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Thermal Shock Resistance of Multilayer Silicon Carbide Receiver Tubes for 800°C Molten Salt Concentrating Solar Power Application

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Abstract. CSP power tower receiver systems during rapid transient weather periods can be vulnerable to thermal shock conditions from rain that which can facilitate the onset of leaks and failures that can have catastrophic consequences. Silicon carbide (SiC) materials have attractive receiver application characteristics for being light weight, having high-strength and excellent thermal shock resistance performance which make them a particularly good fit for receiver absorber materials in CSP. In this investigation, the performance characteristics of Ceramic Tubular Products (CTP) SiC ceramic matrix composite (CMC), multilayered tubes were explored with respect to thermal shock performance for solar receiver applications in next generation CSP plants. Here, thermal shock testing was performed at the Sandia National Laboratories (SNL) Solar Furnace facility using a dynamic stage and thermal shock tube test setup. The tubes tested under incident solar heat flux of 100 W/cm^2 were heated with inner tube temperatures reaching approximately 800 °C, with outer temperatures exceeding or just reaching 1000 □ for the multilayer and monolithic SiC tubes respectively. The tubes were then quenched with simulated rain. The tubes were then cooled and subjected to hoop stress analysis using an Instron device to assess their subsequent mechanical strength. The on-sun study experimental results indicate an average of 24.2% and 97% higher hoop strength for the CMC tubes than those composed of monolithic SiC and aluminum oxide (Al_2O_3) respectively.

INTRODUCTION

Traditional CSP power tower facilities employ stainless steel or high-temperature nickel-based alloys to facilitate molten salt flow and thermal absorption. Metallic receivers partly reflect solar light while strongly emitting infrared radiation when heated under incident solar flux, where spectrally-selective coatings may be deposited on the solar receiver to facilitate low reflectance in the visible and near infrared (NIR) regions for efficient solar radiation absorption [1]. However, high-temperature nickel-based alloys can have costs of three to five times that of lower-strength stainless steels [2], while the added costs of coating labor and reliability, requiring re-application maintenance and downtime can be substantial over the life of the plant. SiC, however is an attractive material for high-temperature solar receiver tubes due to its high strength, high thermal conductivity, low thermal expansion, high solar absorptance and chemical inertness at high temperatures [3]. However, monolithic SiC tubes can have challenges related to brittleness, which can cause failure due to thermal or mechanical shock. Ortona et al. [4] demonstrated the fabrication of SiC fiber-reinforced SiC matrix composites (SiC_f/SiC) for toughened high-temperature tubular receivers, which exhibited toughening as a result of crack deflection, crack bridging, fiber pull-out and delamination mechanisms at the interface between the fibers and matrix. In addition to SiC, Sani et al. [5] proposed using other ultra-high-temperature ceramics (hafnium and tantalum carbides) for use in high-temperature receiver applications. Their investigated carbides had melting temperatures above 3200 K and good thermophysical properties. Their results showed that the total hemispherical emissivity of the hafnium and tantalum carbides ranged from 0.2 to 0.6, which was considerably lower than the measured total hemispherical emissivity of silicon carbide (0.6 to 0.8).

To achieve higher-temperatures, with improved reliability, particularly with respect to thermal shock, SiC CMC's are proposed. SiC CMCs are a high-temperature class of materials that have been gaining interest as a lower-cost alternative to high-temperature metallic alloys. SiC CMC materials are made up of ceramic fibers that lie in a ceramic matrix phase. Here a SiC/SiC composite is made by having a SiC matrix phase and a fiber phase incorporated together through novel processing methodologies to facilitate a strong, damage tolerant composite structure. Additionally, as in the case of the CTP tubes, these CMC materials are combined with an inner layer of high density monolithic SiC to assure full containment, with zero leakage, of the heat transfer fluid, whether it be high temperature molten salt, liquid sodium, or a high temperature gas. Such multilayered SiC tubes have been developed and tested for use as Accident Tolerant Fuel cladding for commercial nuclear fuel as reported by Feinroth et. al [6].

