

## **C/SiC LIFE PREDICTION FOR PROPULSION APPLICATIONS**

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### **ABSTRACT**

Accurate life prediction is critical to successful use of ceramic matrix composites (CMC). The tools to accomplish this are immature and not oriented toward the behavior of carbon fiber reinforced silicon carbide (C/SiC), the primary system of interest for many reusable and single mission launch vehicle propulsion and airframe applications. This paper describes an approach and progress made to satisfy the need to develop an integrated life prediction system that addresses mechanical durability and environmental degradation of C/SiC. Issues such as oxidation, steam and hydrogen effects on material behavior are discussed. Preliminary tests indicate that steam will aggressively remove SiC seal coat and matrix in line with past experience. The kinetics of water vapor reaction with carbon fibers is negligible at 600°C, but comparable to air attack at 1200°C. The mitigating effect of steam observed in fiber oxidation studies has also been observed in stress rupture tests. Detailed microscopy of oxidized specimens is being carried out to develop the oxidation model. Carbon oxidation kinetics are reaction controlled at intermediate temperatures and diffusion controlled at high temperatures (~1000°C). Activation energies for T-300 and interface pyrolytic carbon were determined as key inputs to the oxidation model. Crack opening as a function of temperature and stress was calculated. Mechanical property tests to develop and verify the probabilistic life model are very encouraging except for residual strength prediction. Gage width is a key variable governing edge oxidation of seal coated specimens. Future efforts will include architectural effects, enhanced coatings, biaxial tests, and LCF. Modeling will need to account for combined effects.

# C/SiC Life Prediction For Propulsion Applications

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27th JANNAF Airbreathing Propulsion Subcommittee Meeting

December 1-5, 2003

Colorado Springs, CO



## OUTLINE

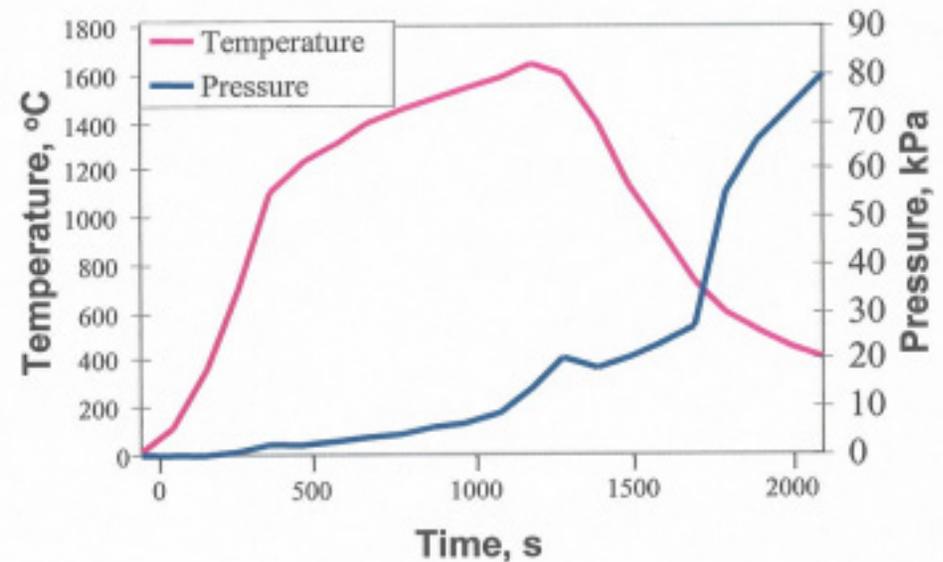
- ◆ Introduction
- ◆ Oxidation Model
- ◆ Stress Rupture
  - Gage width
  - Enhanced materials
  - Effect of Environment
- ◆ Probabilistic Model
- ◆ Steam Effects on SiC Seal Coat and Matrix
- ◆ Concluding Remarks

## PROPULSION

- ◆ High temperatures (~ 3500R)
  - Low and intermediate temperatures can also be a problem
- ◆ High pressures (e.g. to ~ 6000psi)
- ◆ Severe chemical environments
  - Steam
  - Oxygen rich or fuel rich
  - Hydrogen
- ◆ High velocity
- ◆ Exposure cycles from minutes in rockets to ~ hours in some combined cycle approaches
- ◆ Severe thermal transients and gradients

## AIRFRAME

**X-38 Reentry Profile for Body Flap Windward Surface Location**



# Task Objectives

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◆ **Primary goal:**

- Develop and verify a robust methodology for confident determination of the reusable life capability of C/SiC space propulsion hardware.

◆ **Secondary goals:**

- To ground the methodology with mechanism-based descriptions of mechanically and environmentally induced damage.
- To expand the database for C/SiC.
- To directly support flight experiments which use CMC propulsion components.

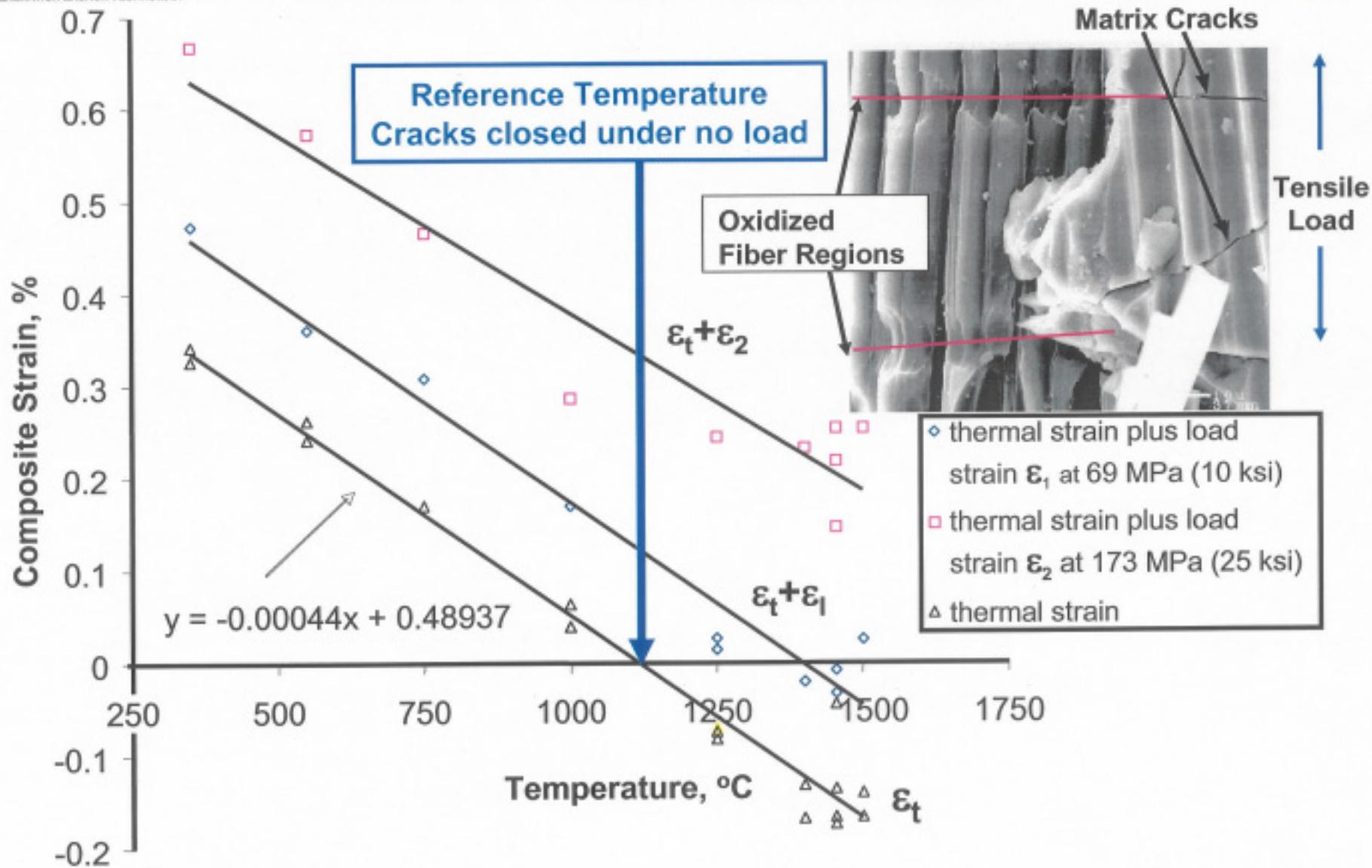
- ◆ **Plain weave C/SiC with seal coat**
- ◆ **Enhanced with seal coat**
  - **plain weave**
  - **5 harness satin weave**
  - **w/wo CBS coating**

## C/SiC Life Controlled by Complex, Interactive Mechanisms

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- ◆ Environmental
  - surface recession due to moisture
  - interface and fiber oxidation
- ◆ Mechanical
  - strains due to thermal and mechanical loads
  - cycling of loads (LCF, HCF)
  - creep

