurred during irradiation.

As part of the process initiated to make SiC temperature monitors available at the ATR, post-irradiation evaluations of these monitors have been previously completed at the High Temperature Test Laboratory (HTTL). INL selected the resistance measurement approach for determining irradiation temperature from SiC temperature monitors because it is considered to be the most accurate measurement [1]. The current process involves the repeated annealing of the SiC monitors at incrementally increasing temperatures with resistivity measurements made between annealing steps. The process is time consuming and requires the nearly constant attention of a trained staff member. In addition to the expensive and lengthy post analysis required, the current process adds many potential sources of error in the measurement, as the sensor must be repeatedly moved from furnace to test fixture. This time-consuming post-irradiation analysis comprises a significant portion of the total cost of using these otherwise inexpensive sensors. An additional consideration of this research is that if the SiC post processing can be automated it could be performed in an MFC hot cell further reducing the time and expense of length decontaminations prior to processing.

Section 2 of this report provides a general description of resistivity techniques currently used to infer peak irradiation temperature from silicon carbide temperature monitors along with some representative data. Section 3 describes the proposed concepts to improve the process of analyzing irradiated SiC temperature monitors. Section 4 describes the completed efforts to prove the proposed concepts. Section 5 describes the future activities. References cited in this plan are listed in Section 7.

2. Background

Since the early 1960s, SiC has been used as a post-irradiation temperature monitor. Several researchers have observed that neutron irradiation-induced lattice expansion of silicon carbide annealed out when the post-irradiation annealing temperature exceeds the irradiation temperature [1]. Some researchers attribute this irradiation-induced swelling with point defect formation, while others indicate that interstitial cluster formation affects this swelling. Peak irradiation temperatures from SiC monitors have primarily been inferred from length measurements after isochronal annealing steps, but several researchers have investigated other techniques, such as x-ray line broadening to detect lattice parameter changes or changes in thermal diffusivity, density, and electrical conductivity. However, Snead et al. [2] recommend using changes in electrical resistivity for improved accuracy, ease of measurement, and reduced costs. Additionally, the researchers recommend using SiC produced via chemical vapor deposition (CVD). The large variation of resistivity observed in CVD produced SiC (from 1 to 10^5 ohm-cm) is primarily dependent on the level of dopant impurities. Hence, the authors stress that the SiC material should be fully dense (3.203 g/cm^3) and stoichiometric. In addition, it is recommended that samples be immersed in a dilute hydrofluoric wash to remove any surface oxidation that would alter the resistivity measurements. Furthermore, because SiC is temperature-dependent, Snead et al. emphasized that measurements should be taken in a controlled environment (within 0.4 °C) for isochronal annealing periods of approximately 30 minutes. Comparisons of temperatures inferred from SiC measurements and thermocouple data indicate that accuracies of approximately 20 °C are possible for dose ranges of 1 to 8 dpa and temperatures from 200 to at least 800 °C. Absolute temperature limits for resistivity techniques are typically stated as 150 °C (an amorphorous threshold) and 875 °C (due to recrystallization), but work by Snead [3] indicates that electrical resistivity techniques may also provide insights beyond these temperature limits.

Figure 2-1 shows data obtained from a SiC sample that had been irradiated in the ATR [4]. The test capsule contained a Rohm Haas CVD SiC sample with a nominal size of 12.5 mm x 1.0 mm x 0.75 mm. The curve in **Figure 2-1** indicates that the SiC temperature monitor resistivity increases at ~ 294 °C. Results in **Figure 2-1** are representative of data obtained from this technique. As indicated in this figure, the peak irradiation temperature is identified when data extend beyond a band corresponding to the maximum and minimum values measured at low temperatures and continues to exponentially increase (or decrease) with increasing temperature anneals.

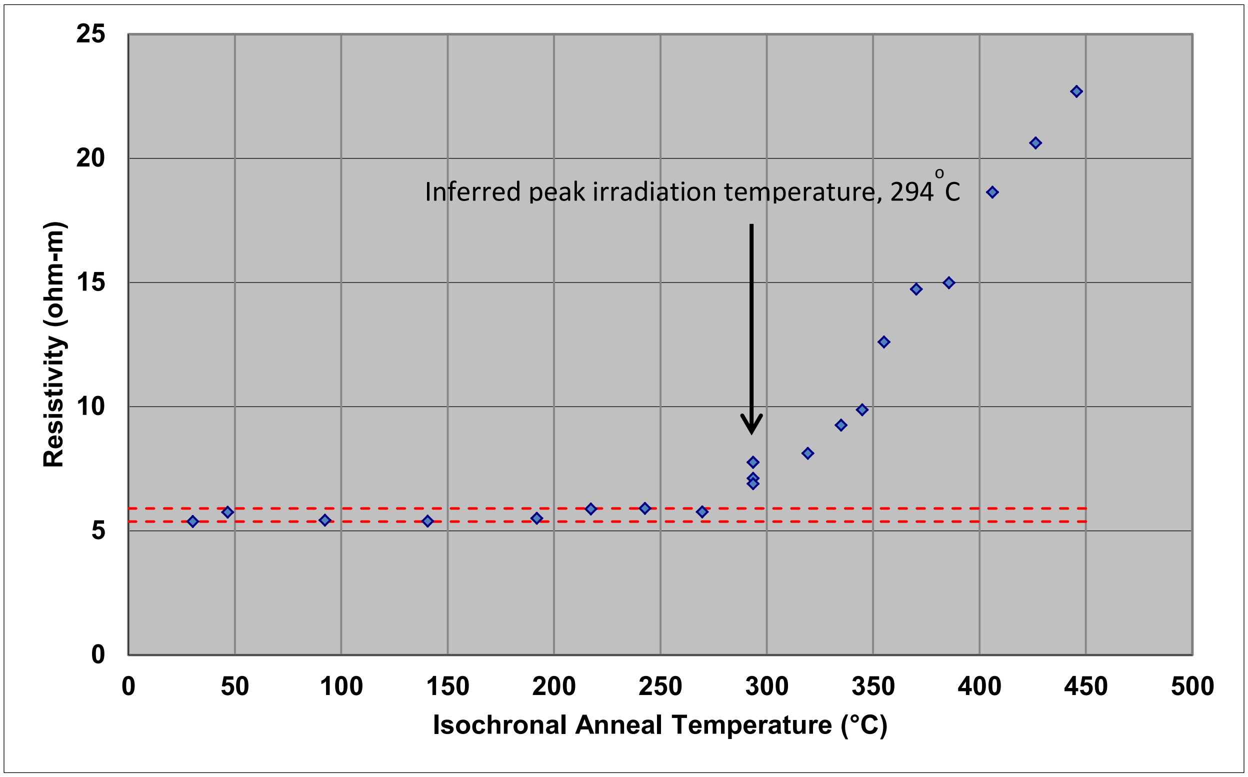

Figure 2-1. Typical electrical resistivity response for post-irradiation SiC monitor temperatures[2].