For the CSP solar receiver application, the matrix composition and texture on the surface of the receiver tubes can be tailored to assure very high absorptivity and low emissivity of solar radiation. These materials exhibit outstanding properties which include high thermal, mechanical and chemical stability while also providing a high

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strength to weight ratio, which is attractive for high-temperature CSP receiver applications, with the goal of reaching heat transfer fluid (HTF) temperatures as high as 800 °C. These CSP systems with thermal energy storage have the potential to produce emissions-free electricity for commercial grid delivery at competitive prices. The high temperature capability leads to improvement in the overall solar to electric conversion efficiency which in turn leads to competitive prices. In this work, Sandia National Laboratories (SNL), as part of a U.S. DOE Small Business Voucher project, completed a series of thermal shock experiments of multilayer SiC materials manufactured by Ceramic Tubular Products to determine their suitability as CSP receiver tubes. The tubes were tested in open air, without any added salt to demonstrate their strength efficacy during a rapid rain transient event that could thermal-mechanically rupture a receiver. The results demonstrated a notable improvement in strength and thermal-shock resistance of the multilayer SiC tubes when compared to monolithic SiC and Al_2O_3 tubes. Additional testing of these multilayered SiC tubes has been performed by Sandia to confirm chemical compatibility with molten chloride salt, mechanical shock resistance, and solar absorptance and emittance properties, as reported by Walker, et. al. [9]

THERMAL SHOCK EXPERIMENTAL DEVELOPMENT

The experimental test system comprised a horizontal tube holder with a SS316 mesh basket to capture all pieces of the tubes that shattered under incident high-flux. The tube holder assembly comprised several thermocouples that were positioned within each respective tested tube to measure axial thermal distributions along each tube. The initial shake-down tests performed with an Al_2O_3 tube determined the optimal height above the coupon needed to provide enough uniform water coverage during quenching. Initial shake-down tests comprised tests at varying solar flux and simulated rain pressure levels to determine the optimal ramp and quenching rates.

An IR camera was used to assess thermal distributions across the sample for use in post-test stress analysis. Before and after each respective test, sub-millimeter scale geometrical measurements, as well as optical measurements, were taken to assess thermal-mechanical deformation. After all post-test analyses were completed a final test was conducted to determine the maximum internal pressure required to induce failure. A polyethylene plug test approach [6] was used to evaluate tubes that survive thermal shock testing to understand how the thermal shock conditions may have impacted its strength. The total thermal shock test assembly was placed on top of a movable stage at the NSTTF Solar Furnace Facility which could be translated in three axes and equipped with a LabVIEW data acquisition (DAQ) system for monitoring temperatures, flux profiles and flow rates. The facility also encompassed a high-speed attenuator system to control the level of incident heat flux on to the samples, as well as a flux gauge to calibrate the flux level. The facility has a peak flux of up to 600 W/cm^2, where for these tests flux levels were only tested up to approximately 500 W/cm^2, which is characteristic of average maximum NSTTF TMY (Typical Meteorological Year) periods. For this investigation, a variety of thermal shock tests were performed with the tube materials listed below where geometrical and surface cracking analysis was performed before and after each respective test.

