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DEVELOPMENT OF HIGH TEMPERATURE TESTING FOR POLYMER MATRIX COMPOSITES, METALS, AND CERAMIC MATRIX COMPOSITES:

VOLUME 1 – CMC High Temperature Testing
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Interim Report

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14. ABSTRACT

The objective of the collaborative research is to increase the temperature capability of standard mechanical test equipment. Three classes of materials were examined under this collaboration; polymer matrix composites (PMCs), metals, and ceramic materials (CMCs). Accessory packages to facilitate these high temperature testing applications were designed and constructed by MTS Systems Corporation (MTS) and tested at the Air Force Research Laboratory, Materials and Manufacturing Directorate, Structural Materials Division (AFRL/RXC). The temperature goals for these three systems were 425°C for PMCs, 1200°C for metallic materials, and 1500°C for ceramic matrix composites. This report covers the CMC testing

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Table of Contents

Section **Page**

List of Figures

List of Tables

1. SUMMARY

Characterizing the performance of materials at ever increasing temperatures is necessary to ensure the durability and performance of Air Force systems. Improved high temperature materials are critical for many aerospace applications including improving the efficiency and performance of aerospace turbine engines. MTS Systems is a major producer of mechanical test equipment and accessories that are widely in use by the Structural Materials Division (RXC) of the Air Force Research Laboratory (AFRL), as well as other AFRL and DoD materials testing facilities. Further, this equipment is also widely used by commercial laboratories and universities.

The objective of this collaborative relationship was for MTS to design, engineer, and produce advanced mechanical test hardware which then would be evaluated by RXC for potential improvement in high temperature testing capability versus the state-of-the-art. This collaboration focused on the development of elevated temperature testing systems targeting temperature ranges (425°C – 1500°C) that are relevant for three key material systems: polymer matrix composites (PMCs), high temperature metals, and ceramic matrix composites (CMCs). The mechanical test systems provided by MTS included: high temperature grips (for cylindrical and flat test coupons), furnaces co-engineered with the grips, and in the case of the PMC system, high temperature extensometery. These systems were intended to facilitate high temperature materials testing with increased ease and accuracy, benefiting the Air Force and other users through enhanced capability and reduced testing costs.

After system delivery, RXC conducted a series of mechanical tests to help assess the capabilities of the developed systems. The systems were evaluated for system durability, alignment, temperature capability and thermal gradients, and ease of use. The Air Force, DoD-related facilities, and commercial testing laboratories are all expected to benefit from these advancements in mechanical testing capabilities.

2. INTRODUCTION

The objective of the collaborative research was to increase the temperature capability of standard mechanical test equipment. Three classes of materials were examined under this collaboration; polymer matrix composites (PMCs), metals, and ceramic matrix composite materials (CMCs). Test equipment packages were designed and constructed by MTS Systems Corporation (MTS) [1] and tested at the Air Force Research Laboratory, Materials and Manufacturing Directorate, Structural Materials Division (AFRL/RXC). The temperature goals for these three systems were 425°C for PMCs, 1200°C for metallic materials, and 1500°C for ceramic matrix composites. Each system solution was comprised of a specifically designed specimen gripping solution and a unique high temperature furnace or chamber. The grips were designed to have enhanced durability and ease of use. These products have hydraulic actuation of the mechanical grips. The approach was to have the gripping surfaces in the hot zone while the hydraulic actuation elements remained outside of the high temperature zone. Furnaces or environmental chambers for heating to the high temperatures were designed and constructed by MTS/Mellen and tested by the AFRL/RXC. Also, a solution using non-contact strain measurement similar to digital image correlation (DIC) was demonstrated for elevated temperature testing of the PMC materials that greatly improved test times.

This report will detail the systems, their thermal and structural capabilities, and use for only ceramic matrix composites. The systems for PMCs and metals will be covered in a separate report.

3. CERAMIC MATRIX COMPOSITE TESTING NEEDS

Testing Ceramic Matrix Composites at temperatures at and above 1200°C requires very specialized equipment. The main objective was to design hot grips that could operate for long times at 1000°C. This would prevent CMC specimens from failing at intermediate temperatures between 800°C and 950°C. Two attempts were made to develop the high temperature grips. A custom high temperature furnace designed to work with the high temperature grips was also evaluated. The furnace is a unique three zone design that allows the center zone to operate at 1500°C for long periods. The goal for the furnace was to maintain the temperature along the entire gage length of the CMC test specimen to ±1%.

The objective was to first evaluate the grips in terms of ease of use, gripping force, and alignment. In addition, the furnace was evaluated for temperature profile of a CMC test specimen. Once evaluated, the test plan called for testing a Carbon/SiC and SiC/SiC CMC with the grip/furnace arrangement. The testing would involve conducting creep rupture testing for approximately 100 hours.

The following sections describe attempts to develop the unique high temperature CMC grips and the evaluation of the high temperature furnace.

4. INITIAL CMC HIGH TEMPERATURE GRIP EVALUATION

MTS invested considerable time and resources working through multiple design approaches for the high temperature CMC grips. The goal was to design grips that could operate at up to 1000°C for extended periods of time. The goal was to design grips that could survive in elevated operating temperatures. This allowed the grip tabs to be quite hot (~1000°C) and gage section temperatures up to and sometimes exceeding 1500°C. This was desirable to avoid issues like intermediate temperature embrittlement (ITE) that often plagues testing of some CMC systems. Maximum axial load was designed to be approximately 25 kN at room temperature and 4 kN at 1482°C (~2700°F), and alignment was to conform to the ASTM requirement of no more than 5% bending at 500 micro-strain. [2]

4.1 CMC Grip Version 1

In late 2014, after several design iterations, MTS settled on a final approach and design concept. A schematic of the grip is shown in Figure 1 and is referred to as CMC Grip version 1 (V1). The design leverages the success of the MTS 647 wedge grips and the 1200°C 680.12 grip cooling concept that was designed primarily for metals. However, it utilizes an inverted wedge arrangement as is shown schematically in Figure 2. In this design, the wedge body and the wedges themselves are supported by a stout piston rod that runs through the interior of the grip to the hydraulic fluid chamber that is located outside of the furnace. Hydraulic pressure applies force to the sturdy rod resulting in the wedge cavity moving upward. This motion causes the wedges to press against the wedge cap and results in the closing of the wedges. No vertical translation of the wedges can occur because they are held in place by the wedge cap. The wedge cap screws into an outer sleeve that is mounted firmly to the grip outer body. In addition, when mounting the test specimens, the wedges are pressed upward against the wedge cap by an alumina rod. This rod keeps the wedges pressed firmly against the wedge cap and also applies a splitting force to the wedges keeping them open. There is a spring at the bottom of this alumina rod that applies constant pressure. Finally, there is an insulating alumina tube that surrounds the entire grip all the way from the base up to the top of the wedge cap. Cooling air is run inside the grips to keep the metallic components at or below 1000°C.

The grips and furnace were successfully manufactured. After assembly at MTS, the equipment was boxed up and shipped to AFRL/RXCCP. The grips and furnace arrived near the end of September 2015.

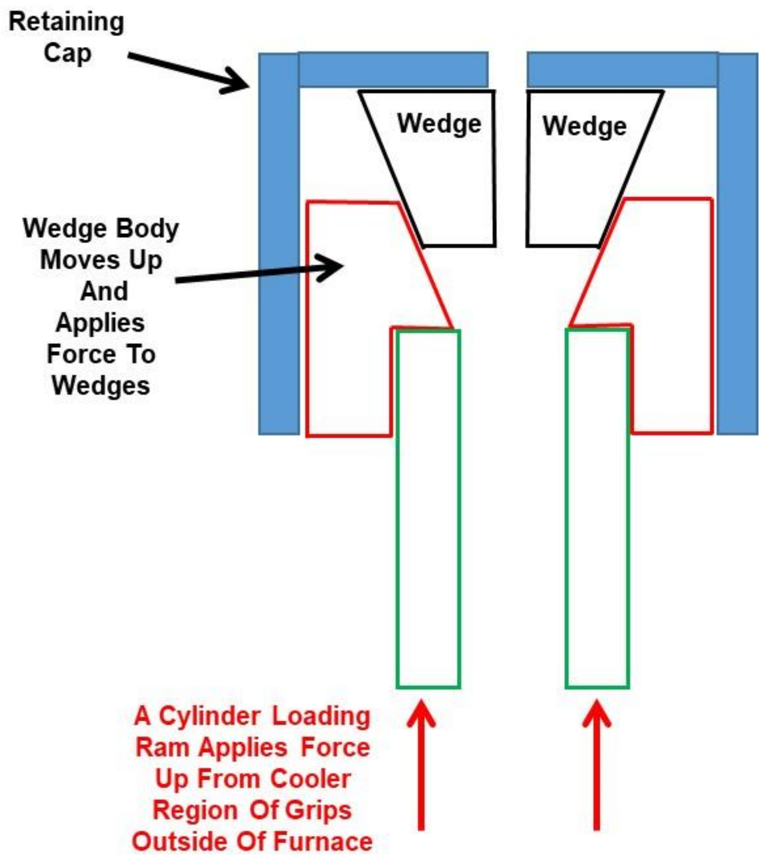

Figure 1. Schematic Of MTS High Temperature CMC Grip V1

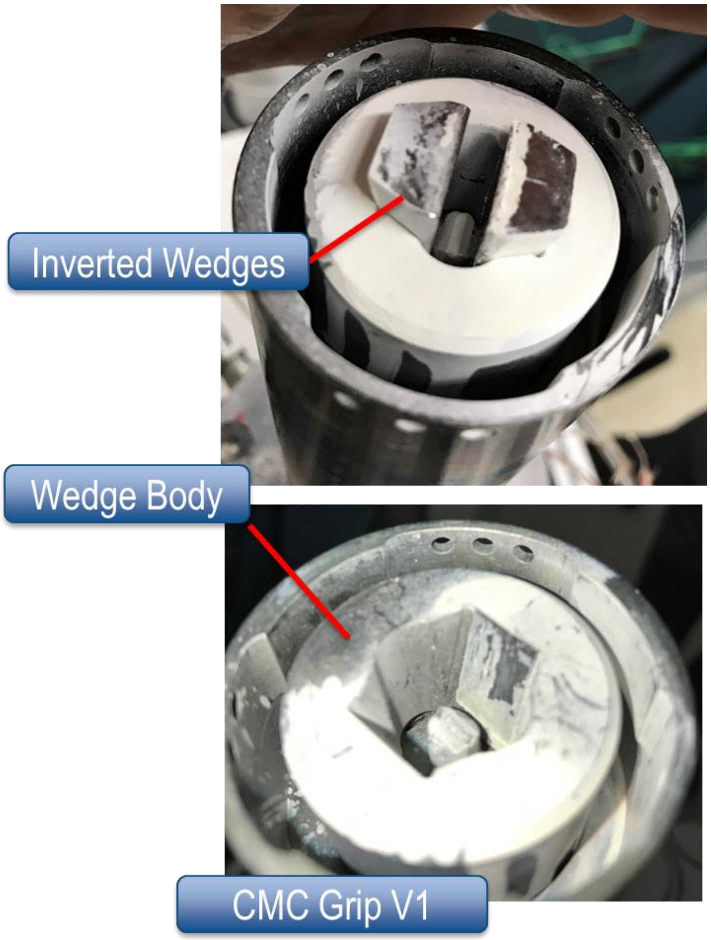

Figure 2. Photo of Wedge Cavity and Wedges for MTS High Temperature CMC Grip V1

4.2 Alignment Check

The initial set of CMC V1 grips were received and thoroughly inspected. Mounting the grips to a servo-hydraulic test frame went relatively straight forward with few obstacles. However, initial alignment checks determined that the grips had issues that were affecting alignment. Repeated attempts to align the grips failed to reach the ASTM standard [2]. It was determined that the alignment appeared to change after each insertion of the alignment specimen, even with set-up by a technician with years of experience with these types of machines and alignments. In addition, when trying to dial in the alignment of the grips using the MTS 609 alignment device, the changes in bending strain often went opposite to what was expected. Experience dictated that there needed to be less than 10-15 micro-strain bending during gripping. In addition, during gripping of an alignment test specimen, the bending strains should be of similar magnitude with opposite signs when the test specimen is rotated 180 degrees in the axial directions. This implies that there is approximately zero machine bending with the measured bending that of the test specimen itself. Overall the desired bending strains due to testing machine alignment is approximately 3% or less.

In what follows, a brief description is given of some of the specific issues identified that resulted in an unacceptable loading condition of the gripped sample. An alignment specimen was placed in the grips and the bending strains recorded. This was done several times. In addition, each time the wedges were also removed and inserted back into their original location. The results are presented in Figure 3. There is considerable scatter for repeated re-gripping of the specimens and the bending strains are too high after gripping. The scatter would often fall primarily on the X-Axis. This is the direction parallel to the width of the specimen. This suggests that the wedges are not properly restrained in the sliding wedge cavity body during initial grip pressurization.

A second study of alignment involved taking the alignment specimen out and paced back into the grips ten times. Bending strains needed for reproducible testing of CMCs typically need to be less than 10-15 micro strain and should be repeatable. In addition, the wedges were removed and inserted in different locations each time, as it is expected that after each test the wedges will need to be inspected and coated with an anti-seize compound. Each time a sample was gripped, a different amount of bending strain resulted, as shown in Figure 4. In addition, there was an effect of which wedge was placed in which location within the grip. Each time their location changed it significantly impacted total bending strains of the test specimen. Since wedges will need to be removed/ cleaned/ re-installed for every test, this issue was of serious concern.

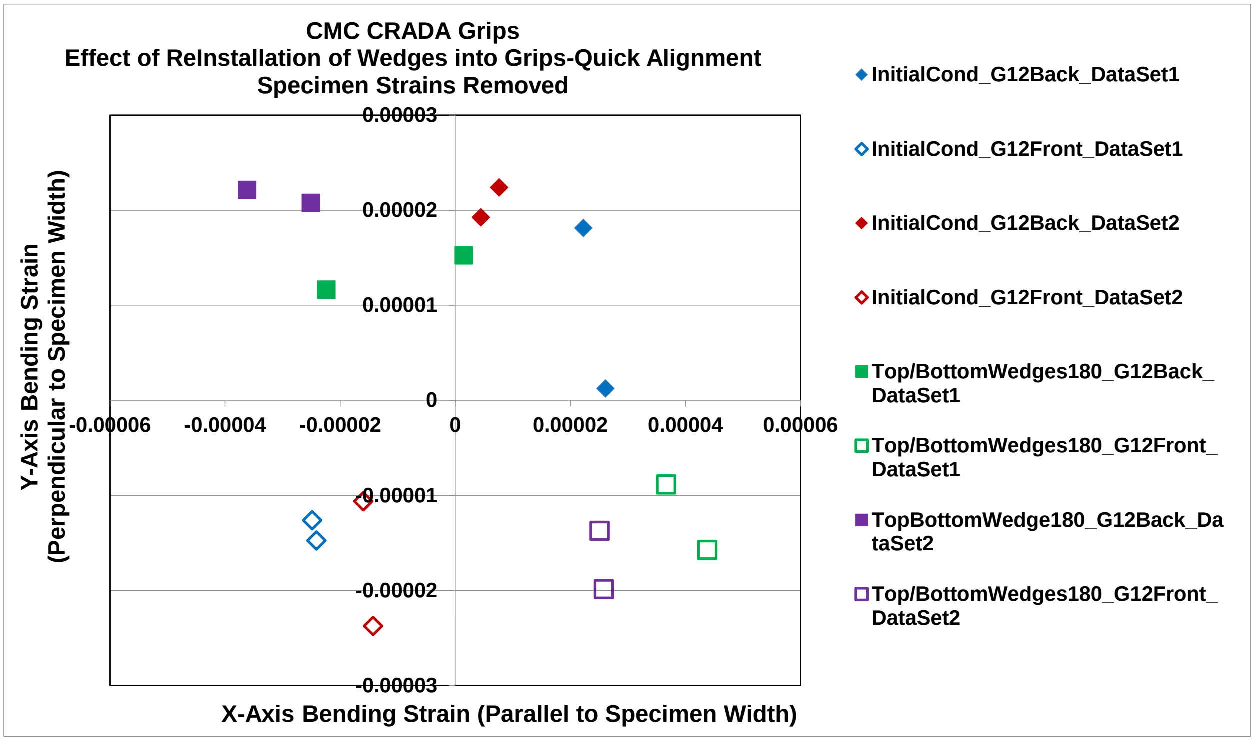

Figure 3. Effect of Gripping a Test Specimen for CMC Grip V1

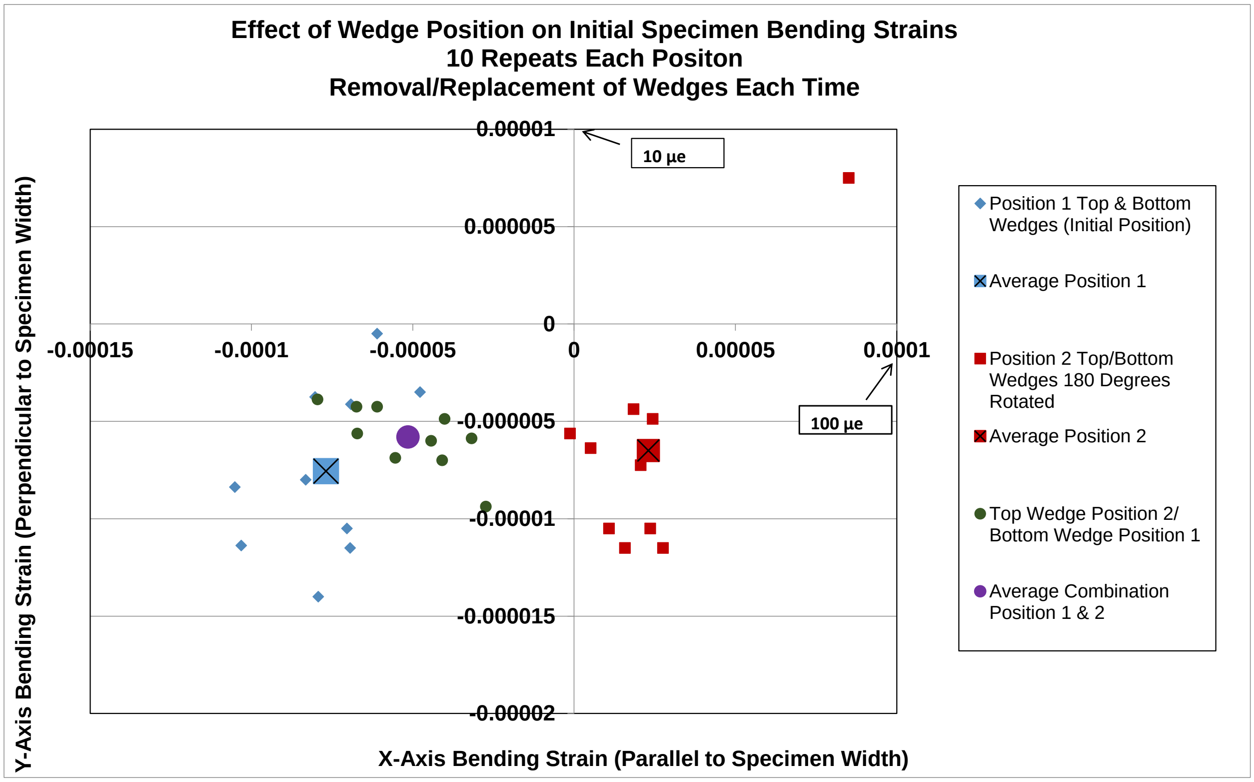

Figure 4. Effect of Wedge Removal on Bending Strains for CMC Grip V1

Grip pressure also had a large effect on alignment. Increasing the grip pressure resulted in more bending strain, as is shown in Figure 5. As the grip pressure increased, the bending strains continue to increase. This study documents that the amount of applied grip pressure affects bending, and contributes to the overall measured bending strains during gripping. Such trends indicate a serious design issue with the grips.