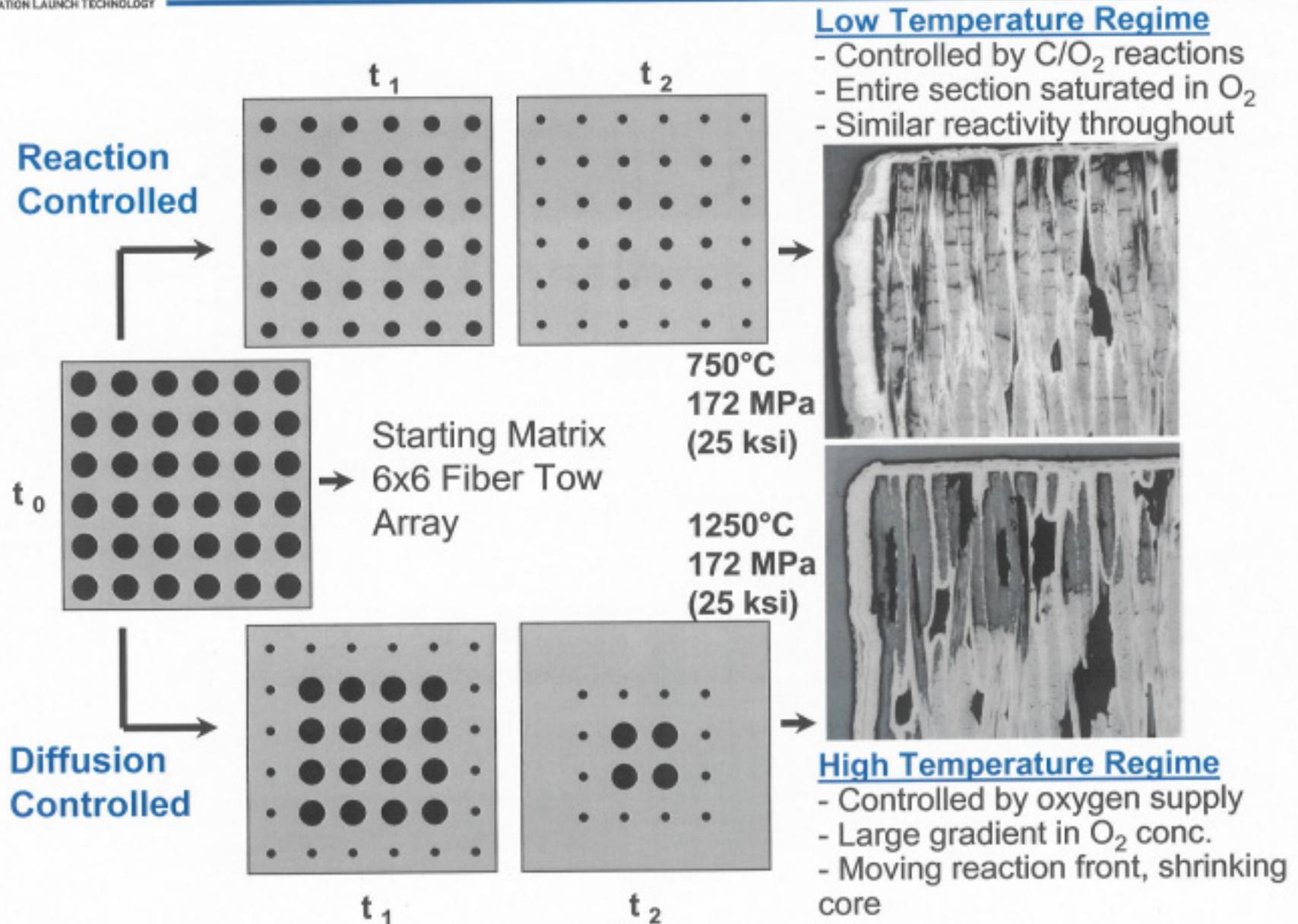
# Crack Opening Determined by Load and Thermal Strain



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- ◆ Introduction
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# Temperature Dependent Carbon Fiber Oxidation Mechanism