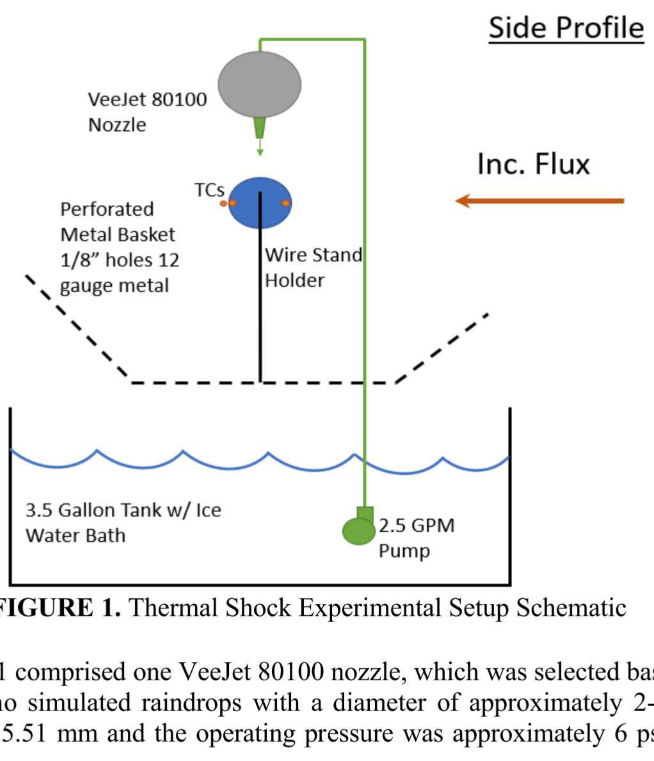


FIGURE 1. Thermal Shock Experimental Setup Schematic

The system as shown in Fig. 1 comprised one VeeJet 80100 nozzle, which was selected based on simulated rain tests by previous investigators who simulated raindrops with a diameter of approximately 2-4 mm [7, 8]. The orifice diameter for this nozzle was 5.51 mm and the operating pressure was approximately 6 psi. The initial shake-down tests with an Al_2O_3 tube helped determine the optimal height above the coupon that provided enough uniform water coverage during quenching. Initial shake-down tests also comprised tests at varying flux and simulated rain pressure levels to determine the optimal ramp and quenching rates. Subsequent tests then used these values for their respective tests. During the experiments, in-situ analysis comprised a pyrometer as well as thermocouple temperature data collection, using type-K thermocouples where test flux levels were determined based on calibration panel measurements, just prior to each respective test.

As shown in Fig. 2, the actual test system was mounted on a small skid that included the water pump, splash shields, 7 thermocouples (indicated with yellow k-type connectors), water tray and Veejet nozzle piping. The pump was connected to a VariAC voltage transformer that was used to set the water pump flow rate to approximately 8 GPM based on literature [4]. For safety a mesh wire basket was placed just below the sample stand to catch any debris, as well as on the pipe inlet to the pump. The nozzle had an adjustment connection to raise or lower the device, where its height was determined to be approximately 7.25 in. based on initial shakedown tests to determine optimal water coverage and positioning.

FIGURE 2. Experimental Setup and Solar Furnace Integration Photos

For this investigation, a variety of thermal shock tests were performed with the tube materials listed below where geometrical and surface cracking analysis was performed before and after each respective test:

1. Aluminum Oxide – used as practice tubes during experimental setup development shake-down tests.
2. Monolithic SiC Hexoloy™ – used as the primary test tubes for investigation. These tests were conducted immediately following success of the shake-down experiments.
3. Composite Type 1: Monolithic SiC Hexoloy™ plus outer composite layer (Nicalon CG fiber + Lancer SiOC matrix) – tubes tested as part of the primary thermal shock tests.
4. Composite Type 2: Monolithic SiC Hexoloy™ plus outer composite layer (Nicalon CG fiber + Starfire SiOC matrix) – tubes tested as part of the primary thermal shock tests.
5. SS304: A low-cost, highly-available material used in CSP molten salt power tower applications.
6. SS347: A high-temperature, high-strength mateiral used in nitrate molten salt applications.

Table 1, presents the test matrix which was executed for the thermal shock investigation where three tubes were not tested on-sun for the purpose of subsequent control base-line hoop stress measurements. The first tests conducted were with Aluminum oxide (Al_2O_3) white test tubes; the third tube was painted black for comparison with a highly-absorbing coating. This was then followed by five tests with monolithic SiC tubes that were tested as a baseline comparison to the multilayer composite SiC tubes which followed next. For these tests, two sets of samples were created from two separate tube manufacturing techniques, listed respectively as “Composite Set 1” and “Composite Set 2.” All tubes were approximately the same length, ranging from 4 to 5 inches and had a diameter of 0.75 in. The flux spot size from the solar furnace was approximately 2 in., centered along the length of each tube tested. Before and after each respective test, sub-millimeter scale geometrical measurements, as well as optical measurements, were taken to assess thermal-mechanical deformation.