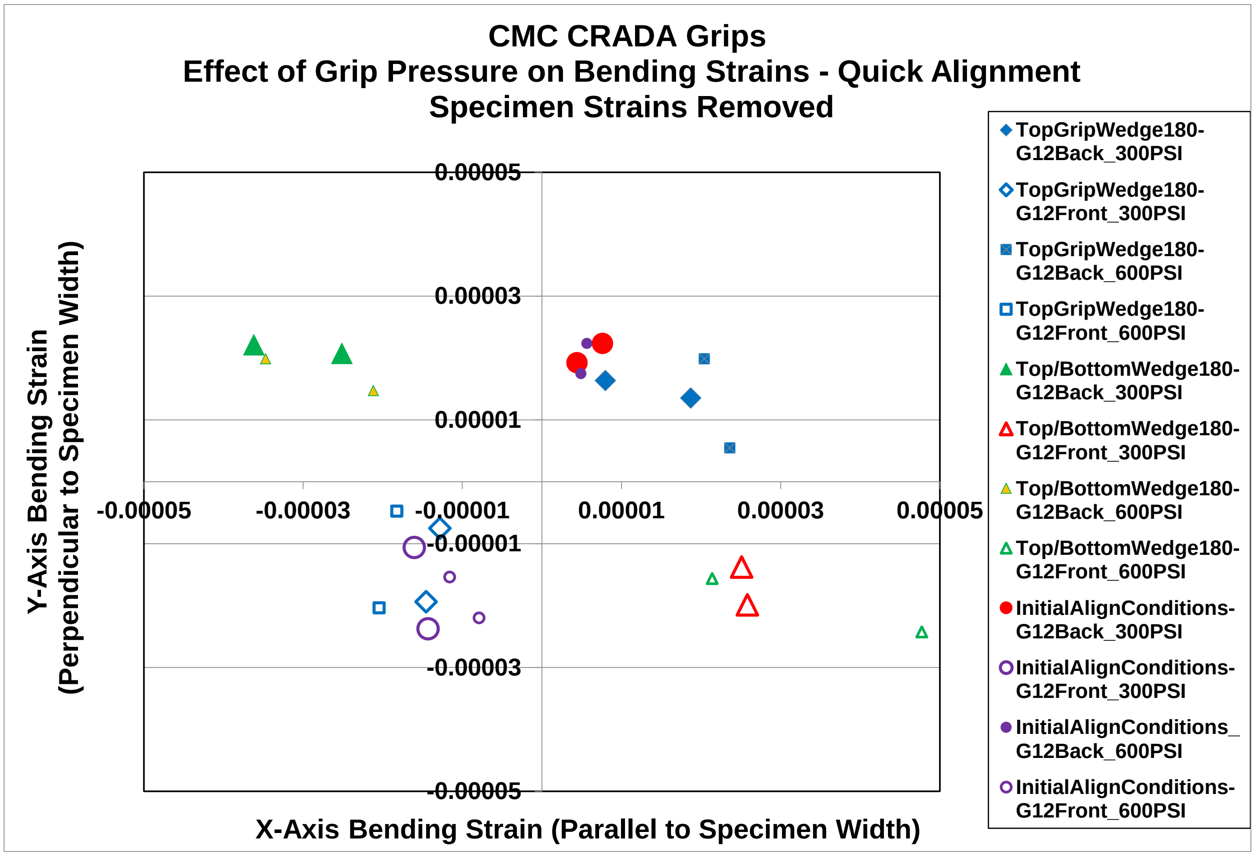

Figure 5. Effect of Grip Pressure on Bending Strains of CMC Grip V1

4.3 MTS Check of Alignment Issues with Initial Grip Set

On 19 October, 2016, MTS sent out an MTS alignment expert, to evaluate the installed grips. Also participating in this review was an alignment expert from UDRI and a MTS field representative. The purpose of this site visit to AFRL/RXCCP was to attempt to find the source of gripping alignment variability for the 1500°C MTS grips at zero axial force and room temperature. The variable increasing bending strains that resulted as a function of increasing grip pressure of the strain gaged specimen resulted in an exceedance of the maximum allowed bending strains. This variable alignment condition makes it difficult to adjust the alignment. The desired bending strains due to testing machine alignment, is 3% or less.

The team started by reviewing the data acquired by UDRI. They then went to the lab where the UDRI expert and MTS field representative showed the MTS alignment expert s the partial disassembly of the grips and how they worked.

The MTS expert set up the MTS 709 alignment system and for the first series of tests they attempted to replicate the data UDRI had collected. Values presented in Table 1 are in micro-stain bending. The results are also presented in Figure 6 as a plot of micro-strain bending versus gripping run. As with the earlier gripping trials, the bending strains during gripping were too high.

Table 1. Bending Strains For Grip V1 Test #1

Start		Top				Bottom
Series 1	με	31				27
Series 2	με	10				15
Series 3	με	11				24

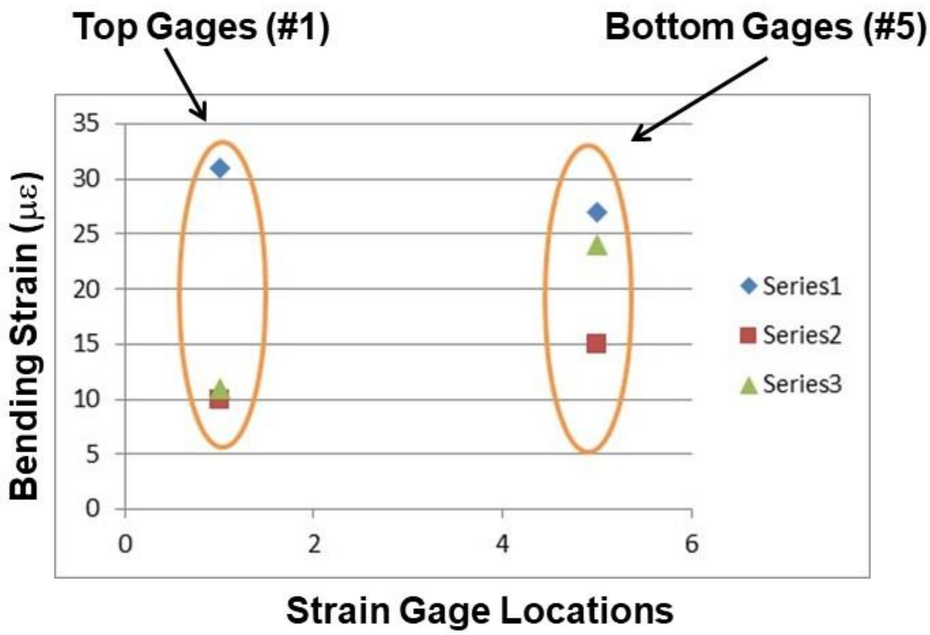

Figure 6. Grip V1 Bending Strain Check #1 by MTS

The team performed an offset of the gage readings at the first grip for each test. This removes the initial bending strains from the variability tests. They then gripped the alignment specimen an additional three times, acquiring bending data after each gripping with the exception of Test 4, which was gripped five times.

During Test 1, they observed that the metal covers of the grips, top and bottom, rotated from an initial state. This was seen at the set screws (see Figure 7, Figure 8, Figure 9). This implies that there was unacceptable movement within the grip assembly.

The team decided to acquire data while physically holding the metal covers first clockwise (Test 2, Table 2, Figure 10), then counter clockwise (Test 3, Table 3, Figure 11).

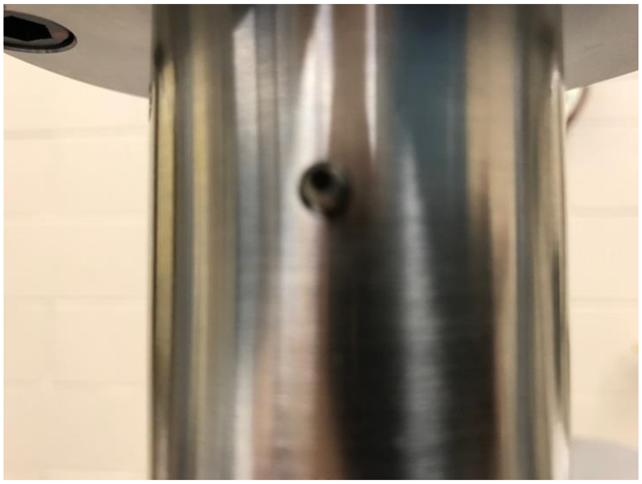

Figure 7. The Initial Condition of Grip V1 Showed the Set Screw Roughly in the Center of the Hole.

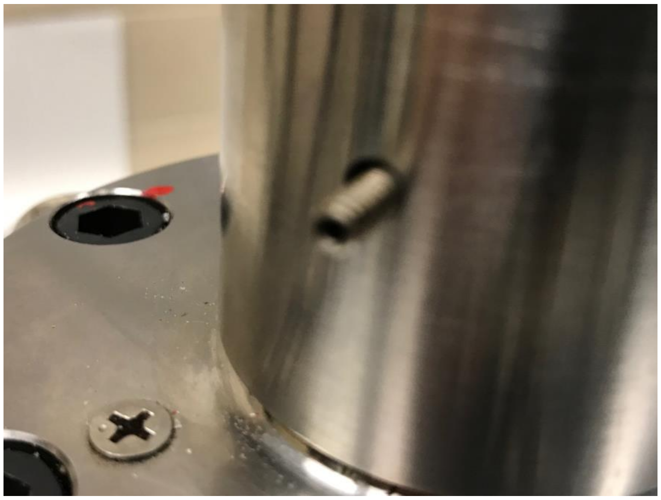

Figure 8. After First Grip Of Grip V1, The Screw Was To The Right Side of the Hole.

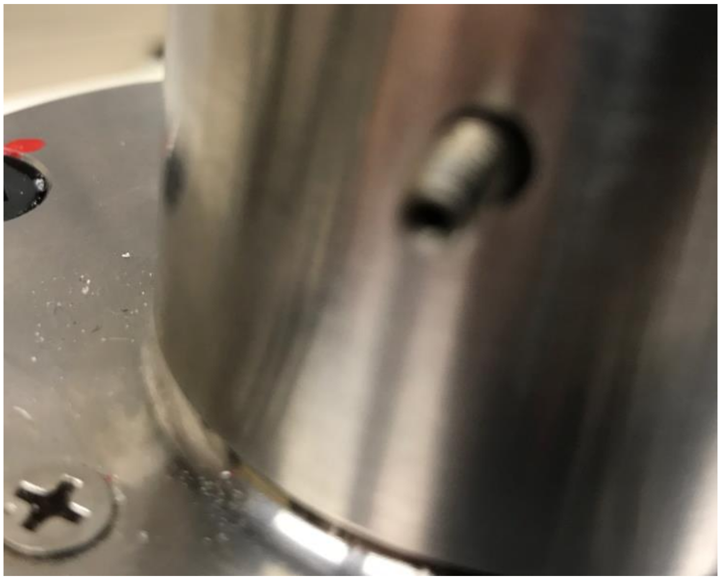

Figure 9. After First Grip Of Grip V1, Screw Was Held To The Left Of The Hole.

Table 2. Test 2 Of Grip V1 With Metal Covers Top And Bottom Held Clockwise

Regrip 2		Top				Bottom
Series 1	με	13				13
Series 2	με	13				19
Series 3	με	12				29

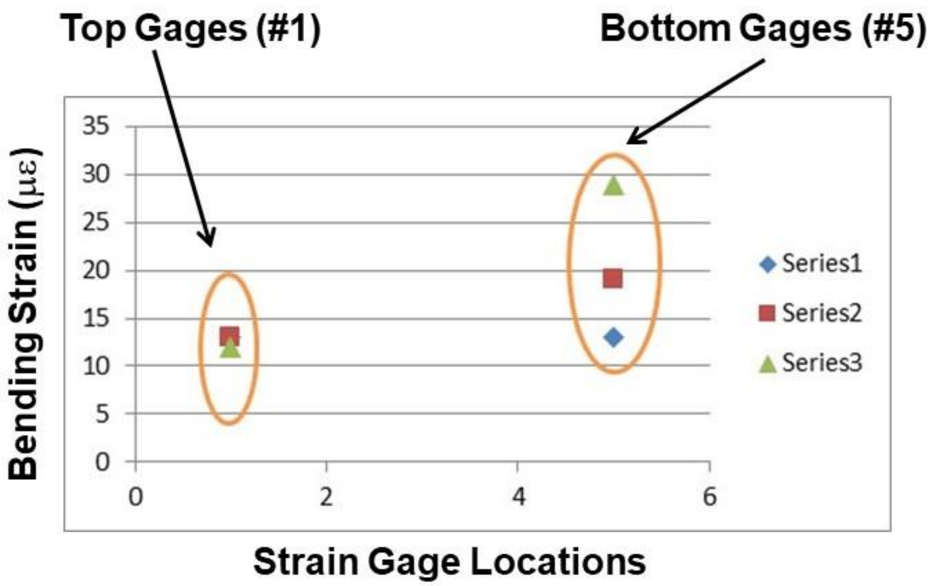

Figure 10. Plot of Bending Strains For Grip V1 Test 2 With Metal Covers Top And Bottom Held Clockwise

Table 3. Test 3 Of Grip V1 With Metal Covers Top And Bottom Held Counter Clockwise

Regrip 3		Top				Bottom
Series 1	με	10				31
Series 2	με	17				31
Series 3	με	11				33

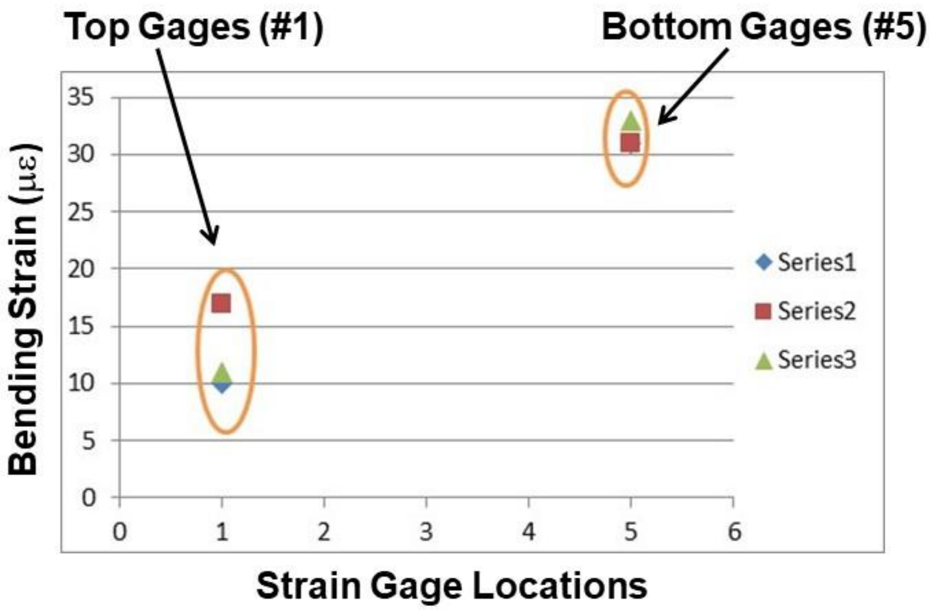

Figure 11. Plot of Bending Strains For Grip V1 Test 3 With Metal Covers Top And Bottom Held Clockwise

Test 2 and 3 show tighter grouping of the series 1 through 3, especially when the covers were held in the counter clockwise position. However, the bending strain values all exceed the allowable.

At this point the team decided to take the grips apart to try and determine why the covers where moving. Once apart, they noticed that there was significant side to side play in the wedges. It was determined the side to side play was a total of 0.2 mm (0.008") when measured with a feeler gauge.

An attempt was made to reduce the side to side play in the wedges by gluing shims to one side of each wedge. At first the reduced side to side play caused binding of the specimen in the grips once the pressure was released. Therefore, a shim from one wedge for each pair, top and bottom, was removed. This allowed the specimen to come free when the pressure was released. Typical rotation of the covers is shown in Figure 12. Results after adding the shim are shown in Test 4 (Table 4, Figure 13). Adding the shim to the wedges again produced significant rotation or twist in the covers, very visible at the top of the bottom grip. There was no physically holding of the covers in either direction for Test 4 results. Once again the bending strains were too high.

Figure 12. Photograph Showing Grip V1 Twist of Wedge Retaining Cap

Table 4. Test 4 Of Grip V1 Bending Strains For Wedges With Shims

Regrip 4		Top				Bottom
Series 1	μƐ	27				34
Series 2	μƐ	35				18
Series 3	μƐ	22				15
Series 4	μƐ	10				6
Series 5	μƐ	32				20

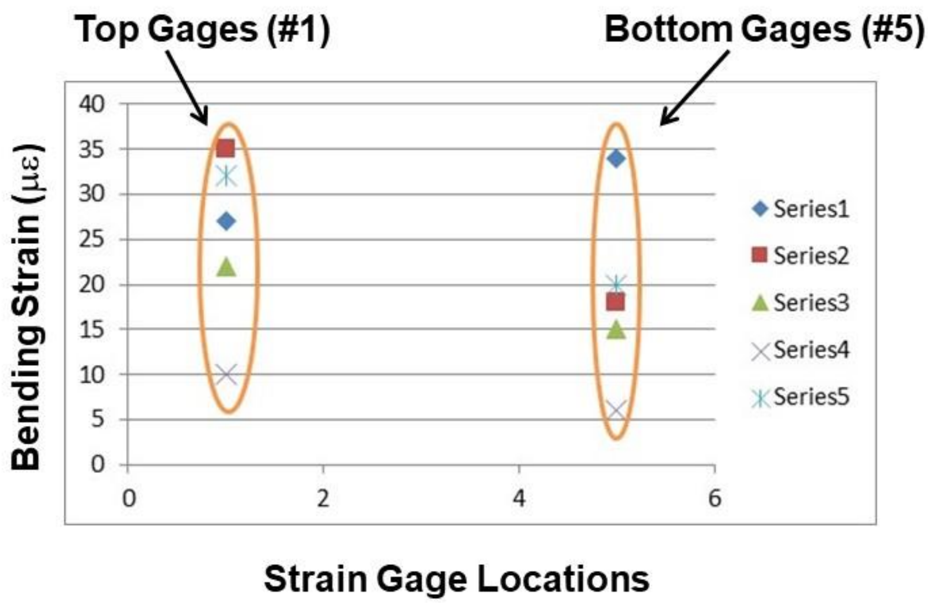

Figure 13. Plot Of Bending Strains For Test 4 With Shims On Wedges

Additional comments:

AFRL/RXCCP utilizes a precision fixture to position the specimen in the grips. This fixture ensures that the distance, top and bottom of the specimen, is consistent on one side of the specimen with respect to the sides of the metal grip covers. There is no method to ensure repeatable vertical placement of the alignment specimen. The actuator is positioned and repositioned with the testing system LVDT readout. The grips are actuated with a single hand pump supply.

