AFRL-RX-WP-TR-2014-0168

CONFIGURATION AND CALIBRATION OF HIGH TEMPERATURE FURNACES FOR TESTING CERAMIC MATRIX COMPOSITES

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AFRL/RXCC

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OCTOBER 2014
Interim Report

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REPORT DOCUMENTATION PAGE

Form Approved
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1. REPORT DATE *(DD-MM-YY)*
October 2014

2. REPORT TYPE
Interim

3. DATES COVERED *(From - To)*
21 January 2010 – 2 September 2014

4. TITLE AND SUBTITLE
CONFIGURATION AND CALIBRATION OF HIGH TEMPERATURE FURNACES FOR TESTING CERAMIC MATRIX COMPOSITES

5a. CONTRACT NUMBER
In-house

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
62102F

6. AUTHOR(S)
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5d. PROJECT NUMBER
4347

5e. TASK NUMBER

5f. WORK UNIT NUMBER
X0S7

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

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Wright-Patterson AFB OH 45433

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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Air Force Research Laboratory
Materials and Manufacturing Directorate
Wright-Patterson Air Force Base, OH 45433-7750
Air Force Materiel Command
United States Air Force

10. SPONSORING/MONITORING AGENCY ACRONYM(S)
AFRL/RXCCP

11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S)
AFRL-RX-WP-TR-2014-0168

12. DISTRIBUTION/AVAILABILITY STATEMENT
Distribution Statement A. Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES
PA Case Number: 88ABW-2014-4465; Clearance Date: 22 Sep 2014. This document contains color.

14. ABSTRACT (*Maximum 200 words*)
A dedicated experimental test program aimed to characterize the material behavior of seven different ceramic matrix composite (CMC) materials at room and elevated temperature for thermal protection systems is described. This report describes how to configure a furnace, set up a thermal profile, and conduct a thermal profile for testing CMCs at elevated temperatures up to 1300°C.

15. SUBJECT TERMS
ceramic matrix composites, thermal protection systems, temperature profile, thermal calibration specimen, thermocouples, thermal probes, high temperature furnaces

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT:	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON (Monitor) / 19b. TELEPHONE NUMBER *(Include Area Code)*
Unclassified	Unclassified	Unclassified	SAR	51	Larry P. Zawada / 937-255-1352

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FOREWORD

The work described in this report was performed by the Composite Performance Research Team of the Composites Branch, Structural Materials Division of the Materials & Manufacturing Directorate, Air Force Research Laboratory (AFRL/RXCC). Larry P. Zawada was the principal investigator and program manager. This report describes the objective of a research effort to evaluate the durability of ceramic-matrix composites (CMCs) as thermal protection systems for hypersonic applications. The in-house testing was performed under contract with the University of Dayton Research Institute (UDRI). Philip Blosser was the lead engineer with UDRI and responsible for all in-house experiments.

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EXECUTIVE SUMMARY

The U.S. Air Force (USAF) has requirements for efficient space access and hypersonic flight. Applications span the spectrum from expendable missiles to reusable access-to-space vehicles. At hypersonic velocities above approximately Mach 6, innovative high-temperature materials such as ceramic-matrix composites (CMCs) will be required to replace conventional metallic thermal protection systems (TPS) materials in order to protect the internal systems from the extreme thermal environment.

Air Force (AF) researchers (AFRL/RXCC) conducted a multi-year in-house program focused on evaluating the high-temperature properties and mechanical behavior of CMC materials targeted for structural TPS. The goal of this in-house research is the development of a high-temperature performance knowledge base that can be shared with the entire TPS community. Establishment of this knowledge base will provide a better understanding of the behavior and life-limiting mechanisms of high-temperature CMCs. Such information is required for materials technology maturation, behavior modeling, and for weapon platform manufacturers to conduct trade studies involving TPS conceptual system designs.

The materials selected for initial study are made by COI Ceramics, Inc.[1] and HyperTherm HTC[2] (now Rolls-Royce High-Temperature Composites, Inc.). The materials include: three oxide-oxide systems made by sol-gel processing (N610/AS, N720/AS, and N720/A), two silicon-carbide (SiC)-based systems made by polymer infiltration and pyrolysis (PIP) processing (S200H or SiC/SiNC and S400 or C/SiC), and two SiC-based systems made by chemical vapor infiltration (CVI; SiC/HYPR-SiC™ and C/HYPR-SiC™). These CMC systems represent a range of processing types, temperature capabilities, price points, and maturity levels. Other CMC systems will be evaluated in later phases of this research.

The test matrix for the program was developed with the main focus on the needs for short-term hypersonic strike applications. However, the durability tests were also extended in terms of test time to evaluate the CMCs for reusable TPS for high-speed platforms. Characterization and mechanical behavior testing was broken down into several main test matrix elements: baseline properties, durability behavior, and physical properties. The combination of these tests and microstructural studies will provide weapon system manufacturers with the information required to conduct conceptual design trade studies for a wide range of CMC material systems.

Seven different CMC systems were selected for the initial evaluation following a thorough review of the CMC industry. The material systems selected for study were purchased and delivered as finished test specimens that were designed by the researchers at AFRL. Test specimen geometry was carefully selected with the testing methods and expected material behavior in mind. All seven CMC systems purchased met a critical strength criterion that was verified using witness test specimens. Use of witness coupons was determined to be critical for this evaluation project and is recommended for all CMC material evaluation projects. It is also emphasized that it is important to document the pedigree of each CMC system capturing as many processing details about the

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[1] COI Ceramics, Inc. is an ATK Space affiliate, 9617 Distribution Avenue, San Diego, CA 92121
[2] Hyper-Therm High-Temperature Composites, Inc, now Rolls-Royce High-Temperature Composites, is a wholly-owned subsidiary of Rolls-Royce North America, 18411 Gothard St., Units B&C, Huntington Beach CA, 92648.

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CMC as the manufacturer will release. Such details are extremely critical for being able to reproduce the CMC system at a later time or for comparing different sets of test data.

This report focuses on the work performed to develop test techniques for testing ceramic-matrix composites at elevated temperatures. Extensive detail is presented on how to configure two types of furnaces for testing CMCs at temperatures up to 1300°C for this study. Once the furnaces were configured correctly for long-duration tests, several detailed thermal maps were performed. The results of these thermal profiles studies, along with the procedure developed, are presented in great detail.

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1.0 INTRODUCTION

The Air Force Research Laboratory (AFRL) is leading critical science and technology efforts to develop and mature robust, comprehensive technology options for High-Speed Strike and Intelligence, Surveillance, and Reconnaissance (ISR)/Strike capabilities. The objective is to develop a suite of materials and processing (M&P) that, when combined with advanced designs, will enable future United States Air Force (USAF) hypersonic systems. Therefore, AFRL has extensive activities focused on establishing a comprehensive M&P knowledge base. Such an understanding is required in order to quantify risk associated with structure and thermal protection systems (TPS) for next-generation hypersonic platforms. A segment of this M&P task has involved an in-house research program focused on evaluating the high-temperature durability of seven ceramic-matrix composite (CMC) materials targeted for structural TPS. Establishment of this knowledge base will provide a better understanding of the behavior and life-limiting mechanisms of high-temperature CMCs in order to promote technical maturation of materials technology and provide input for both behavior modeling and conceptual design studies. A series of individual reports document research performed under this study. This report focuses how to conduct testing of CMCs at elevated temperatures and specifically addresses furnace type, operation, and thermal profiling.

1.1 Background

The USAF requires advanced capabilities for efficient space access and hypersonic flight. The notional diagram in Figure 1 illustrates the high-level goals for future hypersonic systems for the USAF. Applications range from expendable missiles to reusable access-to-space vehicles with hypersonic velocities ranging from Mach 6 to Mach 20. At these speeds, the aero-heating produces surface temperatures that are too high for conventional metal alloys. Advanced ceramic and high-temperature material systems will be required for leading edges, propulsion flow path, and as large-acreage panels to protect internal systems from extreme thermal environments during hypersonic flight [1-4]. AFRL has identified TPS as a prime example where CMC materials are a key enabling technology.

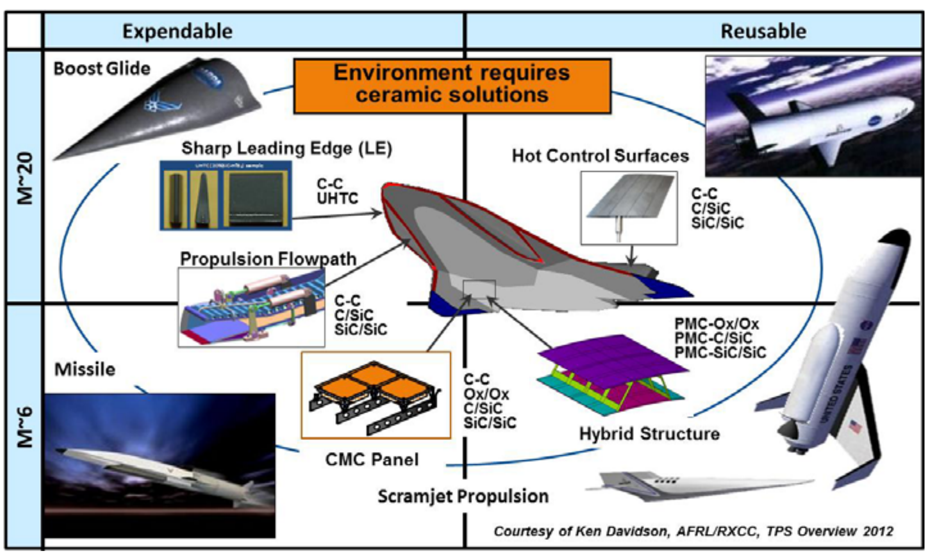

Figure 1. *Notional hypersonic vehicle platforms and material requirements*

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There are a wide range of CMC systems, including C/SiC, SiC/SiC, and oxide/oxides. Each CMC system exhibits unique properties such as specific strength, durability, and affordability. CMCs can operate at very high temperatures and are one-third the density of metal alloys, making them the primary focus on Air Force-led TPS materials technology development and maturation programs. A near-term hypersonic application is the high-speed strike weapon (HSSW) missile concept, which is an expendable platform with both high-speed and long-range cruise capabilities. The intent of such a platform would be to quickly reach any point on the surface of the earth. For example, at a speed of Mach 10, it would take approximately 100 minutes to travel halfway around the earth. At these speeds and times, the surface temperatures exceed the capability of metallic TPS. Reusable platforms are further out and require TPS materials that can survive multiple missions. As such, researchers at AFRL/RXCC are conducting an evaluation program on high-temperature CMC material technologies for structural TPS with primary focus on needs for expendable hypersonic platforms and secondary focus on limited-life reusable TPS applications.

A major barrier to insertion or use of CMCs in hypersonic applications is the lack of available design data on the current state-of-the-art CMC materials. The major issues involve the evolving nature of the material processing, the expense of materials for testing, the challenges with performing relevant elevated-temperature testing, and the proprietary nature of the information. Therefore, it is the goal of this in-house AF program to address these issues. Only the most advanced CMC material systems with pedigree information were selected for study. The intent is to make this highly specialized data available to the CMC and hypersonics community. Industry can then proceed to make informed CMC material selections based on their needs and also conduct conceptual design studies.

In contrast to traditional metal alloys, the pedigree of the most current CMC materials has continued to change over time, and this pedigree is not generally documented sufficiently in the literature with the property database. Even the most mature CMCs systems continue to evolve year after year as performance, cost efficiency, and scale-up are explored and implemented. Currently, there is no established certification process for CMCs, so none of the manufacturing processes have been locked down. Although much research has been published on the behavior of CMC materials, it is rare that the exact pedigree is known and documented. In many cases, the existing technical data are for CMC systems that can no longer be manufactured because of disappearing vendors and evolving processing technologies. For the past 30 years, researchers at AFRL have been collaborating on and leading maturation efforts both in-house and with external programs with CMC manufacturers. Therefore, AFRL has a significant knowledge base of the constituent materials, their manufacturing practices, evolution of the different classes of CMCs, and how changes to the CMC systems over time have impacted behavior.

CMCs are only produced in small batches and require complex processing techniques. These two issues limit the availability of CMC materials to the technical community, not only universities and small businesses, but large corporations as well. CMC materials are generally expensive to procure for study because of limited production capability. It is cost-prohibitive for the AF to fund each weapon systems manufacturer to procure and generate databases on multiple CMC systems. The costs of CMC materials is high, and the costs for characterization are relatively high as well. Elevated-temperature testing is challenging and few facilities are equipped with the hardware and expertise to produce high-quality material behavior and design data. AFRL can

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maximize the investment in evaluating these materials in-house by leveraging their knowledge base of the materials and their state-of-the-art testing capabilities. In addition, the AF is not restricted from sharing all of the information from this program with both CMC manufacturers and the TPS community. This is a more efficient use of research funds and allows for each weapon system manufacturer to have access to current state-of-the-art test data on multiple CMC systems for use in their TPS conceptual design trade studies.

The significant challenge with design data being available to the hypersonics community is that test data are now considered company-proprietary information. There are several threads to this – there is the competitive nature of the CMC community, the aerospace turbine engine manufacturer's needs for CMCs, and the competition between the various weapon system manufacturers. The proprietary nature of material property data information has escalated due to the competitive nature of manufacturing and the recent competing need for CMCs in turbine engine propulsion applications. These two elements in the CMC market have created an environment where large turbine engine manufacturers have purchased many of the primary manufacturers. This makes those companies vertically integrated, but further promotes exclusivity to information and has severely limited access to CMC materials and their performance and behavior data. The other aspect is the competing airframe companies. It is not expected that one company would invest in the material and the required characterization to gain the advantage and then share their intellectual property.