During the experiments, temperature measurements were taken at seven locations along the tube test specimen using thermocouples as shown in Fig. 3. Heating of the tubes took place along the “front” side of the tubes, as this was the side facing the incident solar flux. Of the seven thermocouples used for each test, only the “center backside” thermocouple was positioned on the exterior surface of the tube.

TABLE 1. Thermal Shock Test Matrix

Test Number	Sample ID	Material	Shatter or Melted During Thermal Shock Test	Composite Set Number
1	Tube 1	Al_2O_3 - White	No	N/A
2	Tube 2	Al_2O_3 - White	No	N/A
3	Tube 3	Al_2O_3 - Black	Yes	N/A
4	Tube 4	SS304 - Polished	No	N/A
5	Tube 5	SS304 - Black	Yes	N/A
6	Tube 6	SS347 - Polished	No	N/A
7	Tube 7	SS347 - Black	Yes	N/A
8	Monolithic SiC Tube 1	Monolithic SiC	Yes	N/A
9	Monolithic SiC Tube 2	Monolithic SiC	Yes	N/A
10	Monolithic SiC Tube 3	Monolithic SiC	Yes	N/A
11	Monolithic SiC Tube 4	Monolithic SiC	Yes	N/A
12	Monolithic SiC Tube 5	Monolithic SiC	Yes	N/A
13	17-001-01-C	Composite S	No	1
14	17-001-02-B	Composite S	No	1
15	17-001-02-C	Composite S	No	1
16	17-001-03-C	Composite S	No	1
17	17-001-04-C	Composite L	No	2
18	17-001-05-B	Composite L	No	2
19	17-001-05-C	Composite L	No	2
20	17-001-06-C	Composite L	No	2

The other six thermocouples were all positioned along the interior surface of the tubes. A 1.39 µm pyrometer was also used to measure the tube "front" center temperature, but these are not as reliable as the thermocouple measured temperatures. A high speed camera was used to film each thermal shock test.

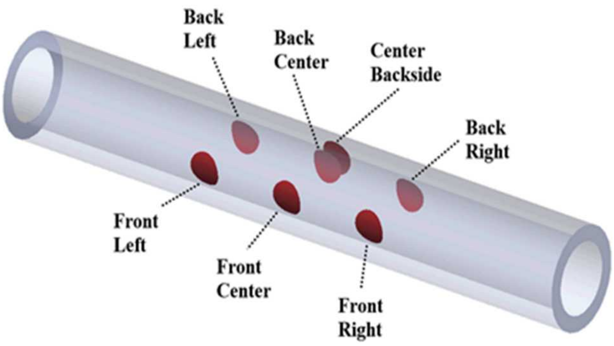


FIGURE 3. Relative thermocouple positions along tube test specimens

After all thermal shock post-test evaluations were completed a final test was conducted to determine the maximum pressure required to induce failure. This hoop strength analysis was conducted through a polyethylene plug test approach [3] using an Instron tester setup shown in Fig. 4, to evaluate tubes that survived thermal shock testing and to understand how the thermal shock test may have impacted their strength. An Intron stress test apparatus, Fig. 5 at SNL was used to measure the effective max hoop stress values from the tested samples.

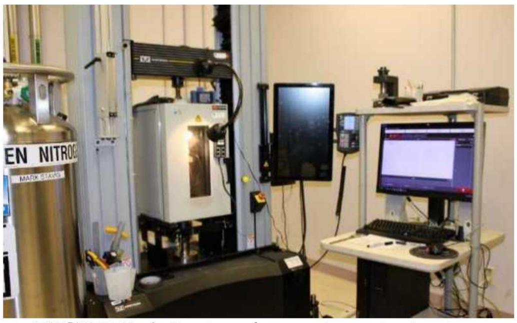


FIGURE 4. Instron hoop stress test setup.