Conclusions:

1) Values for bending strain obtained during gripping of an alignment test specimen by MTS were similar to those obtained by AFRL/RXCCP and were all too high and exceeded the allowables.

2) It is believe that some of the bending variability due to gripping can be improved if modifications to the grips can reduce the movement and or rotation of the metal covers. It was surmised that the rotation is caused when the wedges meet the underside surface of the cover. Reducing the side to side movement of the wedges will likely improve this condition.

3) The team was able to reduce the scatter of the bending strains by physically holding the covers. This was most noticeable in Test 3. However, it is important to note that every series of data was taken relative to a starting condition where all bending was offset to zero. It is likely that bending is taking place due to some other effect. This effect would indicate that some seating of the wedges is needed by multiple gripping. The team did not have time to investigate this further.

4) It was thought that it might be able to reduce the variability of gripping by using two pressure sources for gripping the specimen. This may also provide for a better method to ensure vertical placement of the specimen.

5) The current repeatability performance of the grips, if the initial bending of the specimen were at zero, would likely meet ASTM E1012 Class 5 with force applied. However, since the testing requires better than 3% bending, the current performance will not meet expectations.

Additional observation: The MTS 609 alignment adjusting fixture was installed in the testing machine approximately 90 degree off from the recommended configuration when using with the MTS 709 Alignment Wizard. This does not cause problems for AFRL/RXCCP.

4.4 Determination of Grip Pressure Versus Axial Load

It was important to determine the axial load capability of the grips. The grips were designed to be able to provide up to 25kN of axial load at room temperature. The effective load capacity is limited to what can be coupled into the specimen through friction at the grip wedge to specimen interface. Practically, it can also be limited by the maximum pressure that a specimen can sustain in the short transverse (thickness) direction without being crushed or sheared off.

It was important to determine the axial load capability of the grips. A series of experiments were run to evaluate the maximum axial load that could be applied for different specimen thicknesses with relationship to applied grip pressure. For each test the maximum axial load that could be achieved before the specimen started slipping in the grips was recorded. Specimens were loaded in load control. Care was taken to monitor the test and determine the exact load at which the load started to go flat which indicated slipping. This point is identified as convergence in the plots to follow. The results were all plotted together and are shown in Figure 14.

Pressure sensitive tape was placed behind the wedges to monitor how well the wedges contacted the wedge body. The pressure sensitive tape between the wedges and the wedge body proved to be very informative. Studying the tape revealed that the grips, when using surfalloy coated wedges, were optimum for specimens that were 3.18 mm (0.125") in thickness. It was found that the grip pressure versus force window of thickness was very small. None of the tests on the 0.125" thick specimens could achieve more than 14-16 kN of axial load, and MTS has calculated the grips should be good for up to 25 kN of axial load. The 0.094" thick specimens could only achieve approximately 8 kN of axial load while the much thicker 0.152" thick specimen could only achieve 10 kN of axial load.

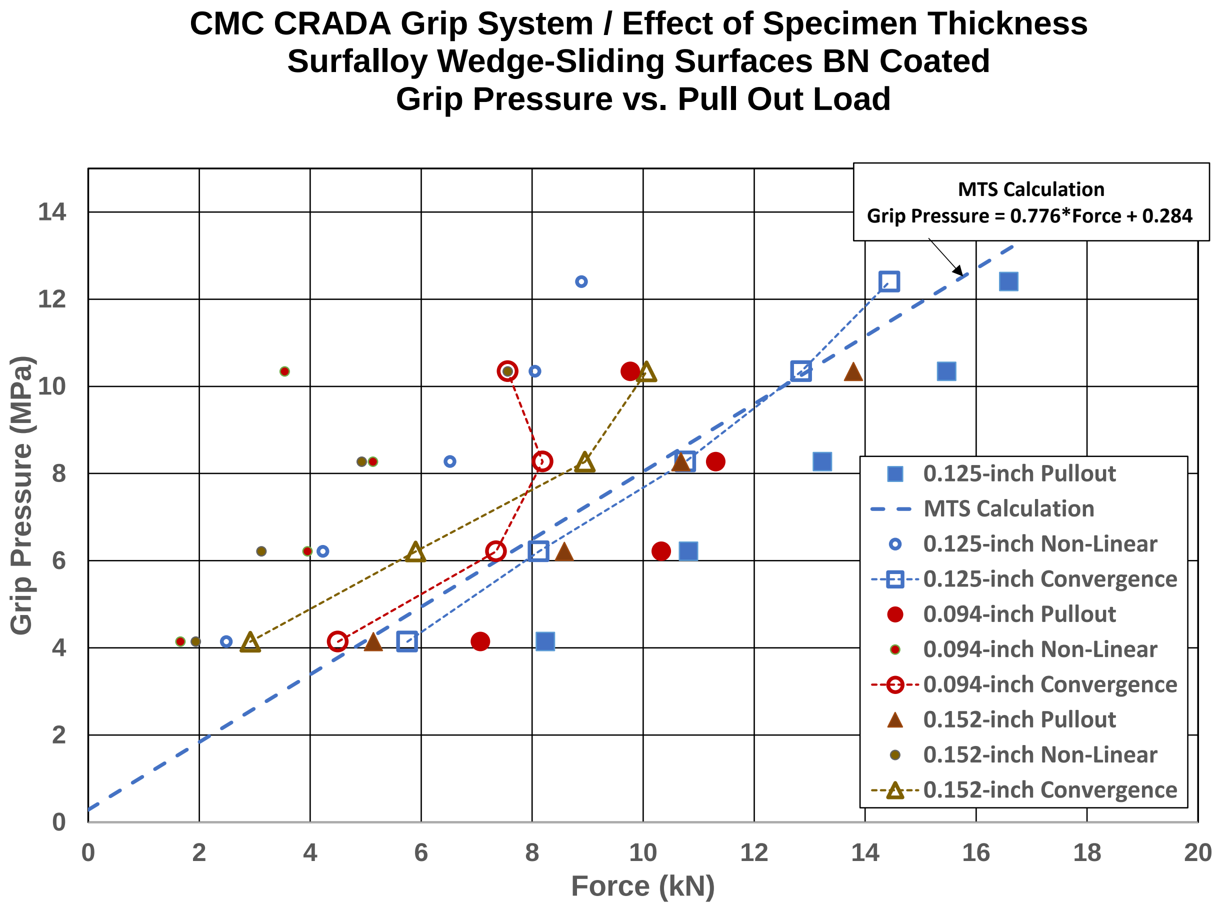

Figure 14. Plot of Grip Pressure Versus Axial Load for CMC Grip V1

4.5 Summary of Version 1 CMC High Temperature Grips

After considerable evaluation the team concluded that the Version 1 CMC Grip would not meet requirements for performance and usability and it was decided to take a different design approach with a second generation design version 2 (V2). The chief findings related to the V1 grip are outlined below.

The V1 grip alignment was unsatisfactory. The unique inverted wedge design minimized design stresses but did not constrain the relative motion of the mechanical elements sufficiently to deliver repeatable gripping and acceptable alignment. Variation in alignment performance also occurred with disassembly and reassembly of the wedges in the grip body. This latter observation was problematic since the grip must be disassembled periodically for cleaning and (high temperature) lubrication. Significant efforts were made by the AFRL/RXCCP, UDRI, and MTS team to improve the alignment capability of the V1 design. Several experiments were conducted to tighten clearances and constrain unintended motion of mechanical elements during the griping action. These activities improved alignment anecdotally but were not workable as a standard test procedure.

As stated above, the ASTM Standard for alignment, (ASTM E1012-14) [2] specifies that there should be less than 5% pending at 500 micro-strain. The best alignment that could be obtained is shown in Figure 15. At 500 micro-strain, the bending is approximately 20% and significantly higher than the ASTM required 5%. It appears that there was a stack-up of machining tolerances fighting the alignment. A large number of alignment checks were performed. The effect of repeated gripping and also removal and reinstallation of the wedges all resulted in high bending strains as shown in Figure 16.

The grips could not come close to applying the desired axial load of 25 kN. The maximum axial load that could be achieved was only 18 kN. In addition, the maximum axial load was very dependent on the thickness of the test specimen.

The CMC Grip V1 design had several ergonomic and functionality deficiencies that were noted. Assembly of the wedges in the wedge body was difficult, especially for the upper grip due to gravity – it was difficult (but possible) to insert the wedges in the upper grip body and tighten down the grip cap with only one person. The wedges were pressed against the wedge cap during assembly by the spring-loaded alumina rod and seemed to rotate slightly as the cap was tightened down. The wedges did not always actuate smoothly. In particular the spring-loaded alumina rod did not reliably open the wedges. Gripping pressure was not uniform on the wedge faces due to variability in positioning of the wedges in the grip body. This impacted alignment performance and may have limited the maximum loads that could be obtained without specimen pullout.

It was ultimately determined that the novel grip body design, intended to lower design stresses and prevent binding after high temperature operation, introduced too much variation in the gripping action. This resulted in poor grip alignment, especially as the wedges interacted with the grip cap. Additionally, there were a number of challenging ergonomic issues associated with the assembly of the grip after cleaning and lubrication when installed in a load frame.

After multiple studies, it was concluded by AFRL and MTS that this grip design was not functionally feasible. With this finding, MTS made the decision to go back to the drawing board and redesign the grips.

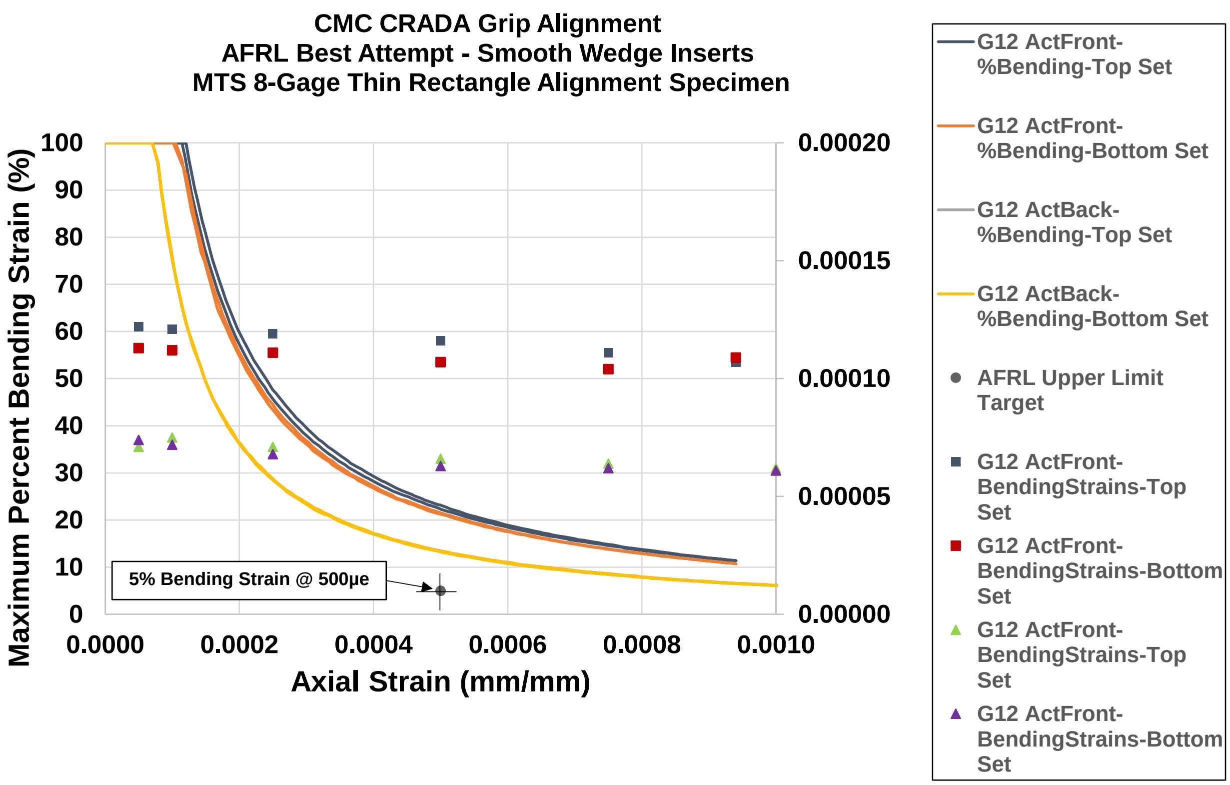

Figure 15. Maximum Percent Bending Versus Applied Axial Strain for CMC Grip V1

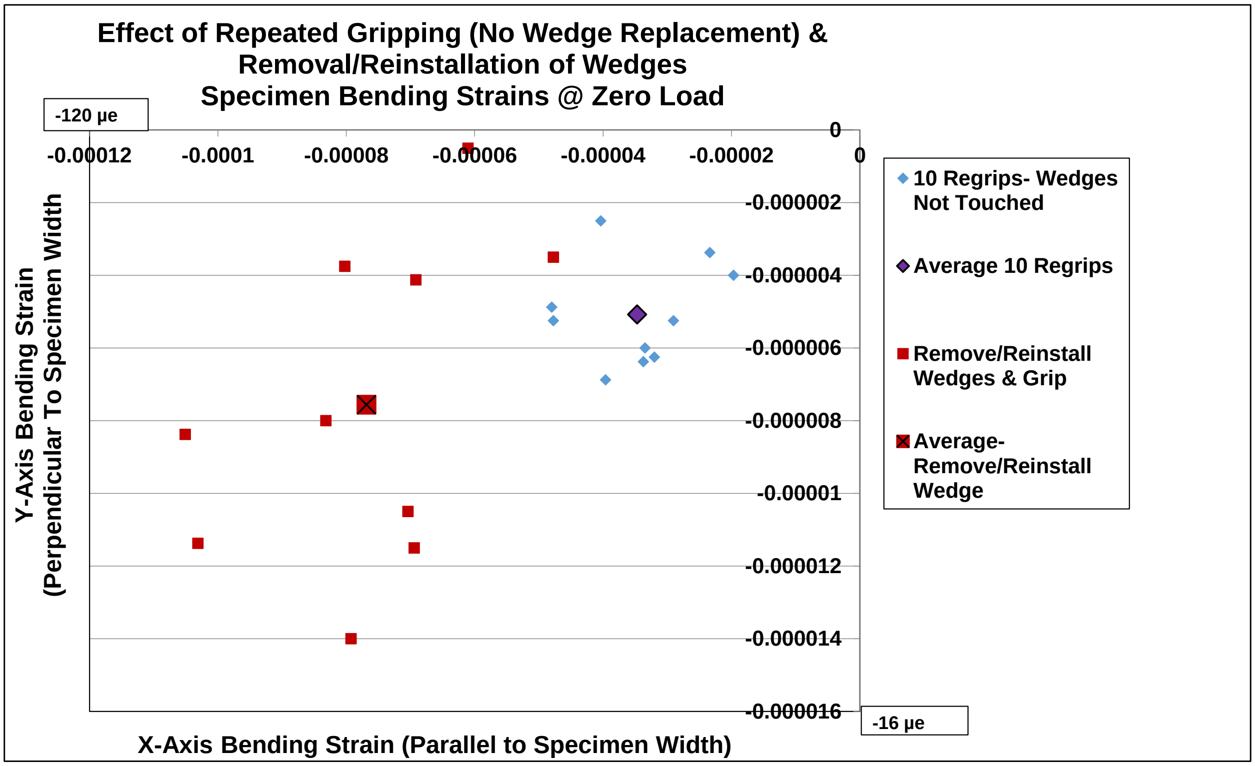

Figure 16. Bending Strain Trials For Repeated Gripping And Wedge Removal

5. REVISED CMC HIGH TEMPERATURE GRIP EVALUATION

A new grip design (V2) was developed based on the previously reported evaluation results. A generic schematic of the CMC Grip V2 design is shown in Figure 17. The grips operate very similar to the 647 Wedge Grips. The grips operate in a similar fashion to the proven 647 Wedge Grips, which improves alignment, operability, and ergonomics. The revised V2 grips retain the advanced cooling concept from the earlier design allowing them to operate at extremely elevated temperatures.

5.1 CMC Grip Version 2

In this revised design the wedges are now facing upwards similar to the 647 Wedge Grips. The wedges rest on a solid non-moving support rod that is anchored to the centerline of the grips. The wedge body sits on top of a hydraulically actuated outer tube. This outer tube and the wedge body, when actuated pulls the wedge body down onto the wedges forcing them to close, gripping a specimen. In this design the grips cannot translate vertically, only horizontally, to clamp the test specimen. A photograph the detail of the wedges, wedge chamber, and loading cylinder is shown in Figure 18. A photograph of the wedges inserted in the loading cylinder is presented in Figure 19. After the wedges are installed, the wedge body is installed and the cap is screwed into place as shown in Figure 20. There is an outer alumina sleeve that slides over and protects the metallic grips (see Figure 21). Air cooling is used to maintain the metallic hardware to 1000°C or lower.