There are several limitations associated with the use of SiC temperature detectors. As discussed above, temperatures are inferred by post-irradiation detection of changes in the stable defect population within SiC monitors that were incurred during irradiation. As noted above, experience indicates that results have an increased accuracy when the monitors are obtained from fully dense (3.203 g/cm^3), CVD SiC and when monitors are irradiated at a constant temperature. Additional research [5] cites several specific examples where errors could be inferred from SiC monitor measurements:

- *Irradiation temperatures rising during the latter part of irradiation.* SiC swelling saturates at low fluence. For damages greater than > 0.1 dpa, the increasing temperature will anneal out defects that occur at the lower irradiation temperature, while creating stable defects at the higher temperature. When isochronal annealing is performed, lower temperature defects are removed, and the recovery curve will be smeared to somewhat higher temperatures. If the temperature increase during irradiation is not great, or the time at higher temperature not too long, then the original departure from linearity will still give the earlier irradiation temperature.
- *Irradiation temperatures decreasing during irradiation.* This decrease will lead to defects being created and frozen-in at the higher-temperature, while continuing to create lower temperature defects. The isochronal anneal will then give an indication of the lowest irradiation temperature (in this case at the end of the irradiation period) and the recovery curve will be smeared because it will continue to anneal higher and higher temperature stable defects.

- *Upward or downward temperature spikes during irradiation.* Depending on the time at temperature the effect will be to smear the recovery curve.

Note that there are no American Society for Testing and Materials (ASTM) standards governing the evaluation of SiC temperature monitors using resistivity techniques.

3. SiC Temperature Monitor Process Improvements

Two techniques have been identified that have the potential to improve on the currently used method for analyzing irradiated SiC temperature monitors. The primary purpose of each of these methods is to reduce the analysis time by automating the analysis process. This will be accomplished by performing the analysis "in-situ." That is, the sample will be placed in an annealing furnace and analyzed during the annealing process, eliminating the step-wise process currently used. This will have the secondary effect of eliminating the error sources introduced by repeated movement of the sensor from furnace to test fixture (i.e. misalignment of the sample, oxidation of the sample surface, etc.).

3.1 Real Time Resistance Measurements

The first method under development is simply a "real time" application of the current technique. Electrical resistivity of the SiC sample will be measured and used as the irradiation temperature indicator, but in this case the measurement will be made while the sample undergoes annealing in the furnace. A specialized, high temperature, test fixture will be used. The SiC monitor will be installed into the fixture for the duration of the analysis, allowing measurements to be made during the annealing cycle. The power analyzer currently used for making the electrical measurements allows for analysis of a maximum of four samples simultaneously. As such, it is envisioned that the "real time" process will allow for analysis of multiple samples, further reducing the necessary analysis time. It is possible that a reference sample may be required, to highlight changes in the temperature dependent electrical resistivity of the irradiated samples, but further testing is needed to verify this and will be completed in future evaluations. If this reference is needed, the current equipment will allow for the simultaneous analysis of at least three irradiated SiC monitors, but equipment could be duplicated should more simultaneous analysis be required to meet NSUF program needs.

Figure 3-1. Benchtop furnace continuous resistivity monitoring processing concept.

3.2 Real Time Visual Measurements

The second method under development relies on real-time visual examination of the dimensional changes of the SiC sample during annealing. As the sample is annealed, the defects that induced the material swelling will be repaired and the sample will change size. This visual analysis, or optical change detection, utilizes a transparent furnace that allows the sample to be photographed with a high resolution camera or microscope during the annealing. As the sample is heated and subsequently cooled the dimensional changes for each temperature will be quantified by a computer-based measurement program to locate the transition temperature at which defects are annealed out of the sample. If successful, it is expected that this visual analysis method could be coupled with the electrical resistivity measurements as a method for real-time validation by correlating separate effect measurements.

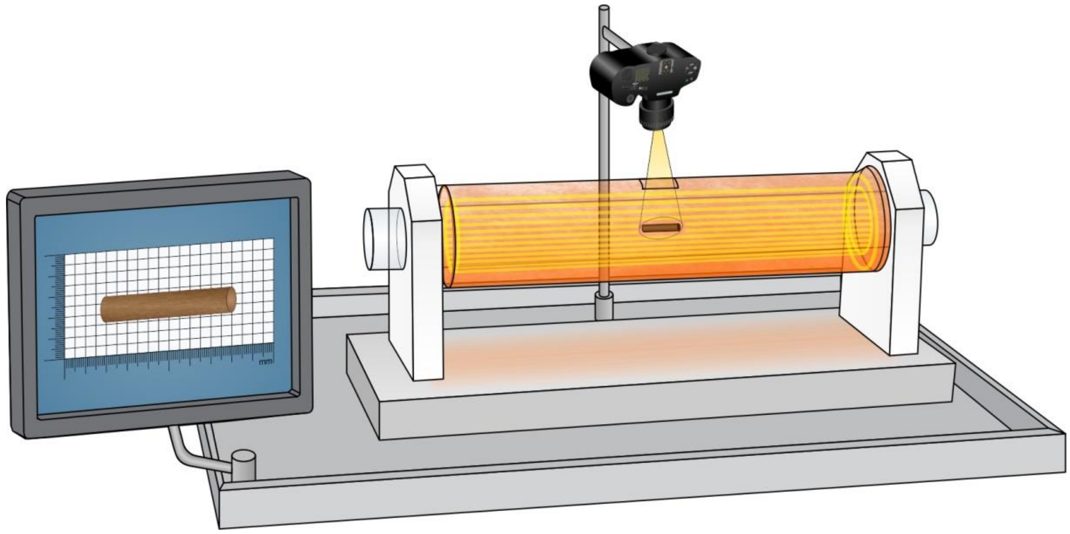

Figure 3-2. Optical change detection-based SiC processing concept.

4. Research Activities

Efforts focused on the design and development of the automation system, including the design, selection, and procurement of the hardware, the assembly and integration of the system components, the development of a preliminary SiC monitor fixture, the design of a technique to automate the data analysis and processing, the design and development of the communication, coordination, and user software, and the execution and troubleshooting of test experiments using the box furnace. The following sections will describe the automation system architecture, and the performed test run experiments. In addition, the transparent tube furnace was received and installed.

4.1 Automation System Architecture

The objectives of the targeted system are that it should be able to read the experiment plan, execute the experiment according to the plan, acquire and store real-time data, enable a user-friendly interface, and execute multiple experiments simultaneously. The system design was performed in the initial stage of this effort, and is shown in **Figure 4-1** . A desktop computer (operator station) was connected to a controllable furnace, a thermocouple interface module, and a resistivity measurement device through a USB and RS-485 serial connection. The following sections will describe the hardware and software designs of the system.

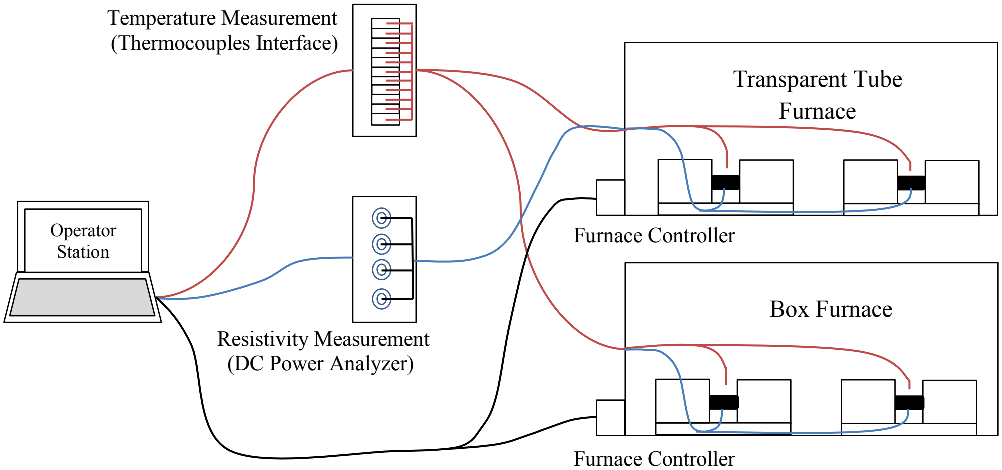

Figure 4-1. The proof-of-concept experimental system layout.