Testing to determine the burst strength of ceramic and composite wound ceramic tubes was done with a unique test that involved internally loading a short length of ceramic tube with a polyurethane plug. The test setup is shown in

Fig. 5. The setup includes two 0.5 in. threaded rods that are attached to an Instron load frame, two steel platens, and a 2" long polyurethane (two 1 in. long lengths of Arathane 5750 were used). Because there was a range of sample inner diameters (0.478 in. to 0.507 in.) two sets of steel platens (0.47 in. and 0.50 in. diameters) and three sets of polyurethanes plugs (0.45 in., 0.47 in., and 0.48 in. diameters) were made to accommodate all the samples. The ends of the 0.5 in. threaded rods that go inside the tubes were also turned down to 0.45 in. diameter to be able to fit into all samples. The tests were run at a crosshead speed of 0.1 in/min until failure.

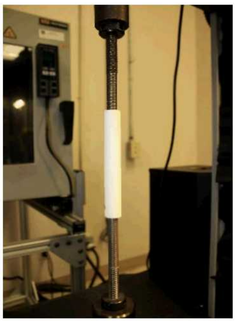
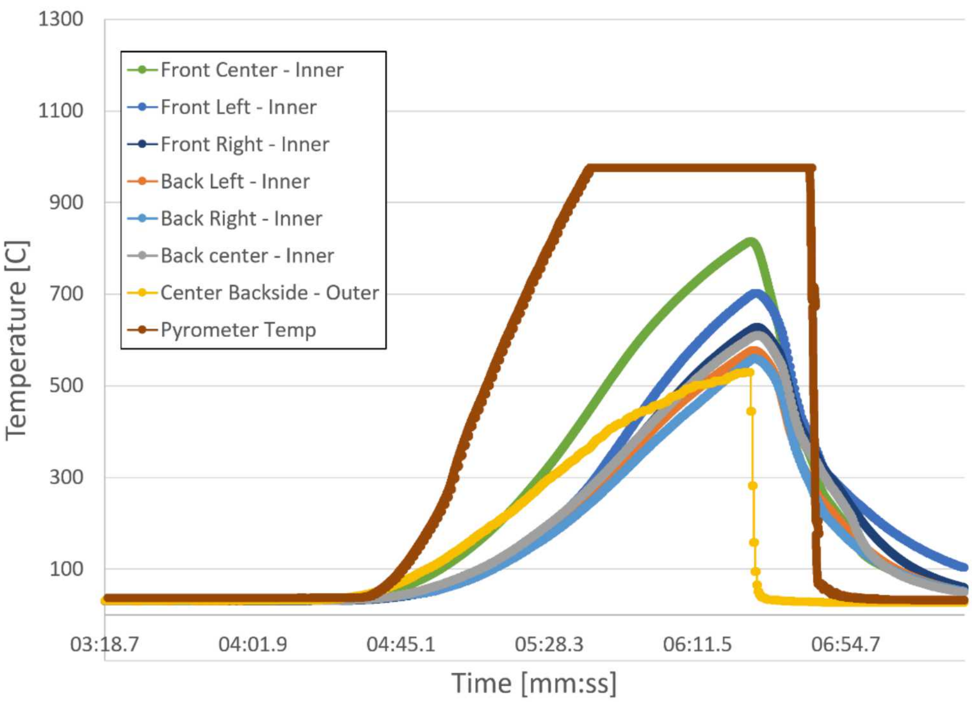
a.  **b.** 

FIGURE 5. a. Test setup with ceramic Al_2O_3 sample, and b. setup showing polyurethane plugs and steel platens that are inside of tubes.

