Measuring Ablation Rates of CMCs and EBCs from Exposure to High-Speed Water Vapor

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John Hayes Cooper
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Technical Project Team Members
John Cooper

On my honor as a University Student, I have neither given nor received unauthorized aid on this assignment as defined by the Honor Guidelines for Thesis-Related Assignments

Signature: John Hayes Cooper Date: 5/13/2021

Approved: Harsha K Chelliah Date: 5/13/2021

Introduction

The efficiency of an aircraft engine can be increased primarily by two methods: 1) increasing the maximum temperature it can burn at, and 2) decreasing the weight of the engine itself. Traditionally, jet engine manufacturers have used nickel-based superalloys that can withstand fairly high temperatures for extended periods of times (Perrut et al., 2018), but new ceramic-based materials have been gaining traction as a higher temperature, lightweight, and more durable alternative. The Achilles' heel of these ceramics is that they deteriorate fairly quickly in the presence of water vapor, a common byproduct of combustion reactions. To prevent this, they can be coated with a thin layer of a more water-resistant material, called an environmental barrier coating (EBC). My technical thesis project focuses on a particular ceramic composite and coating material developed by the project's sponsor, and aims to measure the rate at which it deteriorates when exposed to a high temperature steam jet.

Background

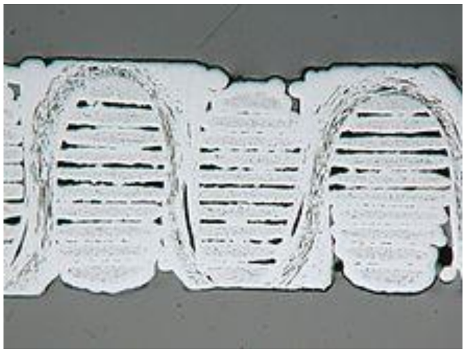

Figure 1 - SEM image of a woven ceramic matrix composite

The ceramic material being tested is called a ceramic matrix composite (CMC), and is composed of a grid-like ceramic matrix, through which ceramic fibers are woven. Figure 1 shows a microscopic view of a typical CMC. The particular CMC used in this study is a silicon carbide fiber reinforced silicon carbide matrix (SiC/SiC CMC), meaning both the matrix phase and fiber phase are silicon carbide.

SiC/SiC CMC Production

With the growing popularity of matrix composites and the rapid developments made in materials science in recent years, countless methods of producing SiC/SiC CMCs have been developed. Four common processes used in SiC/SiC CMC production are briefly described below, but it’s important to note that many variations and combinations of these processes are used to create the desired final product.

- Melt infiltration (MI) – A fibrous preform of SiC is coated with carbon, then a slurry of pure silicon is melted into the preform where it reacts with the carbon to form the SiC matrix. This process is popular as it yields a low final porosity, but it also leaves excess silicon in the matrix which can weaken the final part.
- Polymer impregnation pyrolysis (PIP) – A low viscosity polymer matrix precursor is infiltrated into the SiC fiber preform (impregnation) and then forms the SiC matrix after heating (pyrolysis).
- Plasma sintering (PS) – Similarly to PIP, a SiC fiber preform is infiltrated with a liquid particle slurry, but is then spark plasma sintered to solidify the matrix. Sintering alone cannot fully densify the slurry, though, so this method is always combined with others.
- Chemical Vapor Infiltration (CVI) – In this process, an empty preform is first infiltrated with a gaseous phase of SiC, which slowly solidifies to create the SiC fibers. Then, the preform is again infiltrated with gaseous SiC which solidifies to create the matrix phase.

By creating a braid of ceramic strands and layering many of these braids on top of one another, a composite material is produced that has high temperature resistance, high hardness, and low thermal expansion, the hallmark of most ceramics, but can also stand up to high stresses, is low density, and is incredibly resistant to crack propagation – one of the major downfalls of traditional ceramics (Golden, 2017).