4.1.1 Hardware Selection and Design

The system was designed to fit in a compact portable cabinet as shown in **Figure 4-2**. The target was to have a standalone system that is capable of running the whole experiment with minimal user interaction.

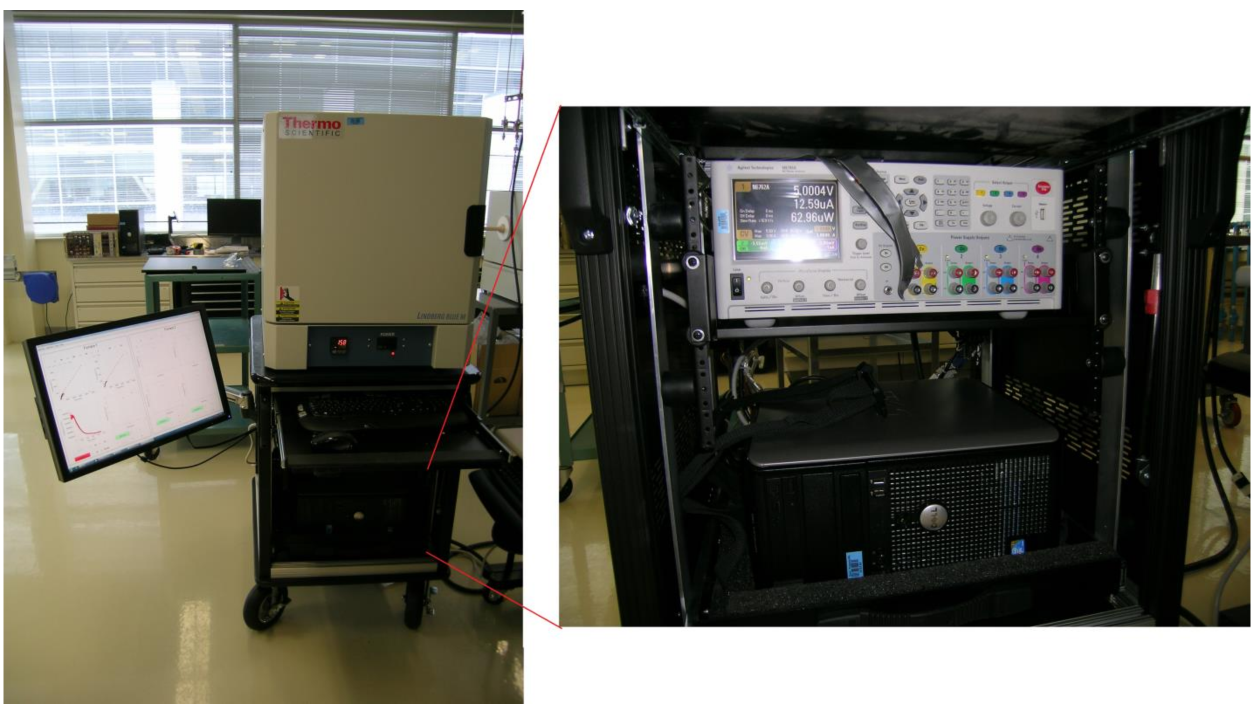

Figure 4-2. The automation system cabinet.

Box Furnace

The Thermo Scientific Lindberg/Blue M™ Moldatherm™ box furnace was selected due to its high heat-up and cool-down rates, and its ability to maintain a uniform heat distribution. It is equipped with a Yokogawa UP150 temperature proportional–integral–derivative (PID) controller. The controller was connected to the operator station through the furnace RS-485 port with an RS-485 cable, and an RS-232 to RS-485 converter. The furnace upper vent was used as a pathway for the thermocouples and wiring.

Resistivity Measurement Device

The resistivity of SiC can vary significantly based on the monitor design. At low temperatures, it is expected that the resistance would be significantly high. As temperature increases to the desired 800 °C, the monitor resistance will drop in orders of magnitude. It is also recommended that SiC resistance measurement be performed at voltages between 2 and 8V [6]. These requirements favored a device that has a very wide range for current measurement, specifically from fractions of a microampere (μA) to several milliampere (mAs) at the recommended voltage. The Keysight N6705A DC power analyzer, which was used in earlier manual measurements, met these requirements. The device is equipped with four modular channels that are equipped with N6762A Precision DC power modules. In a 4 wires setup, these modules fix the voltage on two wires and measure the corresponding current on the other two wires to determine the monitor resistivity.

Temperature Measurement and Interface Device

The main requirement for the temperature measurement instrument was the capability to measure the targeted maximum temperature of 800 °C, and allow a significant temperature safety margin. As a result, it was decided to use Omega K-Type sheathed thermocouples. The thermocouples signals are acquired by a National Instruments USB 9213 device. The use of this dedicated thermocouple measurement acquisition device ensured high measurements accuracy. The device has 16 channels and was connected through a USB cable to the operator station. It was planned that each experiment will acquire three thermocouples measurements through three channels and one channel will be kept as a spare. Though the current system was developed with one setup in the box furnace, the system is designed to accommodate two setups per furnace. The two setups will facilitate comparing irradiated and non-irradiated SiC monitors in the same furnace, i.e. under the same conditions at the same time. This targeted design resulted in the total 16 channels being utilized in the final system.

SiC Monitor Fixture

The sample holding fixture was designed to meet four requirements. The fixture is required to tolerate a temperature that exceeds 800 °C by a safety margin, it should be electrically nonconductive except for the SiC monitor holding part, it should have a very low temperature expansion coefficient, and should facilitate easy placement and removal of SiC monitors. The last requirement was set to enable placement and removal of the SiC monitor if placed in a radioactive environment or hot cell that requires placement with manipulators. The initial design of the fixture is shown in **Figure 4-3**. Quartz was chosen for the fixture structure since it can tolerate high temperatures, has a low thermal expansion coefficient, and has negligible electrical conductivity. Nickel (Ni) was chosen for the conducting rods due to the same reasons except that it has good electrical conductivity. The side holes were designed to allow thermocouples with 1.6 mm sheath to pass through the fixture. This was designed to place the thermocouple in proximity of the actual monitor for accurate temperature measurement at the monitor middle part and at its contacts with the Ni holders. Due to manufacturing restrictions, the manufactured fixture was modified to the one shown in **Figure 4-4** during the production process. The thermocouples were inserted as shown in **Figure 4-5**. However, a significant distance was preserved between the thermocouples and the fixture to account for the thermocouples thermal expansion and the undesired possibility of the thermocouples touching the SiC monitor.