THERMAL SHOCK RESULTS & DISCUSSION

During thermal shock tests on-sun, all tested tubes were thermally ramped up to prescribed max temperature of 800 ºC at the inner surface of the tubes for two minutes. Each test was concluded once the tube temperatures reached the ambient temperature. This ramp time was determined from preliminary empirical tests to ensure all tubes tested would not shatter prematurely before quenching. The results indicated that all multilayered SiC and Al_2O_3 tubes did not shatter, while all monolithic SiC tubes, as well as black Al_2O_3 tubes did. Representative temperature measurements are shown for a SiC composite S tube sample and a SiC monolithic tube sample in Figs. 6a and 6b respectively. These results indicate that although the front inner surface temperature was higher, the temperature gradients were found to be larger radially between front and back surfaces versus axially along the tubes.



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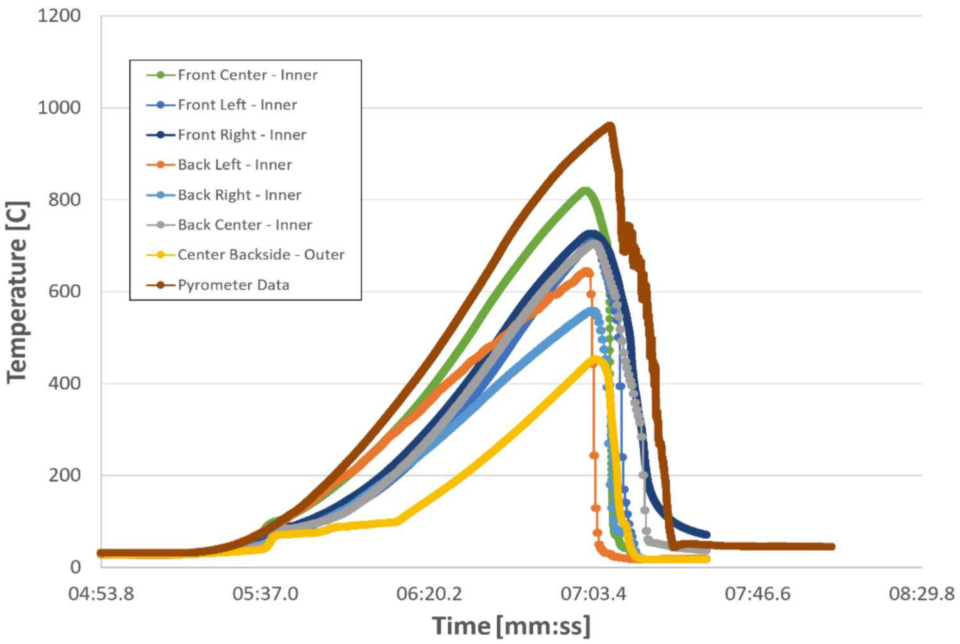

b.

Figure 6. a. Measured temperatures for a. SiC composite tube, and b. a SiC monolithic tube.

For each experiment, a high-temperature pyrometer (capable of temperatures of up to 975 °C) was used for measuring temperature on the front side of the tube. The measured temperatures were not factored into post-processing analysis due to the rounded front surface, that wasn't geometrically adequate for an accurate reading with the equipment. The results qualitatively indicated potentially very high front side temperatures, where for one of the composite tubes, extrapolated values were found to be as high as approximately 1280 °C. The flat portion of this measurement was due to temperature saturation limitations for the pyrometer. The results from the 20 total thermal shock tests are shown in Table 1. All of the 8 SiC composite tubes were found to survive severe thermal shock without shattering while all 5 of the SiC monolithic tubes shattered during the test. No difference was found between the performance for the two types of composite samples. In addition, as shown in Fig. 7, although composite 1 tubes did not have hoop strength improvement between pre- and post-thermal shock exposure, the average failure pressure values for composite 2 tubes were found to be approximately 2.4% higher for the tested tubes, than the one that didn't have solar exposure. From the figure the cross-hatched bars represent tubes that were not tested on-sun. The data suggest that SiC Composite 1 tubes appear to have a higher hoop strrength than the monolithic SiC overall, and the strength of both Composite sets were not degraded by thermal shock testing. Additionally, the strength of the SiC Composite 2 tubes appear to be similar to that for the monolithic SiC, but lower than SiC Composite 1 tubes.