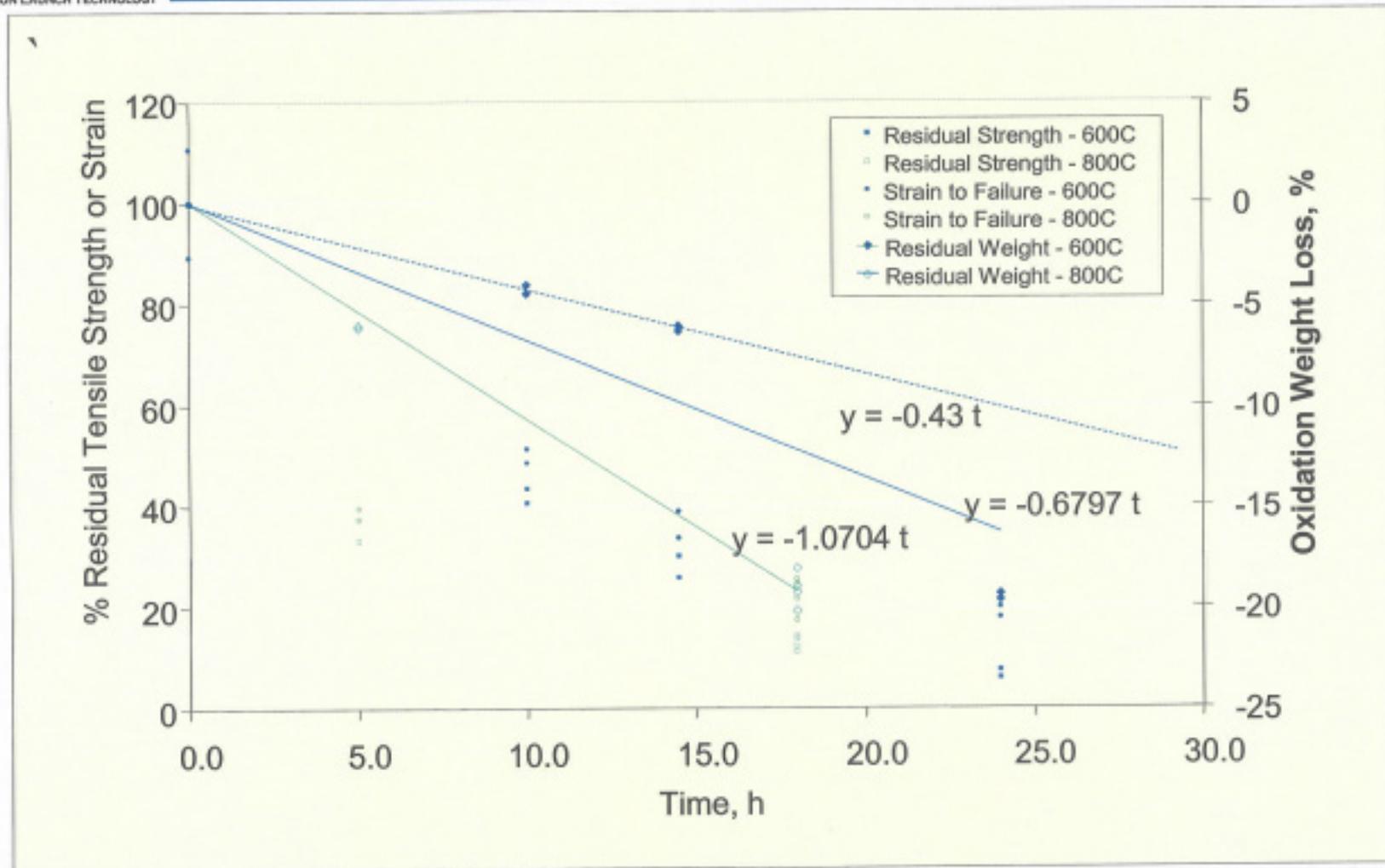
## Status of the Oxidation Model

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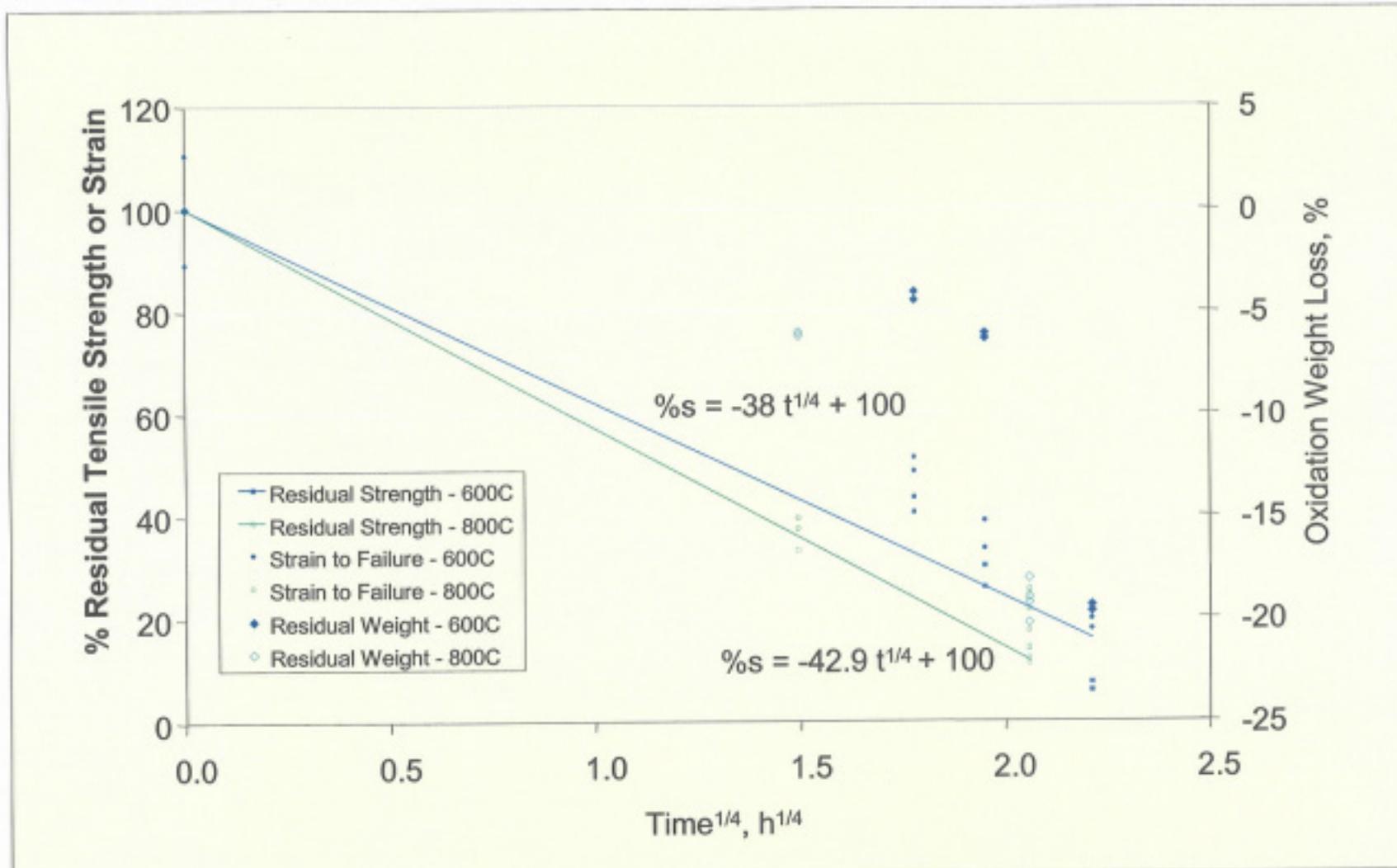
FY'03 effort:

- ◆ Activation energy and a pre-exponential constant for the Arrhenius reaction rate equation were obtained.
  - Thermogravimetric analysis (TGA) tests were conducted for T-300 carbon fiber in flowing oxygen focusing on 6 temperatures in the reaction controlled regime (450°C - 600°C).
  - Values are used as inputs into the model.
- ◆ Strength reduction correlated with a quantified loss of carbon.
  - 12 C/SiC tensile coupons were exposed to furnace oxidation exposures in air at temperatures of 600°C and 800°C.
  - Coupons were allowed to oxidize until 4-20% composite weight loss.
  - Residual tensile strength tests were conducted.
- ◆ Oxidation Model has been optimized.
  - More accurate inputs: geometry and tortuosity factors to match tensile coupons, to allow for different edge effects, and oxygen ingress into individual carbon fiber tows.
  - Current efforts focus on matching the model to the experimental results.

# Residual Composite Properties and Weight Loss Due to Oxidation



Reaction controlled model predicts greater differences between 600 and 800°C weight loss results than observed.



Exponent of 0.25 fits strength loss data, but rate constant difference is smaller than expected from model.

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## Summary of Mechanical Testing of C/SiC in Support of Life Model Development

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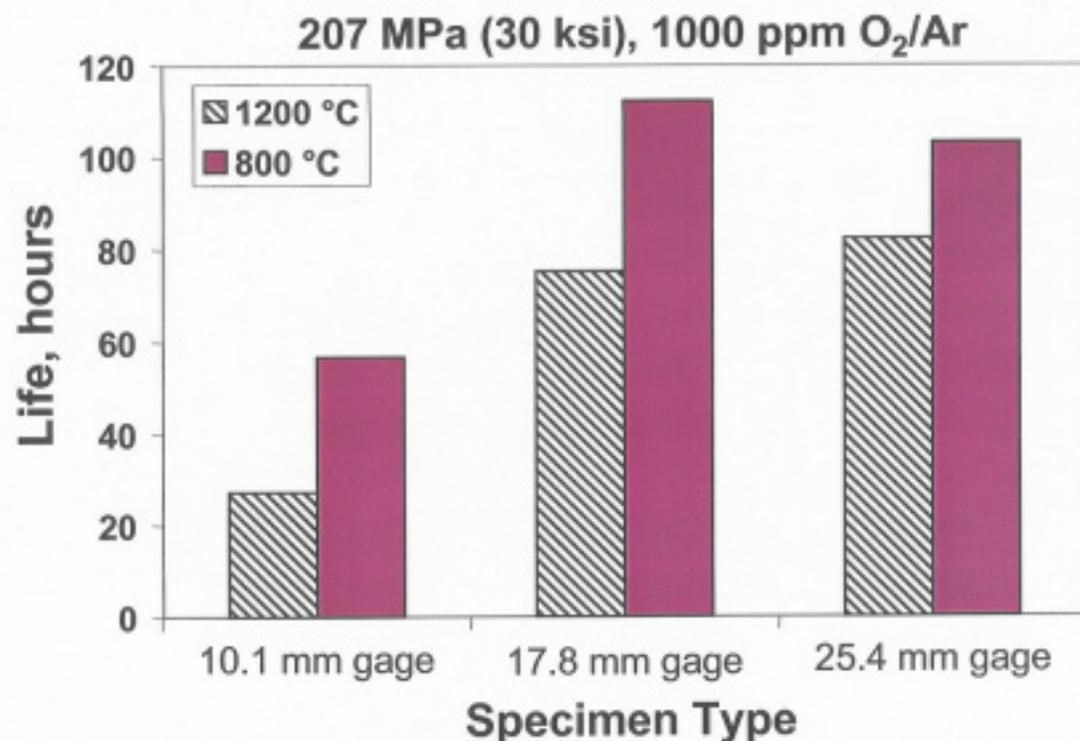
### Tests performed to date on plain weave C/SiC

- ◆ Stress-rupture tests under various environments (steam, air, vacuum).
- ◆ Rupture and tensile tests at 800 and 1200°C:
  - Twenty tensile tests at in a reduced partial pressure of oxygen.
  - Twenty-six stress-rupture tests in a reduced partial pressure of oxygen.
  - Twenty stress-rupture tests at a single stress.
  - Twenty interrupted stress-rupture tests to measure residual strength.
  - Stress-rupture testing of wide specimens to assess dependency of specimen area on life.

### Testing under way or completed in CY 2003

- ◆ Test thick specimens under rupture conditions
  - Quantify specimen volume effects in material that is 2X thicker than material tested to date.
- ◆ Testing of Enhanced C/SiC and CBS coated materials and 5 HS weave Enhanced C/SiC.
  - Compare to standard C/SiC.
  - Determine rupture behavior of Enhanced C/SiC and CBS-coated materials in different environments (air, steam, 1000ppm O<sub>2</sub>/Ar).

# Stress Rupture Life Is a Function of Specimen Width for C/SiC




**standard**  
(10.1 mm wide gage)



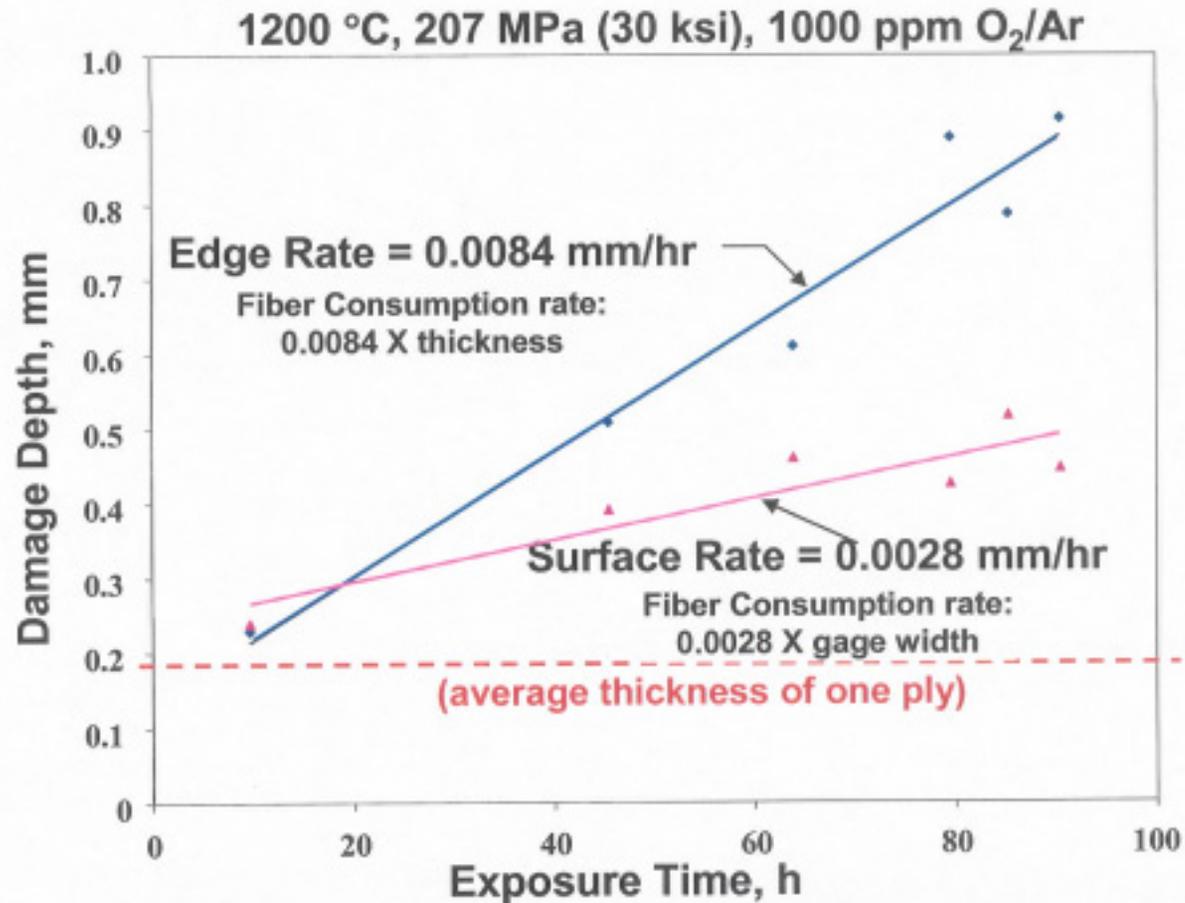
**chubby**  
(17.8 mm wide gage)