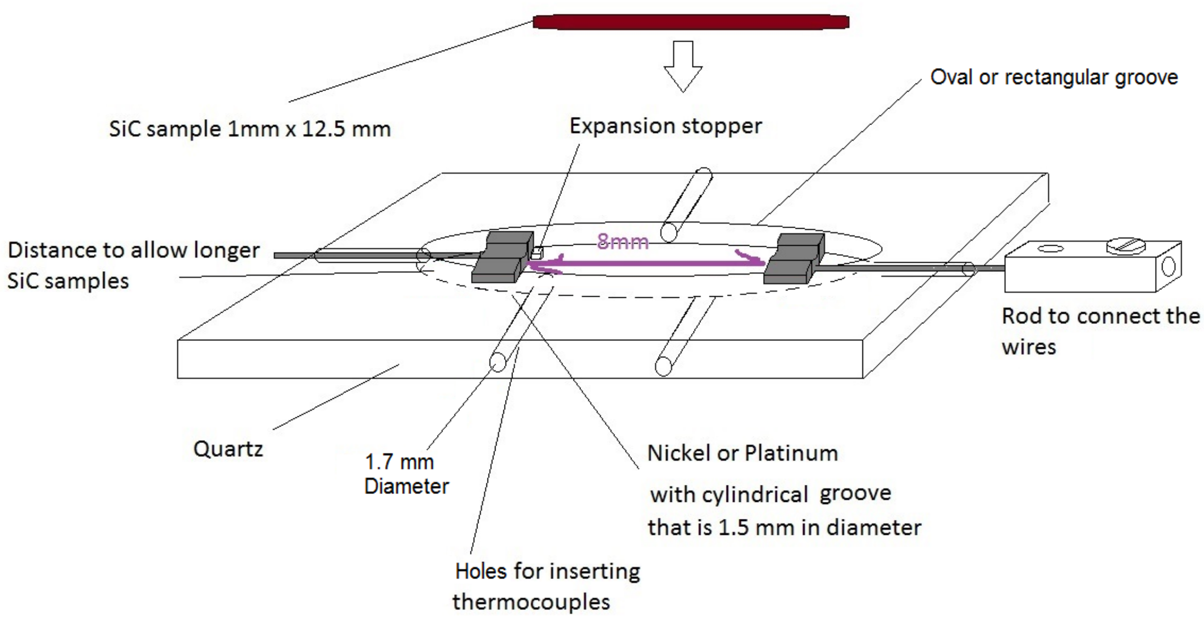

Figure 4-3. Preliminary fixture design.

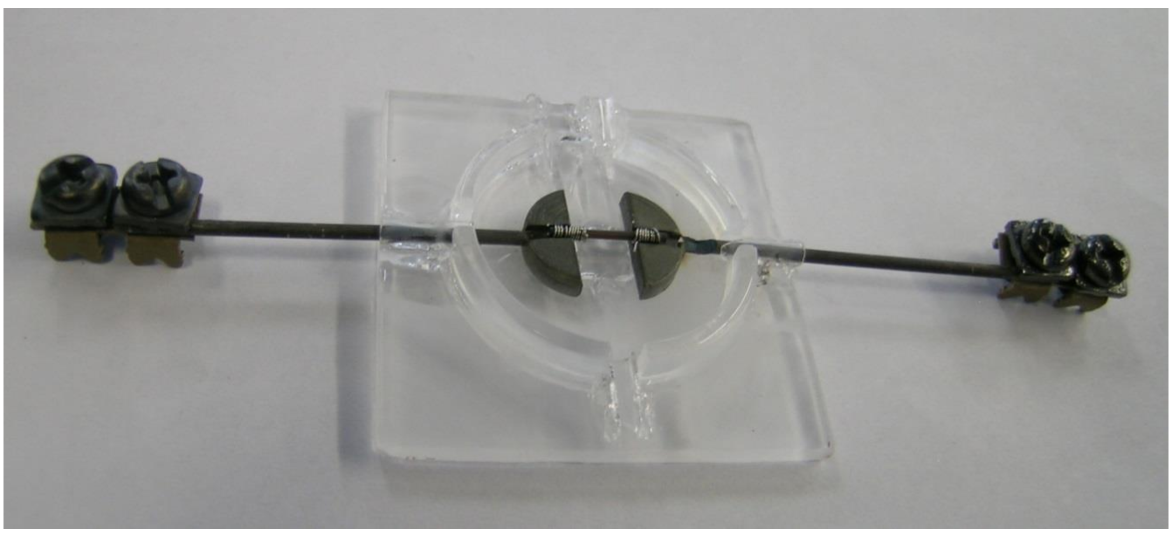

Figure 4-4. Manufactured fixture.

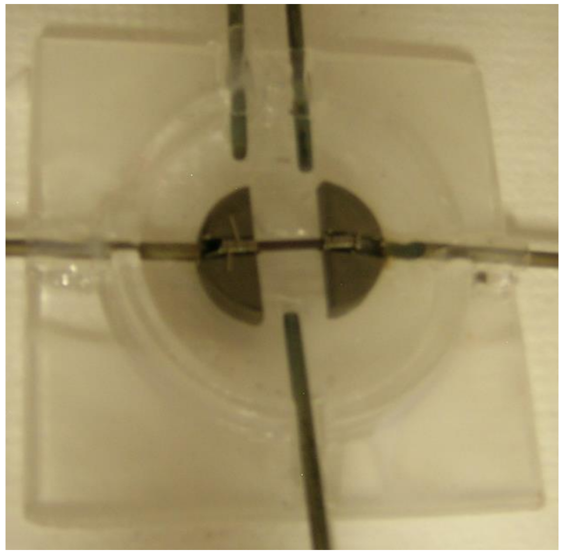

Figure 4-5. Thermocouples placement into the fixture.

4.1.2 Software Design

The diversity of equipment vendors used in the project favored the internal development of the software to accommodate the various communication methodologies, and facilitate significant design flexibility. The software was developed in the C# programming language to perform two tasks that are split on two layers (shown in orange in **Figure 4-6**). The first is the operator layer, which focused on enabling the desired functionality in a user friendly manner. The second layer is the equipment layer. This layer incorporated the code to communicate to the box furnace, the temperature measurements device, and the resistivity measurement device.

Operator Layer

This layer facilitates the graphical user interface (GUI) of **Figure 4-7**. The figure is split into two areas to interface to two furnaces. Each furnace has two columns of plots, one for every SiC monitor measurement. The top plot in each column is the temperature plot as a function of experiment time. It includes the temperature measurement from the furnace, the three thermocouples, and the furnace plan (temperature set points). The lower plot is the resistance measurement as a function of time. The furnace parameters are shown above the plots, while the specific fixture temperature measurements are shown below the plots. The two buttons per experiment, located below the plots, allow the user to stop and start each experiment separately. The menu items on the top of the window are used to load an experiment plan, view the plan, plot the data, store the data on demand, and access the equipment documentations. The experiment plan is a table of desired furnace temperatures as a function of time. For simplicity, it was decided that the most convenient method to define an experiment plan is to enable it to be prepared in one of the common tables software packages, i.e. Microsoft Excel, then enable the Excel file to be loaded into the GUI. After a plan is loaded, the start button is enabled, and the experiment can be started. During an experiment, the operator interface buffers the measurements received from the equipment layer then uses the data to update the GUI. Once a minute, the layer stores the data and associated time stamps into a set of Excel files located in the application folder. Data storage can be forced at any moment of time using the Data menu item. The data menu item also enables the user to view an experiment in a detailed plot as shown in **Figure 4-8**. This window includes details such as the furnace set point and output, and facilitates simple plot manipulation such as zooming in and out, and storing the plot in a graphical format.

