

Material Response Characterization of Low Density Carbon-Phenolic Ablators in High-Enthalpy Plasma Flows

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Vrije
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Brussel

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Outline

- 1 Introduction
- 2 Ground testing in Plasmatron facility
- 3 Results: Ground Testing
- 4 Numerical Tools Development
- 5 Application to Mission Design
- 6 Conclusions and Perspective

Outline

- 1 Introduction
 - Motivation
 - Problem Statement
 - Research Goals
- 2 Ground testing in Plasmatron facility
- 3 Results: Ground Testing
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Ablative Thermal Protection Systems (TPS)

Start of space flight
High-speed reentries



*Apollo 10 capsule
(May 26, 1969)*

→ until today



*Soyuz capsule
(1967-today)*

All European missions:
ablative heat shields



*Atm. Reentry Demonstrator
(1998)*

Ablative Thermal Protection Systems (TPS)

Start of space flight
High-speed reentries



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(May 26, 1969)*

→ until today



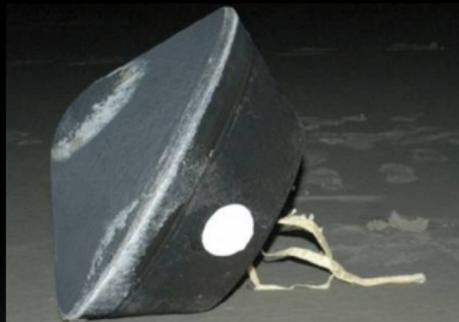
*Soyuz capsule
(1967-today)*

Future: Sample returns
High-speed reentries



*Mars Science Laboratory (2012)
Courtesy: NASA*

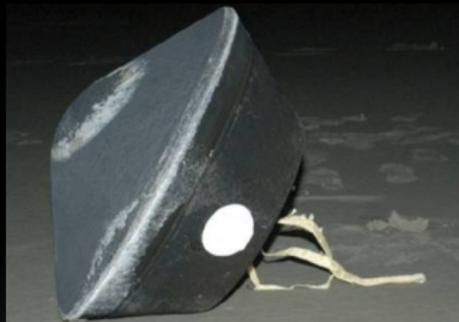
New Porous, Lightweight Ablators



Stardust probe (2006, 12.9 km/s, [1])

- New low weight materials (PICA, ASTERM) [2, 3]
- New missions (Asteroid / Mars sample return)

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Modeling tools inherited from Apollo program (1960s) [4]

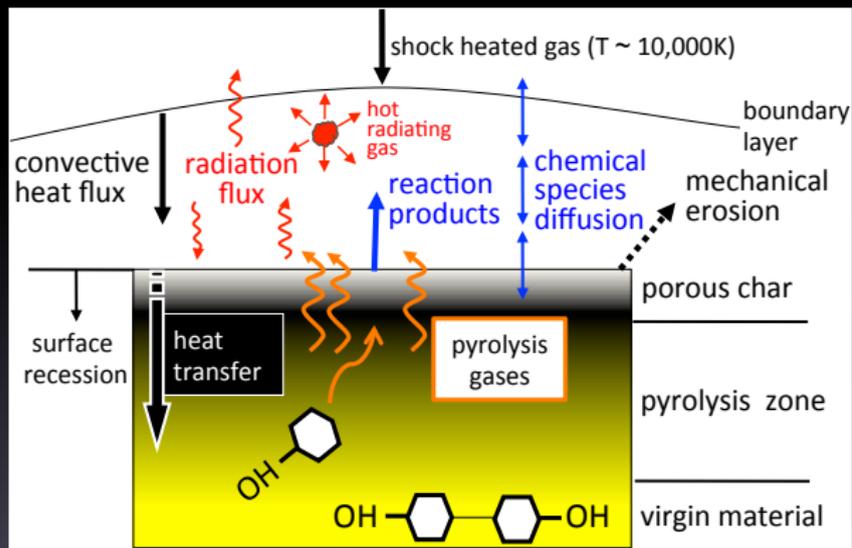
- new material response models [5]
- qualification of materials & validation of models required [6]

Complex Multiphysics - Multiscale Problem

Radiative and convective heating



Pyrolysis of phenolic resin
 C_6H_5-OH ($>200^\circ C$)



Complex Multiphysics - Multiscale Problem

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 C_6H_5-OH ($>200^\circ C$)

Ablation

Chemical mechanisms

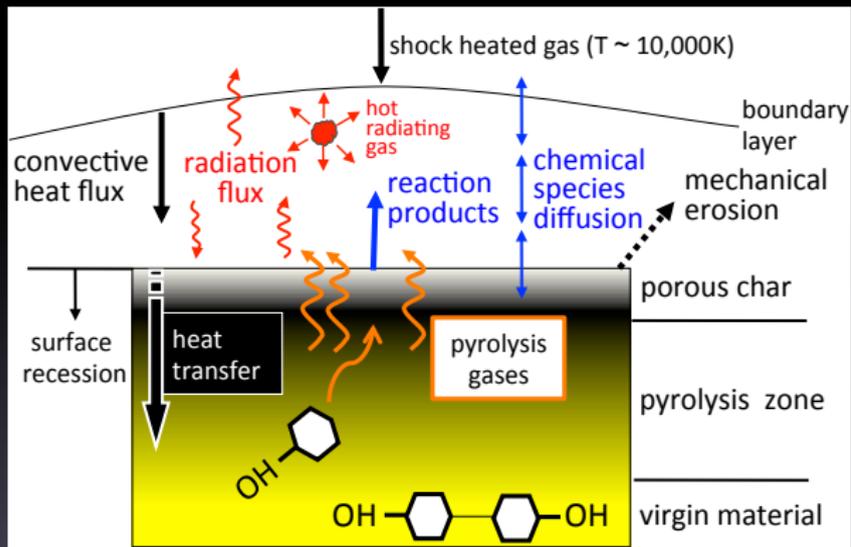
oxidation (CO , CO_2),
nitridation (CN)