**extra chubby**  
(25.4 mm wide gage)

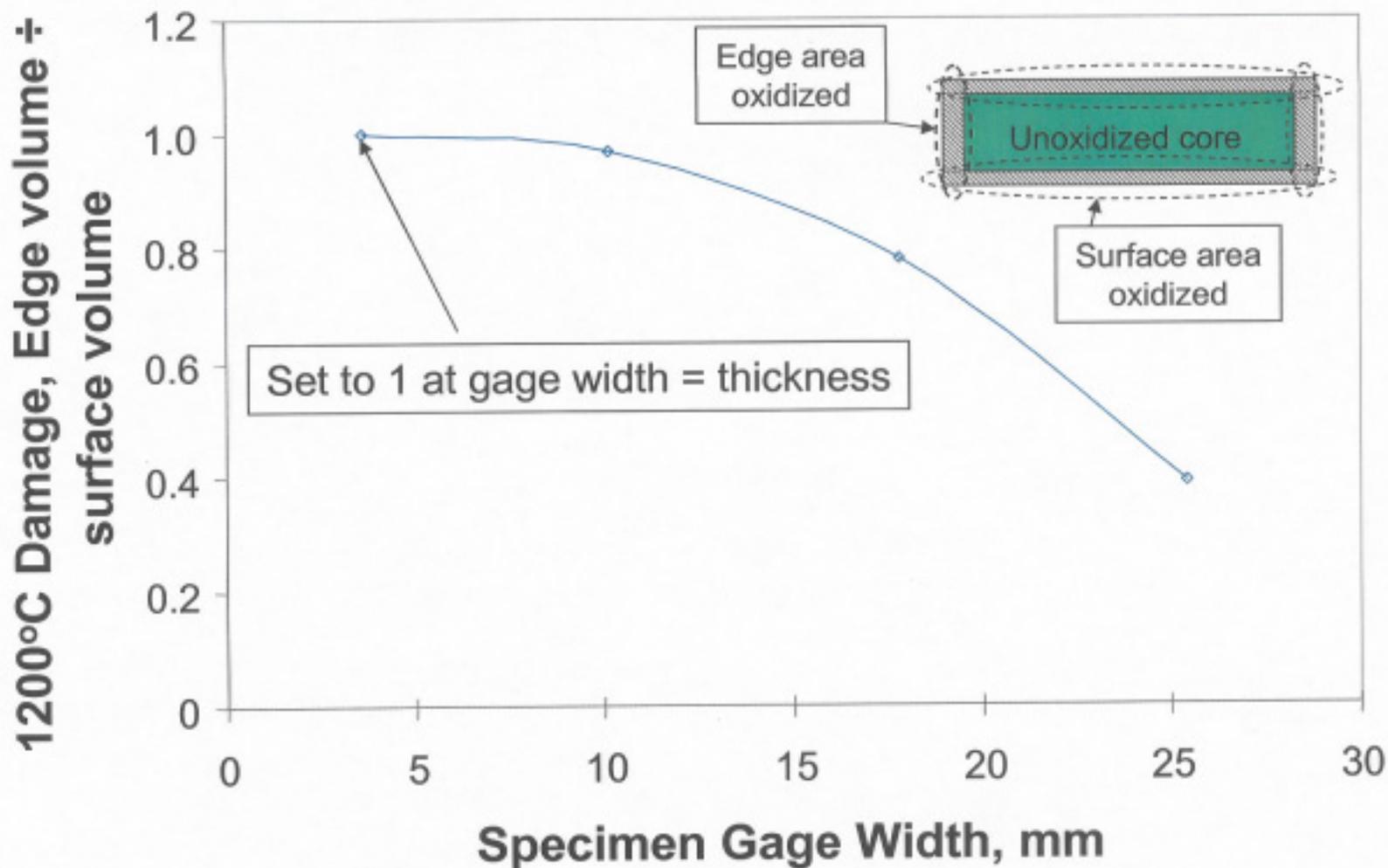
- ◆ Material volume effects need to be incorporated in life prediction models.
- ◆ Average life at 800°C is about 1.5 times longer than at 1200°C.
- ◆ 17.8 mm wide specimens resulted in about a 2.5 increase in life compared to 10.1 mm wide specimens.

# C/SiC Oxidation Damage Rates



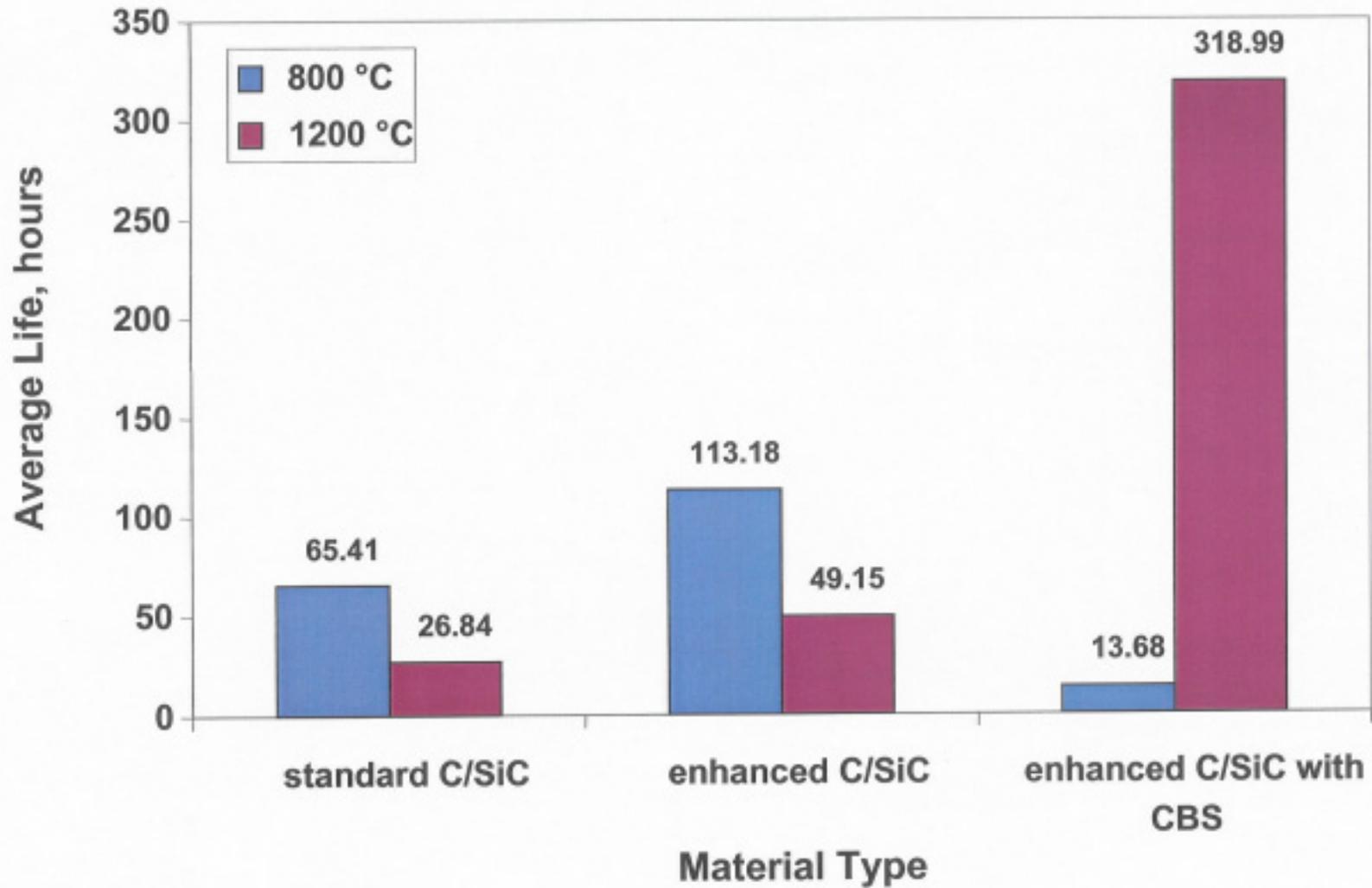
- ◆ Surface damage penetration rate is 1/3 edge penetration rate
- ◆ Highest volume of carbon fiber is consumed by surface penetration