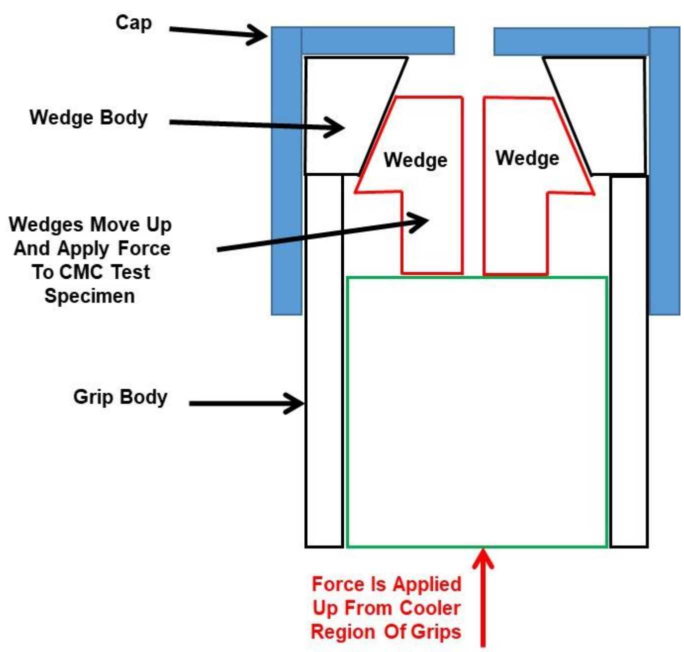

Figure 17. Schematic Of MTS High Temperature CMC Grip Version #2

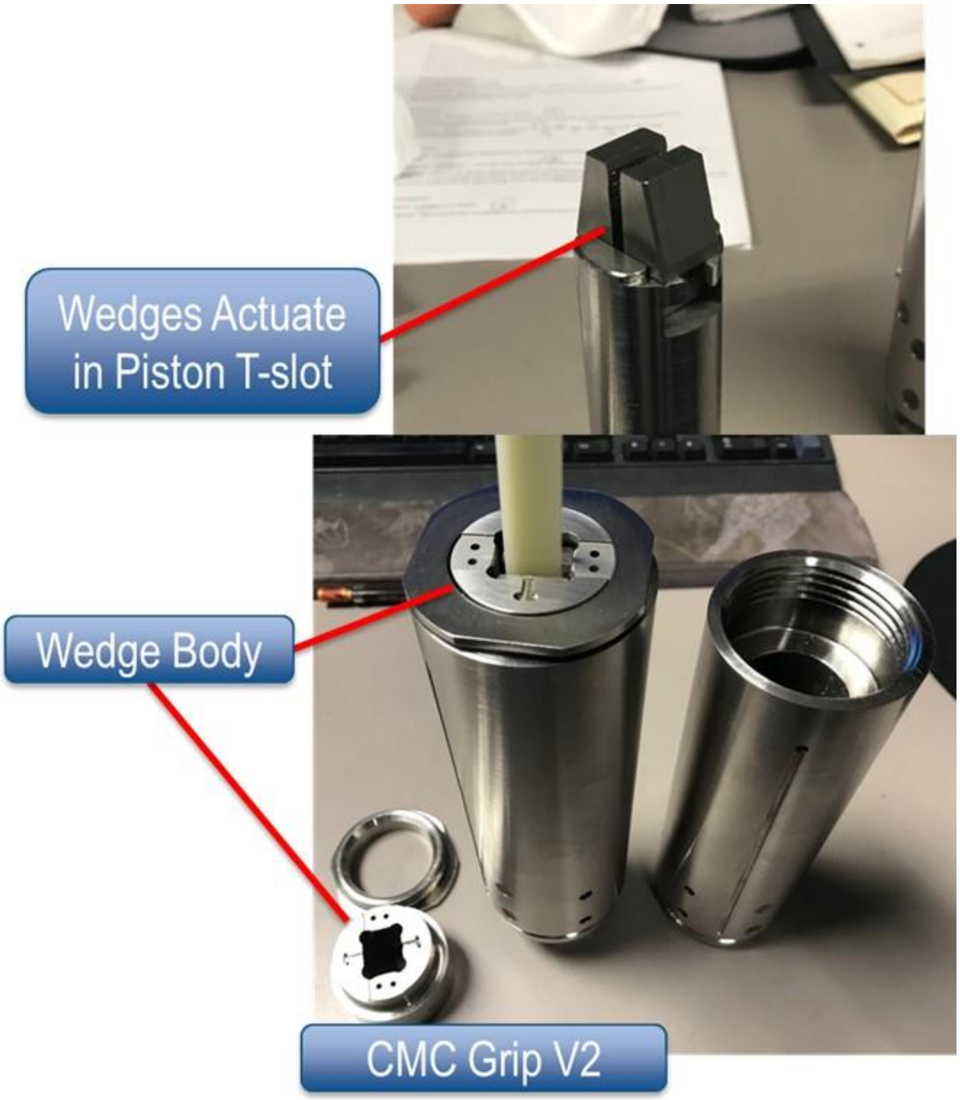

Figure 18. Photograph Of MTS High Temperature CMC Grip Version #2

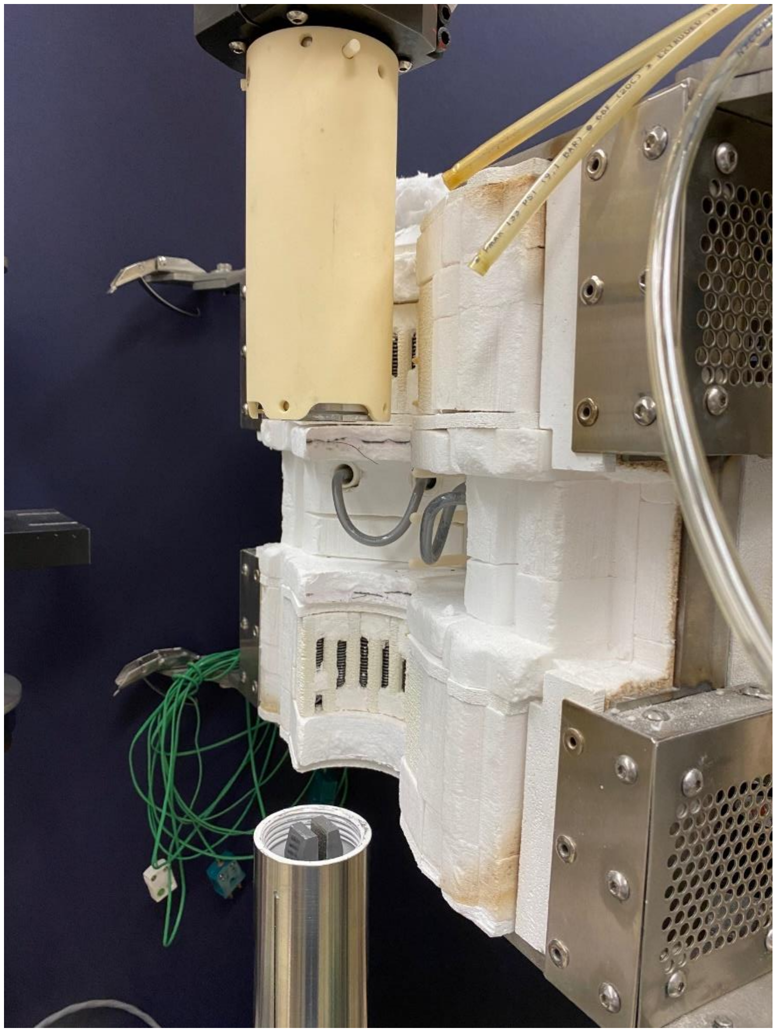

Figure 19. Photograph of CMC V2 Grips Showing Wedges Placed In Grips With Wedge Body Removed

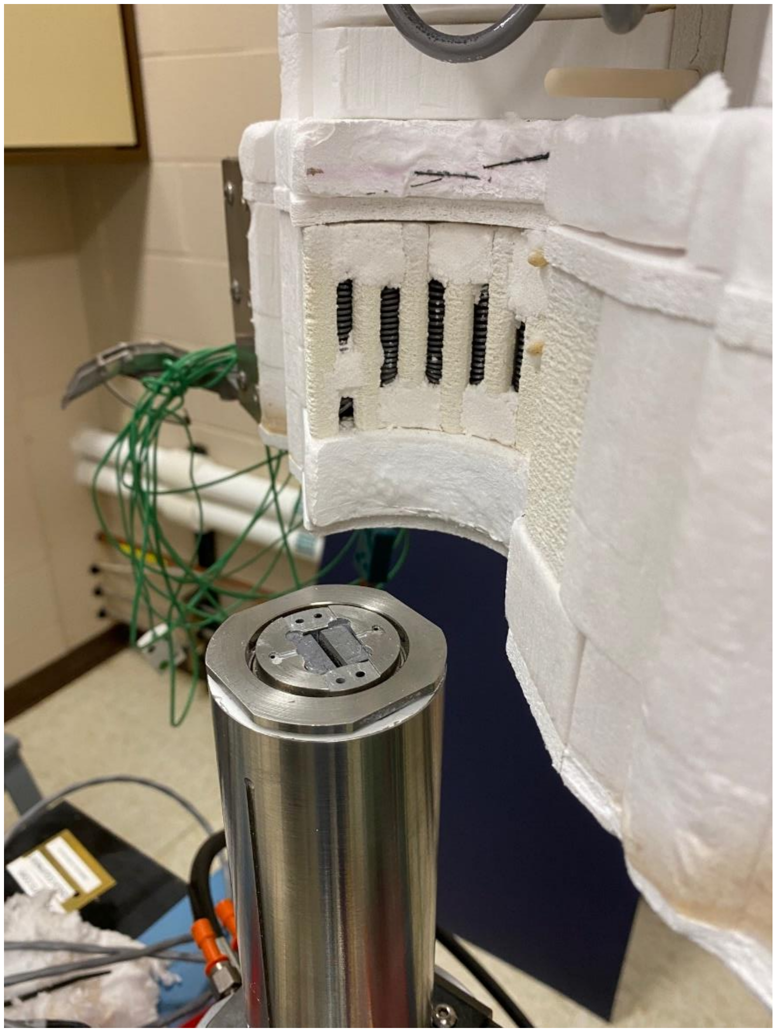

Figure 20. Photograph of CMC V2 Grips Showing Wedges Placed In Grips With Wedge Body And Cap Screwed In Place

Figure 21. Photograph of Revised CMC Grips V2

After manufacturing the prototype V2 grips they were installed on a Landmark servo-hydraulic load frame at MTS Systems for evaluation of alignment prior to shipment to AFRL. The results of these alignment trials as shown below in Figure 22 and Figure 23. Alignment was significantly improved over the CMC Grip V1, and significantly better than ASTM E1012 class 5. The grips and supporting hardware were then shipped to AFRL/RXCCP and arrived on 22 March, 2018

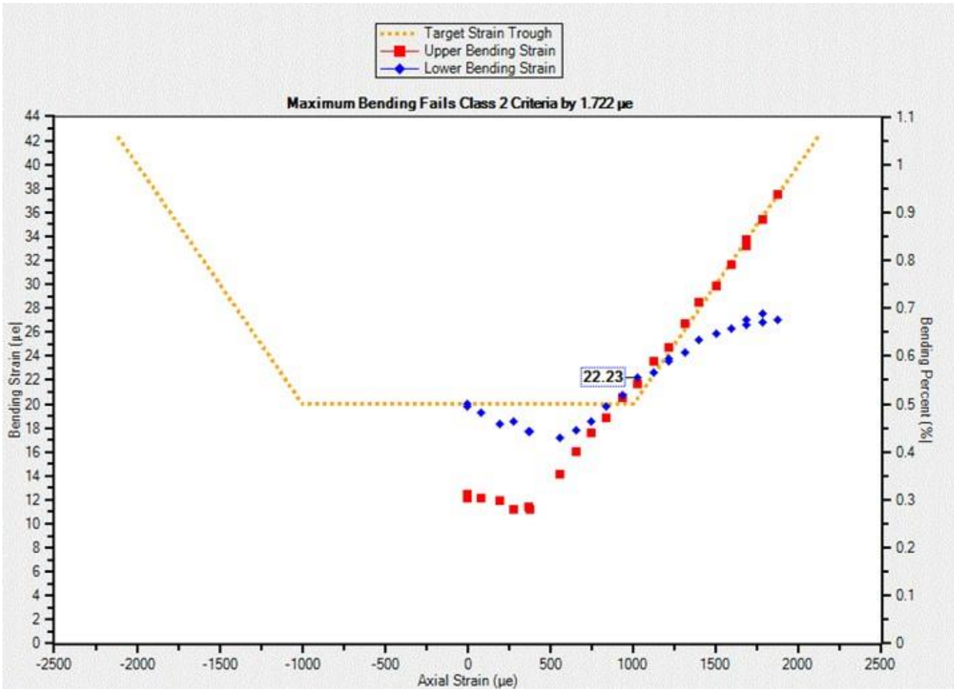

Figure 22. Bending Strain Versus Axial Strain For Revised CMC Grips V2 (Check #1)

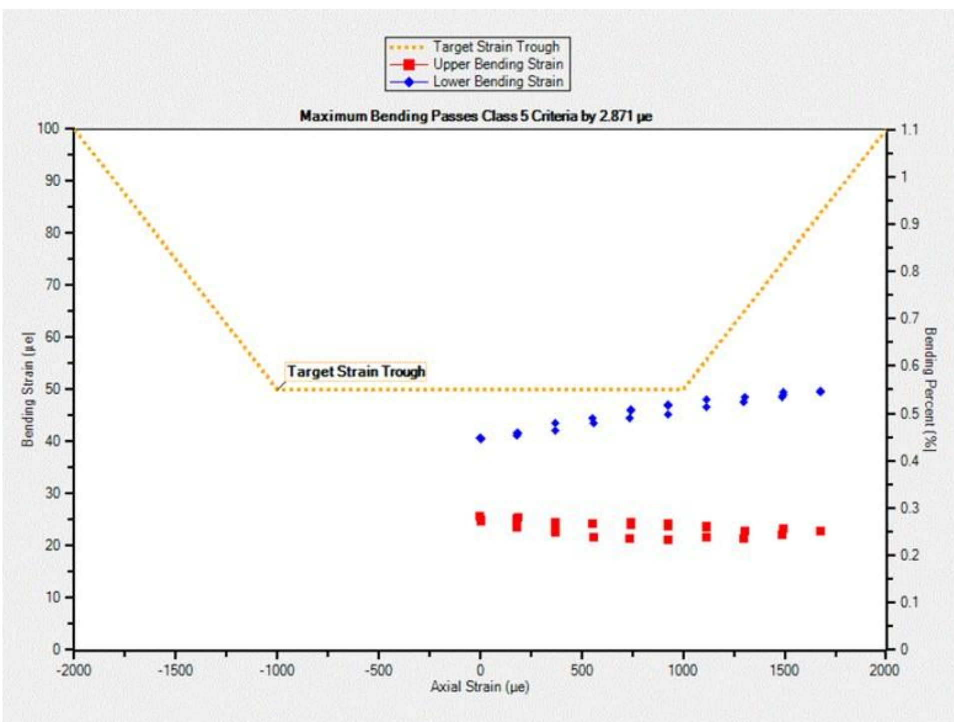

Figure 23. Bending Strain Versus Axial Strain For Revised CMC Grips V2 (Check #2)

5.2 Alignment Checks At AFRL

As stated above, MTS assembled the grips in their laboratory and performed alignment checks before shipping them to AFRL/RXCCP. The MTS alignment data was converted into percent bending and was replotted as percent bending strain versus axial strain (see Figure 24). At 500 micro-strain the bending strains were approximately 7%. The grips meet all of MTS requirements for alignment and were close to the ASTM requirement. Therefore, the grips were packaged up and shipped to AFR/RXCC.

The grips were unpackaged, inspected, and installed in a servo-hydraulic test frame. Significant time was spent trying to align the grips. Each time the alignment specimen was paced in the grips and grip pressure applied the bending strains would be different. Figure 25 presents bending strains for the top and bottom strain gages for three consecutive specimen installations. These were taken after the grips were aligned to produce the lowest bending strains that that could be achieved during gripping. The individual strain gages had an average range of 84 $\mu\varepsilon$, and a maximum range of 105 $\mu\varepsilon$. Well aligned grips normally produce bending strains of ~10 $\mu\varepsilon$ when the specimen is gripped. This means the CMC V2 grips exhibit 10x more bending strain than those measured on properly aligned MTS-647 wedge grips during gripping. At zero applied axial load, the CMC V2 grips are significantly out of alignment and each attempt to put in the alignment specimen generated random bending strains. This means there is no reproducibility in alignment when inserting test specimens. No alignment checks using axial loading were performed because of the repeatability issue with inserting test specimens.

It is important to emphasize that any removal of wedges for replacement or cleaning would likely have a significant effect on the integrity of the alignment.