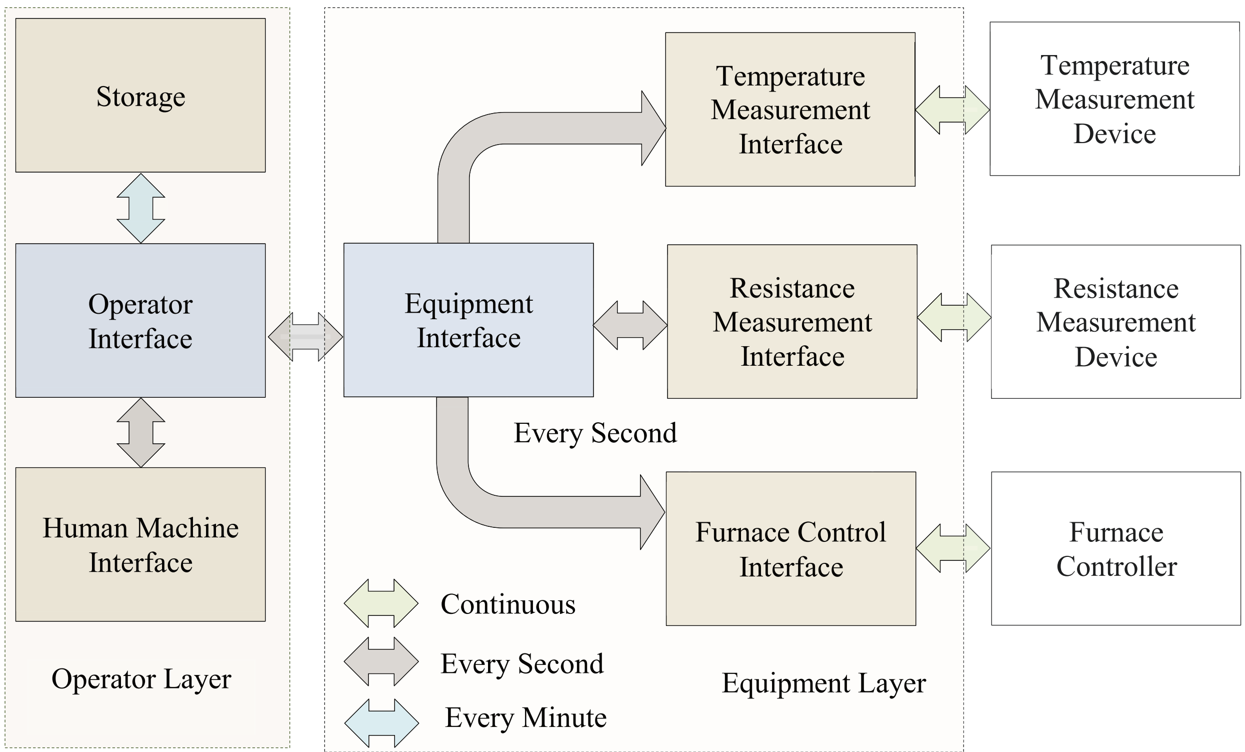

Figure 4-6. Software layers and communication pathways.

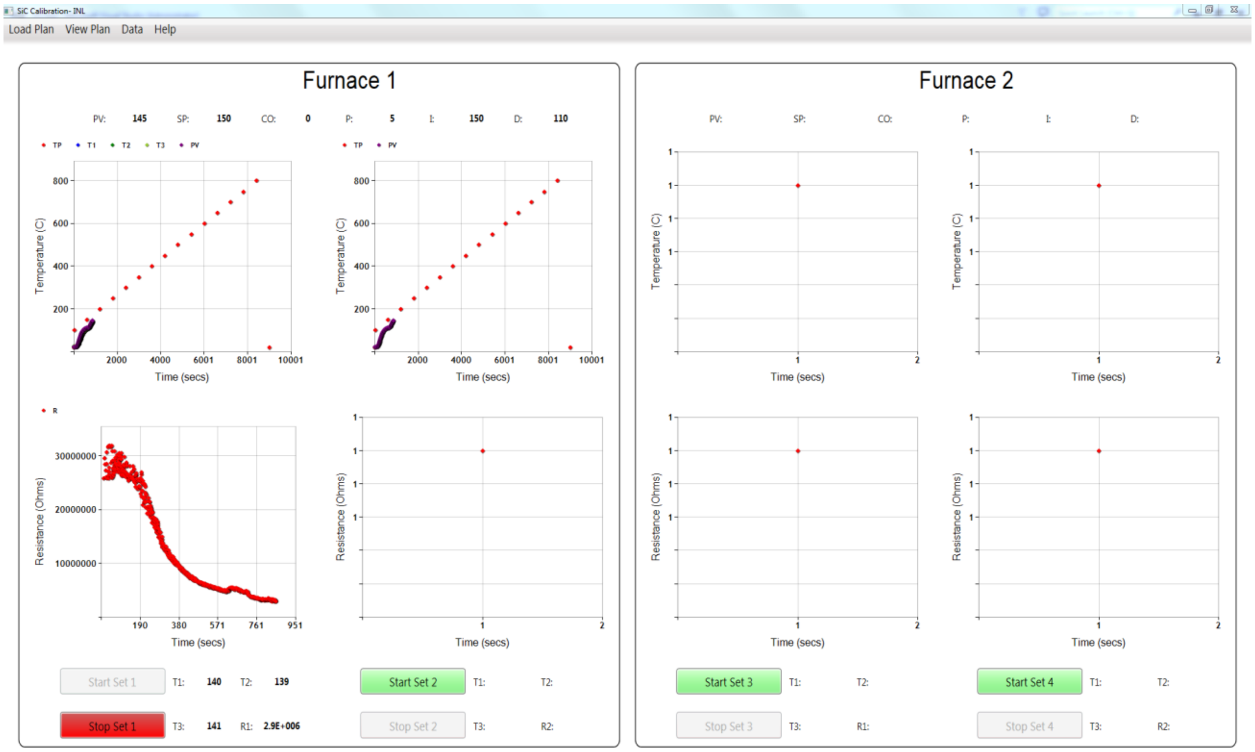

Figure 4-7. GUI layout.

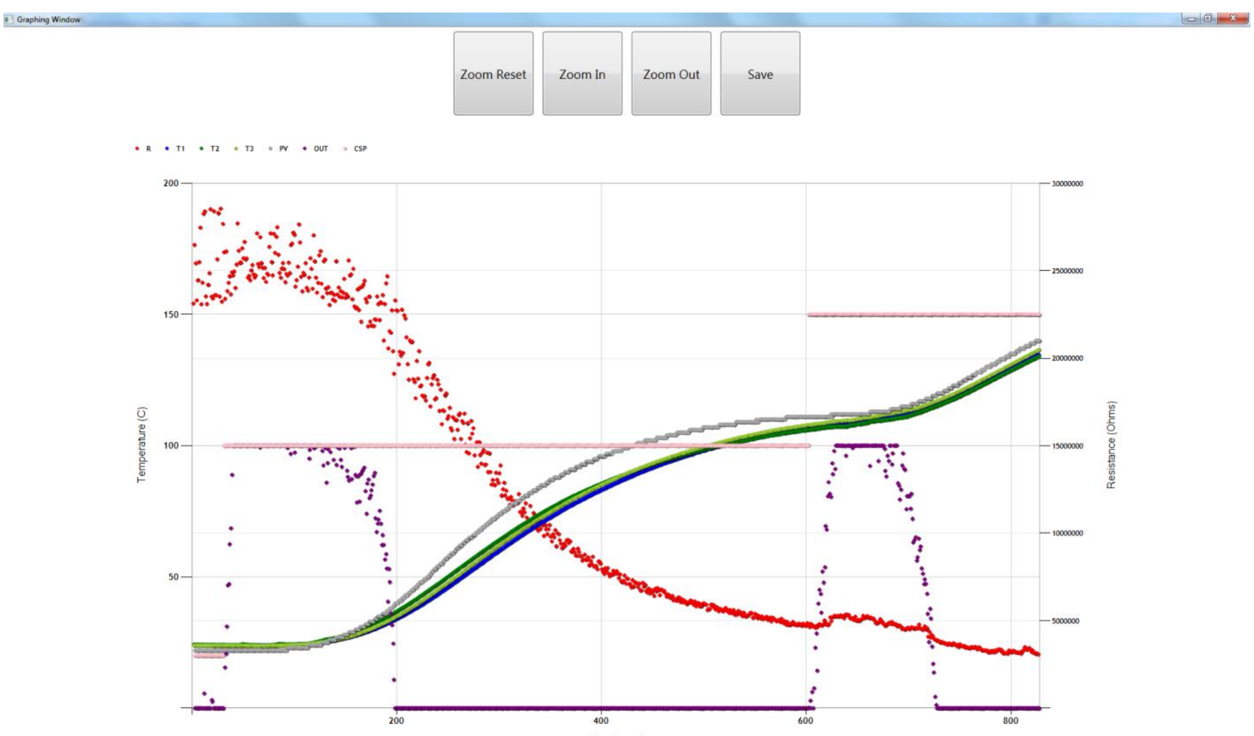

Figure 4-8. Detailed data plot.