1.2 Program Objectives

The objective of the in-house portion of the TPS M&P program is to establish baseline high-temperature mechanical behavior and thermal properties for several state-of-the-art CMC materials that are relevant for TPS applications. The intent of this investigation is not the generation of a design database; instead, the main thrust is the establishment of high-temperature performance envelopes for several CMC systems with respect to the requirements for expendable and multi-use TPS platforms. The technical goal was to be able to identify both strengths and weaknesses of the various CMC systems and provide a ranking of performance. An understanding of the material performance will be presented along with the necessary mechanical behavior characterization. Such information will serve three important purposes. First, performance data will allow the CMC manufacturers to baseline their material and determine how their systems perform with respect to a wide range of CMCs. Second, such information will allow the weapon system design community to effectively and more accurately interrogate multiple CMC systems for their specific applications. Third, this performance data will allow for the initial development of a basic lifing methodology and allow for conceptual design studies without the need for each company to generate their own expensive CMC data sets.

The specific technical objectives are as follows:

1) Validation of mechanical and physical properties for the most current pedigreed CMC materials at room and elevated temperatures to address model input requirements and to provide a baseline for durability assessment and damage progression.
    a. Measure and document the conductivity and thermal expansion.
    b. Establish stress-strain behavior as a function of projected CMC use temperature.
    c. Measure and document off-axis tensile properties (shear) and matrix-dominated properties (transthickness tension).

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2) Assess the durability limits of the most relevant TPS CMC materials under extreme loading and temperature conditions, by:
    a. Studying the time-dependent deformation behavior (under sustained load).
    b. Studying cyclic deformation behavior and coupled creep-fatigue interactions using both fatigue and dwell fatigue loading conditions.
    c. Studying the relationship between time, temperature, stress, and environment.
    d. Establishing sensitivity to environmental effects and environmentally assisted damage progression.
    e. Establishing temperature limits on constituent stress-strain behavior.
3) Qualification and characterization of key material properties and microstructure features for model input and trade studies of various conceptual TPS designs by weapon system manufacturers.
4) Providing materials science observations and insight into the relationship between various CMC constituents and mechanical behavior performance.

1.3 Materials Description

Seven different CMC material systems were selected for this initial evaluation following a thorough review of the industry. These CMC systems represent a range of processing types, temperature capabilities, price points, and maturity levels. Table 1 provides details for each CMC system selected for this study.

Table 1. Listing of the Seven TPS Materials and Their Descriptors

Trade Name	Company	Processing	Fiber	Fiber Coating	Matrix	Filler	Exterior Coating	# Plies	Weave
N610 / AS	COIC, Inc.	Sol-gel Prepreg	Nextel™610 99% Al2O3	none	Aluminosilicate	Alumina	none	14	8HSW
N720 / A	COIC, Inc.	Sol-gel Prepreg	Nextel™720 85% Al2O3 + 15% SiO2	none	Alumina	Alumina	none	12	8HSW
N720 / AS	COIC, Inc.	Sol-gel Prepreg	Nextel™720 85% Al2O3 + 15% SiO2	none	Aluminosilicate	Alumina	none	12	8HSW
S200H (SiC/SiNC)	COIC, Inc.	PIP	Hi-Nicalon™ Si-C Low O Fiber	BN +Si3N4	SiNC	Si3N4	none	8	8HSW
S400 (C/SiC)	COIC, Inc.	PIP	IM7 PAN based C	BN +Si3N4	SiC + oxidation inhibitors	SiC	SiC	8	5HSW
HYPER-SiC (C/SiC)	Hyper-Therm	CVI	T300-1K C	Pyrolitic Carbon	SiC	B4C	HYPER-Coat 1300™ glaze SiC	24	Plain
HYPER-SiC (SiC/SiC)	Hyper-Therm	CVI	Hi-Nicalon™ Si-C Low O Fiber	Pyrolitic Carbon	SiC	B4C	none	9	Plain

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COI Ceramics, Inc. (COIC) manufactured the three oxide-ceramic-matrix composite materials, N610/AS, N720/AS and N720/A, as well as the SiC-based S200H and S400 SiC materials. The oxide CMC materials are manufactured via sol-gel technology, are based on alumina and alumino-silicate matrix chemistry, and are reinforced with fiber that is commercially available from 3M[3] including Nextel™610 (alumina) and Nextel™720 (mullite) fibers. The S200H and S400 materials were manufactured using a polymer infiltration and pyrolysis (PIP) process. S200H is the trade name for COIC's material that is an amorphous silicon nitrocarbide (SiNC) matrix reinforced with Hi-Nicalon™ (low oxygen-containing silicon carbide) fibers. S400 is the trade name for Hexcel IM7 carbon-reinforced silicon carbide.

The other two materials studied on the TPS Program were made by Hyper-Therm HTC. The materials of interest were densified using CVI and are classified in their "HYPR-SiC™" product line. The HYPR-SiC materials have an oxidation-inhibited SiC matrix that is produced by discrete matrix layering of the primary matrix constituent phases with secondary inhibitor phases during the CVI process. One of the HYPR-SiC materials has Hi-Nicalon silicon-carbide fiber and the other has T300-1K fiber. Both have the layered CVI matrix that includes SiC and B_4C.

Both cross-ply (0/90) and bias-ply (±45) material architectures were chosen in order to study both fiber- and matrix-dominated behavior.

Details on the material selection process for this Program and more in-depth descriptions of each material studied are documented in AFRL-RX-WP-TR-2013-0212, "Durability Evaluation of Ceramic Matrix Composites (CMCs) for Thermal Protection System (TPS) Application: Description of Test Program" [1].

1.4 Test Plan

The test matrix and test specimen requirements were developed by the engineers of AFRL/RXCC, using extensive input from the TPS community. Primary focus was on the needs for short-term hypersonic strike applications and secondary focus was on the needs for reusable TPS. A summary of the finalized test matrix is given in Table 2. The test matrix was repeated for all seven of the materials selected. All tests were performed on the cross-ply ([0/90]) material, while and bias-ply ([±45]) material were only tested in tension. A more detailed discussion of the test matrix is documented in AFRL-RX-WP-TR-2013-0212.

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[3] 3M Products, 3M Corporate Headquarters, 3M Center, St. Paul, MN 55144-1000

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Table 2. Test Matrix for the In-House Testing Effort on the TPS Program

Test Type	Specimen Geometry	Material Layup	Stress Parameter	Temperature			Spares
				RT	1200 C	1300 C	
Baseline Properties							
Tensile	7" AFRL DB	[0/90]s	Failure	3	3	3	3
ILT	1" Disk	[0/90]s	Failure	10			2
In-Plane Shear	7" AFRL DB	[± 45]s	Failure	3	3		2
Durability Behavior							
Creep Rupture	7" AFRL DB	[0/90]s	6-8 stress levels		6		2
Dwell Fatigue	7" AFRL DB	[0/90]s	6-8 stress levels		6		2
Fatigue	7" AFRL DB	[0/90]s	6-8 stress levels		6		2
Thermal Properties							
Thermal Diffusivity	0.5" x 0.5"	[0/90]s & [±45]s	na	1	1		2
Thermal Expansion	2" x 0.25"	[0/90]s & [±45]s	na	1	1		2

1.4.1 *Baseline Properties*

Baseline property testing using monotonic loading involved both in-plane and through-thickness loading to study fiber- and matrix-dominated behavior and properties. In-plane tensile tests generate the basic properties such as modulus (E), ultimate tensile strength (UTS), proportional limit (PL), strain at failure (εf), as well as stress-strain behavior. Each material was tested at room temperature, at the maximum use temperature for the CMC system (as determined by the CMC manufacturer), and then at 100°C above the maximum use temperature.

The interlaminar tension (ILT) test applies a tensile load through the thickness of the composite, testing the tensile strength of the matrix and the fiber-matrix bonds.

Bias-ply (±45) material architectures were tested in order to investigate matrix-dominated properties. The in-plane shear tests provide qualitative information regarding the toughness of the CMC matrix material and are a practical technique for determining how the matrix material performs at elevated temperature.

1.4.2 *Durability Behavior*

The tests described under the Durability Behavior portion of the test matrix are designed to test a materials' ability to endure conditions of relevant temperature, environment, stress, and time. Testing regimes included Creep, Dwell Fatigue, and Fatigue. Tests were run at the maximum use temperature and at several stress levels. Any specimens that reached 20 hours of thermo-mechanical exposure were cooled to room temperature and then tension tested to measure retained strength. These retained strength tests allow for a measure of degradation that has occurred to the CMC.

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1.4.3 *Thermal Properties*

Thermal/physical properties were also measured to aid in the design of TPS structures. For each CMC system, thermal diffusivity, thermal conductivity, and thermal expansion were measured. Both cross-ply (0/90) and bias-ply (±45) material architectures were chosen in order to study the properties in-plane and off-axis to the warp fiber direction. Measurements were made in the through-thickness direction as well.

More details on the test matrix development for this research can be found in AFRL-RX-WP-TR-2013-0212, “Durability Evaluation of Ceramic Matrix Composites (CMCs) for Thermal Protection System (TPS) Application: Description of Test Program” [1].

1.5 Report Organization

This report is one in a series of reports that document research performed under the TPS M&P evaluation effort. Specifically, this report documents the configuration and calibration of two high-temperature furnaces that were designed specifically for testing CMCs at elevated temperatures. In this report, Section 2.0 outlines the details and considerations that must be given to the application of furnaces to high-temperature testing. Section 3.0 details equipment modifications, furnace and specimen configurations, as well as test methodologies. Section 4.0 identifies significant findings related to the effect of specimen material in defining furnace operating conditions. In addition, appendices have been included that addresses specimen thermal conductivity, procedures for avoiding temperature overshoot, and effects of furnace repair on temperature profiles.

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2.0 HIGH-TEMPERATURE FURNACE CONCEPTS

AFRL has been testing CMCs at elevated temperatures (1000-1200°C) for many years. With the introduction of new ceramic matrices, potential operating conditions have increased beyond 1200°C, with requirements now approaching 1500°C. The furnaces currently used at AFRL are manufactured by Amteco and use silicon-carbide (SiC) heating elements. Photographs of this furnace are shown in Figures 2 and 3. In this case, the furnace is mounted in a horizontal test frame, as mounting horizontally eliminates the "chimney effect" of heat rising inside. These furnaces are very compact, allowing for testing of 150 mm test specimens and are very robust. However, the SiC heating elements limit the maximum operating temperature to approximately 1300°C when used for long periods of time, even with the addition of extra exterior insulation, as shown in Figures 2 and 3.

NASA Glenn Research Center in Cleveland, Ohio collaborated with a furnace manufacturer (Mellon) to develop a capability to test CMCs at temperatures up to 1800°C using molydisilicide heating elements. This compact furnace, shown in Figures 4 and 5, was purchased by AFRL and placed into operation in a vertical test frame. This report summarizes lessons learned and knowledge gained during the process of installing and operating both types of furnaces for testing CMCs.

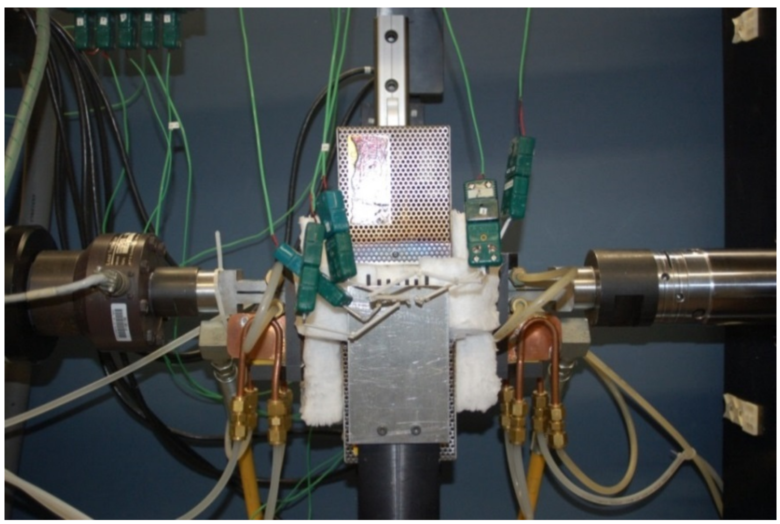

Figure 2. *Front view of Amteco furnace mounted in a horizontal position. Furnace is shown with extra insulation, water cooling for Enterpac grips, and additional thermocouples for temperature mapping.*

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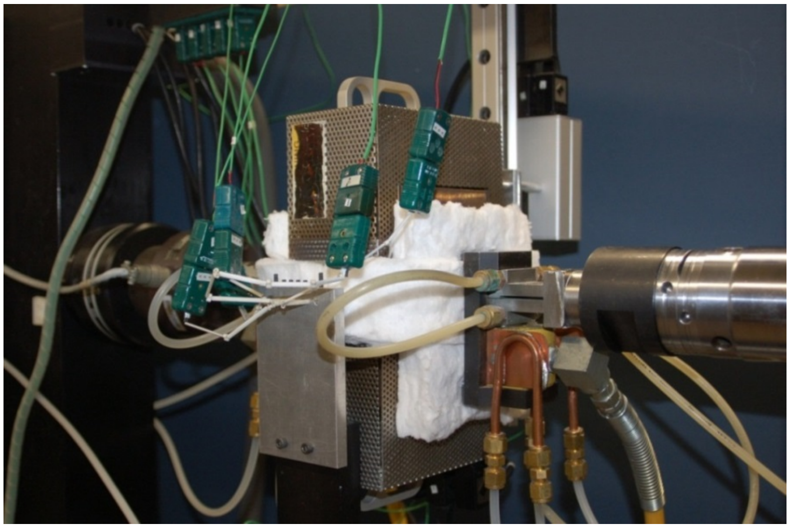

Figure 3. *Side view of Amteco furnace showing custom-built gripping system developed for flat CMC specimens*