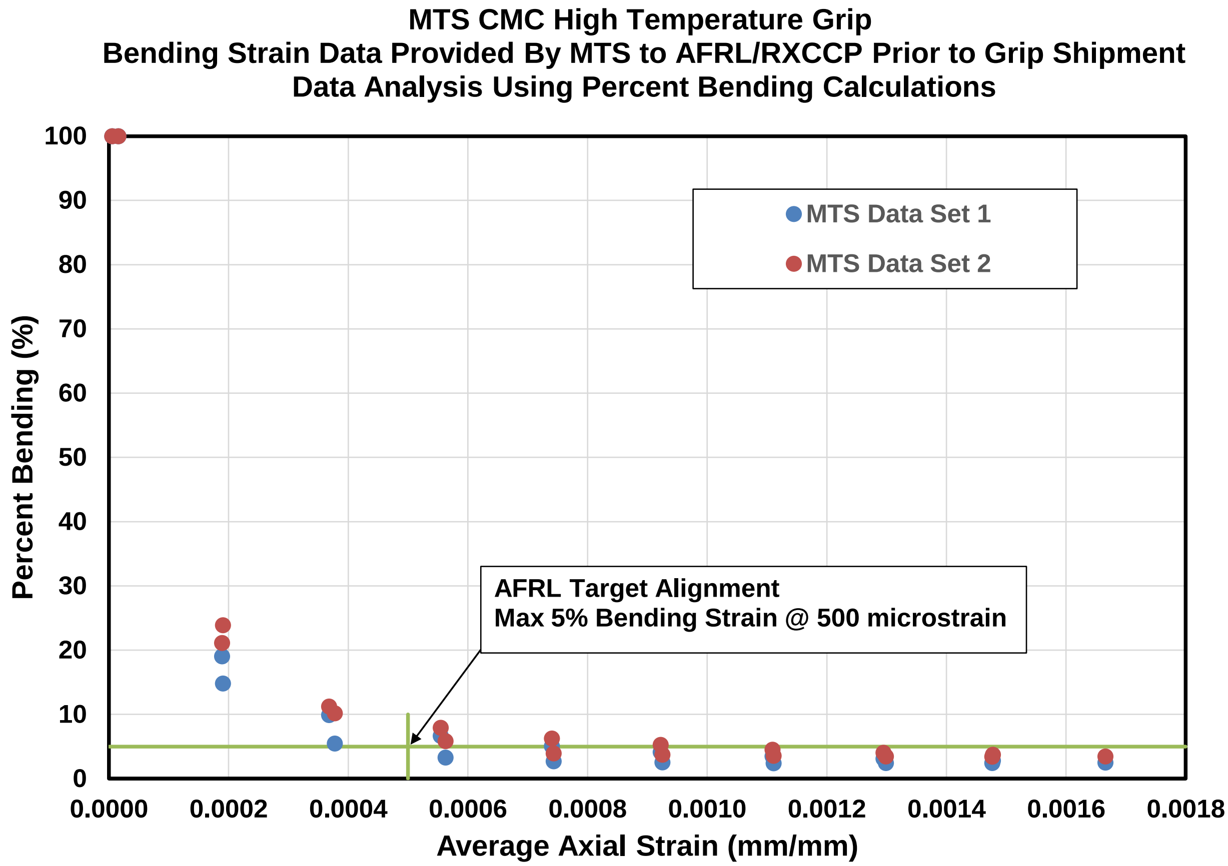

Figure 24. MTS Alignment Data Plotted as Maximum Percent Bending Strain Versus Axial Strain for Revised CMC Grips V2

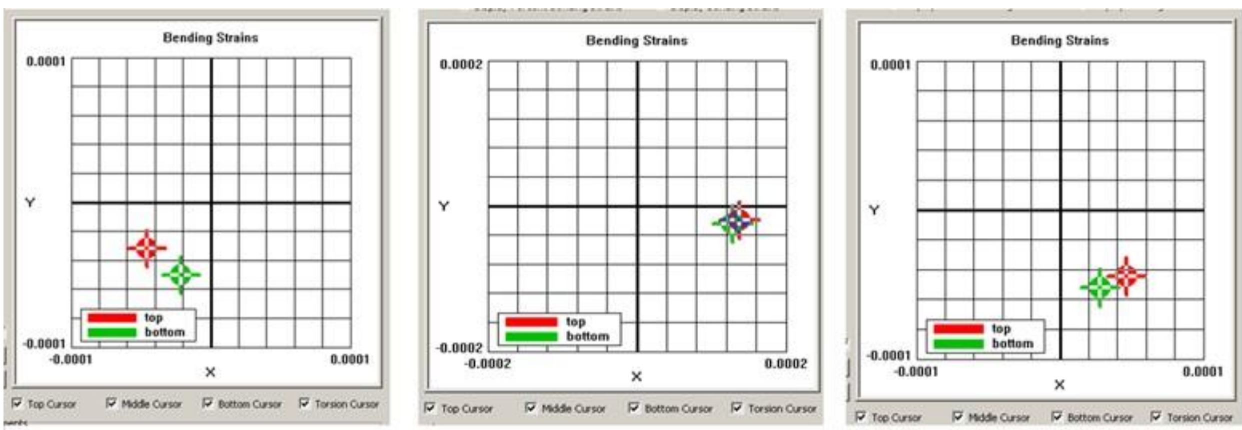

Figure 25. Bending Strains During Gripping For CMC Grips V2

5.3 Determination of Grip Pressure Versus Applied Axial Load

After checking alignment, the maximum load capacity of the CMC V2 grips was determined. MTS had performed calculations relating grip pressure to maximum achievable tensile load and those estimates are presented in Figure 26. Even though the alignment was not satisfactory, it would not affect the verification of the MTS predictions. The grips were designed to operate with pressures as high as 3000 psi.

Specimens of different width were placed in the grips, and different grip pressures were applied. The load was then increased until the specimen slipped in the grips. For all of the tests, the grips performed as designed. The grips are rated at 25 kN axial load, and they were able to meet that load level for both tension and compression, as well as for a much wider range of specimen thicknesses compared to the initial CMC V1 grip set.

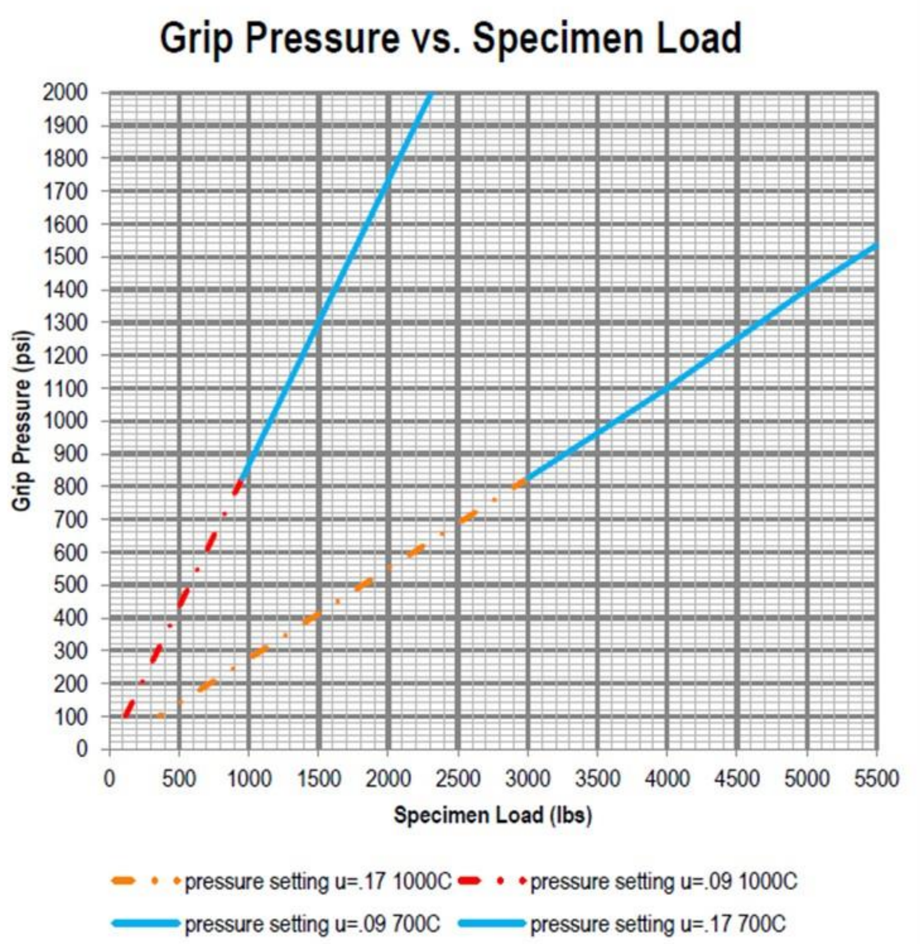

Figure 26. Estimated Grip Pressures Versus Specimen Load for Revised CMC Grips

5.4 Summary Of Version 2 CMC High Temperature Grips

The revised CMC V2 grips are greatly improved over the initial CMC V1 grips. The grips can apply the required axial loads. However, alignment of the grips remains an issue. The team decided that the design needed to be refined so that the grips can be aligned repeatedly. In addition, the grips have only seen limited usage, and there is no time at temperature on these grips. MTS agreed that a redesign was warranted. A CMC version 3 (V3) grip set was designed and constructed. However, time expired on the project before AFRL could evaluate the new redesign.

6. FURNACE EVALUATIONS

Considerable time was spent by AFRL/RXCC evaluating the new three zone furnace manufactured by Mellen for MTS. In this section, the thermal profile of a standard single zone short furnace (Manufactured by Mellen) and the three zone furnace will be discussed. The single zone furnace is a short zone furnace intended to be used with cool grips such as the MTS-647 hydraulic grips. The thermal profile data for the single zone furnace was thoroughly reported earlier in report number AFRL-RX-WP-TR-2014-0168 [AFRL-RX-WP-TR-2014-0168, "Configuration and Calibration of High Temperature Furnaces for Testing Ceramic Matrix Composites", Larry Zawada, Ken Davidson, and Phillip Blosser], so only a short summary will be provided. It is important to note that the Mellon single zone furnace produces an excellent thermal profile and has been used very successfully to test CMC specimens at elevated temperatures.

However, the objective here is to evaluate the new three zone furnace with the hot grips to see if the thermal profile can match or improve on the thermal profile obtained from the single zone furnace. Those earlier thermal profile results for the single zone furnace will be discussed and then followed by a through temperature profiling of the new three zone furnace produced by Mellen. This three zone furnace is intended to be used with the MTS Hot Grips.

6.1 Procedure for Temperature Profile Measurement

Thermocouple placement (location and method) on the CMC specimen used for thermal profiling is critical. CMC test specimens are not normally tested with TCs attached to them to avoid any chance of a reaction taking place between the CMC and the metallic TC wire. Therefore, it is critical to know in detail the thermal gradient profiles, control temperatures, and resulting witness temperatures prior to actual testing. AFRL always uses an actual CMC test specimen to conduct this profile process for each CMC material system tested. For the Mellen furnace, three thermocouples (S-type material) are attached to the specimen at the CL and both ends of the gage section, with the TC bead placed in contact with the specimen.

Upon securing the TC leads to the specimen using platinum wire ties, the bare wire is coated with ceramic cement to prevent degradation and to bond the TC beaded tip to the CMC. Once the calibration specimen is mounted in the test frame, two additional TCs are brought in contact with the edge of the specimen and located at the locations where the extensometer rods would contact the specimen. In addition, there are the three furnace supplied TCs encased in alumina tubes. An actual CMC test specimen (usually a dogbone shape) should always be used for the furnace profiling. However, if an actual test specimen is not available, then a straight-sided piece of the same CMC material can be used. It was found that the shape of the test specimen (straight-side, dogbone, or hourglass) is not critical. However, a similar length is important to insure that symmetrical thermal gradients outside the hot zone are achieved. Use of the same CMC material for thermal profiling as targeted for mechanical testing is critical as the heat transfer properties of various materials can have significant differences.

In addition to calibrating the furnaces using fully instrumented thermal profile specimens, the Mellen furnace was also profiled using a single probe commonly used in many laboratories. For purposes of this study, an additional test fixture was designed to hold either a single ceramic probe or a test specimen and precisely move it vertically (axially) throughout the Mellen furnace.

Specimens were positioned so that the specimen centerline and furnace control probe were at the same vertical position, designated “0 mm” in the presented data.

The thermal probe is always placed parallel to the furnace control probe, with this position defined as zero. Displacement towards the top of the furnace was defined as a negative. The displacement interval was 5 mm, dwell between positions of five minutes, and a data collection rate of every 15 seconds was used. Data averaging of the last eight points for each temperature defined the temperature at position. Single-point mapping was performed over the entire furnace length to define the gradient between the hot zone and room temperature. In cases where a specimen replaced the probe, profile locations above the furnace cavity were not possible. This is because the specimen would need to project up through insulation placed on top of the furnace to simulate typical operating conditions. Profile locations below the furnace cavity were achievable and, thus, more data was obtained. Following initial single-probe profiles, the specimen-holding device was modified to accept CMC test specimens. Multiple specimens having different material and processing conditions, containing both single and multiple thermocouples, were evaluated.

6.2 Mellen Single Zone Furnace Temperature Profiles

A photograph of the Mellen single zone furnace is shown in Figure 27, and a photograph of the furnace operating at 1300°C is presented in Figure 28. The furnace was originally made in one piece to minimize heat loss, but this made it extremely difficult to load a specimen in the test machine. Therefore, a “pie cutout” modification to allow front access was made. This modification allows for easy specimen access, requires only one person to mount specimens in the test frame, and is now used by Mellen when manufacturing this single zone furnace.

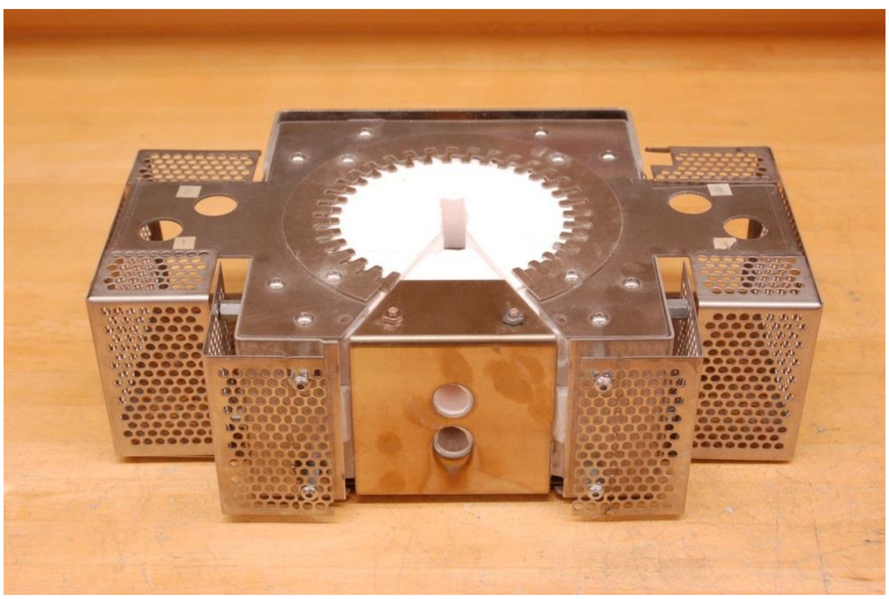

Figure 27. Photograph of Mellen Single Zone Furnace

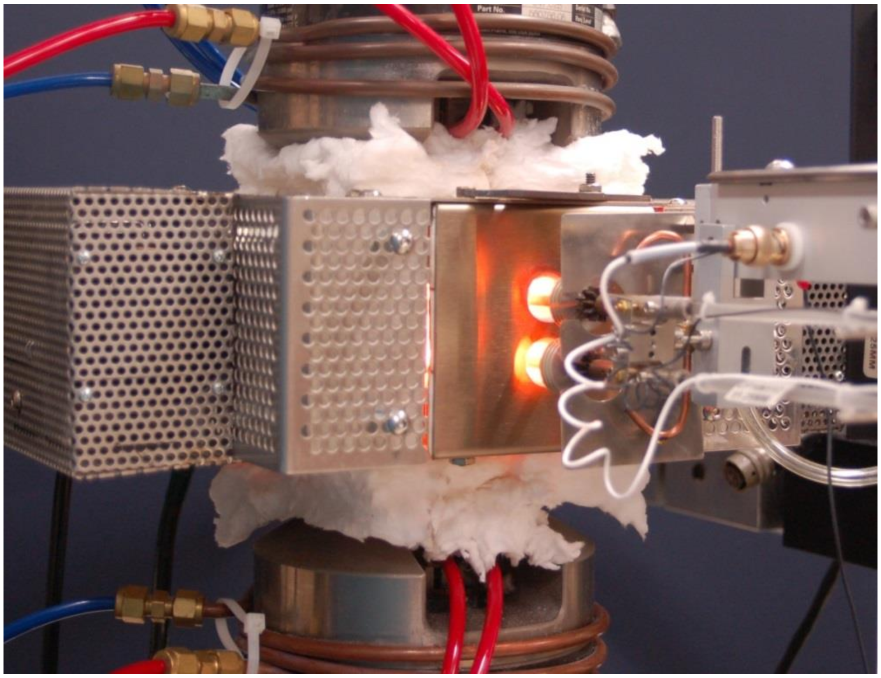

Figure 28. Photograph of Mellen Single Zone Furnace at Temperature

Using the thermal profiling procedure outlined above, the thermal profiles for seven different CMCs for a 25 mm extensometer gage length were measured and are presented in Table 5. In this table the percent error at the center of the specimen was always approximately less than 1%. However, at the edges of the extensometer the temperature was off by roughly 3-4% at the top of the gage length and roughly 4-10% at the bottom of the gage length. These profiles are very good for a single zone furnace, but do highlight that using a short furnace and cold grips does result in slightly larger thermal deviations from the desired test temperature than what ASTM recommends (±1%).

Table 5. Temperature Profiles for Seven CMCs for Single Zone Mellen Furnace

Mellen Furnace							
Ceramic Matrix Composite	**Thermal Profile Temp. (C)**	**Thermal Couple Location On CMC Specimen**			**% Deviation**		
		Top	**Center**	**Bottom**	**Top**	**Center**	**Bottom**
		12 mm Above CL	**CL**	**12 mm Below CL**	**12 mm Above CL**	**CL**	**12 mm Below CL**
Nextel 610/AS	900	922	908	918	2.4	0.9	2.0
	1000	**1013**	**1006**	**945**	**1.3**	**0.6**	**-5.5**
	1100	**1101**	**1100**	**968**	**0.1**	**0.0**	**-12.0**
	1200	1190	1196	1063	-0.8	-0.3	-11.4
	1300	1279	1293	1165	-1.6	-0.5	-10.4
Nextel 720/AS	900	864	902	850	-4.0	0.2	-5.6
	1000	966	1004	959	-3.4	0.4	-4.1
	1100	**1058**	**1101**	**1058**	-3.8	0.1	-3.8
	1200	**1160**	**1203**	**1164**	-3.3	0.3	-3.0
Nextel 720/A	900	897	907	844	-0.3	0.8	-6.2
	1000	992	1005	941	-0.8	0.5	-5.9
	1100	1090	1106	1037	-0.9	0.5	-5.7
	1200	**1179**	**1198**	**1126**	**-1.8**	**-0.2**	**-6.2**
	1300	**1271**	**1291**	**1218**	**-2.2**	**-0.7**	**-6.3**
S200H	900	893	910	866	-0.8	1.1	-3.8
	1000	988	1006	962	-1.2	0.6	-3.8
	1100	1088	1107	1064	-1.1	0.6	-3.3
	1200	**1185**	**1205**	**1163**	**-1.3**	**0.4**	**-3.1**
	1300	**1277**	**1298**	**1256**	**-1.8**	**-0.2**	**-3.4**
	1400	1367	1389	1347	-2.4	-0.8	-3.8
Carbon/HYPER-SIC	900	892	910	812	-0.9	1.1	-9.8
	1000	987	1006	906	-1.3	0.6	-9.4
	1100	1085	1105	1006	-1.4	0.5	-8.5
	1200	**1186**	**1207**	**1110**	**-1.2**	**0.6**	**-7.5**
	1300	**1285**	**1307**	**1211**	**-1.2**	**0.5**	**-6.8**
	1400	1397	1397	1302	-0.2	-0.2	-7.0
SiC/HYPER-SIC	900	863	905	825	-4.1	0.6	-8.3
	1000	964	1005	928	-3.6	0.5	-7.2
	1100	1067	1106	1031	-3.0	0.5	-6.3
	1200	**1167**	**1206**	**1133**	**-2.8**	**0.5**	**-5.6**
	1300	**1268**	**1307**	**1236**	**-2.5**	**0.5**	**-4.9**
	1400	1368	1407	1339	-2.3	0.5	-4.4
S400	900	892	912	830	-0.9	1.3	-7.8
	1000	986	1009	930	-1.4	0.9	-7.0
	1100	1081	1105	1029	-1.7	0.5	-6.5
	1200	**1173**	**1199**	**1128**	**-2.3**	**-0.1**	**-6.0**
	1300	**1266**	**1293**	**1225**	**-2.6**	**-0.5**	**-5.8**
	1400	1362	1389	1326	-2.7	-0.8	-5.3

As stated earlier, a single-probe ceramic-coated thermocouple was transitioned through the furnace and temperature measurements were made (Figure 29). From the figure, one can make two related observations. The first is that the probe matches closely to the center and top furnace TC, but is substantially off for the lower furnace TC. The temperature falls off substantially faster below than above the furnace cavity because of the heat convection upwards.