Equipment layer

This layer establishes an independent computer thread to communicate with each piece of the equipment. It is responsible for establishing the communication link, exchanging the data, and closing the link if the application is closed or the data acquisition has stopped. The layer acquires one measurement every second (or more) from every equipment driver, which is continuously acquiring data from the equipment. As soon as a measurement is acquired, a time stamp is created and stored with the measurement as a data point. Time stamps are equipment specific. They are created for each piece of data acquired from each device to ensure accurate time mapping of data. The communication was designed to be persistent and robust. This implies that disconnecting a device would not terminate the communication link or affect the link to other equipment. As soon as the device is restored, the communication is restored too.

The developed interface drivers were specific to the equipment due to their specific nature of communication. The box furnace communication was based on ModBus. The link continuously acquires specific registers containing information about the furnace state and sends the relevant data to the operator layer. The mapping of the registers to information can be found in the Yokogawa UP 150 documents [7]. The furnace controller set point is set through ModBus too. It is read from the experiment plan, then tunneled through this link to drive the furnace. The set point is only sent to the controller if it is significantly different than the last sent set point. The communication used the cyclic redundancy check (CRC) error detection algorithm, which is part of the serial Modbus standard protocol package.

The interface driver of the temperature measurement device was based on the data acquisition library of the equipment vendor. This link implemented a recursive continuous call for data that is only terminated by the termination of the experiment or application. Every time a reading is requested by the equipment layer, ten readings per channel were acquired in less than a second and an average was sent back.

The resistivity measurement device had its own communication library; therefore, a specific interface was developed to link it to the operator station. The interface initially defined the measurement parameters such as the measurement voltage and measurement type. It, then, continuously acquired measurements and stored them in a buffer that is read by the equipment interface.

4.2 Test Run Experiments

In order to test the performance of the system, a test run was performed on one SiC monitor. The initial testing of the SiC monitor resistivity revealed a contact issue. The SiC monitor was not in good contact with the Ni holder. Applying external pressure improved the contact and reduced the resistance. This, however, indicated that the current gravity-based placement methodology is not suitable at the current stage. To overcome the contact pressure issue, it was decided to wrap the monitor with Ni wiring as shown in **Figure 4-9**. This process increased the surface contact of the monitor with the holder groove. In addition, as the temperature of the monitor increased, its radius increased due to thermal expansion. This increased the monitor experienced pressure by the Ni wiring and improved the contact.

The results of the first run are shown in **Figure 4-10**. The improvement of the contact due to thermal expansion is reflected in a rapid reduction of noise as shown at the beginning of the experiment. During the monitor heating process, it was observed that the monitor lost contact with the holder thus creating the random data shown in the solid red circles of **Figure 4-10**. This is due to the Ni wire expanding before the SiC monitor due to its higher thermal expansion coefficient thus deteriorating the contact. The contact was self-restored as the SiC expanded and came in touch with the wire again. During the monitor cooling process, it was observed that another loss of contact occurred. This can be explained by the monitor caused mechanical and thermal strain on the Ni wire, which caused it to sustain some damage and shrink at a lower rate than it expanded. To confirm these behaviors, another experiment was conducted, the results of which are shown in **Figure 4-11**. The results of the second experiment demonstrated similar results. The heating driven loss of contact was not self-restored, which required a manual external enforcement of the monitor back into contact. The loss of contact data of the solid red circle was omitted from the figure for clarity. The similarity of the two results indicated consistent loss of contact, which indicated that the holder design requires further research and development. Another observation that could be associated with the mechanical behavior of the monitor and holder is the huge hysteresis effect. This is demonstrated through the blue dashed lines of **Figure 4-10** and **Figure 4-11**. The resistance measurement of the element during the heating process differed over an order of magnitude from the resistance measurement at the same temperature during the cooling process. This behavior could be associated with the permanent damage experienced by the Ni wires. These issues are not included in the automation scope of this phase and remain to be investigated in future efforts.

Figure 4-9. SiC monitor wrapped by Ni wiring.

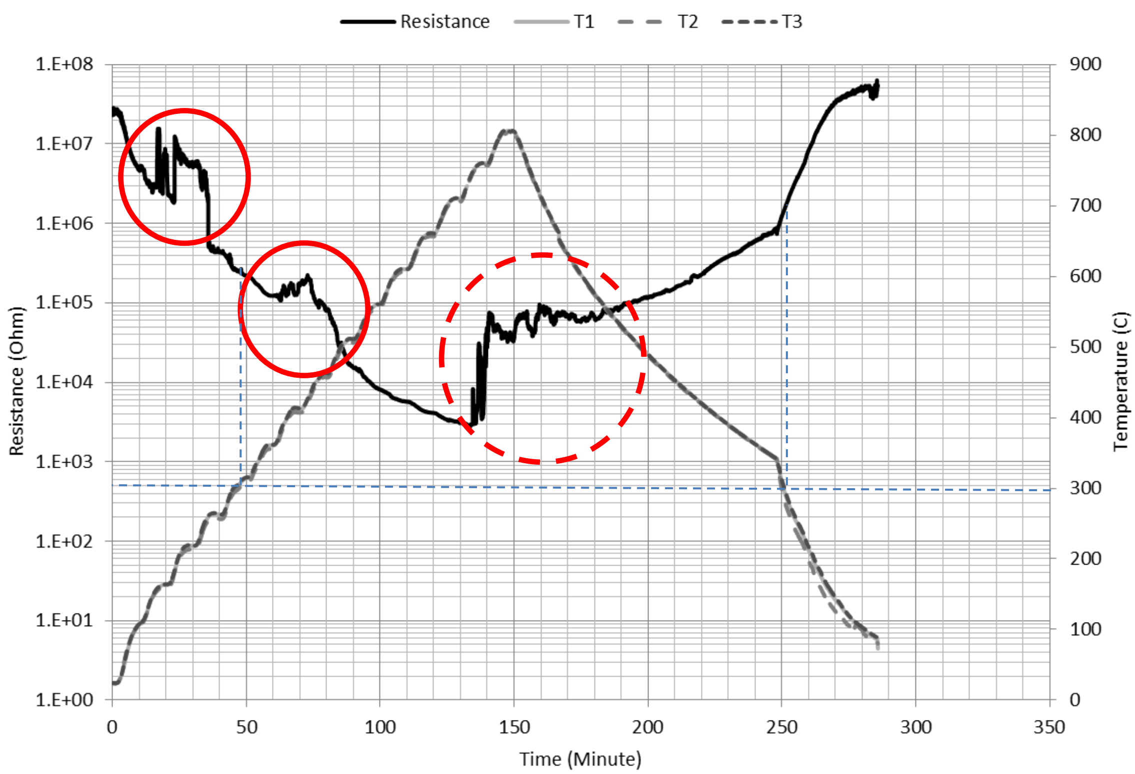

Figure 4-10. Results of the first test run experiment.