# Edge Damage Effects Insignificant for Large Panels

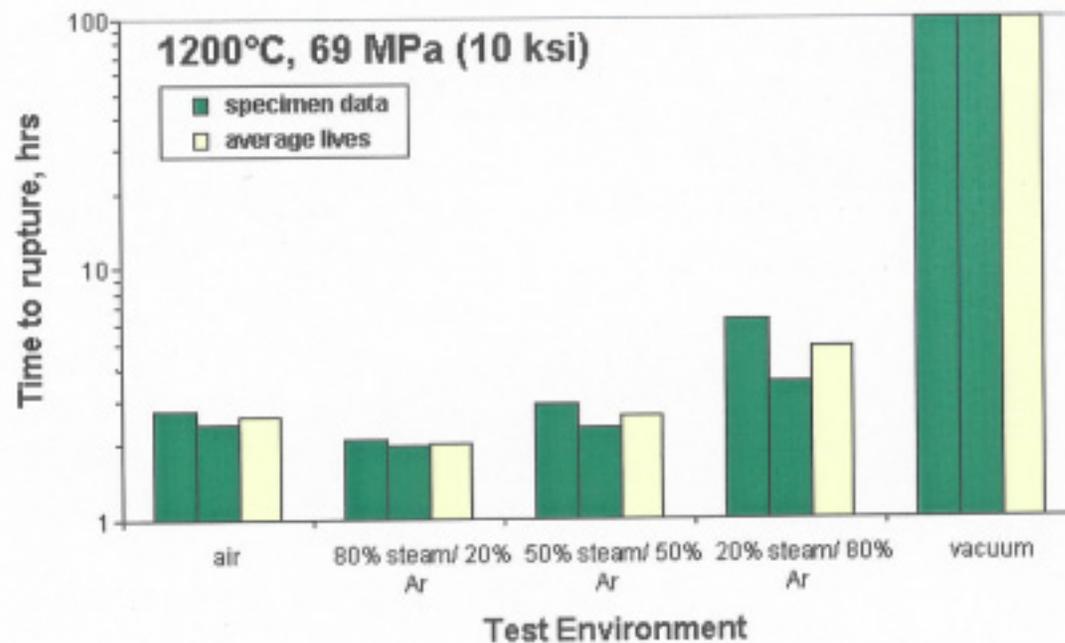
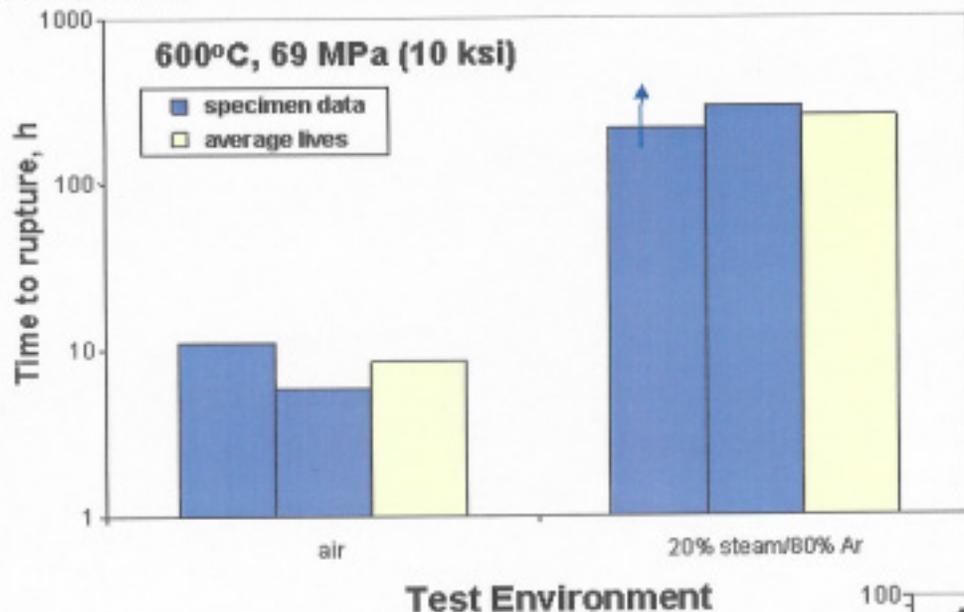


# Stress Rupture Lives of Various C/SiC Materials

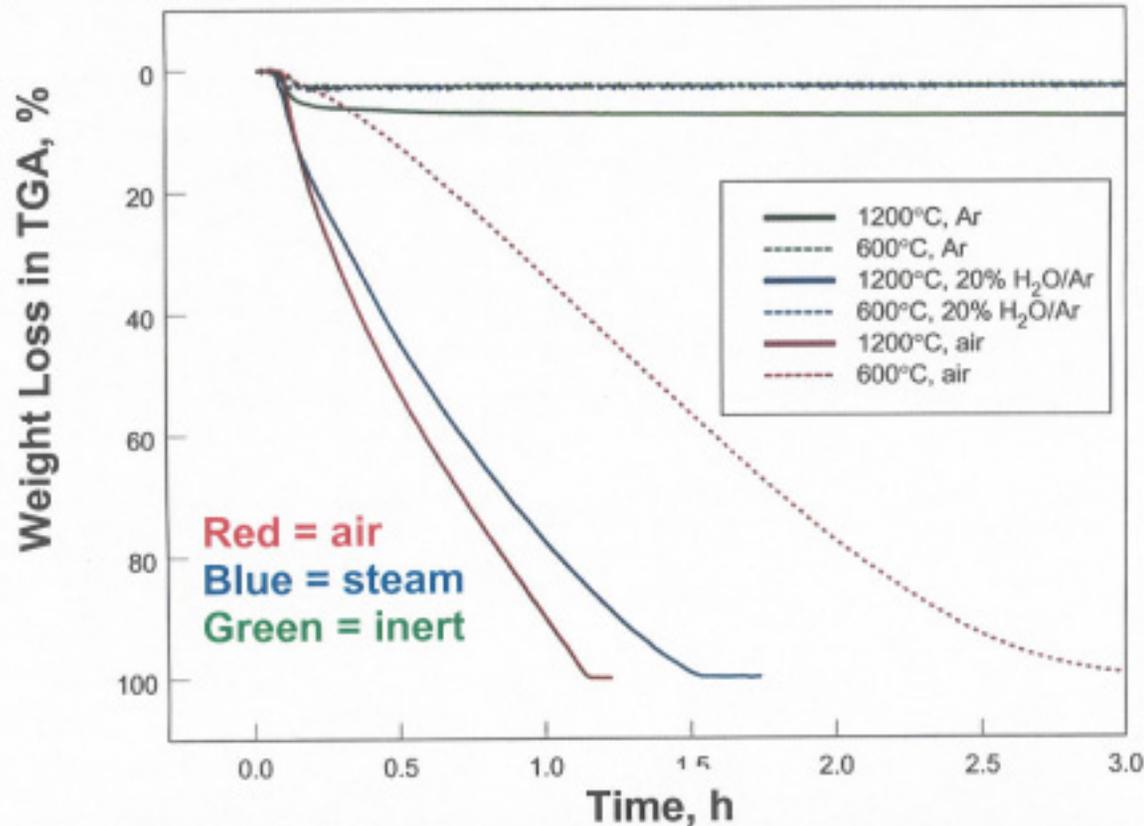
(stress = 207 MPa (30 ksi), 1000 ppm O<sub>2</sub>/Ar)