Following the probe study, actual CMC thermal profile specimens were next transitioned through the furnace using the same procedure as the ceramic probe. Both a straight-sided S200 CMC specimen and a dogbone Nextel 720/AS specimen were used. The results are shown in Figure 30 and compared to the ceramic probe results. Several observations can be made from the thermal profile data presented in the figure. The first and most significant is that, in the furnace cavity, both the ceramic probe and the furnace TC read substantially lower than the TC on the CMC thermal profile specimens. This is a very critical finding and highlights the importance of using an actual CMC test specimen to perform the thermal profiles. It also clearly demonstrates how both radiation and convection contribute to the measured temperature. The second observation is that there is a reasonably good match between the straight-sided CMC and dogbone specimen for inside the furnace cavity. However, below the furnace hot-zone cavity, a large difference between the two CMC specimen geometries is observed. It is suggested that the difference is attributed to the type of specimen material and not the specimen geometry. The S200H is more conductive than the Nextel 720/AS. Also, both the N720/AS and the ceramic probe match closely below the hot-zone cavity region of the furnace. In this region there is only heat conducting along the length. Both the Nextel 720/AS and the probe are mostly alumina, so it is expected that they would have similar thermal profiles outside of the furnace. Above the furnace, the thermal profile was found to be location dependent and not specimen dependent.

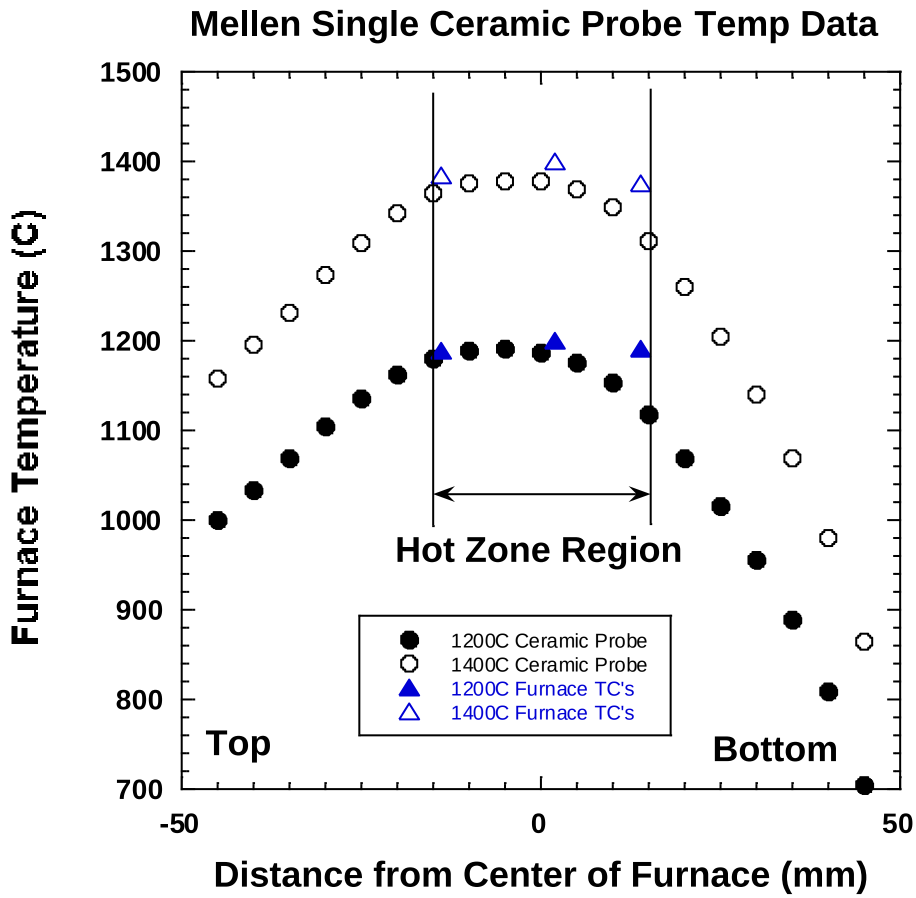

Figure 29. Mellen Single Zone Furnace Thermal Profile Using Single Probe

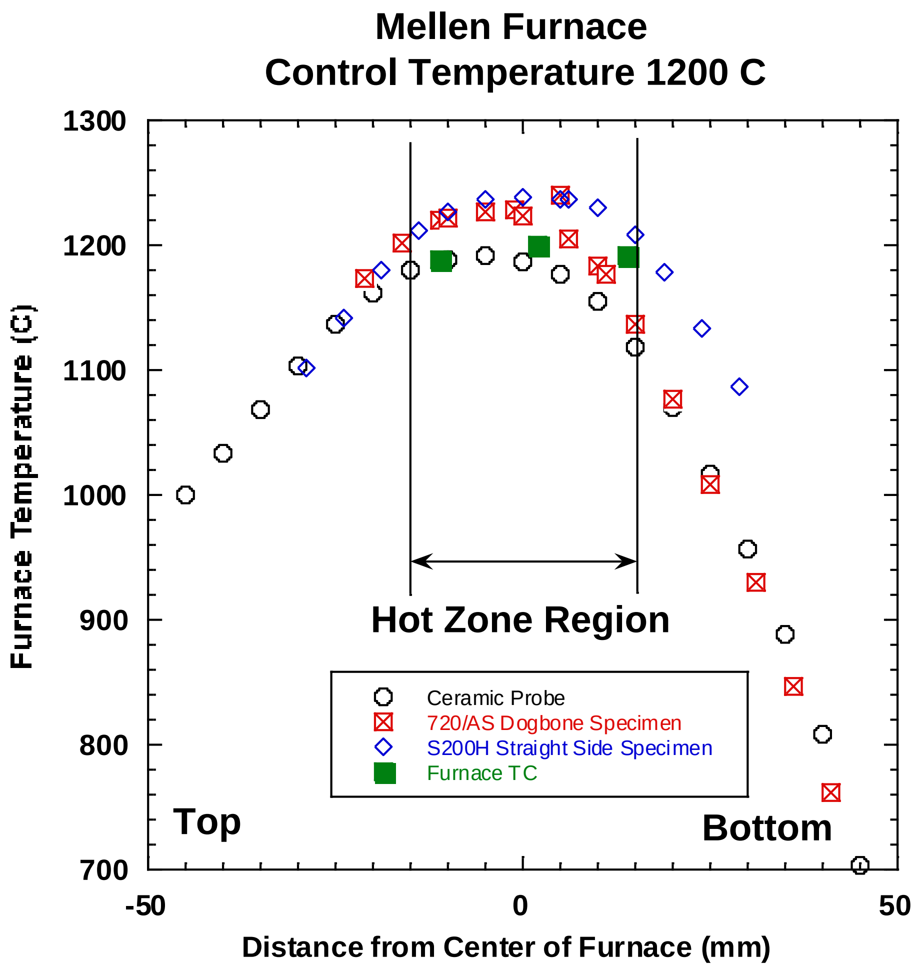

Figure 30. Effect of CMC Type on Thermal Profile of Mellen Single Zone Furnace

6.3 Mellen/MTS Three Zone Temperature Profiles

A schematic of the three zone furnace built by Mellen for MTS is presented in Figure 31, and a photograph of the as-manufactured furnace is presented in Figure 32. The furnace is opened so that one can see the three independent zones. The center zone is insulated from the top and bottom zones to minimize heat transfer between the ~1500°C center zone and the cooler outer zones. The furnace uses standard silicon carbide heating elements in the top and bottom zones, and four molybdenum disilicide heating elements in the center zone. As with the Mellen single zone furnace, there are three control thermocouples in the center zone. A schematic showing how the three zone furnace mounts in a standard two pole servo-hydraulic test frame is presented as Figure 33. A photograph of the three zone furnace and the CMC V2 grips mounted in a servo-hydraulic test frame is presented in Figure 34.

Several thorough thermal profiles were performed on the furnace using thermocouples in all three zones. Results are presented in Figure 35 for a thermal profile performed that started at room temperature and then held at 1400°C. In this case the center zone was set at 1400°C, while the top and bottom zones were held at approximately 1000°C. The maximum thermal capability of the single zone furnace is ~ 1400ºC and therefor this was the hottest temperature at which the performance of the single zone and three zone furnaces could be directly compared.

With time, the top zone does experience some additional temperature rise because of the "chimney effect" of heat rising from the extremely hot center zone. After a number of runs, AFRL/RXCC was able to determine the optimum settings and cooling air to achieve the desired temperature profiles. After several heating cycles it was determined that it was relatively easy to maintain the center zone at extremely high temperatures while maintaining the outer zones at or below 1000°C.

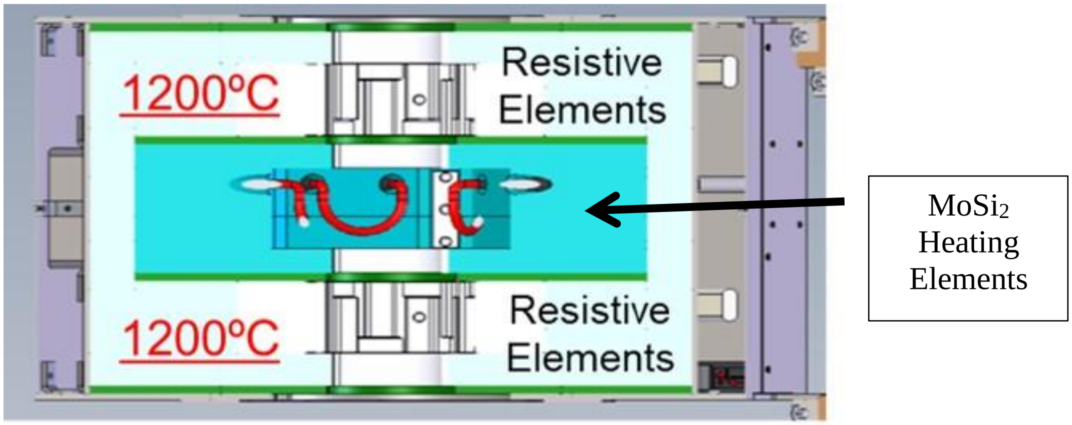

Figure 31. Schematic of Mellen Three Zone (New MTS 657.15) Furnace Configuration Showing The Three Zones – Two Resistive Elements and one Central $MoSi_2$

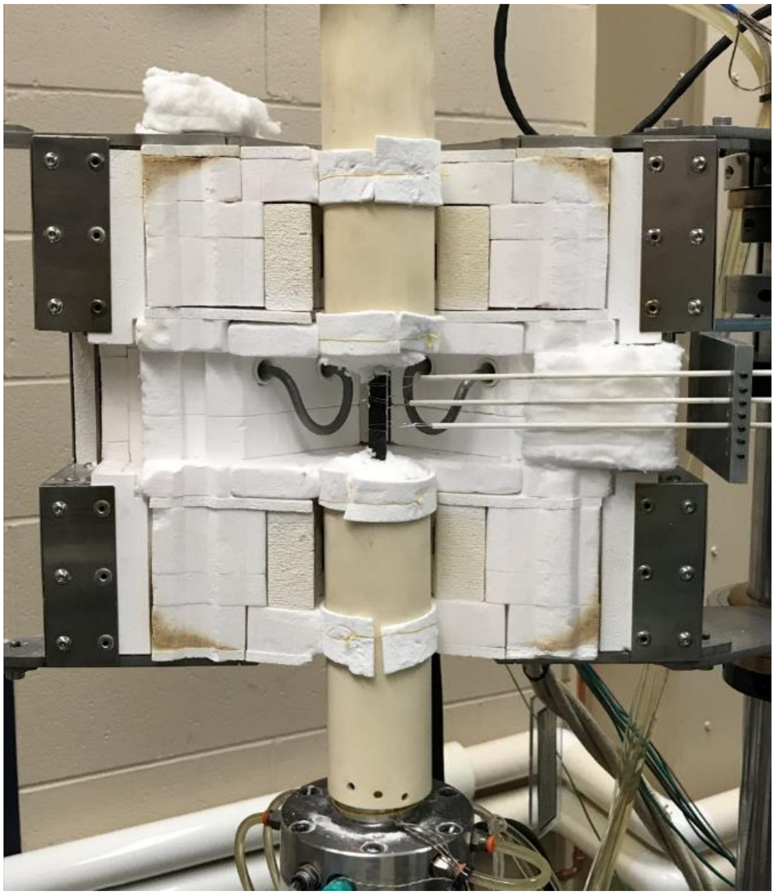

Figure 32. Photograph of Mellen Three Zone Furnace

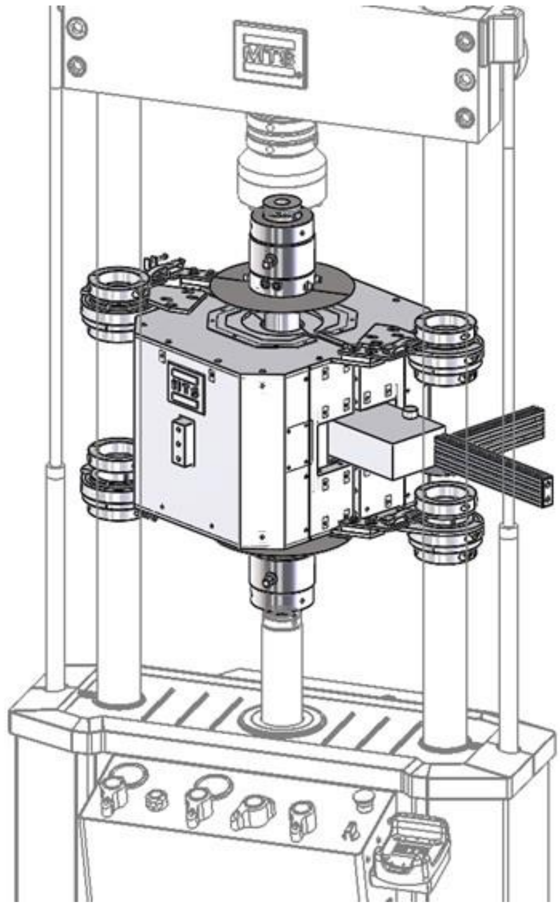

Figure 33. Schematic Showing Three-Zone Furnace Mounted On a Servo-Hydraulic Test Frame

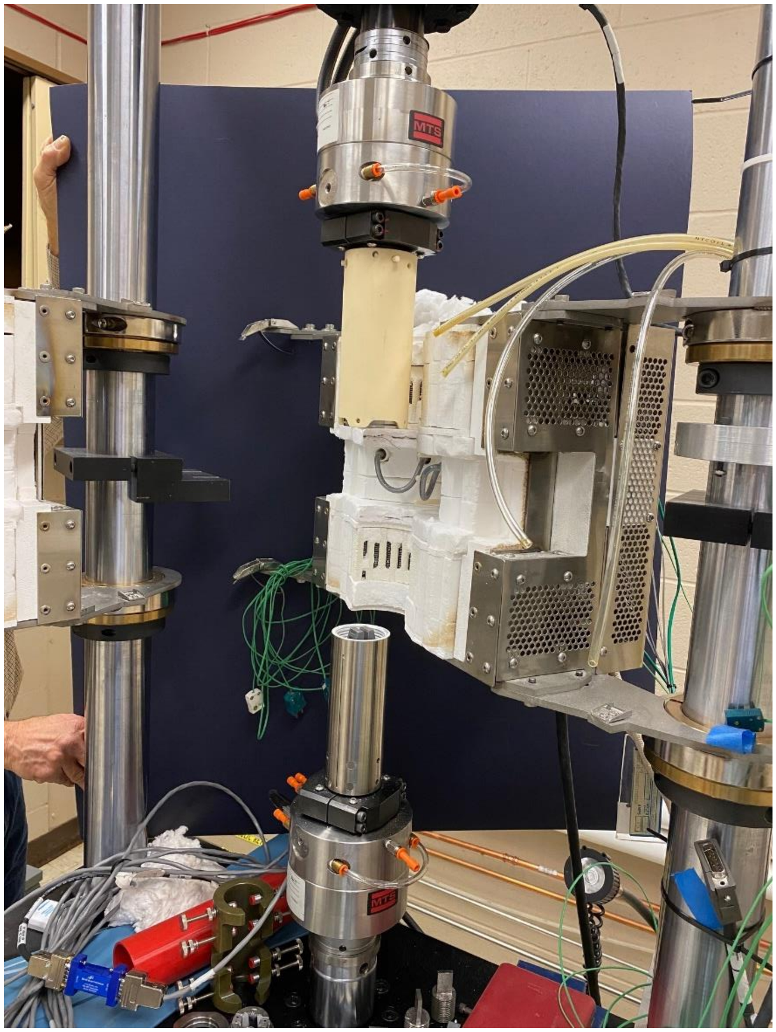

Figure 34. Photograph Of Three Zone Furnace And CMC V2 Grips Mounted In A Servo-Hydraulic Test Frame

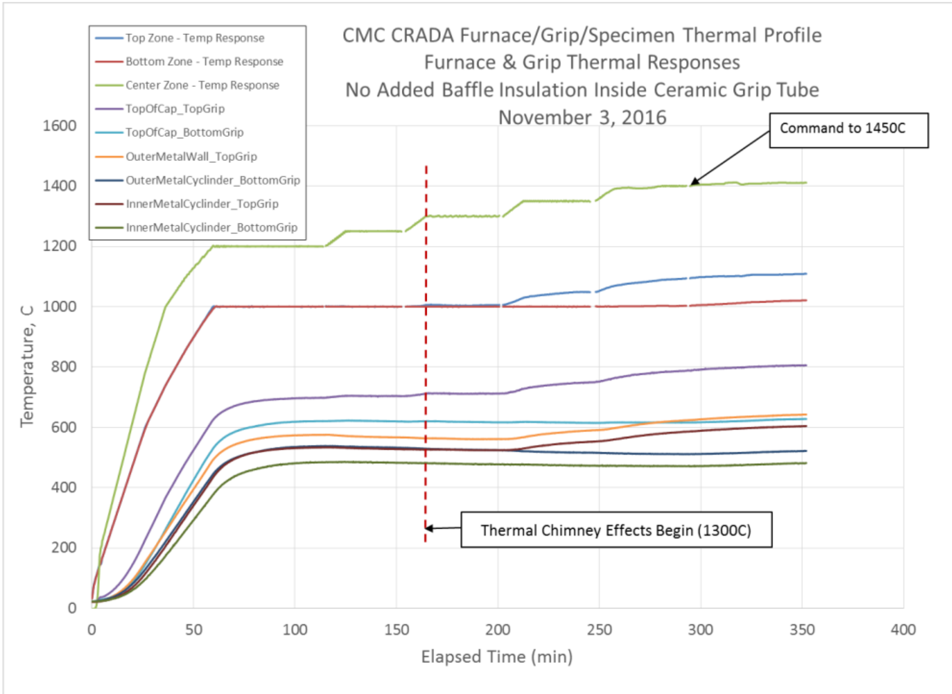

Figure 35. Thermal Profile for Mellen Three Zone Furnace up to 1400°C

There was interest in documenting how much power was required by the furnace to reach the required very high test temperatures. Therefore, the furnace was operated at multiple temperatures and the power requirements were recorded. The results from this study are presented in Table 6. The furnace uses only approximately 100 amps to achieve 1400°C. However, to reach 1495°C, the furnace power requirements jump 45% to 145 amps.