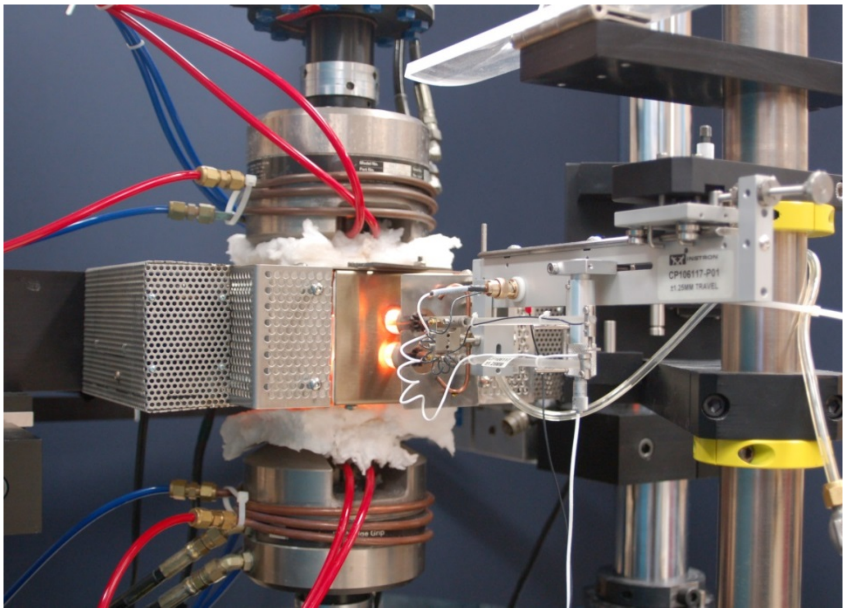

Figure 4. *Mellen furnace with MTS 647 wedge grips and Instron extensometer*

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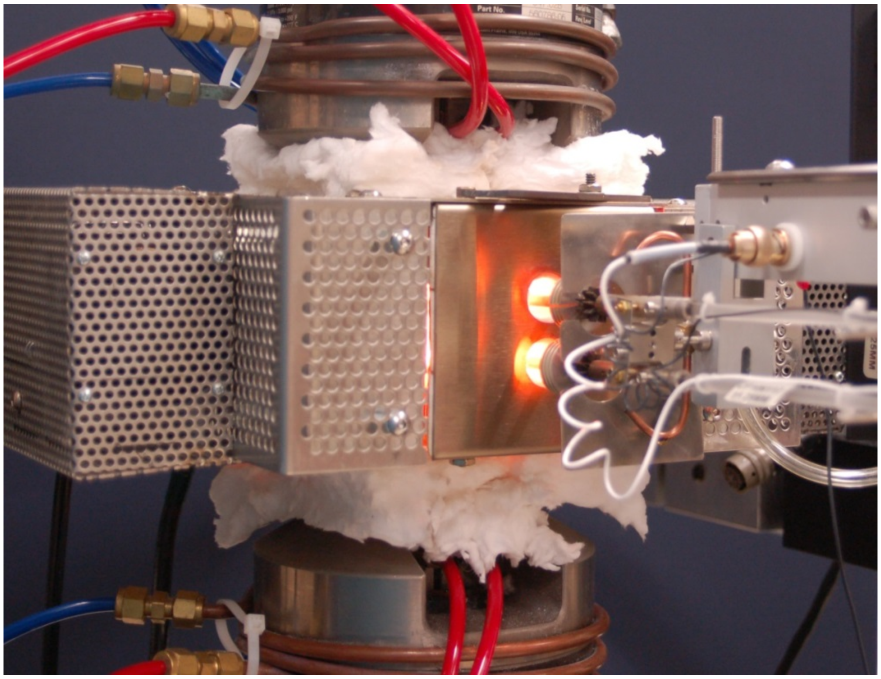

Figure 5. *Close-up of Mellen furnace showing added insulation, water cooling of grips and wedges*

A primary concern for testing advanced materials at high temperatures involves the thermal profile of the furnace hot-zone cavity. Specimen thermal profile is especially important as thermal gradients increase with increasing temperature, and also depends on if the furnace is mounted in a horizontal or vertical position. A secondary concern involves temperature gradients as the specimen transitions between the furnace hot zone and the gripped ends that are typically at room temperature.

AFRL has traditionally conducted furnace calibrations using an actual test specimen of similar manufacturing conditions and bonded beaded thermocouples (TCs) at strategic locations on the specimen. The specimen is then exposed to test temperatures to determine furnace parameters necessary to achieve desired test specimen temperature profiles. This process is time-consuming and expensive (test coupons, TC materials, and labor). Alternative methods, utilized by some test houses, involve a single thermocouple within a ceramic rod that is moved throughout the furnace. This method involves significantly less material and can be automated. Comparison studies were made using both methods, as well as studying the effect of using different materials as the test substrate for calibrating the furnaces.

The choice of material selected to mount the TCs was found to be critical to the furnace profiling process. This is most evident within the actual hot-zone cavity where conduction and convection, along with radiation, contribute to the total specimen temperature. Outside the hot-zone region, thermal results obtained from different methods converge since the measured temperature results only from heat that is conducted along the length of the thermal profile specimen.

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3.0 EQUIPMENT CONFIGURATION

Two Mellen furnaces were purchased by AFRL for the specific purpose of developing a high-temperature test capability up to a minimum of 1500°C. These furnaces were constructed using designs prototyped in partnership with NASA and consisted of the furnace housing unit, ceramic insulation, heating elements, and a Eurotherm 2408 temperature control unit. A photograph of the as-delivered furnace is shown in Figure 6. A mounting assembly was fabricated in-house and consisted of Thomson rods and pillow blocks for ease of movement. The original installation plan called for a rail system where the furnace could be slid into and out of the load train in attempt to always keep the furnace in the same position from test to test. A one-piece furnace concept was selected to achieve maximum thermal insulation by minimizing the amount of openings in the furnace. Thus, the design involved a single unit with openings only at the top/bottom for specimen insertion and two holes in the front for an extensometer to measure strain. Although this design minimized heat requirements, specimen installation was difficult and involved at least two persons, and often times, three. One person would slide the furnace into position while another held the specimen and a third operated the hydraulic grips.

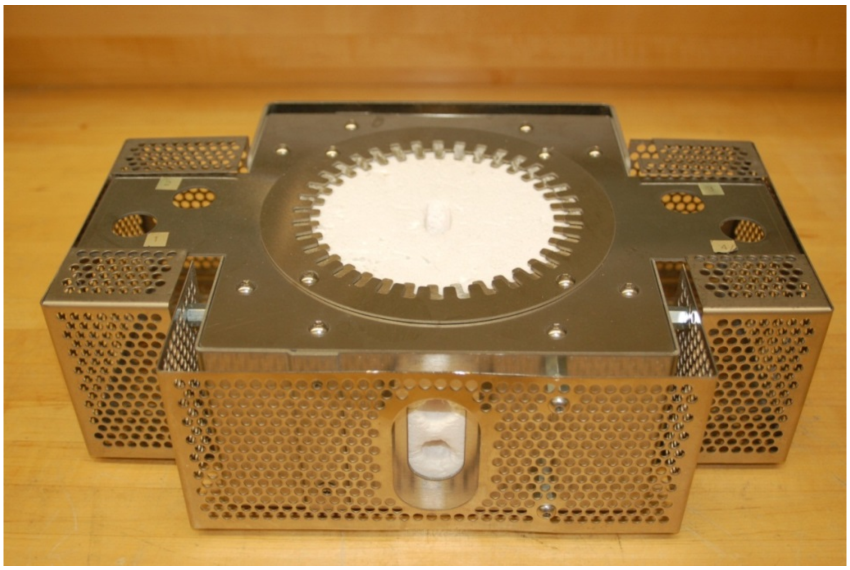

Figure 6. *Original-designed one-piece Mellen furnace*

3.1 Modifications to Mellen Furnace

The specimen positioning challenge made it evident that there was a need for front access to the furnace. In collaboration with Mellen and NASA, a "pie cutout" modification (Figure 7) allowing front access was proposed. The pie segment was taken from the front where the extensometer enters the furnace because the heating elements are on both sides and the TCs are fixed to the back. A thermal profile of the furnace was conducted both before and after this modification using a N720/AS CMC test specimen with three TCs bonded to it. A bar chart showing the results of the thermal profiles before and after the modifications is presented in Figure 8. The data clearly shows that no measurable differences were observed as a result of the modification. This modification allows for easy specimen access, requires only one person to mount specimens in the test frame, and is now used exclusively by Mellen when manufacturing this furnace. Initial use of the furnace to conduct temperature-mapping studies resulted in several additional modifications. The main change was the addition of an alumina element support to prevent

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deformation in the heating elements. A detailed schematic of the current furnace configuration is shown in Figure 9 and the additional changes are discussed in more detail in the following section.

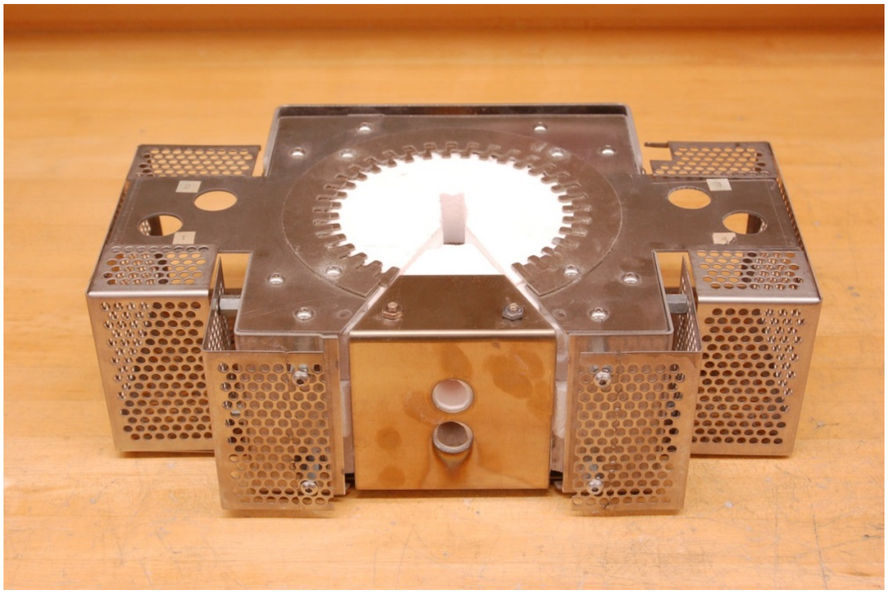

Figure 7. *Modified "pie-cutout", Mellen furnace*

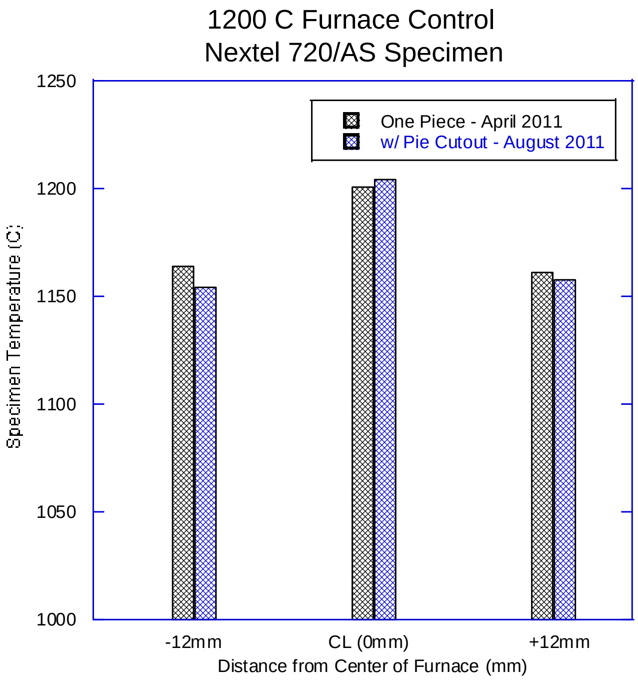

Figure 8. *Mellen furnace thermal profile before/after pie cut-out*

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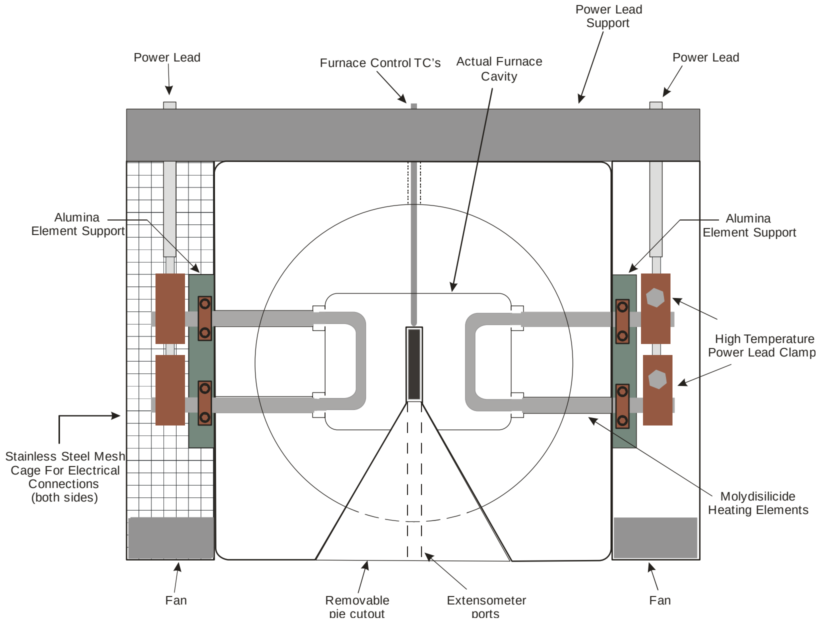

Figure 9. *Top-view illustration showing current modifications of Mellen furnace*

The furnace, as shipped, utilized a bare-bead B-type TC junction for measuring and controlling temperature. Two additional TC locations, approximately ¾-inch above and below the furnace center line (CL), were used to control upper temperature (top TC) and provide a secondary "witness" (or verification) temperature during testing. With use, the TC metal was observed to degrade and actually deposit on the specimen. Ceramic closed-end tubes (Omegatite 450 Protection Tubes) were installed to protect the TC bead from additional degradation. Although TC degradation was eliminated, this "encapsulation" of the TC resulted in a slightly slower response from the controller, creating thermal "overshoots" as the control temperature increased. Further studies showed that the radiant-heat effect had a greater impact on specimen thermal overshoot and is addressed in Appendix B. Current testing conditions at AFRL do not require a specific rate of rise to the test temperature (thus, allowing for time compensation required to prevent an "overshoot" condition), only that the test temperature be reached within approximately 30 minutes.