# Effect of Environment on Stress Rupture of C/SiC



# T-300 Carbon Fiber Oxidation Kinetics

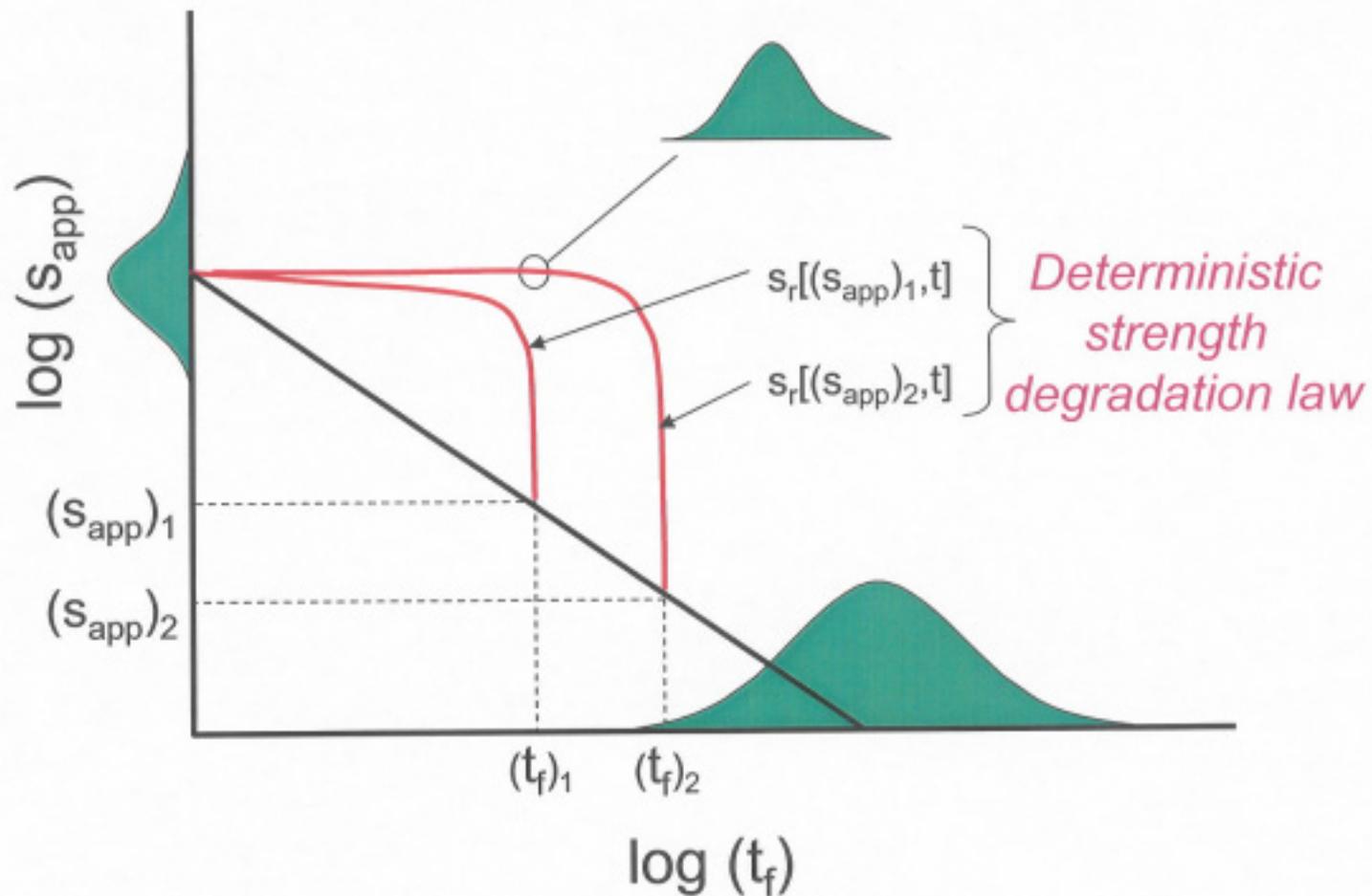


- ◆ Weight loss in inert environment (argon) is negligible.
- ◆ At 1200°C, carbon fiber weight loss kinetics in air and steam due to oxidation are rapid; fibers are consumed in 1.5 hours.
- ◆ At 600°C in steam, weight loss is negligible, while in air, rapid weight loss occurs.

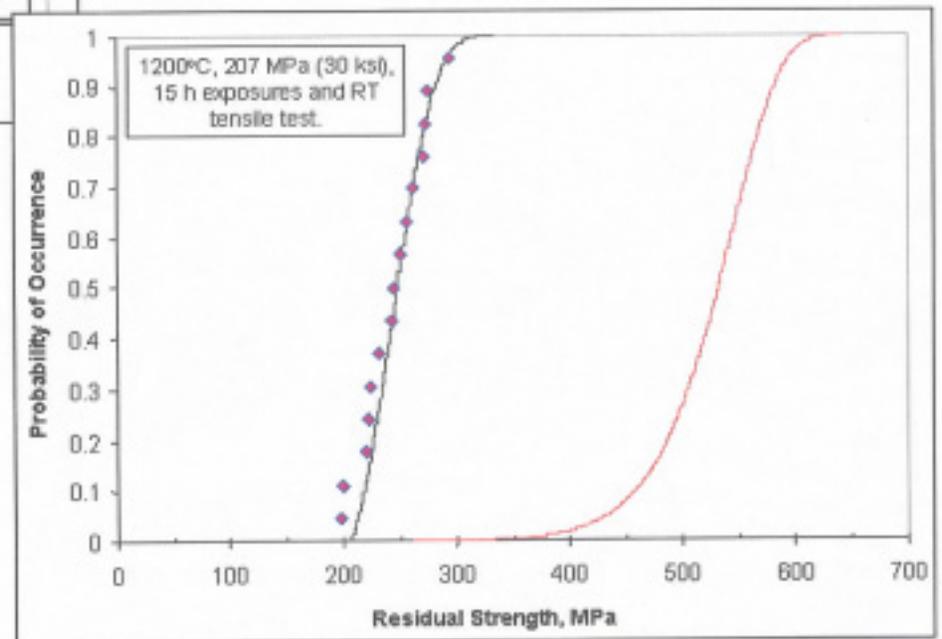
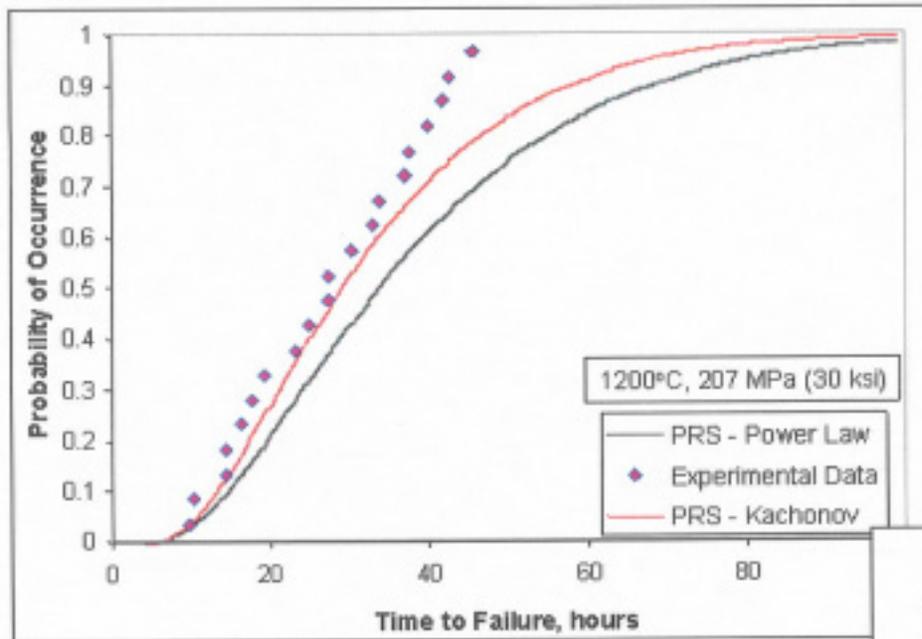
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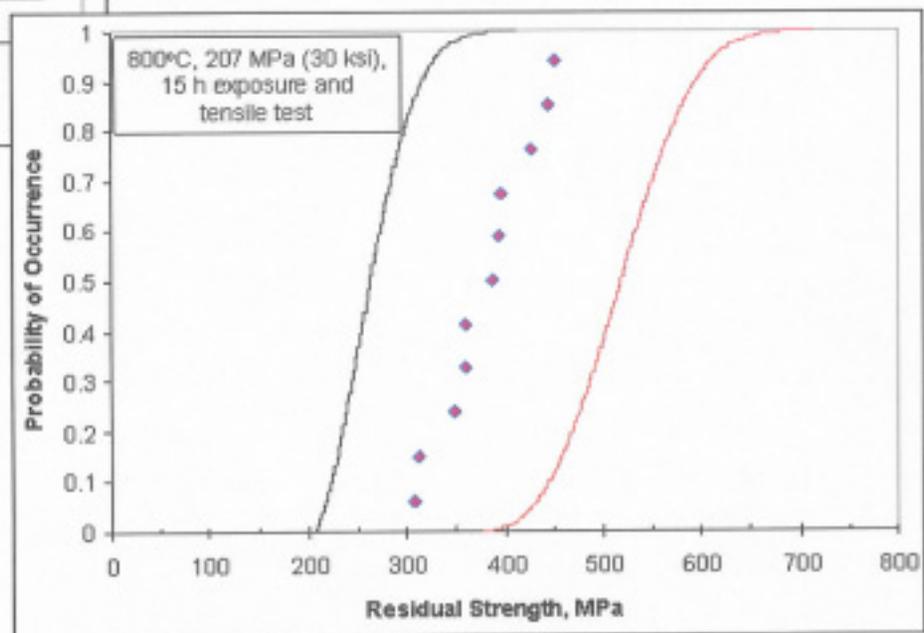
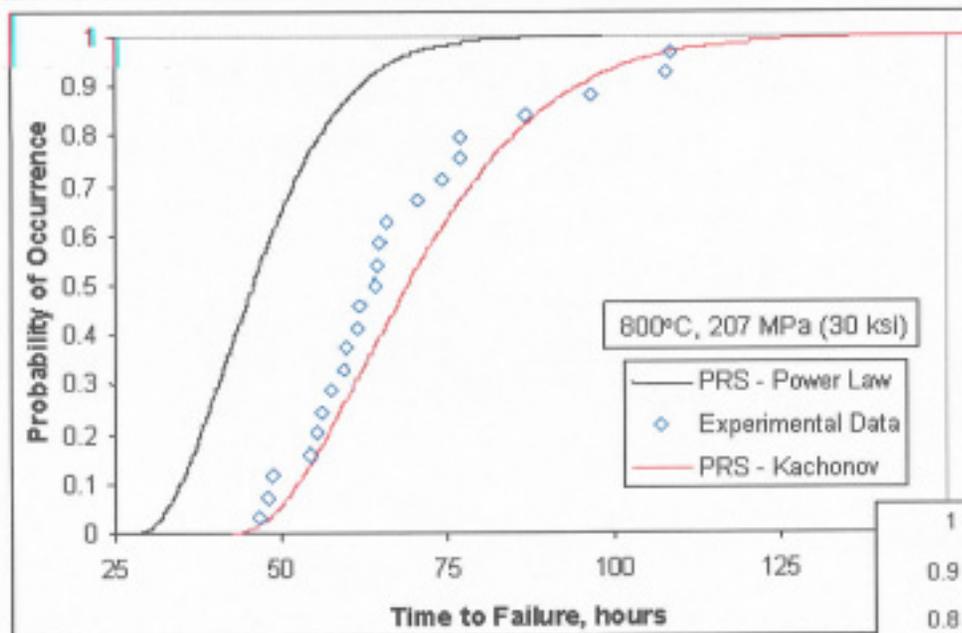
# Probabilistic Residual Strength (PRS) Modeling Approach