Table 6. Power Output Requirements for Mellen Three Zone Furnace

Center Zone Heating Elements		
Control Temperature, C	Duty Cycle, % Output	Electrical Current Usage, Amps
1000	40-49	60-80
1200	56-58	75-85
1300	60-70	80-92
1400	66-72	93-114
1450	68-78	82-130
1495	71-82	112-145

Thermal profiles were performed with CMC specimens and with probes. The first profiles were performed with a CMC specimen. A photograph of a CMC test specimen instrumented with five thermocouples bonded to it is presented in Figure 36. This was performed using the procedure outlined above. Results from one of the profile checks is presented in Table 7. For temperatures between 1000°C and 1495°C the temperature along the entire 25 mm center gage section of the specimen never exceeded 1%. This is an excellent profile and far exceeds that which was achieved with the Mellen single zone furnace. The results from the table are presented graphically in Figure 37.

It is important to establish actual test specimen temperatures with respect to the furnace control thermocouples. This is shown graphically in Figure 38 for a CMC test specimen at 1200°C. The temperature of the test specimen is 1204°C while the furnace control temperature is at 1180°C. This highlights that significant error can be made if one only used the furnace control temperatures vs specimen temperatures as mapped from previous experiments.

Figure 36. Photograph of a C/C Specimen Mounted With Five Thermocouples for Thermal Profile

Table 7. Thermal Profile Values for Mellen Three Zone Furnace and C/C Specimen

Furnace Temperature, C	Max Temperature Gradient, Specimen +/- 20.5mm, C	Max Temperature Gradient, Specimen GL +/- 12.5mm, C	Percent Temperature Gradient, Specimen +/- 20.5mm	Percent Temperature Gradient, Specimen GL +/- 12.5mm
1000	22	3	2.2	0.3
1200	31	8	2.6	0.67
1300	26	9	2	0.69
1400	21	6	1.5	0.42
1450	35	7	2.4	0.48
1495	31	3	2.1	0.2

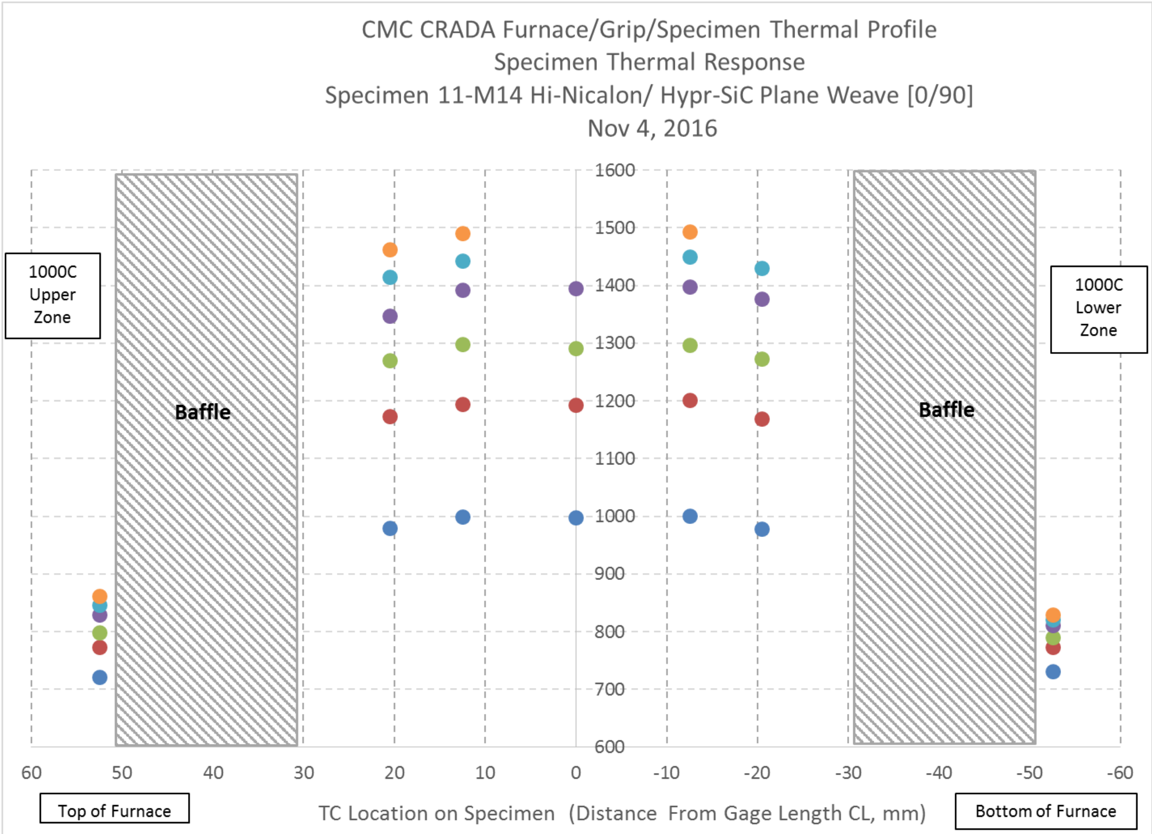

Figure 37. Thermal Profile of a CMC in the Mellen Three Zone Furnace

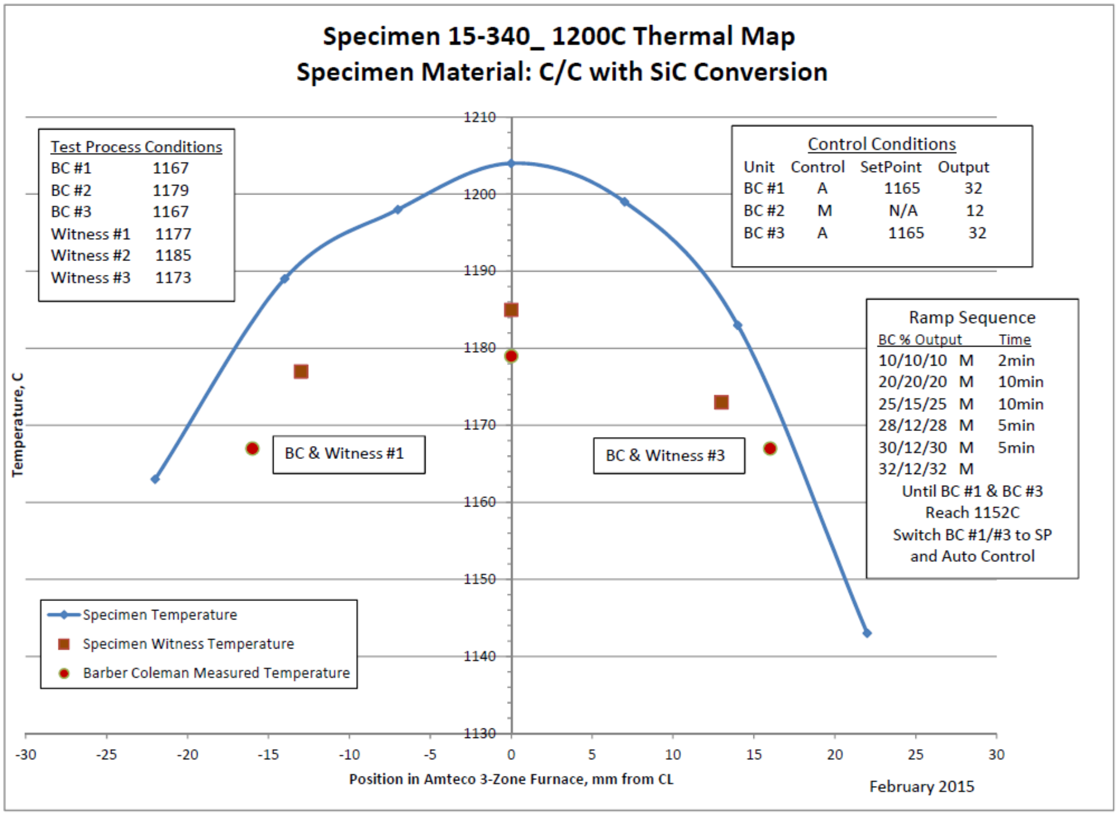

Figure 38. Furnace and CMC Thermal Profiles for Mellen Three Zone Furnace

Thermally profiling the Mellen three zone furnace with the probe technique required the construction of a new probe arrangement. This was required because of the long length of the furnace and the limited travel of the MTS actuator. Therefore, a design was incorporated that used three probes that were different length. A schematic of the probes is shown in Figure 39 and a photograph of the probe fixture in the furnace is presented in Figure 40. The furnace was profiled at 1200°C (see Figure 41) and at 1400°C (see Figure 42). At both temperatures the thermal profile within the 25 mm center gage section show very favorable (small) gradients. The profiles are essentially flat out to ±20 mm. This favorable gradient performance allows for larger gage sections in the CMC test specimens, allowing a larger volume of material to be characterized per test. In addition, the outer zones are at or above 1000°C. The top zone is slightly hotter than the bottom zone. This, for a 178 mm long specimen, the entire specimen should be above 1000°C and hence avoid any chance of intermediate temperature embrittlement occurring.

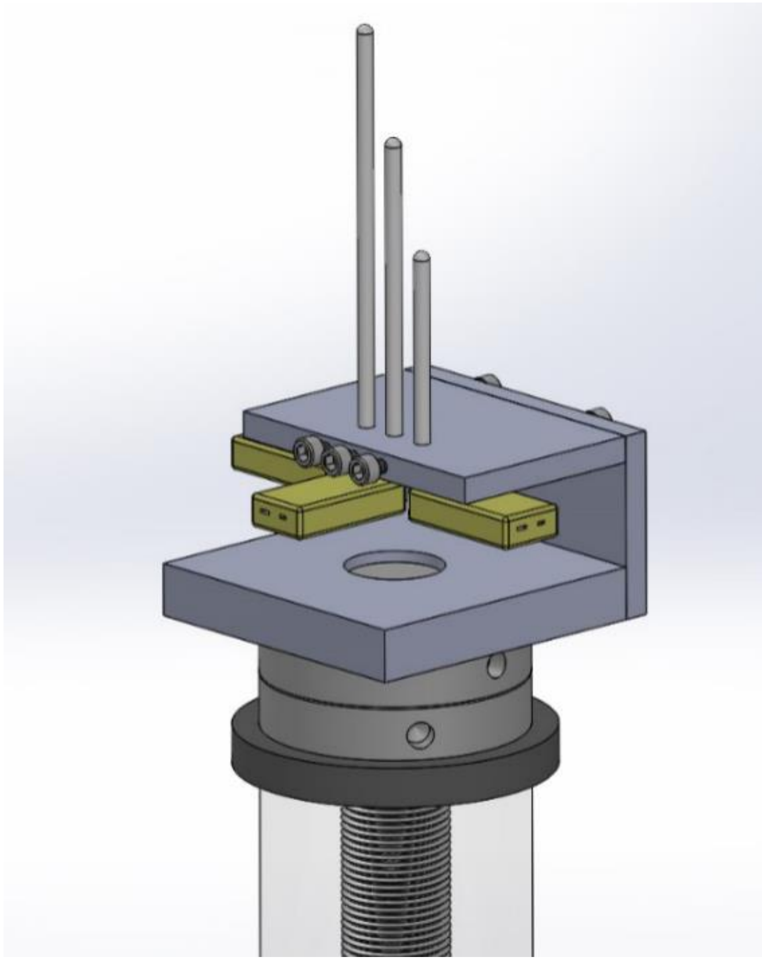

Figure 39. Schematic of Three Thermocouple Thermal Profile Probe Assembly

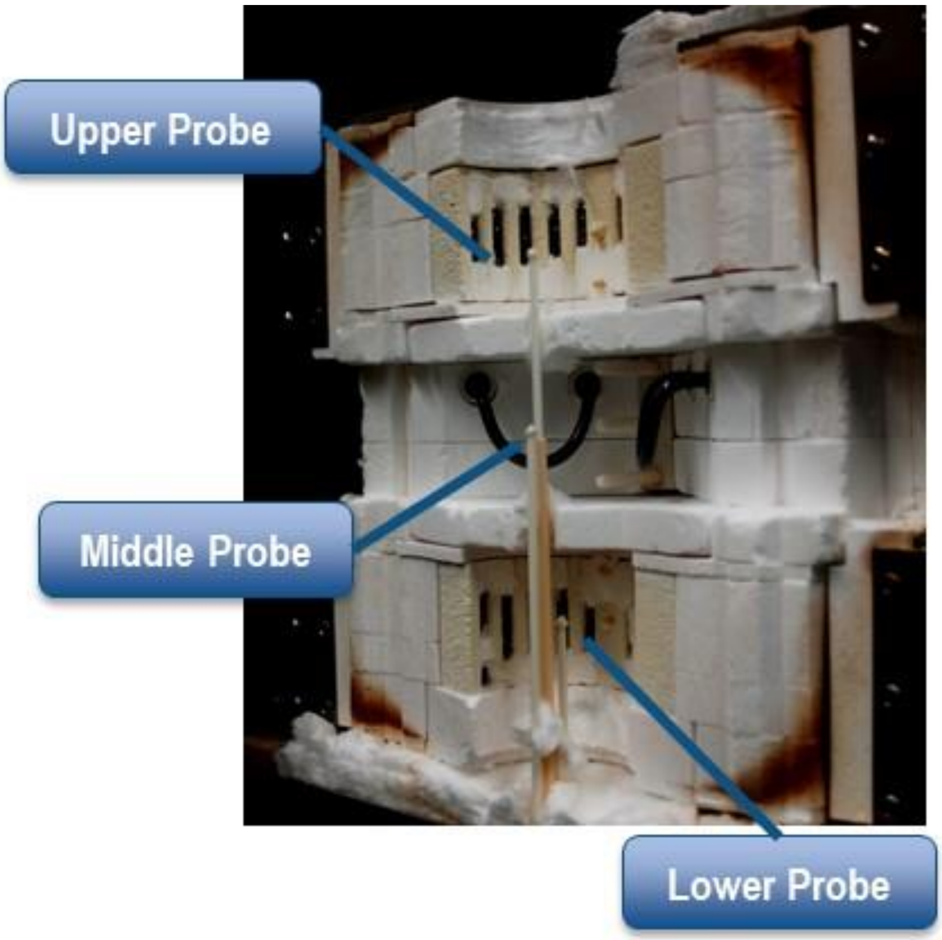

Figure 40. Photograph of Three Probe Assembly in Mellen Three Zone Furnace

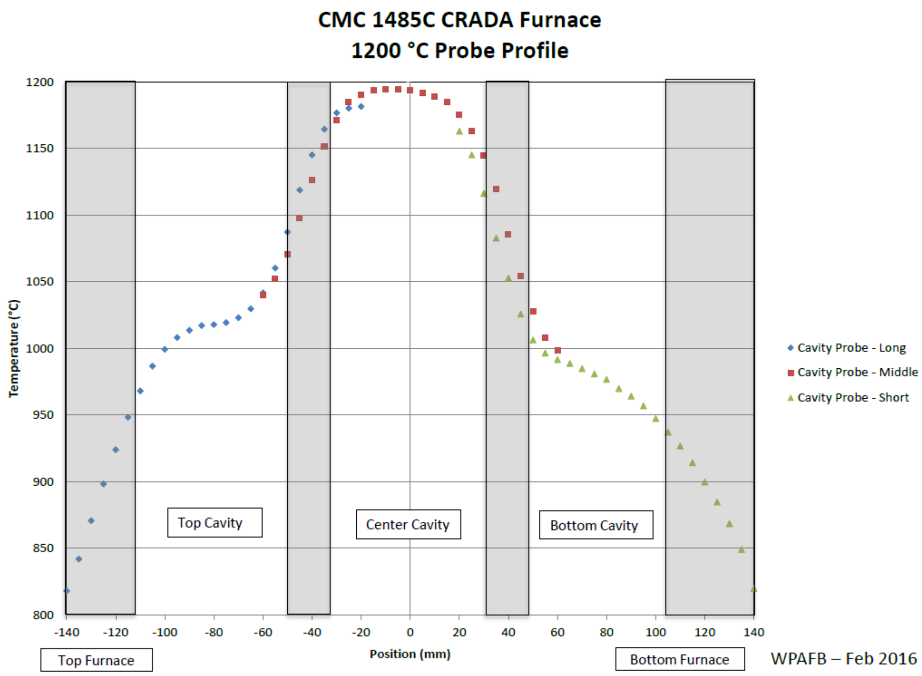

Figure 41. Plot of Thermal Profile of Mellen Three Zone Furnace at 1200°C

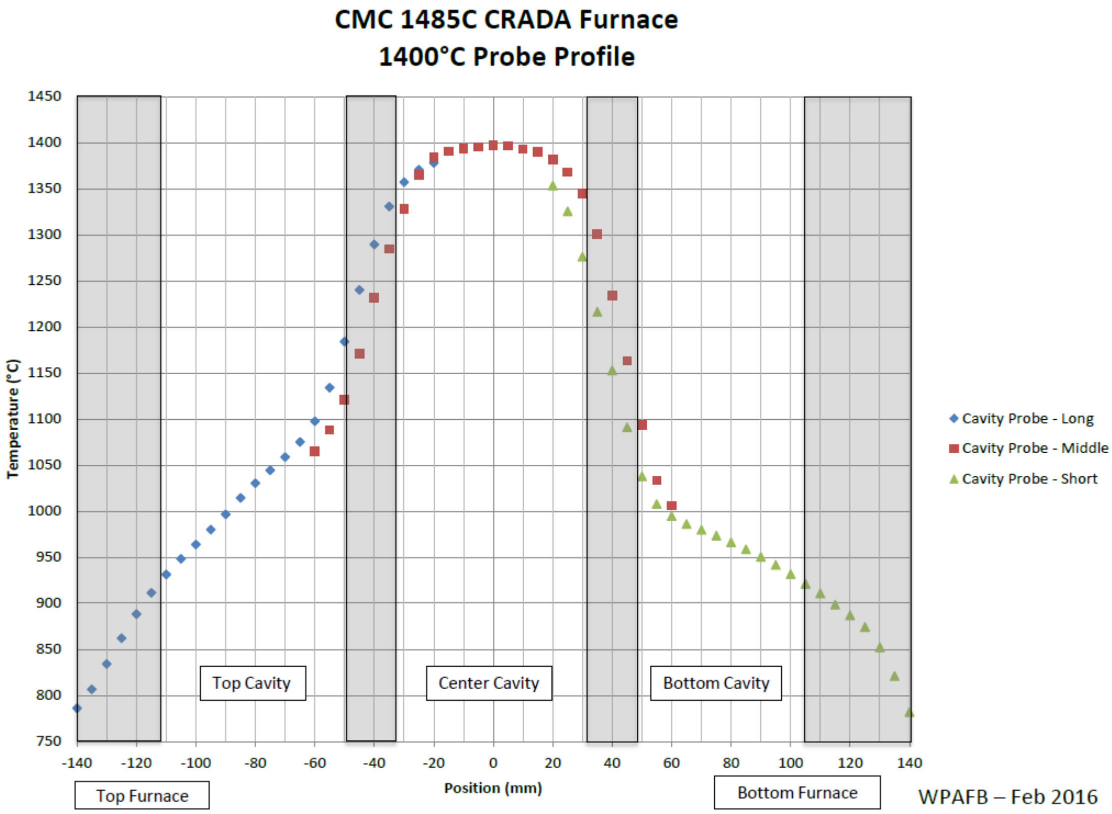

Figure 42. Plot of Thermal Profile of Mellen Three Zone Furnace at 1400°C

6.4 Heating Rates For The Mellen/MTS Three Zone Furnace

When using furnaces to heat specimens, it is typical to use the fastest possible heating rate available. This can improve test throughput and also limit environmental degradation (which may or may not be relevant to the test). It is critical to note that using very fast heating rates can result in the specimen temperature overshooting during heat-up.