During continued use of the furnace for testing of CMCs, it was observed that the thermal profile gradient changed with time and became non-symmetrical, with the lower portion of the specimen cooler than previously measured during initial calibration. Inspection of the heating elements found that they had sagged considerably. It was suspected that this significant deformation was a result of the low creep resistance of the molydisilicide heating elements at such high temperatures, coupled with the weight of the power cables attached to them. An alumina element support was created (Figures 10 and 11) and the power cables were re-routed, as shown in Figure 10, to minimize their gravitational effects. The addition of this alumina element support and re-routing of the power feeds eliminated deformation of the heating elements. It was also observed that care must be taken when selecting the route for the power cabling. The high power loads running through the cables will generate electromagnetic interference (EMI). It was found that such high power loads were affecting the digital control (DC) signals, such as load and extensometer outputs. Selective coiling and physical separation of the two wires minimized these effects.

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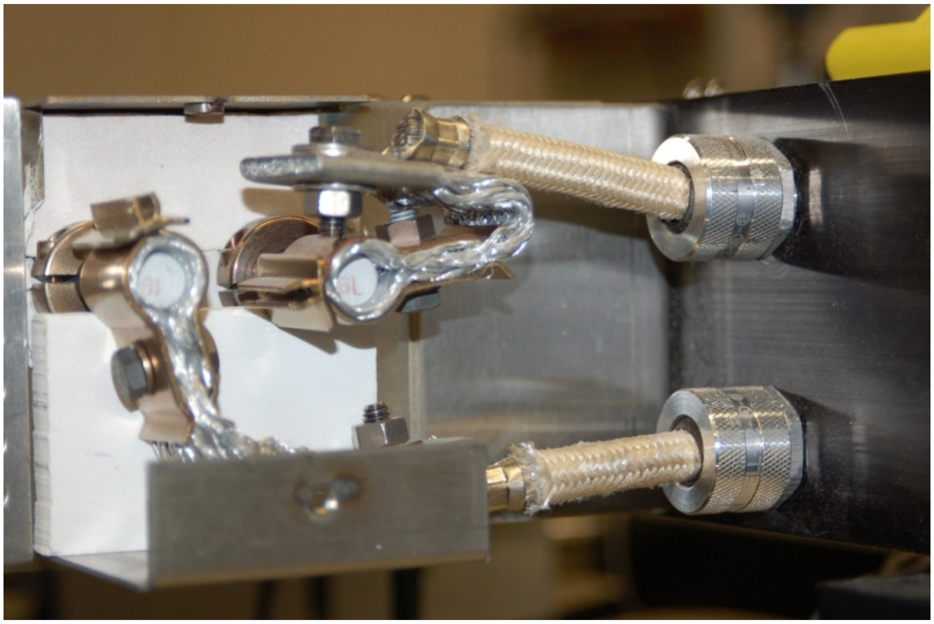

Figure 10. *Front view furnace element support with re-routed power feeds*

Figure 11. *End view of alumina block furnace element support*

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During a periodic maintenance inspection of the furnace, corrosion at the connections between the power leads and heating elements was discovered, as shown in Figure 12. In the figure, one can clearly see that the copper connector lug that attaches the power cable to the heating element strap has severely corroded, along with significant corrosion of the power cable. Inspection of the connectors revealed they were standard-grade copper lug. Several changes were made to eliminate this degradation. First, a small cooling fan was installed and the cooling cage was modified to keep the connectors cooler. The copper electrical leads and copper connector lugs were changed to high-temperature nickel-coated versions. These simple changes completely eliminated the corrosion problem. Dialogue with Mellon has resulted in them manufacturing furnaces with similar modifications. All of the changes to the Mellen furnace were in place before conducting the thermal profile studies presented in the following sections.

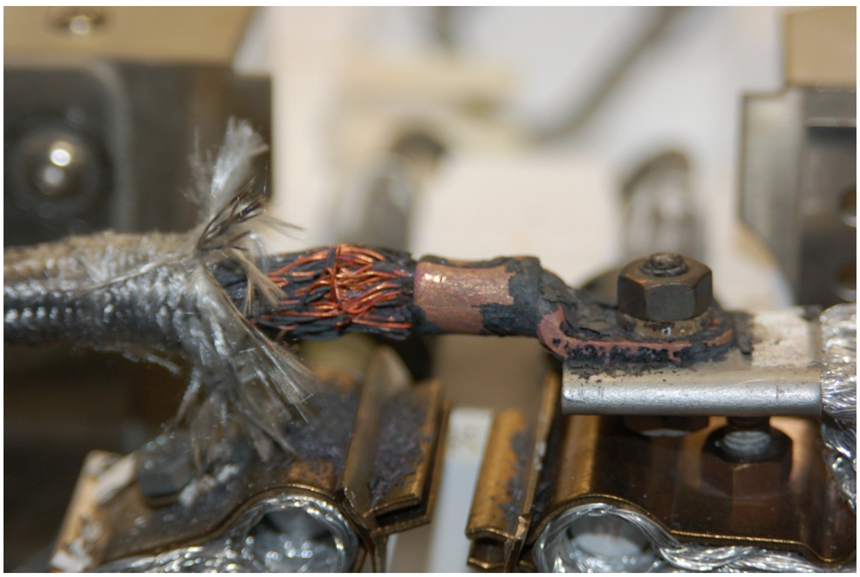

Figure 12. *Heating element corrosion caused by incorrect selection of connectors*

3.2 Configuration of Furnaces and Thermal Calibration Specimen

Furnace positioning is critical and should always be mounted in the load frame such that the center line of the furnace is aligned with the specimen center line. This ensures that the furnace control TC is centered with respect to specimen gage length. Changes to specimen length require the furnace to be repositioned. Currently, AFRL uses 178 mm-long specimens; however, previous testing has involved a 150 mm specimen length.

Minimization of heat loss during testing, as well as maintaining the integrity of hydraulic components in close proximity of the furnace, is of significant importance. Upon installation of the specimen and placement of the furnace, specimen openings are filled with loose insulation, as shown in Figure 13. Additional insulation material is placed over the furnace top and between the furnace and the hydraulic grips. External cooling is provided for the grip body, as well as utilizing the cooling provided by MTS for the 647.02 wedge grips. Ceramic materials have a low coefficient of thermal conductivity; therefore, cooling of the wedges has no significant impact on the specimen's thermal gradient within the gage section region. Recent studies involving thermal conductivity rates and furnace heat loss confirm these initial findings and are detailed in Appendix A.

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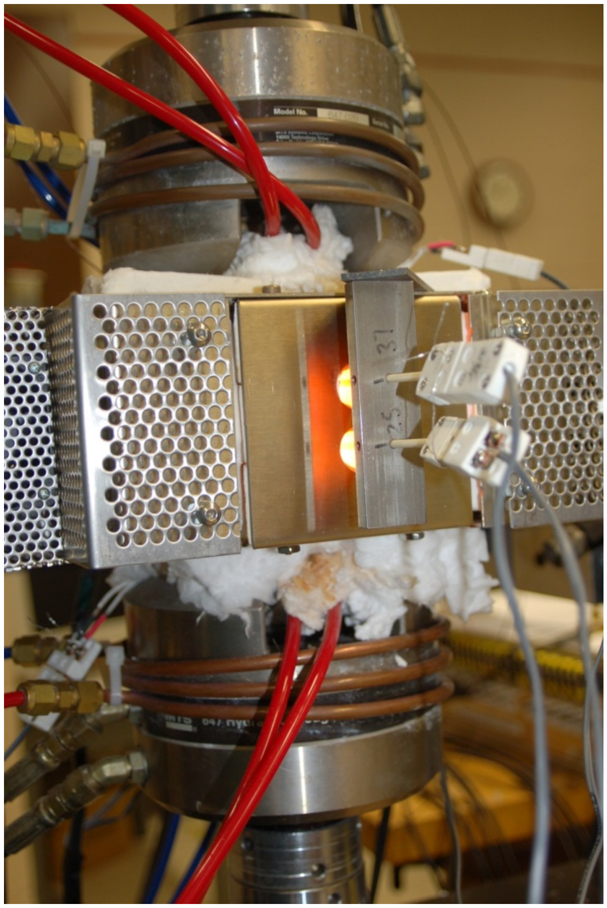

Figure 13. *Mellen furnace showing added loose-pack insulation to minimize heat loss*

Thermocouple placement (location and method) on the specimen used for thermal profiling is critical. CMC test specimens are not tested with TCs attached to them to avoid any chance of a reaction taking place, so 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. A photograph of a fully instrumented CMC thermal profile specimen is shown in Figure 14. 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 supplied furnace TCs encased in the alumina tubes (Figure 15). An actual 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 exact 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. Figure 16 is an optical photograph of the furnace with the pie cutout removed showing the thermal profile specimen installed in the furnace.

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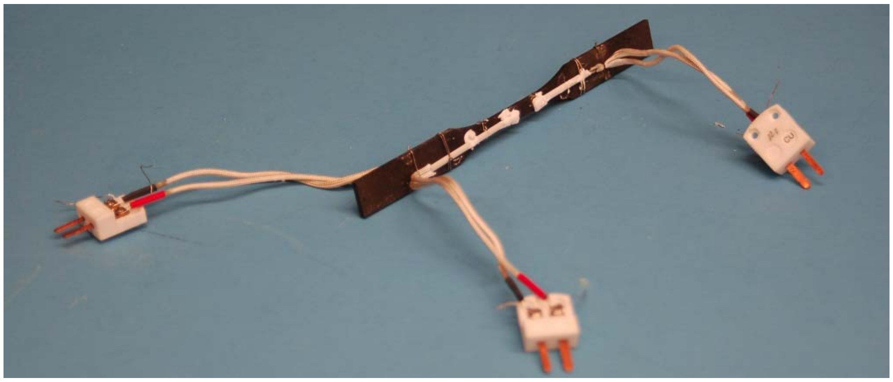

Figure 14. *Example of thermal profile specimen ready for installation in furnace*

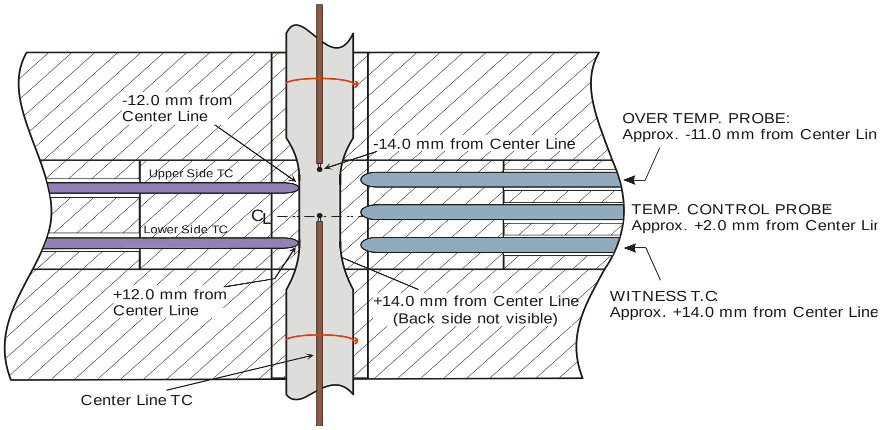

Figure 15. *Schematic illustration showing placement of specimen and furnace control TCs in Mellen furnace*

Figure 16. *Mellen furnace with thermal profile specimen installed*

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This process is similar to that used with AFRL's Amteco furnaces that are mounted horizontally. Figure 17 is a detailed schematic cross-section view of the Amteco furnace which comes with either 2, 4, or 6 heating elements. The AFRL unit shown in the figure utilized 6 heating elements, which were arranged in three heating zones. Each zone was controlled by a Barber-Colman 560 temperature controller. Not shown are the additional TCs that are bonded to the specimen to adequately profile the entire section of the thermal profile specimen inside the furnace. Figures 18 and 19 show the thermal profile specimen installed in the Amteco furnace and the five TCs bonded to the test specimen. The Amteco furnace can be operated with multiple zones that can be defined by the user. At AFRL, the furnace is now run with three zones of temperature control as shown in Figure 17. This is a relatively simple configuration requiring only three feedback signals, and has been found to produce good thermal profiles. Examples of thermal profiles will be provided in the following sections of this document. However, a lot more research would need to be done to fully optimize the number of zones required to produce the best thermal profile. In addition, being mounted horizontally results in a symmetric temperature gradient centered on the middle of the gage section of the test specimen. This is in contrast to furnaces mounted in the vertical position where the lower gage section of the test specimen will always be slightly cooler than the top portion of the gage section because of rising heat within the furnace, and will become evident in the results section of this report.