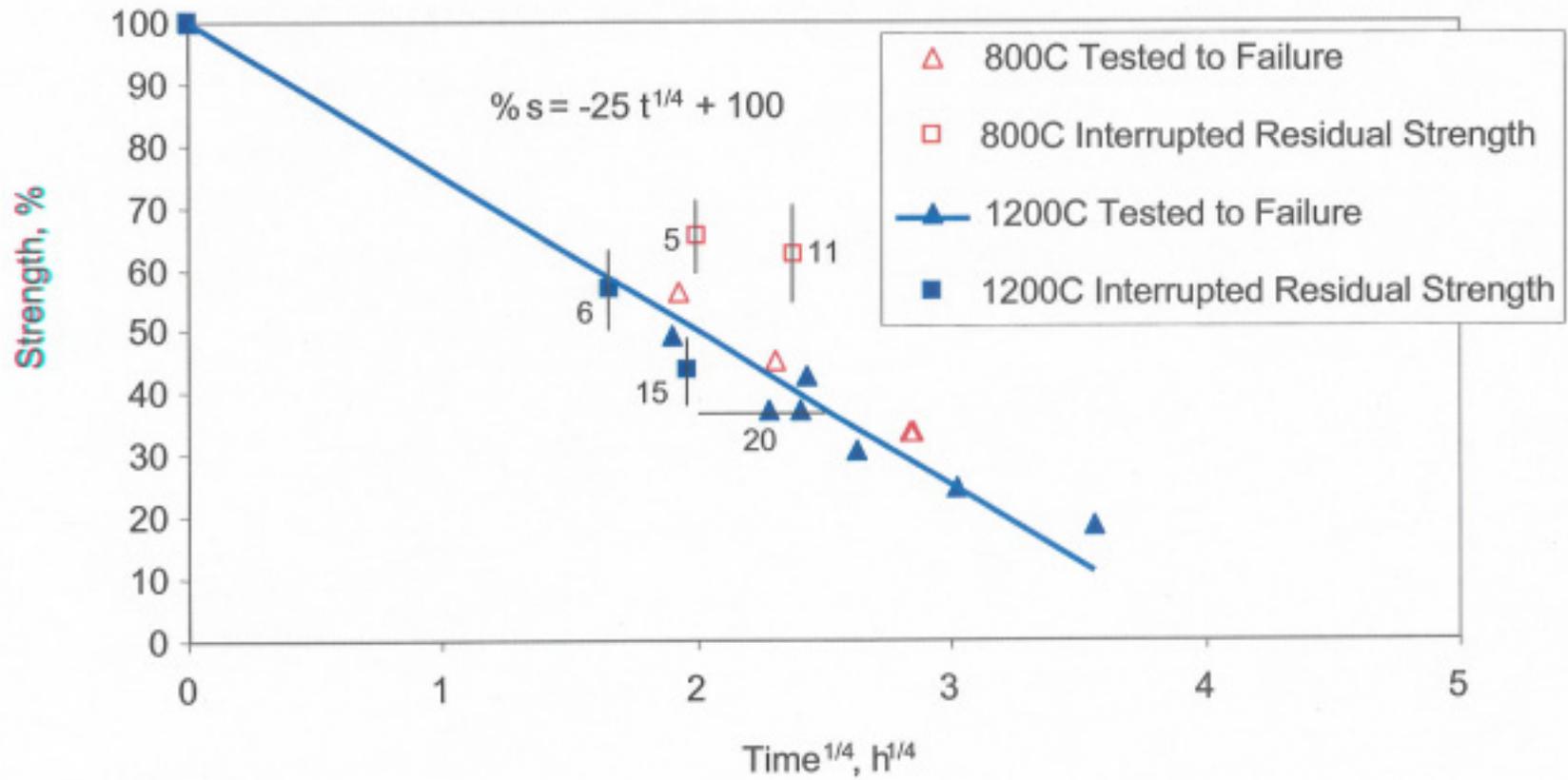
# PRS Model Predicts Behavior at 1200°C



# PRS Model Predictions at 800°C



# Power Law Behavior for Stress Rupture Strength and Residual Strength



Exponent of 0.25 fits stress rupture and residual strength loss data

## OUTLINE

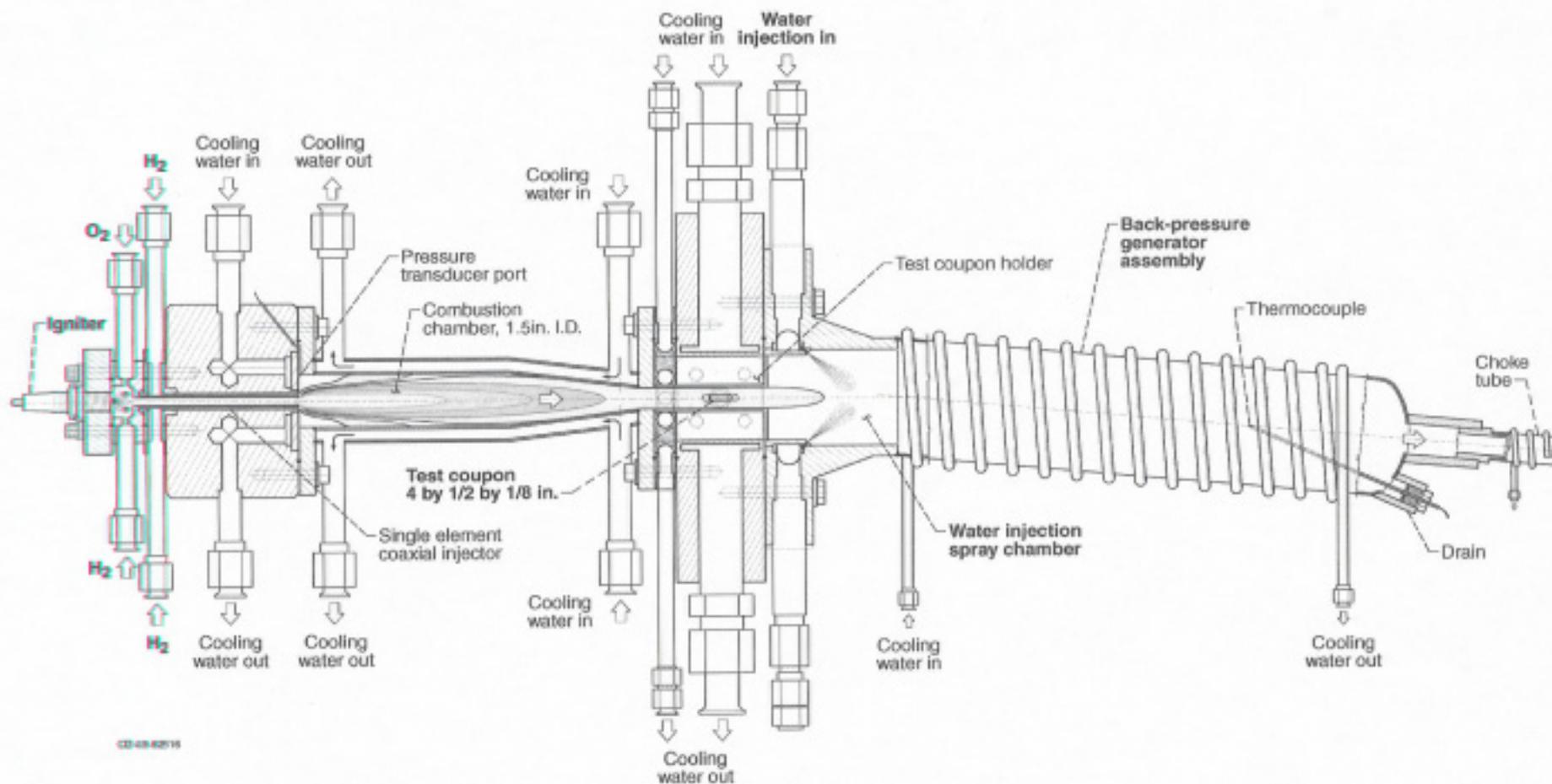
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## Status of Steam Environment Model