Thermal and Environmental Barrier Coatings

When heated above ~1000 C, the silicon carbide forms a solid silicon oxide (SiO_2) “shell” that encompasses the inner matrix. The SiO_2 shell grows slowly, but is enough to completely protect it from oxidation in dry environments, making silicon carbide ideal for high temperature, non-combustive applications. In jet turbines, though, where combustion abounds, a considerable amount of water vapor is produced. This deteriorates the protective SiO_2 layer more quickly than it can regenerate, causing oxidation and volatilization to occur in the silicon carbide. This effect can lead to erosion rates of up to 1 μm/hour (dos Santos et al., 2011), a considerable amount for blades with lifetimes of up to 10,000 hours.

To combat this erosion, many CMCs are protected by what is called an environmental barrier coating. These coatings are on the order of ~1 mm to ~100 μm, and are composed of complex oxides that are more resistant to water vapor than the SiO_2 that naturally forms on the silicon carbide composite. Environmental barrier coatings (EBCs) were developed to parallel thermal barrier coatings (TBCs), which are applied to traditional nickel-based superalloy turbine blades to insulate them from the inlet temperatures that surpass nickel’s melting point. While the primary function of environmental barrier coatings is to prevent oxidation of the CMC, they also act partially as a TBC in that they also thermally insulate the ceramic material and preventing repeated thermal expansion and contraction, which can cause microscopic crack propagation called thermal fatigue. SiC/SiC CMCs have seen wide use in aerospace applications, and their deterioration rates have been extensively measured under a wide range of conditions. SiC/SiC CMC samples will be tested in this study, but mainly for the purpose of comparing the results against other literature to verify that the testing rig is functioning as expected. The novel data will be from measuring the reaction of different EBC materials when exposed to a high velocity steam jet.

Some studies have been done on deterioration rates of different EBCs at high temperatures, but, as dos Santos et al. point out, many laboratory setups neglect the high velocity of the incoming water vapor in a turbine environment. Our project design surmounts this shortcoming by rapidly heating water vapor through a 1 mm inner diameter platinum-rhodium tube (90% Pt / 10% Rh), causing it to expand quickly and achieve a velocity of ~200 m/s and temperature of ~1400 C, accurately mimicking a turbine environment. This steam jet will then be shot from a distance of ~2 mm at the SiC or EBC test sample over the course of a few days in increments of up to ~8-12 hours at a time. After a total of 24 hours of run time, the test sample will be removed and its surface will be scanned using either laser profilometry or atomic force microscopy to get an accurate measurement of the rate of surface ablation under accurate turbine conditions. At this time, water vapor is the only substance being used to wear down the test samples, but there are plans to incorporate other chemical species that are found in turbine environments in future iterations.

Project Design

The water for the steam jet is supplied from a Teledyne ISCO D-500 syringe pump, which is capable of dispensing water for prolonged periods of time with 0.01 mL/min precision. The water is then fed through an alumina-sand fluidized bed preheater which raises the water to superheated vapor at about 600℃. The water vapor is then fed into a platinum rhodium tube, capable of withstanding up to 1600℃, which then enters the pressure chamber. The pressure chamber is a 2’ diameter, ~3’ long cylindrical black steel vessel, with many sealed NPT fittings for air inlets and outlets, cooling water inlets and outlets, thermocouple wires, heating element power wires, and the test sample holder. Inside the chamber, the platinum-rhodium tube passes through three resistive heating elements to get up to the final temperature, and is then fired at the test sample from a distance of ~1 mm. The sample is at a 45° angle relative the steam jet to mimic the angle of turbine blades. A simplified drawing of the operation is shown below (fig. 2), as well as a close-up representation of the steam impinging on the test sample (fig. 3).