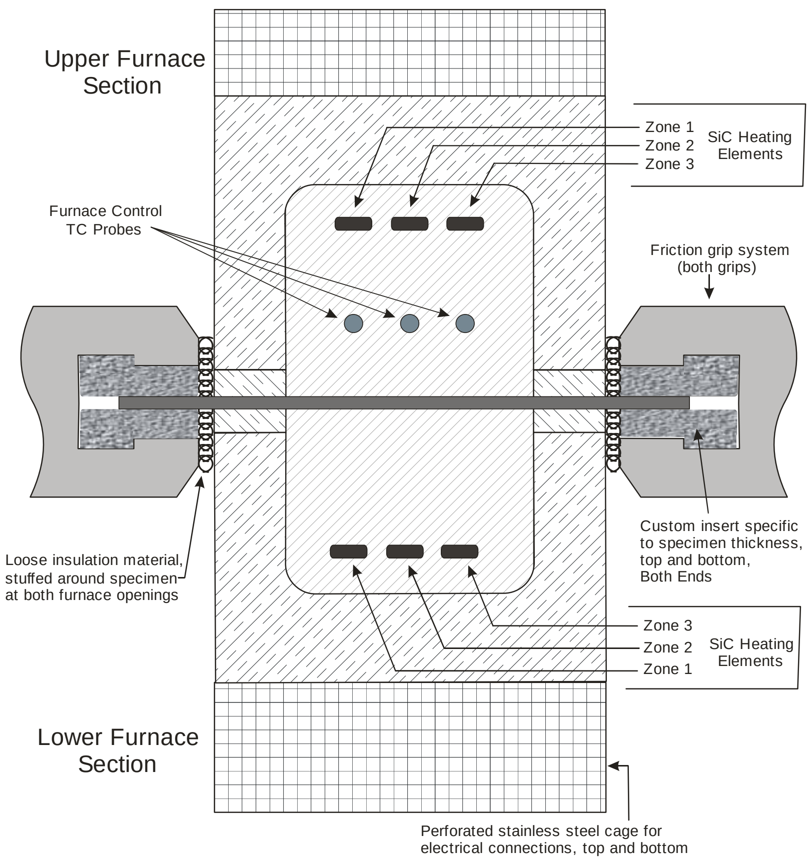

Figure 17. *Schematic illustration of the Amteco furnace with TC locations*

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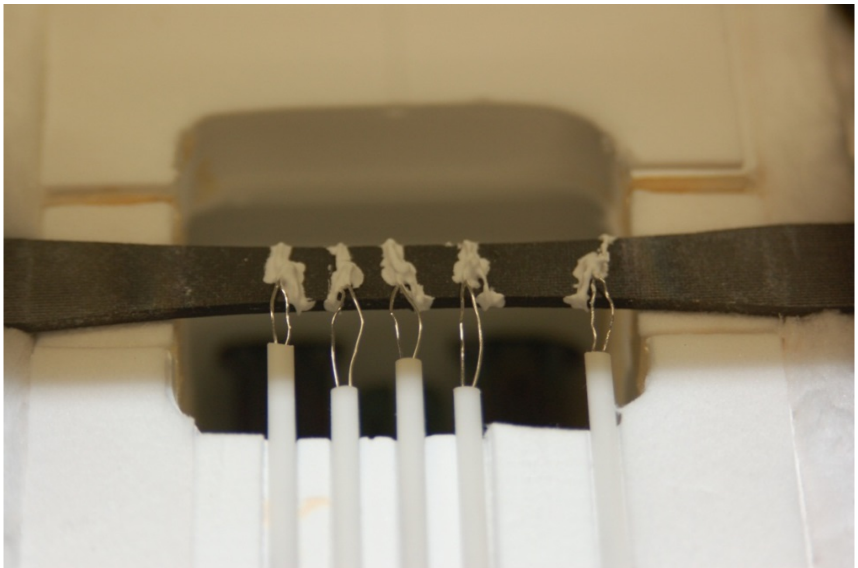

Figure 18. *Thermal profile specimen installed in Amteco furnace (front view)*

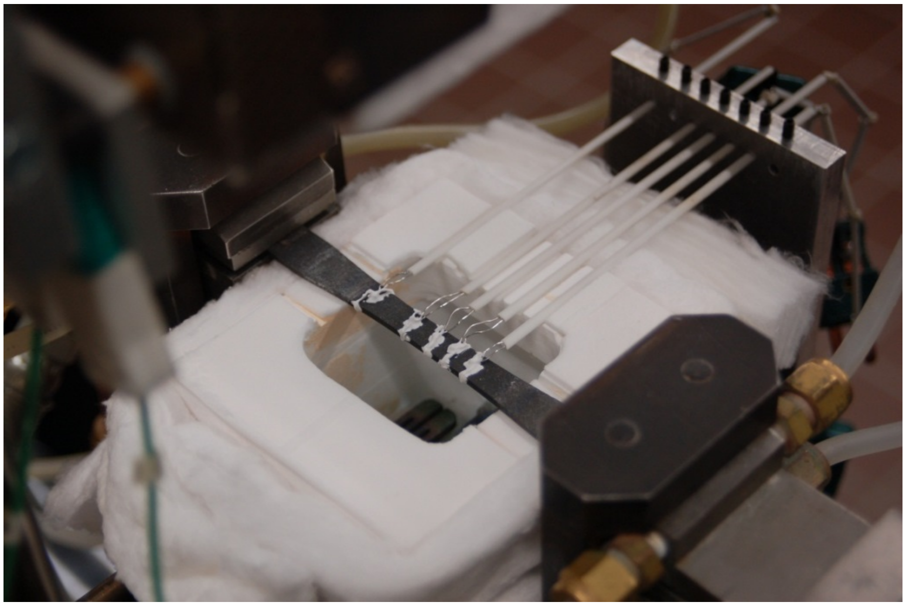

Figure 19. *Thermal profile specimen installed in Amteco furnace (back view)*

Temperature measurement accuracy and precision are maintained through use of equipment calibrated by the Air Force PMEL (Precision Measurement Equipment Laboratory) to NIST standards. Calibration recall is defined by the equipment manufacturer and never exceeds one year. When multiple heating zones are utilized, all control units (Barber Coleman) have the same temperature control accuracy. This is achieved by having a multiple-unit equipment pool and selecting units based on reported accuracies. Care must be taken to insure that equipment calibration of the digital display values also include the Bayonet Neill–Concelman (BNC) 10V output. This is typically not the case for most temperature calibrations; therefore, calibration (standardization) curves based on calibrated digital output and measured BNC outputs must be performed to provide correct temperature data for test collection purposes.

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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 (Figure 20) and precisely move it vertically 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.

All data collected 20 mm above the hot-zone CL utilized this method. In addition to the ceramic probe, one additional thermal profile measurement test was conducted with a standard-profile CMC test specimen, positioned normally and gripped only with the lower grip. This specimen provided thermal data below the hot zone as the actuator was moving away from the furnace.

Figure 20. *Single ceramic TC probe positioned at furnace centerline*

Use of multiple thermocouples on a thermal profile specimen requires multiple-measurement capability through use of multiple single measurement devices or a switchable unit. Both types of devices are utilized by AFRL, thus, requiring human interaction to collect data. Single-probe temperature measurements were automated, reducing the amount of technician time needed to perform a profile. Manual data collection utilizes a spreadsheet identifying both the thermocouple location and associated temperature.

3.3 Procedure for Conducting Thermal Profiles

Thermal profiling for each CMC system was conducted at multiple temperature ranges. Profiling started at the lowest temperature of 1000°C and moved up to 1200°C, and then 1300°C. Initial Mellon furnace thermal studies focused on the response of furnace control, temperature stability, and stabilized thermal differences between the specimen and the control probe temperatures. These data suggested that the default proportional-integral-derivative (PID) control settings provided by Mellon provided excellent control properties. However, extensive studies on specimen thermal response to the furnace thermal ramp parameters suggest that initial temperature ramps must be

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performed in manual control to minimize specimen thermal overshoot. Upon completion of the thermal ramp, the default PID settings provide optimal thermal stability. Specifics of these studies are included in much more detail in Appendix B.

The AFRL procedure when using the multiple zone igniter heaters is to step up to the test temperature in manual increments of power. One could also use automatic ramping to temperature; however, manual increments allow for less chance of overshooting temperature. Due to the thermal mass of the furnace responding slower than the specimen, specimen overheating during the preliminary heat-up is a real possibility. Thus, the standard protocol for AFRL, as well as numerous other test houses, is to select a preliminary temperature (50-100°C) below the desired test temperature for the initial furnace heat-up. Once thermal stability has been achieved at the preliminary temperature, furnace controls are set to the desired final temperature. Final ramp rates and difference between preliminary and final temperatures are dependent on furnace performance and are unique to each test system. The typical goal is to reach the desired specimen temperature in about 30 minutes. Once at the desired temperature, the furnace is allowed to stabilize about 30 minutes before making profile measurements. This same sequence is used with actual specimen testing.

Single-zone furnaces, such as the Mellen furnace, use the Temperature Control Probe, as shown in Figure 15, to control furnace temperature. Therefore, specimen response (temperature) is evaluated after profiling is completed to determine the final furnace control temperature necessary to achieve the desired test specimen temperature.

Multiple-zone furnaces require some amount of manual power adjustments to each zone to optimize the specimen thermal gradient. Thus, thermal profiling for multiple zones initially defines the desired specimen temperature and profile shape, with the operator identifying the optimum power/temperature for each zone to achieve the desired temperature profile on the test specimen. Then, those temperature settings for each zone are recorded and used during the actual tests.

Single-probe thermal profiling utilized the actuator LVDT position to automate the position sequencing, as well as data collection options. A unique specimen-holding device was designed to be secured in the lower MTS 647 grip and position the probe in the vertical centerline of furnace. The thermal probe was 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 to define the gradient between the hot zone and room temperature. In cases where a specimen replaced the probe, profile locations much above the furnace cavity were not possible, as 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 actual specimen materials. Multiple specimens having different material and processing conditions, containing both single and multiple thermocouples, were evaluated.

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4.0 RESULTS OF THE THERMAL PROFILE STUDIES

The current AFRL thermal profile methodology uses multiple thermocouples placed on a CMC thermal profile specimen of identical length and material processing conditions as the specimens to be tested, providing data identical to actual test conditions. Material type and processing conditions affect the thermal response, as specimen temperature is a combination of convection and radiant thermal components. Thermal profiles were conducted using both the Amteco and Mellen furnaces and measured for all seven of the CMC systems described earlier in Section 1.3 and at multiple temperatures.

4.1 Amteco Furnace Temperature Profiles

As stated previously, use of a horizontally mounted furnace and multiple heating zones provides for a uniform and symmetric thermal gradient over the specimen's gage length. Thermal profiles for all seven CMCs were conducted for the Amteco furnace and are shown in Table 3. The funace was set up to use an extensometer with an ~14 mm gage length, so the thermal profile specimen temperatures were measured at the Center line, ±7 mm, and ±14 mm. The goal of the thermal profiling was to keep the temperature variation in the gage length to less than 1% of the test temperature. So for a test temperature of 1000°C, the goal was to have the temperature along the gage length deviate by no more than 10°C. The results in Table II show that for all the CMCs tested the deviation was substantially lower than 1%. Often it was approximately only 0.2%. In order to compare to the Mellen furnace, measurements were also taken at ±14 mm. So for a 28 mm gage length, the average deviation for Nextel 610/AS and Nextel 720/AS was consistently at or within the 1% variation goal.

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Table 3. Thermal profile of seven CMCs using a Amteco Furnace

Amteco Furnace								
Ceramic Matrix Composite	Thermal Profile Temp. (C)	Thermal Couple Location On CMC Specimen						
		Left			Center	Right		
		20 mm Left CL (C)	14 mm Left CL (C)	7 mm Left CL (C)	Center Line (C)	7 mm Right CL (C)	14 mm Right CL (C)	20 mm Right CL (C)
Nextel 610/AS	1000		991	1000	1002	999	992	
Nextel 610/AS	1100		1089	1099	1102	1099	1092	
Nextel 720/AS	1000		993	999	1000	999	993	
Nextel 720/AS	1100		1093	1098	1102	1099	1088	
Nextel 720/AS	1100		1094	1101	1101	1097	1090	
Nextel 720/A	1200	1163		1199	1201	1200		1162
Nextel 720/A	1200	1160		1196	1202	1199		1165
S200H	1200	1184		1198	1202	1199		1196
C/HYPR-SiC	1200	1168		1206	1198	1201		1159
C/HYPR-SiC	1200	1176		1199	1199	1200		1169
SiC/HYPER-SiC	1200	1156		1199	1200	1202		1154
S400	1200	1154		1200	1200	1198		1146

Some of the results from Table 3 are presented graphically in Figure 21. Shown in this figure are the thermal profile results for the Amteco furnace for four different CMCs. The Nextel 610/AS was profiled at 1000°C, the Nextel 720/AS at 1100°C, while both the Nextel 720/A and the S200H CMC systems were profiled at 1200°C. For each CMC system, the test specimen temperature and corresponding furnace control TC temperatures are presented. As stated earlier, the thermal profiles are very symmetric around the center of the gage section of the thermal profile test specimens. In addition, the actual test specimen temperature does not deviate more than 1% from the desired test temperature over the 28 mm gage length of the test specimen. In many cases, the deviation was only approximately 0.5%. In addition, the temperature does not reduce substantially until well into the radius of the specimen at 20 mm. This helps avoid the issue of intermediate temperature embrittlement.