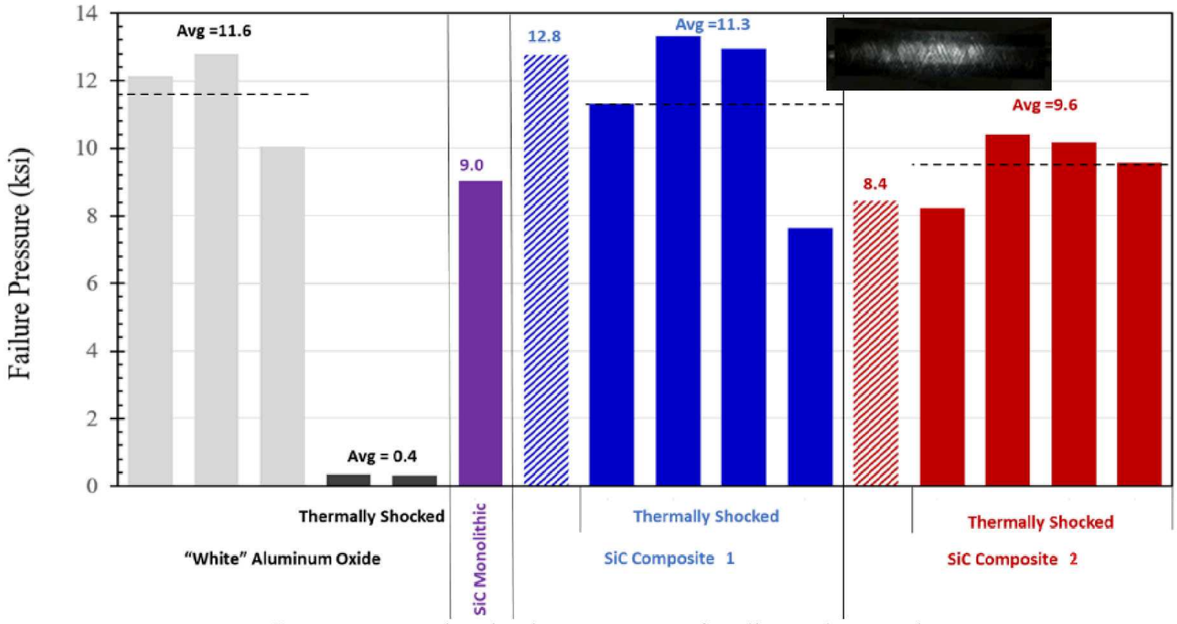

FIGURE 7. Data showing burst pressures for all samples tested.

CONCLUSION

A rigorous analysis was performed with monolithic SiC tubes and multilayer SiC (monolithic SiC plus CMC layer) tubes being considered for the development of a high-temperature CSP receiver. The investigation experimental setup was developed to evaluate the performance of receiver tube materials against the thermal shock that they may experience in service. A series of 20 total experiments were completed where receiver tube samples of various materials were heated to an intenal tube temperature of 800 □ and then thermally shocked when subjected to simulated rain. Through this testing it was found that the multilayer SiC tubes were able to withstand thermal shock, while SiC monolithic tubes were not. Furthermore, the composite tubes that survived the thermal shock were evaluated for post-test damage through hoop stress testing, and were found to have failure pressures consistent with tubes that were not subjected to thermal shock testing. Both types of SiC CMC tube materials survived the thermal shock testing. Instron hoop stress tests indicated that the CMC Composite Set 1 materials had a 17.2% higher hoop strength (or failure pressure) than the Composite Set 2 materials. Although all of the Hexoloy^{TM} monolithic SiC tubes that were tested on sun were destroyed during the thermal shock process, there was one pristine sample that was not put through the thermal shock process which was tested with the Instron tester. This sample exhibited a slightly lower strength compared to both sets of composite tubes, where its measured stress of 9.04 psi was found to be 20.1% and 5.8% lower than the average values of set 1 and set 2 respectively.

ACKNOWLEDGEMENTS

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