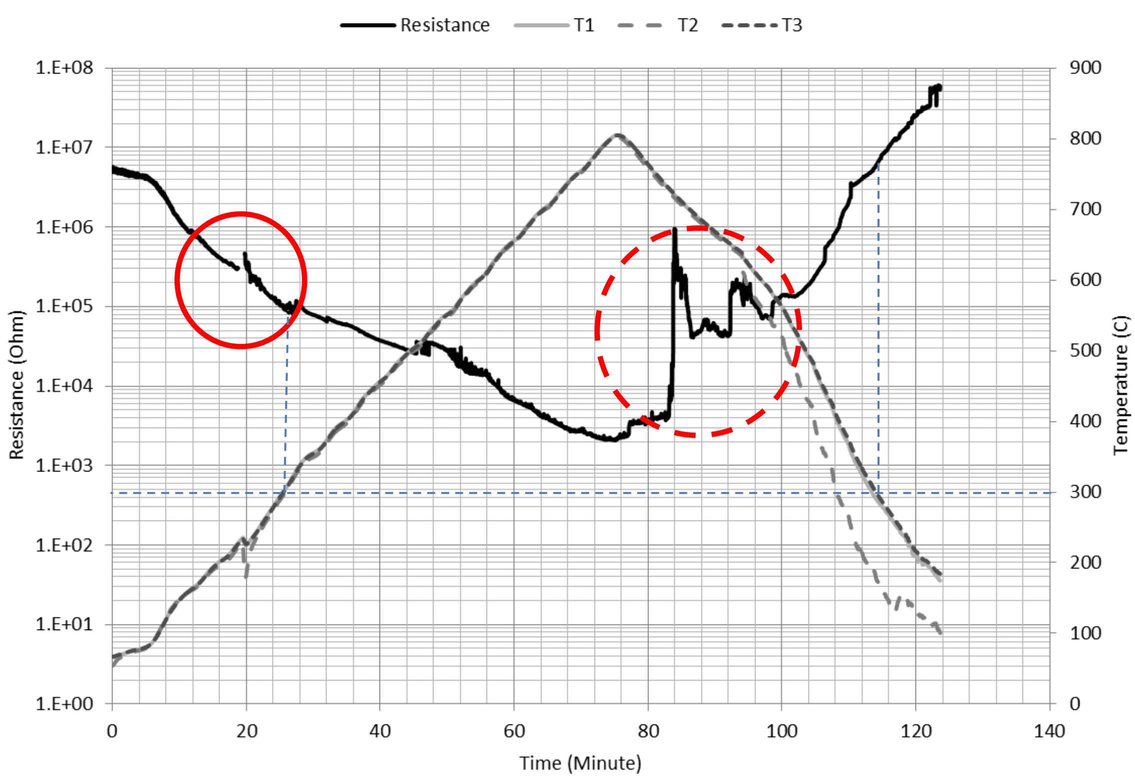

Figure 4-11. Results of the second test run experiment.

4.3 Real Time Visual Measurements

The transparent furnace used for visual examinations is the Thermcraft Trans Temp transparent tube furnace as shown in **Figure 4-12**. This furnace permits visual monitoring of the specimen geometrical deformation due to temperature changes, provides a more uniform temperature distribution than conventional furnaces, and is capable of causing rapid temperature changes. The furnace was ordered with a ~1” x 2” window to allow for visual examination throughout all annealing temperatures.

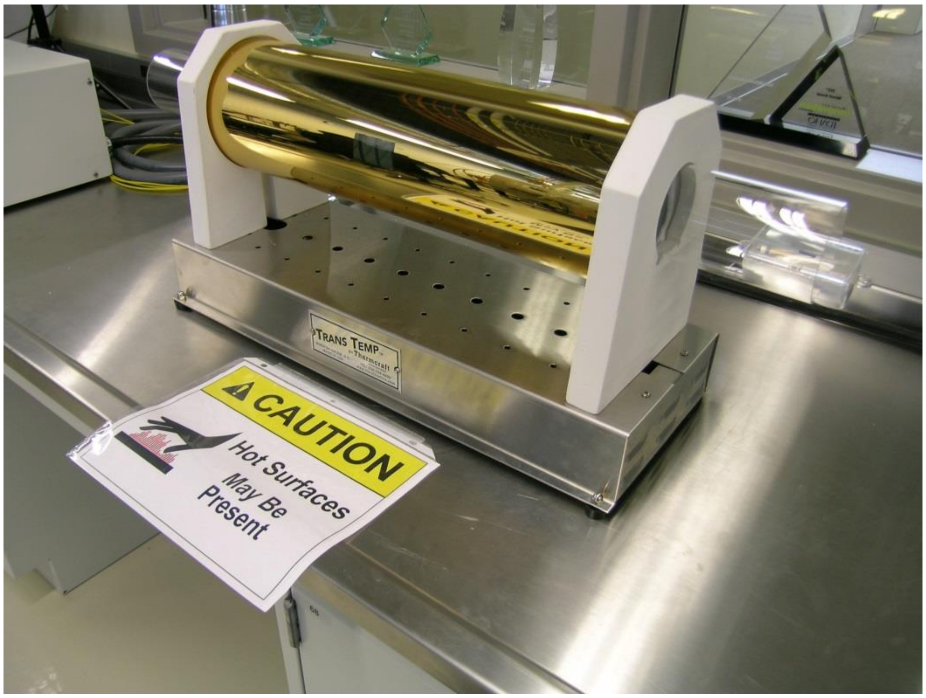

Figure 4-12. Thermcraft Trans Temp transparent tube furnace.

The transparent tube furnace has been installed and set up in the HTTL. The furnace was slowly brought to 1000 °C to observe the transition temperature where the gold reflector became transparent as shown in **Figure 4-13**. This temperature was at nearly 800 °C so the visual examinations will be performed in the specialized viewing window that was added.

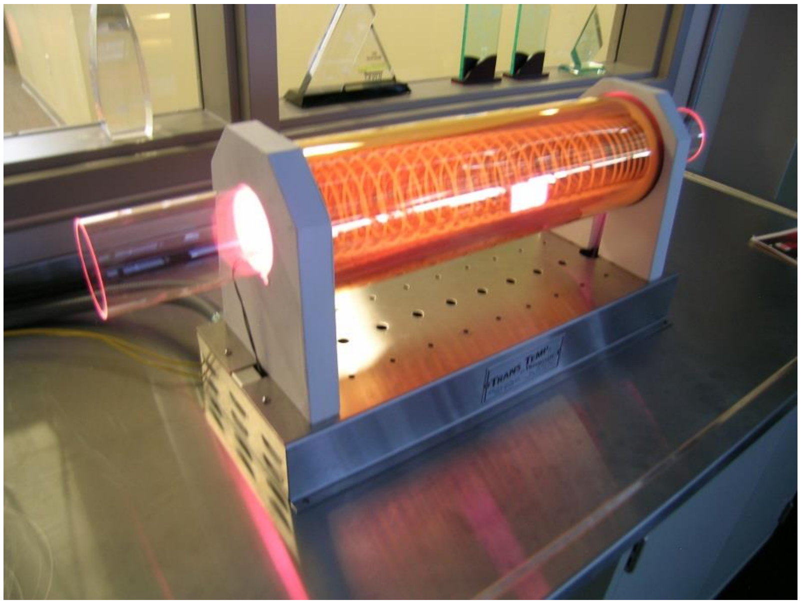

Figure 4-13. Transparent tube furnace during heating.