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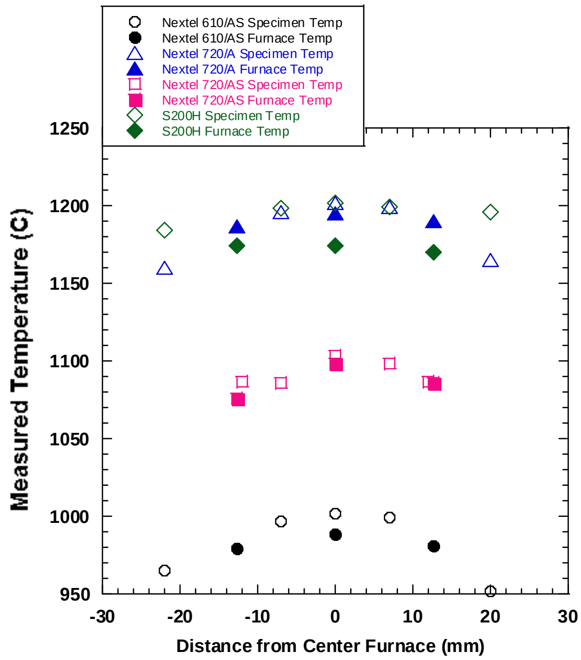

Figure 21. *Amteco furnace thermal profiles for four CMC systems*

4.2 Mellen Furnace Temperature Profiles

In contrast to the horizontally mounted Amteco furnace, the Mellen furnace is mounted in the more standard vertical position. A total of seven CMC systems were profiled in the Mellon furnace using three thermocouples that were located 12 mm above the center line of the thermal profile specimen, at the center line, and 12 mm below the center line. For each CMC system multiple temperatures were profiled, and the results are presented in Table 4. The Mellon furnace is a one zone furnace, so no tuning of the furnace could be performed to alter the profiles as was done with the Amteco furnace. During thermal profiling the procedure was to keep the center of the gage section very close to the target temperature. As stated earlier, the goal was to achieve less than 1% deviation across the gage section of ~24 mm. Therefore, the percent deviation from the target temperature was calculated for each data point and entered into Table 4 for faster interpretation of the results. Also, the data entered in bold are the actual test temperatures used during testing, while the other temperatures were profiled for completeness. The results show that each CMC system behaves uniquely. The S200H exhibited the most uniform temperature profile while the Nextel 610/AS exhibited the largest deviations. Operating the furnace vertically produced a non-symmetric thermal profile. The bottom TC were approximately 3-6% lower than the target temperature, the center line TC were always under the 1% target, while the top TC were approximately 1-2% of the target temperature. In general the percent deviation was surprisingly consistent for the entire temperature range for each CMC tested.

Figure 22 presents graphically the thermal profile results for the Mellon furnace for four different CMCs. Shown are the thermal profile specimen temperatures as well as the furnace probe temepratures. As with the Amteco furnace, the Nextel 610/AS was profiled at 1000°C, the Nextel 720/AS at 1100°C, and the Nextel 720/A and the S200H CMC systems at 1200°C.

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Table 4. Thermal profile of seven CMCs using a Mellon Furnace

Mellen Furnace							
Ceramic Matrix Composite	**Thermal Profile Temp. (C)**	**Thermal Couple Location On CMC Specimen**			**% Deviation**		
		Top 12 mm Above CL	**Center CL**	**Bottom 12 mm Below CL**	**Top 12 mm Above CL**	**Center CL**	**Bottom 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

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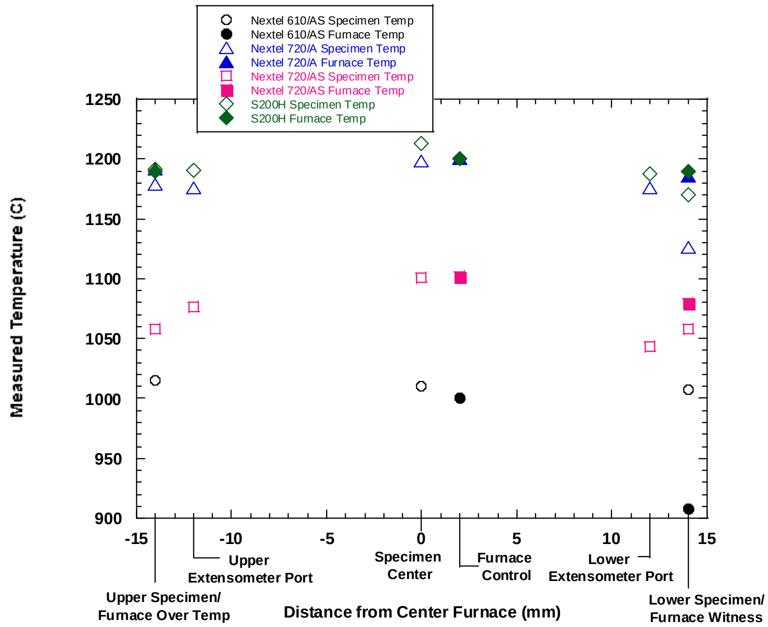

Figure 22. *Mellon furnace thermal profiles for various material types*

From Figure 22, we can see that the thermal profile for each CMC is not nearly as symmetric as those for the horizontally mounted Amteco furnace because of upwards heat convention that occurs inside the furnace. However, with proper use of insulation around the outside of the furnace near each opening, this effect has been minimized. In studying Figure 22, one can clearly observe that the furnace probe temperatures are mesureably different than the temperatures measured on the actual thermal profile test specimens.

4.3 Mellen Furnace Thermal Probe Profiles

As stated earlier, a single-probe ceramic-coated thermocouple was transitioned through the furnace and temperature measurements were made (Figure 23). 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 furnce TC. Following that behavior, 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 24 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

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difference is attributed to the type of specimen and not the specimen geometry. The S200H is substantially 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.

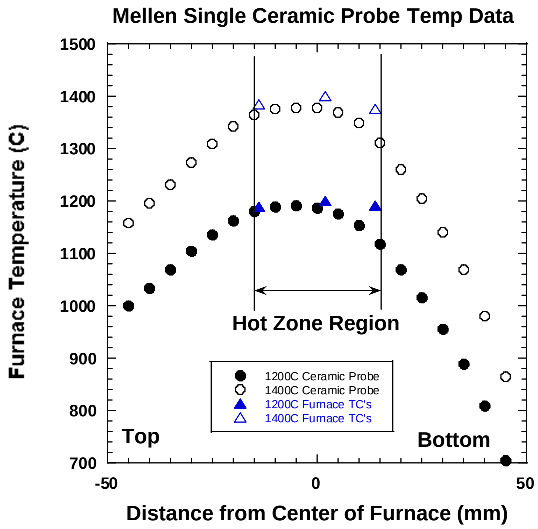

Figure 23. *Mellon furnace thermal profile using single ceramic TC probe*

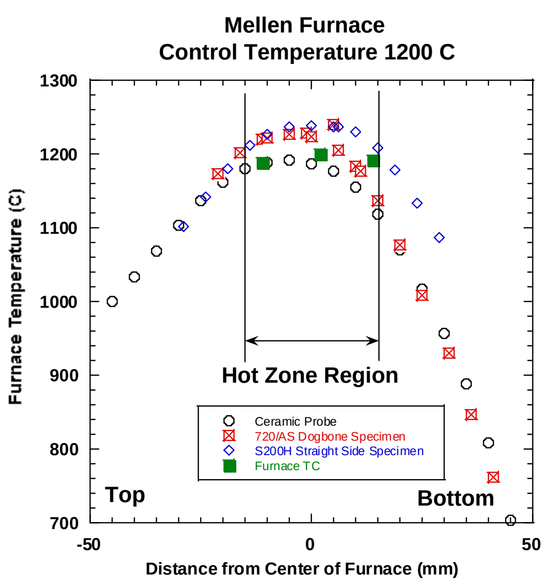

Figure 24. *Effect of material type on thermal profile of furnace both inside and outside hot zone region*

4.4 Considerations for Testing At Temperatures above 1300°C

Increasing test temperatures to 1400°C and above will require careful consideration to the imposed thermal gradients from within the hot-zone region to the outside of the furnace and how these

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sharp transitions affect material performance. Knowledge of thermal profile conditions in the transition region outside the furnace hot-zone will become even more critical. The selection of the proper test method for these increased temperatures will require further study to identify the relationship of specimen conductivity contribution to that of only measuring air temperature. Increasing the test temperature will also make it difficult to use contact extensometry to measure strain. As temperature increases, so does the likelihood of the extensometer rods reacting with the test specimen, creating a damage-reaction zone on the test specimen. Therefore, much work remains on developing high-resolution non-contact strain measurement techniques.

Also, with increasing temperature, there will be additional issues that need to be addressed. Long-term degradation of electronics within load cells and hydraulic seals become a significant concern, along with creep of the heating elements. However, the low-conductivity properties of CMCs become an asset when it comes to gripping the test specimens. It is critical to use grip cooling to protect the necessary mechanical equipment and, fortunately, the low conductivity of the CMCs means that cooling the grips should have an insignificant effect on the measured thermal gradient. The issue of cooled and uncooled grips was studied in detail and the results of the thermal gradient studies are presented in Appendix A.

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5.0 CONCLUSIONS

An instrumented thermal profile specimen identical to the actual test coupons to be tested should always be used to conduct thermal profile studies. Even simple furnace repair or modifications can affect how the specimen is heated, requiring profiling prior to resuming testing. Knowledge of furnace and specimen responses to initial thermal heat-up are vital to insure temperature overshoot conditions do not occur. Temperature characterization outside the hot-zone region is less dependent on the method used, especially with increasing distance from the hot zone.

The important findings from this study are:

- A specimen of similar length and processing conditions to actual test coupons should be used to thermal profile a furnace for each test temperature.
- Thermocouples must make direct contact with the specimen and be coated with protective ceramic cement to accurately determine specimen temperature. Integrity of the TC must be made before each use of the thermal profile specimen, as the ceramic cement may de-bond from the specimen during cool-down.
- Multiple TCs are required on a specimen to define the temperature uniformity across the region of interest (typically the gage section).
- Thermal profiling of the furnace outside the hot-zone region was measured to be independent of specimen material.
- A horizontally mounted Ametco furnace produced a symmetric thermal gradient profile on the thermal profile specimen and the temperature along the gage length typically deviated by no more than ~0.2% for a 14 mm gage length and less than 1% for a 28 m gage length.
- The Mellon furnace operates with only one zone of temperature control. When run in the vertical configuration the temperature along the 24 mm gage length can deviate ~3-6% of the target temperature.
- Careful placement of insulation minimizes heat loss from the furnace and improves the thermal profile of the test specimen.
- Any repair, change, or rebuild of a furnace requires a specimen thermal profile to define the specific control temperatures required to achieve the desired specimen temperature.
- Furnace control TCs measure temperatures lower than the specimen. Therefore, temperature sequencing is recommended to prevent specimen over-temperature conditions
- Cooling of the grips had no effect on the measured thermal gradient along the length of the CMC thermal profile specimens and was attributed to the low thermal conductivity of CMCs tested. This might change for CMCs with much higher thermal conductivity.

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6.0 REFERENCES

[1] Zawada, L. P., Pierce, J. L., and Davidson, K. "Durability Evaluation of Ceramic Matrix Composites (CMCs) for Thermal Protection System (TPS) Application: Description of Test Program" AFRL-RX-WP-TR-2013-0212, 2013.

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APPENDIX A
INFLUENCE OF GRIPS ON THERMAL PROFILES AND GRIP-CMC INTERFACE TEMPERATURES

Testing at temperatures at and above 1500°C requires careful analysis of all aspects of thermal loss from the furnace. Increasing test temperatures affect gripping components, as well as all control and measurement components in close proximity to the furnace. CMC materials remain very expensive, requiring the use of test specimens that are as short as possible, yet suitable for the test frame, gripping method, and heating method. In general, very little space is left between the furnace and the grips. This study evaluated both how different test temperatures contribute to temperatures at the interface between the grips and the test specimen, as well as measured how different gripping conditions contribute to the measured specimen temperature profile.

TEST PROCEDURES

Testing for this study was conducted as an extension to a standard specimen thermal profile in order to utilize the multiple thermocouples already available. Additional TCs were added to the top and bottom wedge grips, top and bottom grip surfaces, and the interface between installed additional external furnace insulation and the grips. Figure A-1 is a schematic that precisely details all of the test components and related TCs. Monitoring data of the cooling water reservoir and air temperature approximately 2 feet from the furnace were also collected. These additional TCs utilized K-type junctions connected to a multiple input/output TC indicator box with analog output collected digitally for every channel.

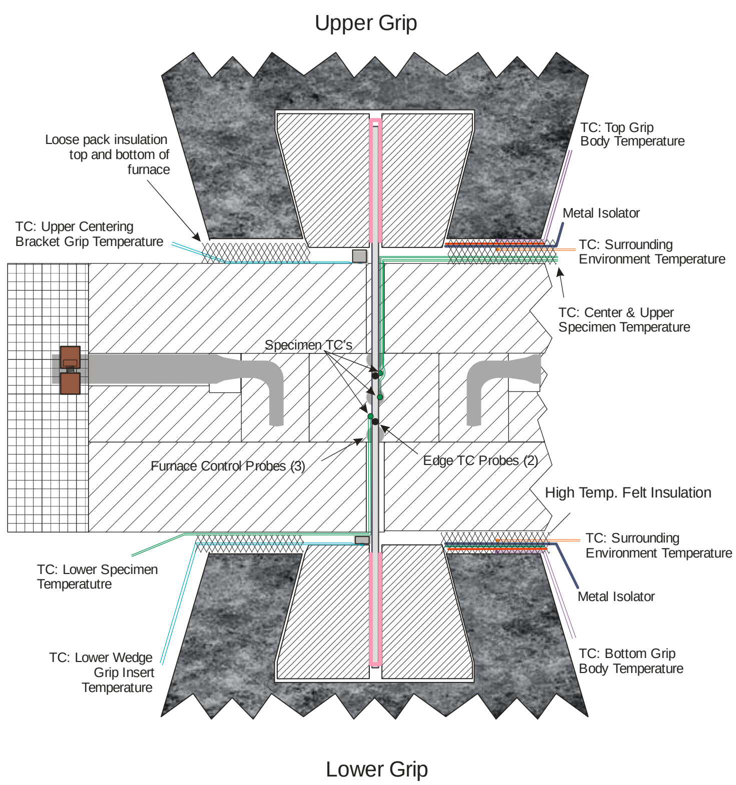

Figure A-1. *Schematic illustration of TC locations for mapping thermal loss from Mellen furnace*

Testing was conducted in two phases. The first investigated the thermal conditions after one hour soaks at 1000°C, 1200°C, and 1400°C. These data were collected at the end of the thermal

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stability portion of the profile and represent normal operating conditions, with focus on how hot the grips actually get at each test temperature. For this study, cooling water was used on both the wedge grip inserts and the cooling tubes mounted around the wedge grip body. The second phase investigated the effect of specimen grip condition on the specimen thermal profile. In this study, the cooling water to the wedge grip inserts was turned off. In addition, measurements were made with the grips both open and closed. Water controls were added to isolate cooling water to the wedge inserts of the grips, while still maintaining cooling integrity for the grip body and load cell components. Thermal stability for each test condition was observed to occur within 5 minutes, with actual testing lasting 15 minutes. This study focused on the impact of external cooling on specimen temperature caused by specimen thermal conductivity of heat along the length of the specimen.