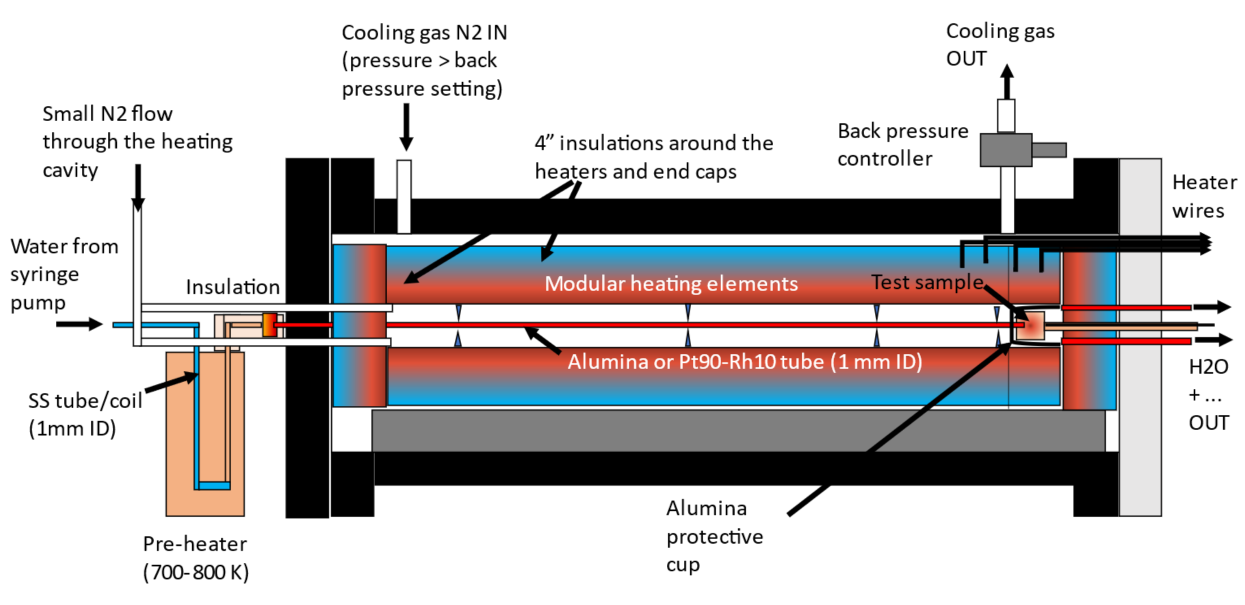

Figure 2 - Schematic showing the preheater and main heating chamber apparatus

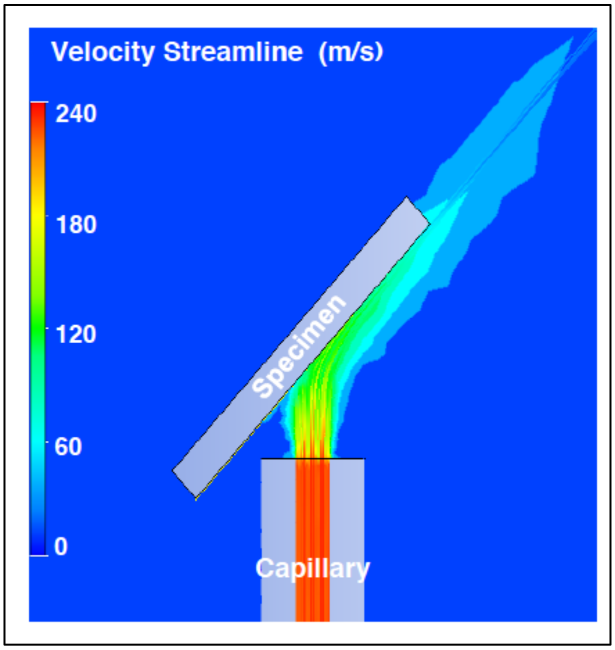

Figure 3 - CFD simulation showing impingement site of water vapor onto test specimen

Design Specifications and Equipment Overview

In order to accurately mimic a jet engine environment, the three critical design constraints for this project are:

- Temperature – The test sample and steam jet must be kept at a consistent 1400℃
- Velocity – The impinging steam jet must cover a range of 100 – 200 m/s, and the flow rate must be precisely controlled
- Pressure – The testing chamber must be air tight, and be able to hold a pressure of 75 psi

Figure 4 – 40 amp heater, ~9.5 inch OD

To achieve the 1400℃ temperature requirement, three resistive heating elements were used: two smaller, ~30 amp models and one larger, ~40 amp model, pictured below (figs. 4 and 5). Each heater is powered by a variable AC transformer which can be adjusted to control the current output based on the heater's demand. A b-type (platinum rhodium) thermocouple is inserted into each heating element and connected to a PID controller. The PID controller then reads the temperature and toggles a solid-state relay on and off to keep the temperature at the desired set value of 1400℃. Other thermocouples are also inserted to monitor the temperature at other locations, including at the exit of the steam jet, in the transfer line between the preheater and chamber, and at the roof of the chamber to ensure the outer walls don't overheat.