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- ◆ Objective: Determine SiC recession rate as a function of P, T, gas velocity, gas chemistry
  - 3 O/F's between 1 and 2
  - 3 pressures: 100, 500, 1000 psi
  - Thin film thermocoupled samples
  - Gas velocity ~ 180 m/s (600 ft/s)
  - Weight, recession measured at intervals for a total exposure time of up to 1 hour at each condition.
- ◆ FY'03: Studied recession of SiC coated C/SiC under simulated rocket engine environments.
- ◆ FY'04:
  - Complete recession study
  - Compare results to SiC recession model predictions developed for aircraft engine applications

## Subsonic High-pressure Coupon Test Configuration Used for Determination Of SiC Recession Due to Moisture Generated by Combustion of H<sub>2</sub> and O<sub>2</sub>



## SiC Coated C/SiC After Exposure to H<sub>2</sub> / O<sub>2</sub> Combustion

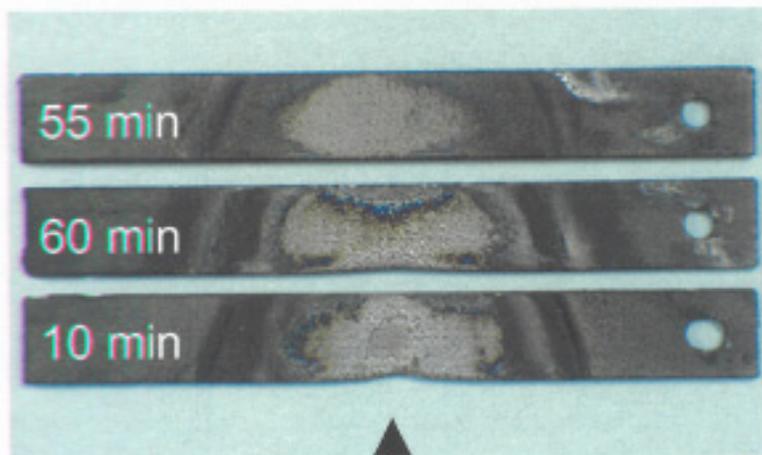
0.625 mm (25 mil) thick SiC seal coat

Total pressure: 6.8 atm

Gas velocity: ~ 180 m/sec

100 psi

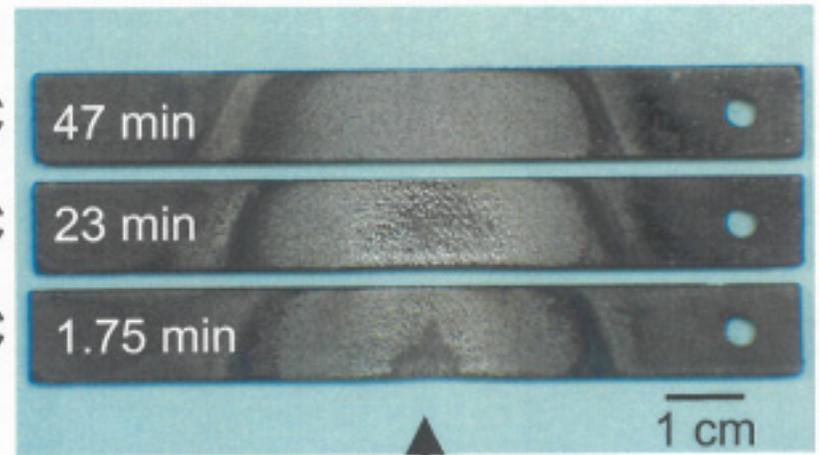
500 psi



1210°C

1380°C

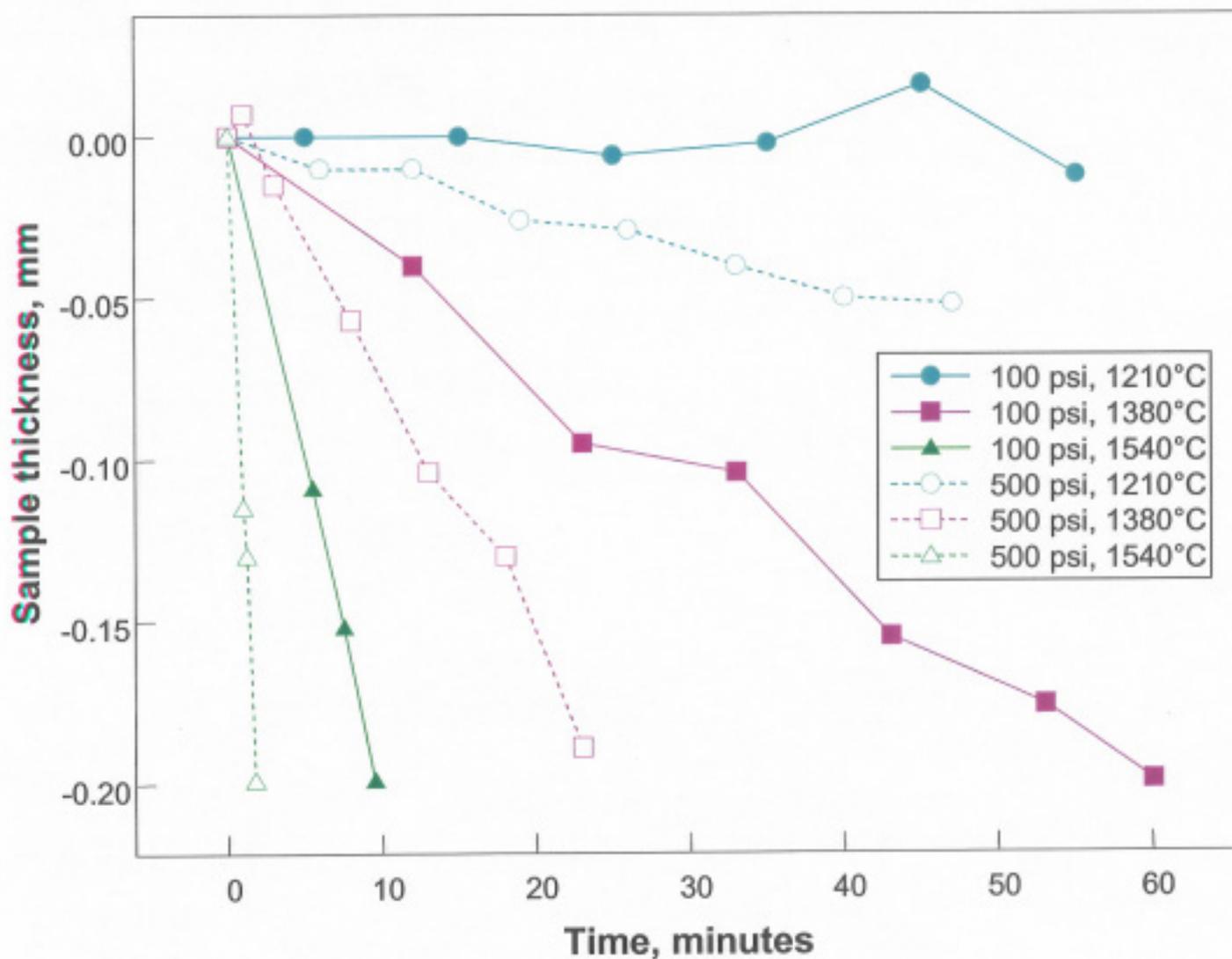
1540°C



↑  
flow direction



## SiC Coated C/SiC Recession Measured As Thickness at Leading Edge After Specimen Exposure to Products of H<sub>2</sub> / O<sub>2</sub> Combustion



## Concluding Remarks

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- ◆ **Fiber oxidation dominates C/SiC behavior**
- ◆ **Stress makes fibers accessible**
- ◆ **Edge effects diminish as specimen width increases**
- ◆ **Coatings and inhibitors greatly extend life**
- ◆ **Water vapor oxidizes carbon like air at high temperature, but is less aggressive at low temperature**
- ◆ **Water vapor strips protective silica scale rapidly at high temperature and water vapor partial pressure. Environmental barrier coating is required.**
- ◆ **Much more effort will be required to develop a physics based life prediction model, and the physics will be unique to a given set of fiber coating, matrix, and external coating constituents**

# Acknowledgements

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The authors thank

- ◆ John Zima for conducting the mechanical tests
- ◆ Dr. John R. Ellis for his many helpful comments during the life of this effort