FINDINGS

Furnace Thermal Loss

The Mellon furnace, as operated at AFRL with the use of additional loose and blanket insulation materials, produces an environment where very little heat escapes the furnace body. The principal heat loss locations are the top and bottom furnace openings where the specimen exits the furnace body to be gripped. The closest surface to the high-temperature is the grip wedges, which are water-cooled (typical 17°C reservoir temperature). As shown in Figure A-1, thermocouples were placed on the surfaces of the wedge inserts in the grip bodies and these thermocouples were near the test specimen. Results of the study are shown in Figure A-2 as a plot of measured wedge insert surface temperature versus furnace control temperature. As shown in the figure, temperatures at the surfaces of the wedge inserts in the grips increase as furnace temperatures increase, but only 15-20°C for each furnace increase of 200°C. In addition, the top wedge inserts were approximately 20°C hotter than the lower wedge inserts and is a good demonstration of the heat-rising “chimney” effect. Cooling coils are wrapped around the actual grip bodies to insure no harm is done to temperature-sensitive hydraulic seals and strain gages within the load cells. Maximum recorded temperature at the face of the grip body was only 70°C, nearly 40°C cooler than the upper recommended operational temperatures, and these temperatures were at the location closest to the furnace, not at the actual critical seal component location. This demonstrates that, for the temperatures studied, the AFRL configuration of the Mellen furnace in conjunction with the MTS hydraulic wedge grips has more than enough operational margin for the grips.

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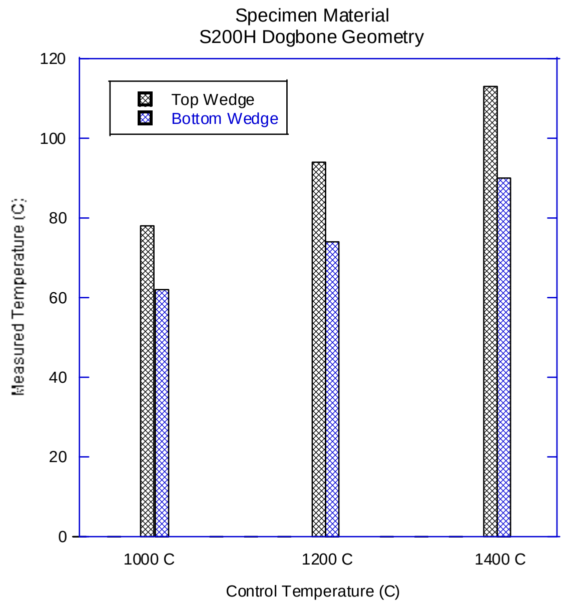

Figure A-2. *Plot of measured temperature at the top of the wedge grip inserts as a function of furnace temperature*

A second study involved measuring the specimen profile at 1400°C while changing water cooling conditions to the wedge grip inserts and also either gripping or not gripping the specimen as follows: 1) Water cooling to wedge inserts on and specimen gripped top and bottom, 2) Water cooling to wedge inserts off and specimen gripped top and bottom, 3) Water to top wedge inserts turned off while top grip is clamped and lower grip is open, and 4) water to top wedge insert turned on while top grip is clamped and lower grip is open. The results from this study are shown in Figure A-3 and clearly show that both wedge insert cooling and actually gripping the specimen have no measurable effect on the temperature profile of the test specimen. This result was somewhat expected and can be attributed to the low thermal conductivity of the CMC test specimen. However, it is important to point out that this might change if a more thermally conductive CMC were to be tested.

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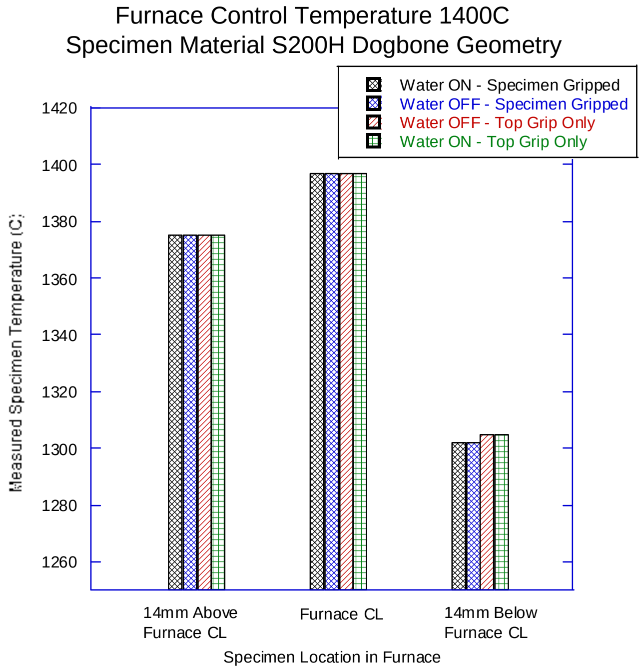

Figure A-3. *Effect of wedge grip insert cooling and gripping method on specimen temperature*

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APPENDIX B
DETERMINING FURNACE HEAT-UP PROFILE TO MINIMIZE SPECIMEN THERMAL OVERSHOOT

There is a specimen thermal interaction to the radiant heat generated by the Mellon furnace that is not captured by monitoring the furnace temperature control thermocouple. This interaction is different for different final temperatures and, most likely, dependent on the thermal emissivity of the CMC specimen. The test data presented in this study were developed using S200H test specimens which consist of Hi-Nicalon silicon-carbide fibers in a silicon-carbide matrix manufactured using a polymer infiltration and pyrolysis method. The final as-produced specimens are black, in contrast to oxide/oxide CMC materials that are very white. The black S200H CMCs should be more sensitive to radiant heat and is more thermally conductive than the oxide-oxide CMCs. Therefore, the heat-up time/temperature profile derived using the S200H specimen was used for all other CMC materials.

Initial furnace PID settings were studied using an instrumented specimen. Initial settings were as follows: proportional band of 16, integral time of 13, and derivative time of 2. The furnace control was set to auto and the control temperature to 1200° on the first heat-up run and 1400°C on the second. A data collection rate of 1 second was used for the hour-long test.

Results from this first heat-up study are shown in Figure B-1. For both temperatures, the furnace control thermocouple data shows little to no thermal overshoot. However, if one looks at the thermocouple data from the S200H test specimen, it is evident that it experienced a significant overshoot of temperature, with 1200°C being more severe than 1400°C. At the very high temperature of 1400°C, there is significantly more heat loss from the furnace, requiring significantly more power to reach the higher temperature, resulting in less overshoot. It is also important to note that the furnace temperature at 1200°C is significantly lower than the specimen temperature which, again, demonstrates the importance of instrumenting an actual thermal profile specimen with thermocouples.

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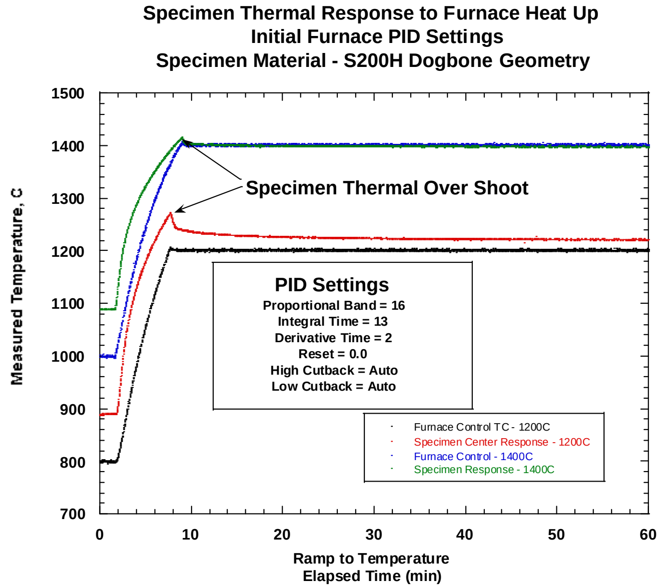

Figure B-1. *Mellen furnace thermal profile with factory setting PID*

Several procedural steps were made in an attempt to minimize the thermal overshoot. Changing the bandwidth or integral values produced no positive results. However, adjusting the temperature at which the power is cut back did improve the amount of overshoot to some degree. A 200°C cut-back temperature was combined with manual stepping of the furnace set-points to limit overshoot while still reaching temperature in a reasonable time period. For each set of experiments, the manual stepping increments were evaluated for the time required for the thermal profile specimen to respond to the furnace change. After several manually adjusted heat-up experiments, a defined ramp procedure that achieved the targeted specimen temperatures with negligible temperature overshoots was established. This ramp procedure is shown in Figure B-2 for the same two temperatures shown in Figure B-1. For both test temperatures, the thermal profile specimen never exceeded the maximum temperature. Table B-I presents an example of a new, modified ramp rate that shows the step-by-step rate increases used to achieve the ideal ramp rate. This ramp can be achieved in about 30 minutes, followed by a 30-minute soak at temperature to allow the specimen temperature to stabilize. Some additional optimization for time efficiency purposes could be achieved at the higher temperatures. It is important to note that this procedure needs to be done for each furnace because each will operate a little differently. Development of an external computer program to automate this process could easily be done. Segment control programs provided by the Eurotherm 2408 temperature controller for the Mellen furnace do not allow for transfer to automated control once temperature is achieved using the manual process; therefore, this route was not pursued further.

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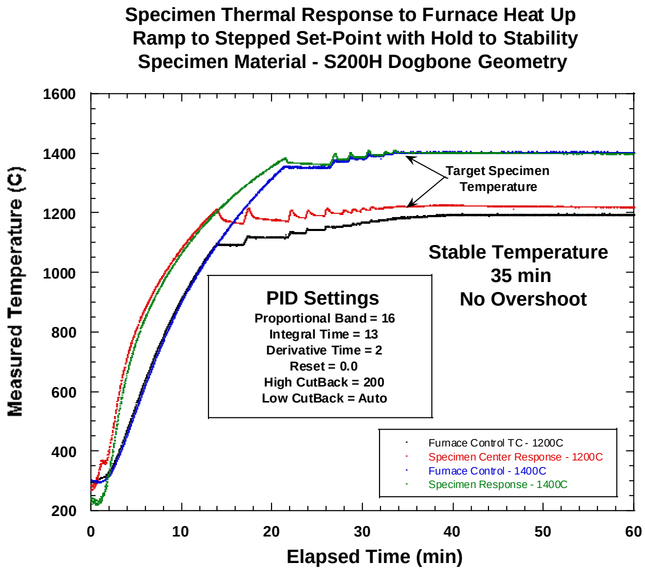

Figure B-2. *Mellen furnace thermal using defined time/temp heat-up cycle*

Table B-1. Example of Heating Ramp Profile Used to Achieve 1200°C

Typical 1200C Furnace Ramp Program	
Furnace Control (Set Point) Temperature	Heat Time (min)
1080 C	15
1105 C	5
1120 C	2
1130 C	2
1140 C	2
1145 C	1
1150 C	1
1155 C	1
1160 C	1
1162 C - 1170C by 2 degree increments	every 1/2 min (Total time 4 minutes)
1171 C - 1190 C by 1 degree increments	Every 1/2 min (Total time 5 minutes)
Final Set Point 1190 C	20

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APPENDIX C
LESSONS LEANED – EFFECT OF FURNACE REPAIR/ REBUILD ON SPECIMEN THERMAL PROFILE

Repair or rebuild of any high-temperature furnace has a significant likely hood of changing the thermal profile conditions of the furnace and the test specimen. This was confirmed during recent work at AFRL on the Amteco furnace shown earlier in Figures 2, 3 and 17. The research involved conducting approximately 25 creep tests on a N720/AS test specimen at 1100°C. For this testing, it was very critical to conduct all tests under the exact same thermal conditions; therefore, detailed thermal profiles were made both periodically during the test series, as well as any time the furnaces required repair.

The Amteco furnace, as used by AFRL, is mounted in a horizontal position to minimize thermal gradients. The furnace consists of three pair of silicon carbide igniter heating units placed directly above and below the specimen, as shown schematically earlier in Figure 17. These igniters, commercially marketed to ignite gas furnaces, have been specially modified to achieve the desired resistance properties. Prior to the start of this series of creep testing, the entire furnace was rebuilt. Igniter heating elements were replaced, furnace control TCs replaced and coated with new cement, and a new thermal profile specimen was assembled. After each thermal cycle, resistance of each element was measured and documented, as experience has shown that changes in the resistance indicate degradation in the heating elements. During the testing period, several “minor furnace repairs” were made, such as repositioning or recoating the control TCs. After each repair, the original thermal profile specimen was used to verify and/or adjust the furnace settings to achieve the same thermal profile as that achieved during the first run. Several additional profiles were conducted periodically throughout the test series just to make sure no changes had occurred to the furnace. These additional tests showed no change in the thermal profile measured by the specimen and confirmed that the primary contributor to changes in the thermal profile resulted from the furnace rebuild efforts.