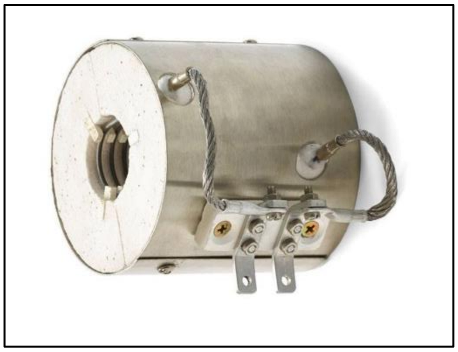

Figure 5 – 30 amp heater, ~4.5 inch OD

As mentioned previously, a Teledyne ISCO 500-D syringe pump is used to reach the 100-200 m/s steam jet velocity requirement. The syringe pump outputs at a constant mass flow rate, not velocity, but using the known pressure, temperature, diameter of the tube, and the ideal gas law, the mass flow rate required to reach a certain velocity can easily be determined. At atmospheric pressure the flow rate is 1.2 mL/min and at 75 psi it's 5.8 mL/min. An individual 500-D syringe pump has a capacity of 500 mL, which would last for about 1.5 hours at the maximum flow rate. Because run times of up to 24 hours were desired, two syringe pumps were used along with an auxiliary device that automatically switches and refills syringe pumps when one runs out. This allows the rig to be run over the course of a day without any operators present, except for the start up and shut down procedures.

Lastly, an industrial air compressor capable of delivering 95 psi at 15 cubic feet per minute was used to pressurize the chamber, provide diluting air around the outside of the heaters, and supply the 30 psi required by the preheater. As mentioned, the testing chamber has many NPT fittings threaded into the outside to provide access to the inner components, to measure the pressure inside, and for two shutoff valves and an emergency pressure relief valve (fig. 7). The two control valves can be opened or closed to reach the desired pressure, and to change the internal airflow so that the outside of the chamber stays cool. The end plate (fig. 6) is attached by bolts to the rest of the chamber, and can be removed to allow easy access to the heater and test sample location.

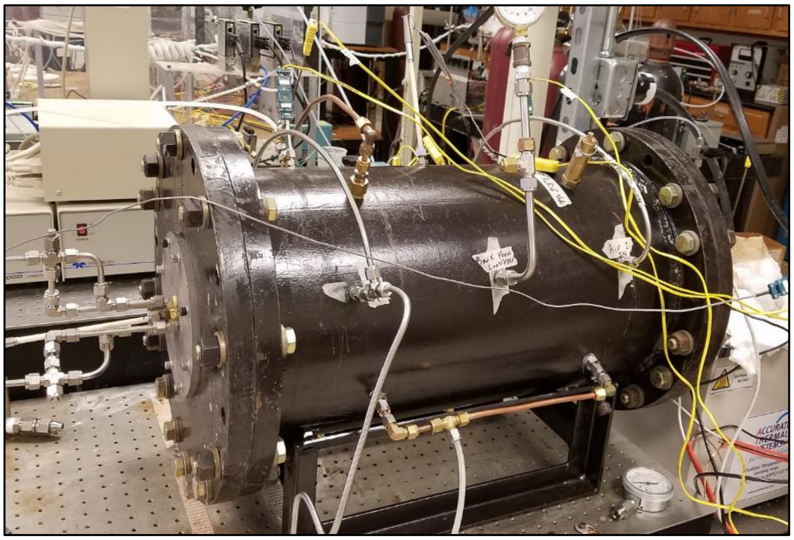

Figure 7 - External view of the pressure chamber

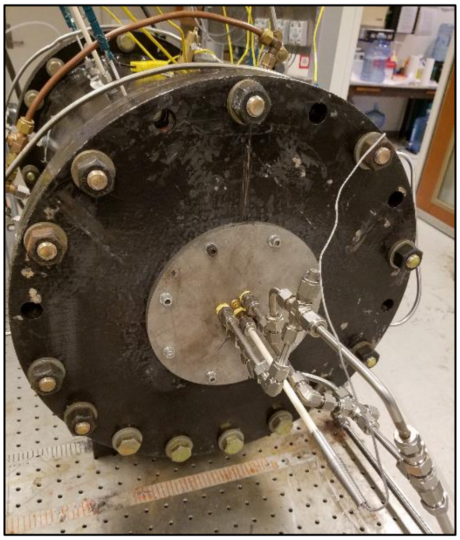

Figure 6 - Removable end plate on the pressure chamber with air and steam outlet pipes