Additional quartz hardware to position the SiC sample in the center of the tube furnace was constructed as shown in **Figure 4-14**. This hardware will allow for accurate placement of the SiC sample in the viewing window of the furnace. Initial setup of the camera has begun as shown in **Figure 4-15**; however, temperature evaluations to quantify the temperature dependence of the SiC sample have not been performed.

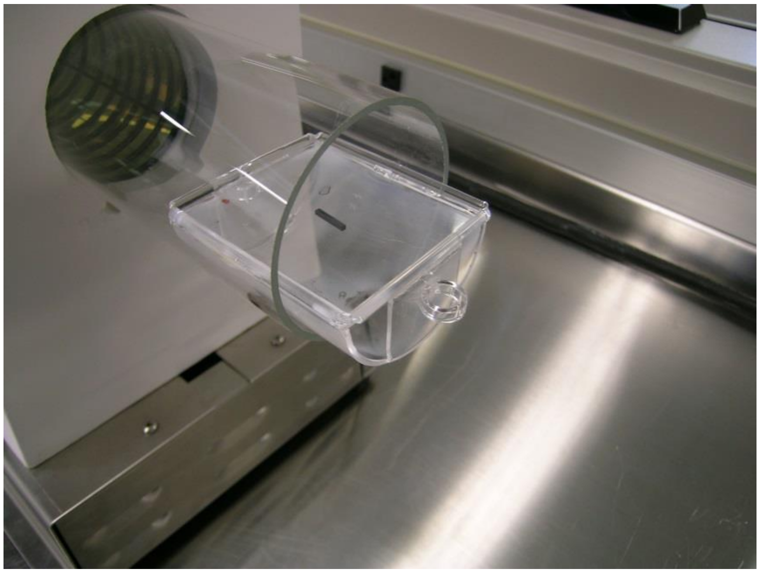

Figure 4-14. Quartz holder for tube furnace.

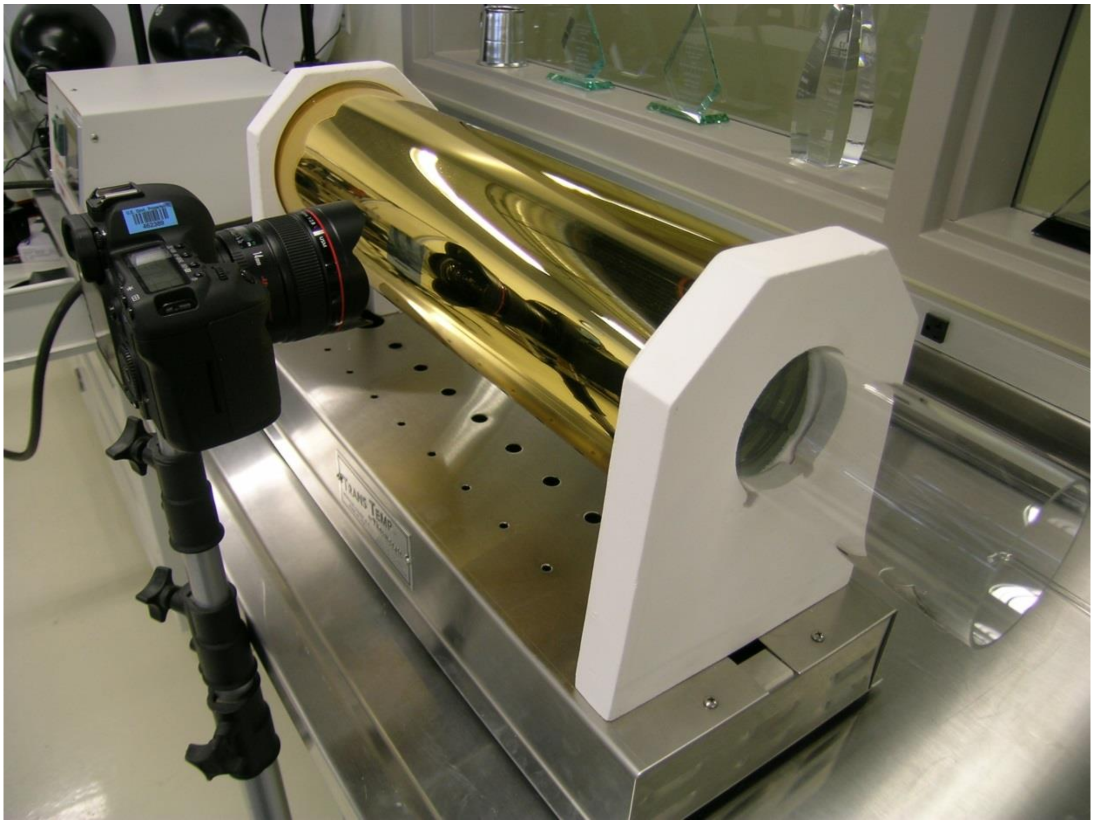

Figure 4-15. Initial setup for visual investigations with camera.

5. Future Work

Though the material selection of the fixture design demonstrated satisfactory behavior, the fixture design will require further research. The lack of good contact between the monitor and the Ni holding structure is a major source of uncertainty. The fixture and/or monitor need to be redesigned to overcome this issue. It is suggested that the next step involve testing of various geometrical shapes of monitors and further development of the current fixture design to improve its contact with the monitor, and improve its resilience to the mechanical and temperature-induced strain.

Once the contact issue is resolved, it is desired that work be carried out to incorporate the second channel of the box furnace using two fixtures, and perform a comparison of behavior between irradiated and non-irradiated SiC samples. The real-time acquisition of both measurements is expected to result in new insights about SiC monitor behavior.

In order to use the developed system for irradiated SiC measurements, it is recommended that future efforts focus on transforming the system into a wireless setup. Ideally, the setup would place the operator station in a distant location from the actual monitor to both reduce the experimenter dose and enable remote monitoring of the experiment.

Additionally, it has been suggested that future work be carried out to compare the geometrical behavior of SiC with the electrical resistivity. This will require integrating the transparent furnace and an optical acquisition system (programmable camera) into the current system in order to run experiments quantifying behavior during heating.

6. Summary

Efforts detailed here succeeded in designing and developing a real-time automated SiC resistivity measurement system, and performed two initial test runs. Activities completed included the evaluation, selection, and purchase of furnaces and data acquisition equipment required for the real-time measurement process improvements as well as the purchase of the SiC CVD samples to allow for experimental investigations. Additional activities carried out include the assembly and integration of the system hardware, the design and development of a preliminary monitor fixture, the design of a technique to automate the data analysis and processing, the development of the communication, coordination, and user software, and the execution and troubleshooting of test run experiments using the box furnace.

Although the automation system performed as required, the designed fixture did not succeed in establishing the needed electrical contacts with the SiC monitor. A workaround using Ni wiring succeeded in overcoming the weak electrical contact, but did not succeed in sustaining the contact through the heating and cooling process.

Future activities will focus on advancing the proof-of-concept to overcome the weak electrical contact of the SiC monitors. This includes advancing the fixture design, and testing various SiC monitor geometries. In addition, future work will focus on optical acquisition of SiC monitor, and enabling real-time comparison of irradiated samples with non-irradiated ones. Future activities could also include transforming the system into wireless communication if it is deemed necessary for hot cell implementation.

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