The principle cause for thermal variations that result from a furnace repair/ rebuild the majority are, most likely, caused by slight variation in the placement of the furnace control TCs. Three thermocouples are placed directly below the upper heating elements about halfway between the specimen and element, as shown in Figure 17. These thermocouples are mounted in 2-holed alumina rods and extend into the furnace cavity approximately 38 mm parallel to the heating element. The beaded TC is then coated with ceramic cement to prevent degradation. During any repair of the TCs, placement is carefully monitored to insure “exact” repositioning within measurement capabilities. Even with the amount of care taken, specimen thermal profiles were found to change after each repair. For the first furnace rebuild, only the control TCs were replaced. The furnace was then profiled using the initial Barbara-Colman settings. The results are shown in Figure C-1 which documents that the thermal profile specimen averaged 7°C lower than the initial profile. This required the temperature controller set-point temperatures to be increased to produce the desired temperature of 1100°C. The furnace was used for a significant amount of testing and then a second repair consisting of new elements and new TCs was performed. Once again, the furnace was profiled using the initial temperature controller settings and resulted in a profile temperature approximately 17°C higher than the previous measurement

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data (Figure C-1). Therefore, if the initial control settings were used to continue our testing program, there would have been a 15-20° variation in our actual specimen temperatures.

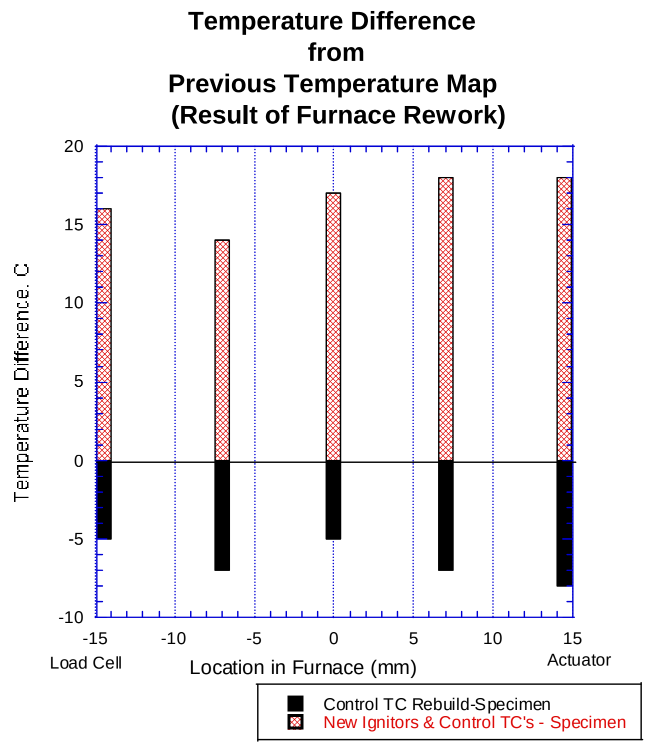

Figure C-1. *Effect on specimen thermal conditions resulting from repair or rebuild of an Amteco furnace*

Throughout the entire program, maximum temperature variation over the gage length of the thermal profile specimen never exceeded 6°C. To achieve these conditions, the actual temperature of the control TCs were adjusted as much as 15°C after each repair. Figure C-2 demonstrates how, throughout the test series, the specimen TC, control TC, and witness TC temperature values changed. As shown in the figure, the control TC were approximately 10-20°C lower than the thermal profile specimen, while the witness TCs were also 20-30°C lower. This is important to note, because even though the witness TCs are in the same plane as the thermal profile specimen and very close to the edge of the thermal profile specimen, they still read significantly cooler. Relying only on the furnace TC or the witness TC would produce significant error in the specimen temperature. Extreme care was taken after each furnace rebuild, as stated earlier, and yet each following calibration demonstrated that new furnace settings were required. This data clearly shows the need for calibrating the furnace after even the most minor of changes to the furnace.

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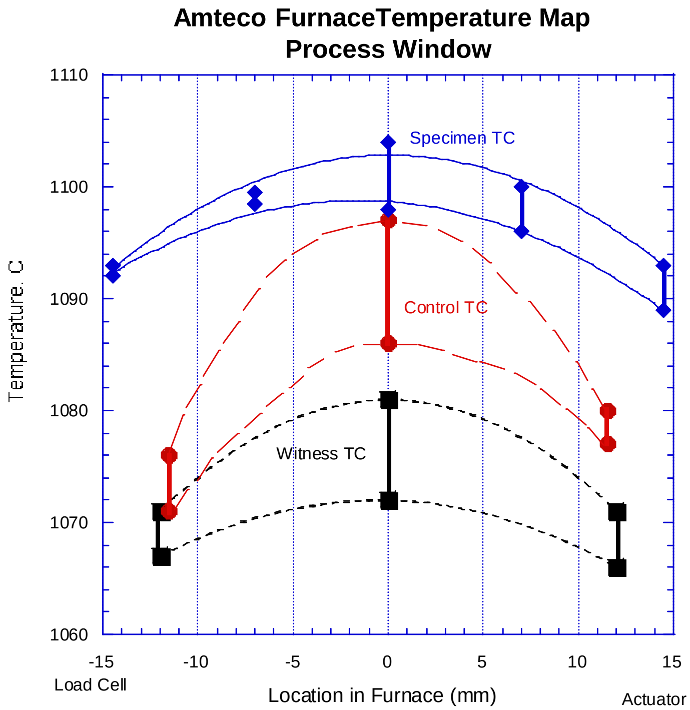

Figure C-2. *Furnace temperature variation resulting from repair or rebuild of an Amteco furnace*

The furnace repairs were often caused by failed heating elements. As was shown in Figures C-1 and C-2, changing heating elements can have a large effect on the temperature of the thermal profile specimen. Detailed studies have shown that it is not only placement of the thermocouples, but also the resistance of the heating elements that affect temperature.

Degradation of the heating elements during extended periods of operation at high temperature has always been a concern, as it affects the amount of power needed to achieve the desired test temperature. Therefore, the Amteco furnace cannot simply be run in manual set power output control. Commercial design requirements for this element involve short cycles (less than a minute) of high output to create a glow necessary to ignite a natural gas flame. AFRL testing typically utilizes lower temperatures, but for many hours. During time at temperature, the elements react with the environment, resulting in an increased resistance and color change from black to grey. The greatest change occurs during the first 50 hours of use. Recently, AFRL has been tracking the resistance of all the elements in the furnace after each test. The results are shown in Figure C-3 as a plot of element resistance change as a function of cumulative test hours at 1100°C. During the first few hours of operation, the resistance changes very quickly by approximately 32 ohms, after which the change is more gradual. Specific placement of the element in the furnace (top or bottom element) has no significant effect on rate of degradation. Previous experience at AFRL has indicated that the longest-life elements are those that have a starting resistance no higher than 70 ohms. This requirement has been incorporated in the purchase requirements for a number of years.

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As stated earlier, the designed use of the elements in gas furnaces requires them to switch on and off many thousands of times without failure. They are specifically designed to withstand extensive numbers of thermal fatigue cycles. Data collected over a two-year period appears to document this behavior. Figure C-4 shows the average resistance for three different sets of elements versus the numbers of hours tested. That same data is plotted in Figure C-5 as a plot of change in resistance versus number of thermal cycles to temperature. We can see that at around 10 thermal cycles, there is an approximately 50-ohm difference between the 2013 reading and the 2014-2 readings. Almost all of the 2014-2 reads are from tests that often exceeded 100 hours. Recently, AFRL has incorporated a 24-hour "burn-in" period at temperature before conducting the first set of thermal profiles. This appears to "age" the elements enough to allow for a more steady power output since element resistance change will affect the power requirements needed to achieve desired temperature. This further validates the need to perform testing using active temperature control instead of simply power output. Neither an absolute upper resistance nor a limit for amount of allowable resistance change has been determined at this time.

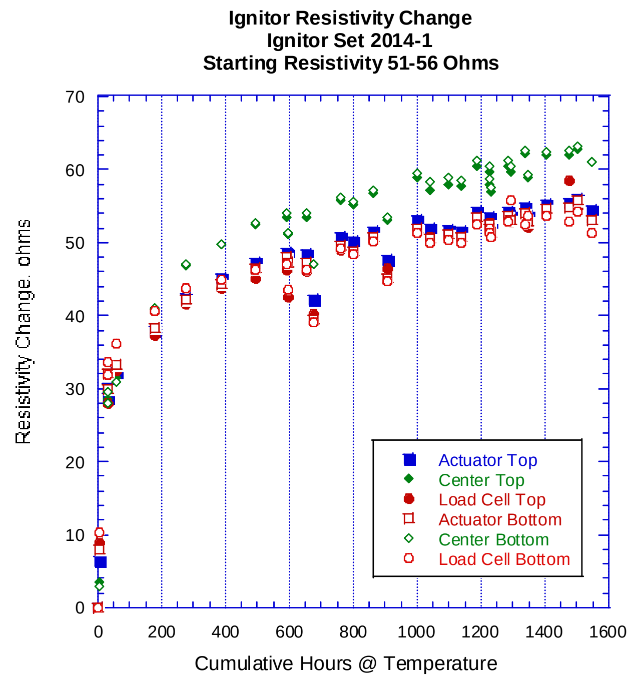

Figure C-3. *Furnace element resistance change during course of testing*

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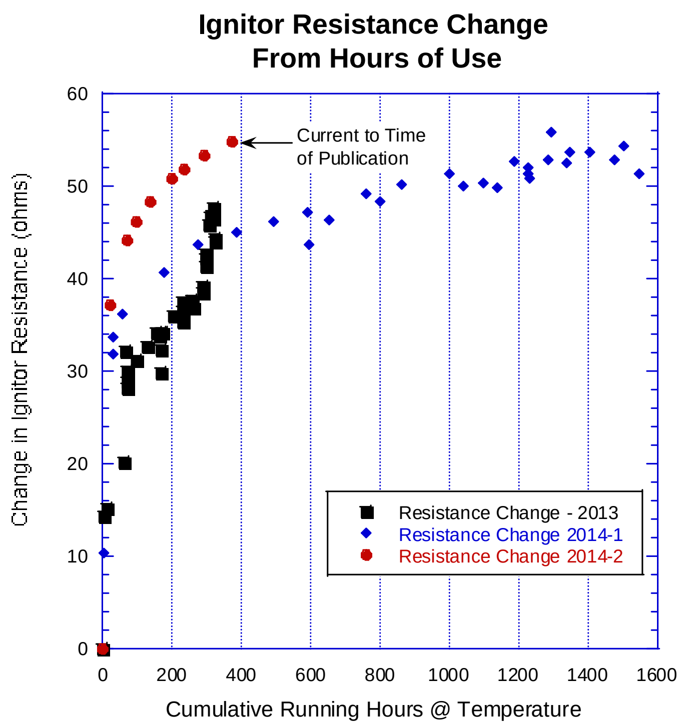

Figure C-4. *Furnace element resistance change due to hours at temperature*

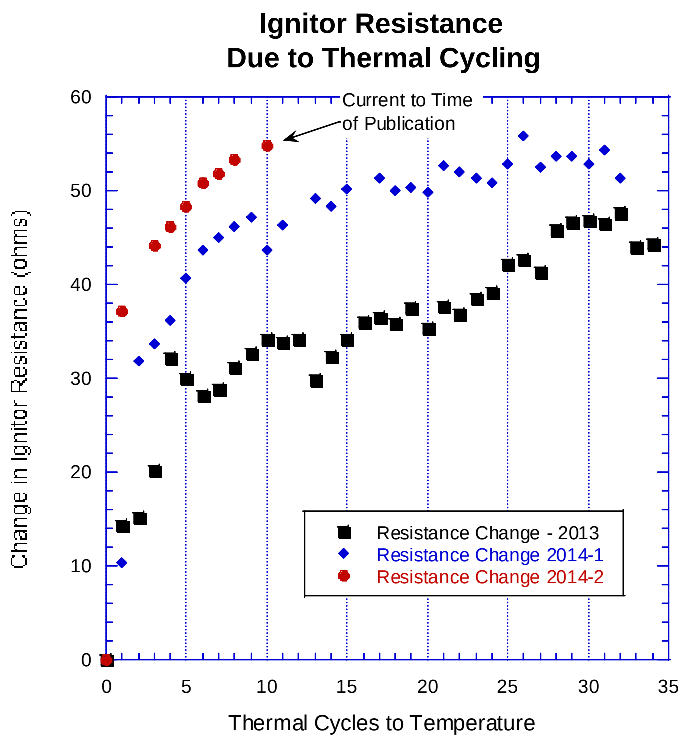

Figure C-5. *Furnace element resistance change due to thermal cycling*

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LIST OF SYMBOLS, ABBREVIATIONS, AND ACRONYMS

AF	Air Force
AFRL	Air Force Research Laboratory
BNC	Bayonet Neill–Concelman
CL	centerline
CMC	ceramic-matrix composite
COIC	COI Ceramics, Inc.
CVI	chemical vapor infiltration
DC	digital control
EMI	electromagnetic interference
HSSW	high-speed strike weapon
ILT	interlaminar tension
ISR	Intelligence, Surveillance, and Reconnaissance
M&P	materials and processing
PID	proportional-integrated-derivative
PIP	polymer infiltration and pyrolysis
RXCC	Composites Branch, Structural Materials Division of the Materials & Manufacturing Directorate
RIDHM	Rapid Development and Insertion of Integrated Hypersonic Materials
SiC	silicon carbide
SiNC	silicon nitrocarbide
TC	thermocouple
TPS	thermal protection system
UDRI	University of Dayton Research Institute
USAF	United States Air Force

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