Different heating elements contribute to overshoot as they have differences in the radiative heat transfer contribution. It was observed that the outer zones respond slower. Therefore, the center zone temperature programing was optimized to eliminate overshoot at the critical test temperature.

The results of two different heating rate tests are presented in Figure 43. The slower heating rate produced no temperature spikes in the test specimen. However, the faster heating rate resulted in a significant temperature spike at 800°C and a smaller over-temperature spike at the target of 1000°C. Therefore, care must be taken in what ramp rates and ramp rate profiles one uses to reach maximum temperature. Many additional heating rate runs were conducted to evaluate the furnace using various ramp rates. When the furnace was delivered, MTS proposed a ramp rate that would take approximately 52 minutes to reach a temperature of 1400°C. After several trials, it was found that a temperature of 1400°C could be achieved in only 25 minutes using a modified profile, as is shown in Figure 44.

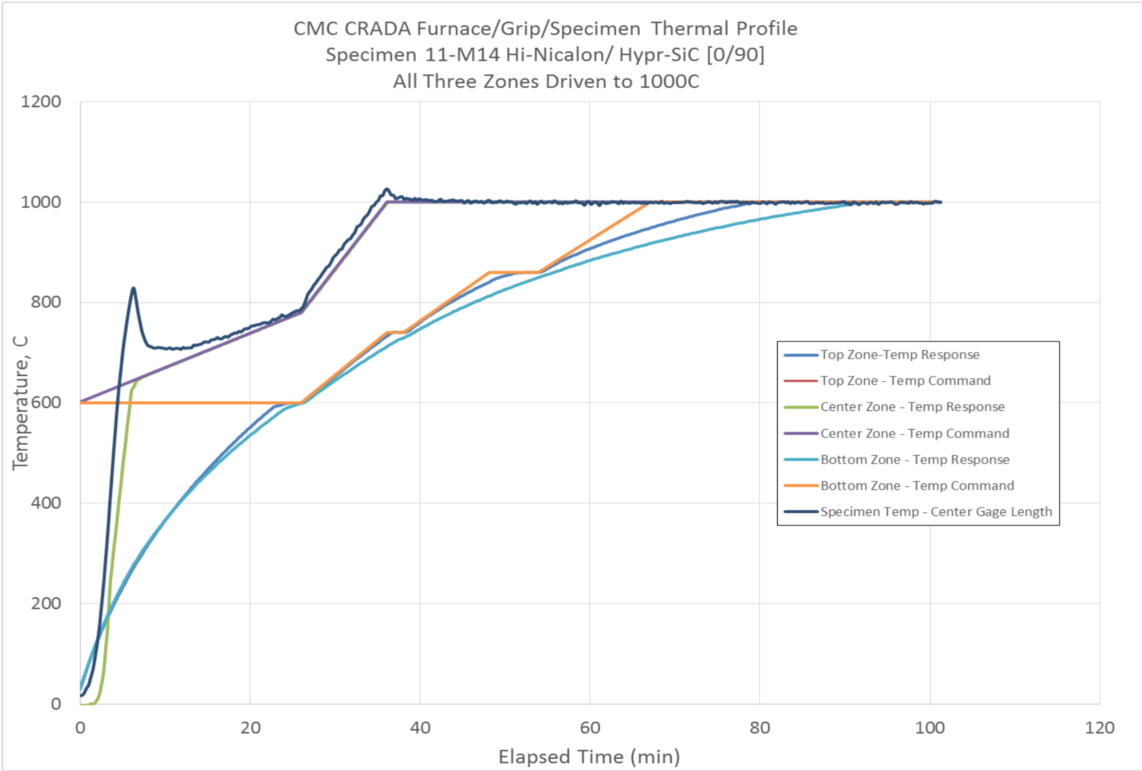

Figure 43. Furnace Temperature Versus Time for Two Different Heating Rates

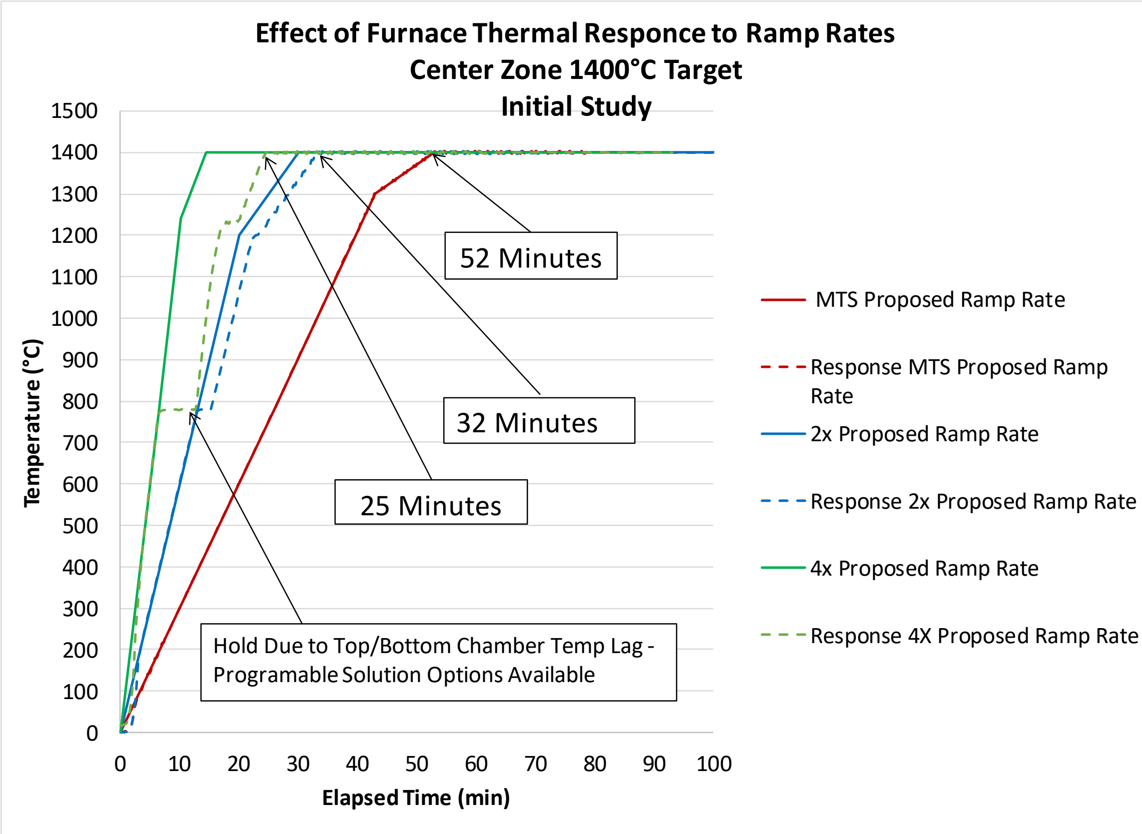

Figure 44. Times to Reach 1400°C Using Different Heating Profiles

6.5 Comparison of Mellen Single Zone Furnace To The Three Zone Furnace

The probe data from the Mellen single zone furnace was compared to the Mellen three zone furnace, and the results at 1200°C are presented in Figure 45 as a plot of temperature versus position along the length of the furnace. The results at 1400°C are presented in Figure 46. From both figures one can immediately observe that the three zone furnace provides two very important features. The first is that the temperature profile in the center 24 mm of the three zone furnace is much flatter and consistent than the single zone furnace. In addition, the temperature drops off much more gradually, especially past ±40 mm. For the three zone furnace the temperature remains above 1000°C out to ±80 mm. This should prove to be extremely beneficial for testing CMC materials that are susceptible to intermediate temperature embrittlement effects.

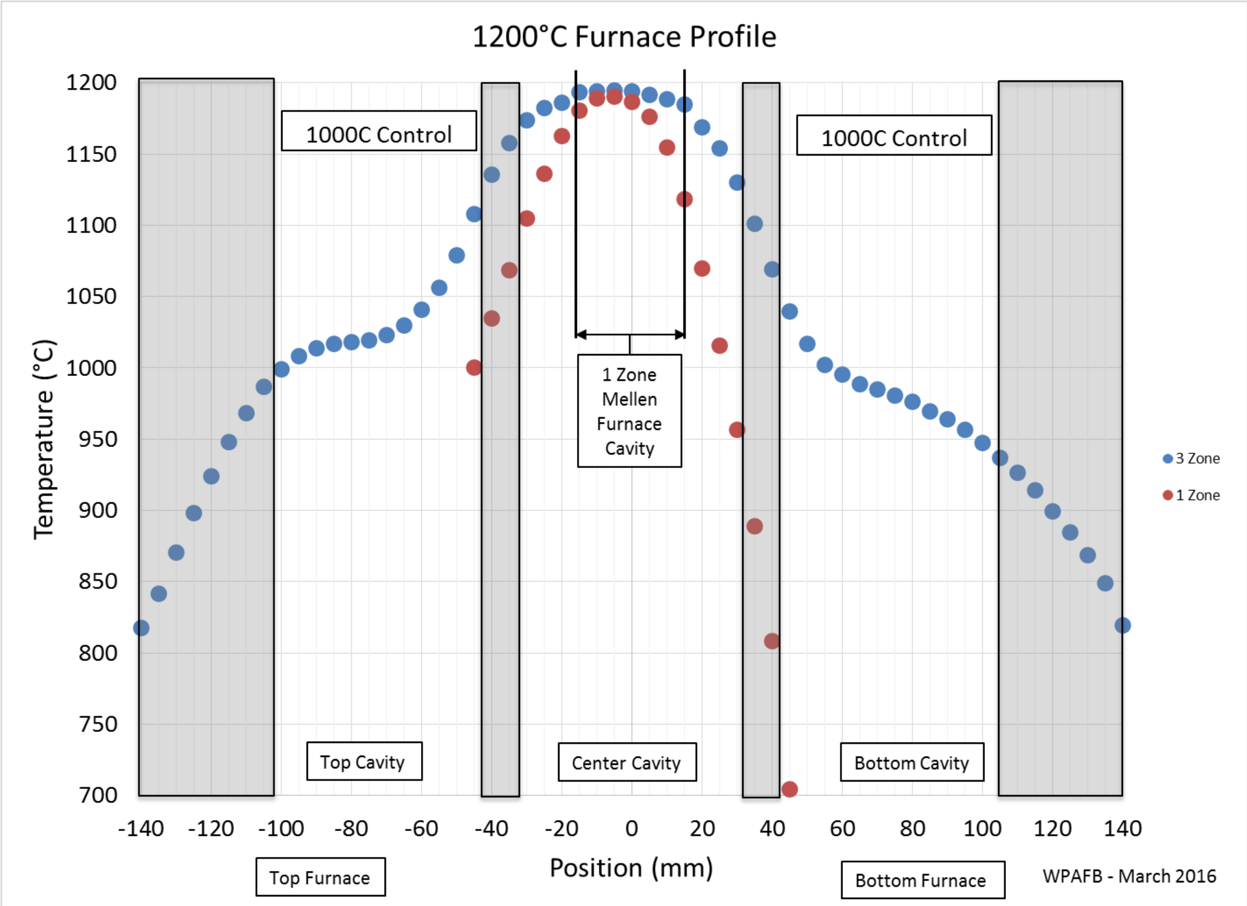

Figure 45. Comparison of Thermal Profile of Mellen Once Zone and Three Zone Furnaces at 1200°C

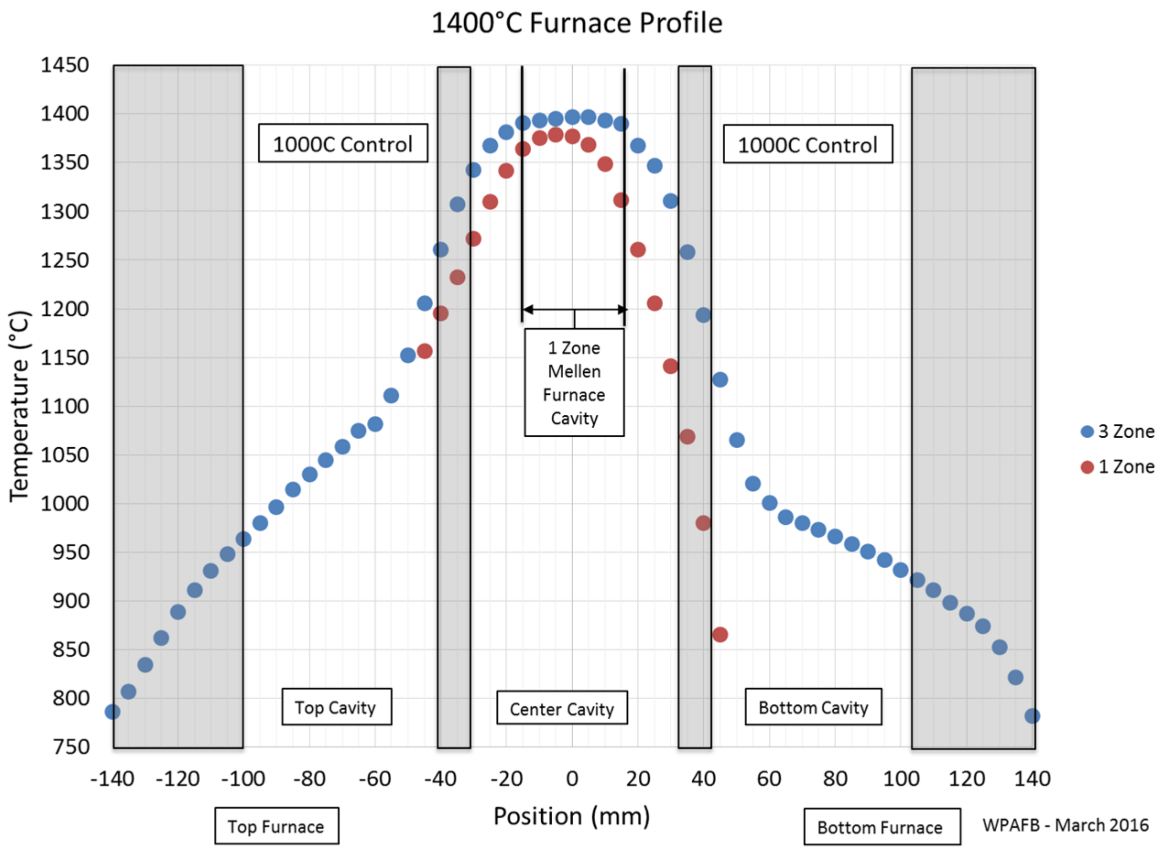

Figure 46. Comparison of Thermal Profile of Mellen Once Zone and Three Zone Furnaces at 1400°C

7. SUMMARY

The CMC Version 1 grips used inverted wedges that referenced the top grip face. This configuration had the distinct advantage of being relatively easy to manufacture. However, as gripping pressure was increased to achieve desired axial loads, the top retaining cap began to bulge. This movement caused the wedges to no longer have a uniform normal force on the specimen. The grips were only able to achieve a maximum axial load of about 8 kN no matter how much grip pressure applied. The most serious issue had to do with alignment. The grips could not achieve the alignment requirements recommended by ASTM and were determined to be unacceptable.

The CMC Version2 grips used wedges positioned similar to current MTS-647 wedge grips. The grips were able to achieve the desired axial load of 25 kN at room temperature. However the wedge body and cap that provided the wedge sliding surface was not rigid enough. There was too much movement of the wedges, allowing the wedges to exhibit non-planar movement. This movement results in a significant bending moment on the specimen just during gripping. There was also an issue with repeatability inserting and gripping the alignment specimen. The alignment changed every single time the alignment specimen was inserted into the grips. The bending strains induced just during gripping were so high that a bending as a function of applied axial strain test was never attempted. This revised grip set was also deemed to be unacceptable.

It is worth noting that MTS took the lessons learned from the V1 and V2 grip sets, and has designed a CMC Version 3 grip. MTS claims these grips minimize wedge non-planer movement and . This version has not been tested by AFRL to validate claims.

The two versions of the CMC grips that were evaluated could not be properly aligned. Therefore, no testing of CMCs was performed. Such testing still needs to be carried out to determine how durable the grips would be during long term creep rupture tests that last typically more than 100 hours.

A three zone furnace manufactured by Mellen was evaluated. It performed extremely well and improved on the thermal profile over their very successful single zone furnace. In addition, a temperature of 1400°C could be reached in as little as 25 minutes with no overshoot. This furnace worked very well with the MTS hot grip design approach.

8. REFERENCES

[1] MTS Systems Corporation, "Model 680.01 High_Temperature Grips," [Online]. Available: http://ww.w.mts-systems.de/cs/groups/public/documents/library/mts_004881~1.pdf. [Accessed 10 April 2019].

[2] ASTM E1012-14, “Standard Practice for Verification of Testing Frame and Specimen Alignment Under Tensile and Compressive Axial Force Application,” ASTM, 100 Barr Harbor Drive, West Conshohocken, PA, USA, July 21, 2014

LIST OF SYMBOLS, ABBREVIATIONS, AND ACRONYMS

AFRL	Air Force Research Laboratory
ASTM	American Society for Testing and Materials
CMC	Ceramic Matrix Composite
DIC	Digital Image Correlation
ITE	Intermediate Temperature Embrittlement
MTS	MTS Systems Corporation
PMC	Polymer Matrix Composite
R	Stress Ratio, $R = \sigma_{min}/\sigma_{max}$
RXCC	Composites Branch, Structural Materials Division, Materials and Manufacturing Directorate
TC	Thermocouple
WPAFB	Wright-Patterson Air Force Base