Challenges with Prolonged Testing

The majority of the work on the project thus far has been on designing and building the chamber, testing out the heaters, pressure supply, syringe pump, and other equipment, and troubleshooting the many problems that have cropped up. Additionally, because many of the materials and products used are very expensive, and because of the extreme risks that come with using a high voltage supply and high temperatures, all aspects of the rig must be run multiple times in different conditions to ensure their safety. The project has only recently reached the stage where it can be safely left to run for ~12 hours without anyone present, and even still problems have arisen that required testing to stop and design changes to be implemented. Some of the major problems that have occurred include: the sample holder falling apart during testing, the outer chamber walls getting too hot and potentially melting wires, difficulties with positioning the sample so it's directly in front of the steam jet, the platinum rhodium tube expanding when heated and pressing against the sample, and the third heating element going over temperature (in excess of 1500°C) due to unwanted internal air flow patterns.

Originally, a "homemade" sample holder that was fabricated at Lacy Hall out of machinable alumina, a type of ceramic that can withstand high temperatures, and Sauereisen paste, a high temperature adhesive, was used for testing. There was some success in using these sample holders as the original design and machining process was improved upon, but the machinable alumina was prone to cracking and delamination, and generally didn't last very long. It was decided to get a new sample holder produced externally that is made out of much less porous alumina and though the new holders haven't been tested yet, they are clearly much more durable than the "homemade" ones.

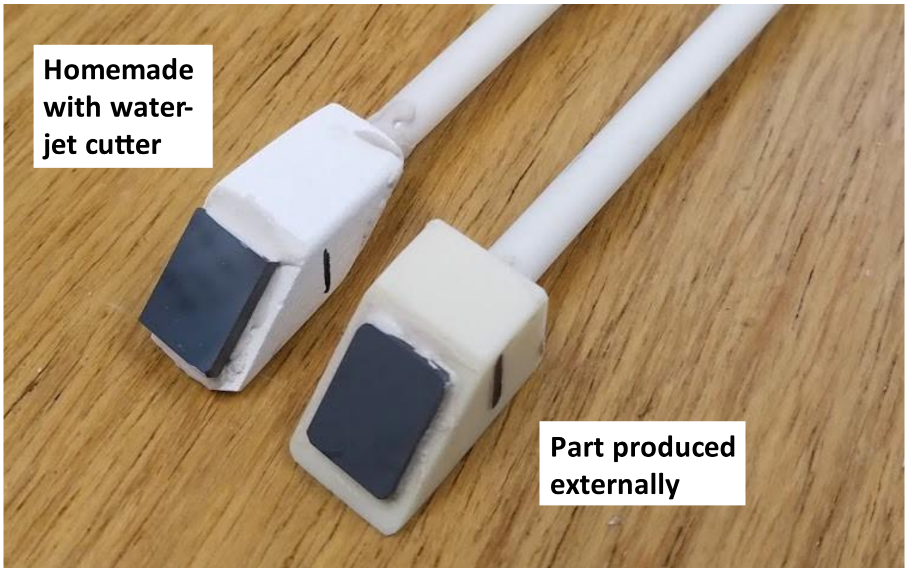

Figure 8 - Two sample holders with CMC test samples cemented onto them

Conclusion

Because this project is still in underway, no results can be shared at this time. That said, the rig has proven its capability in running unmonitored for intervals up to 12 hours. Currently, design work is in progress to add additional cooling methods to the inside of the chamber to prevent the chamber walls from overheating, and to implement a real-time monitoring system using a microcontroller to automatically shut down the rig safely if the pressure, temperature, water flow, or current flow varies outside of acceptable bounds. With the few remaining kinks worked out and additional safety mechanisms put in place, the apparatus will be well on its way to producing thousands of hours of runtime and hundreds of different specimen profiles.

References

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