Phase changes

melting,
sublimation (C , C_2 , C_3)

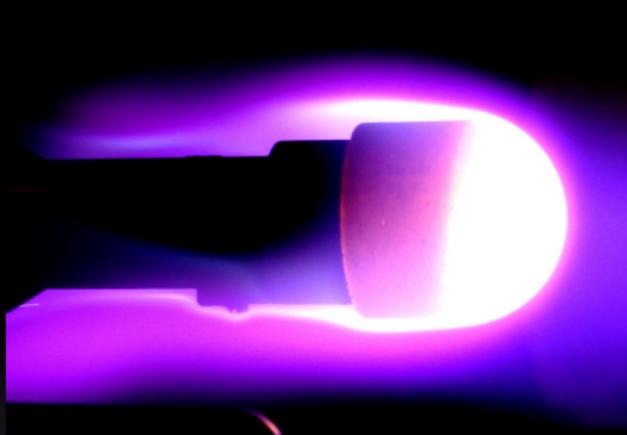
Mechanical removal

spallation, shear stress,
melt removal

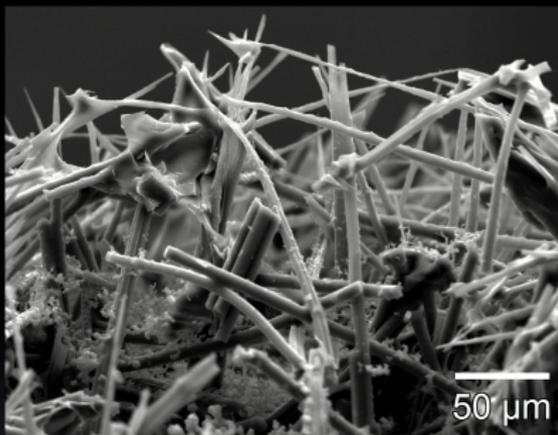


Complex Multiphysics - Multiscale Problem

Research Strategy and Objectives



VKI: Analysis in High-Enthalpy Plasma Flows

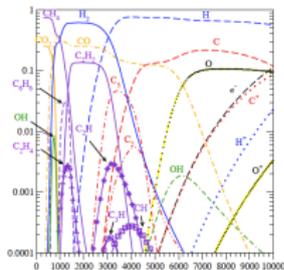


VUB: Multiscale Characterization

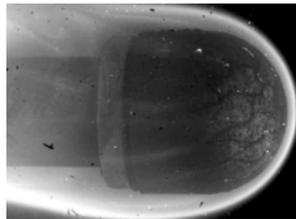
- Gas phase:
 - pyrolysis gas chemistry
 - transport phenomena & radiation in the boundary layer
- Material:
 - thermal performance and internal degradation
 - char ablation zone and degradation of carbon fibers

Research Frame and Goals

- ⇒ Methodology to characterize material response & gas-gas / gas-surface interaction of innovative ablators
- ⇒ Model validation and flight extrapolation



**PHYSICO-CHEMICAL
MODELS**



EXPERIMENTAL DATA



**COMPUTATIONAL
METHODS**



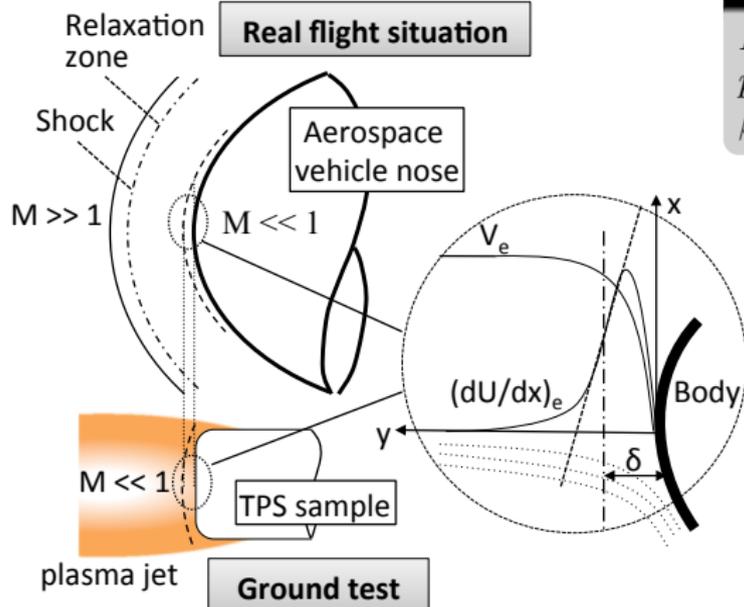
⇒ Combining basic ingredients for prediction in aerospace science

Outline

- 1 Introduction
- 2 Ground testing in Plasmatron facility
 - Local Heat Transfer Simulation: LHTS
 - Plasmatron Facility
 - Measurement Techniques
 - Materials of Investigation
- 3 Results: Ground Testing
- 4 Numerical Tools Development
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Local Heat Transfer Simulation (LHTS)

- Plasmatron design based on LHTS methodology
- Well characterized plasma flow through numerical-experimental procedure



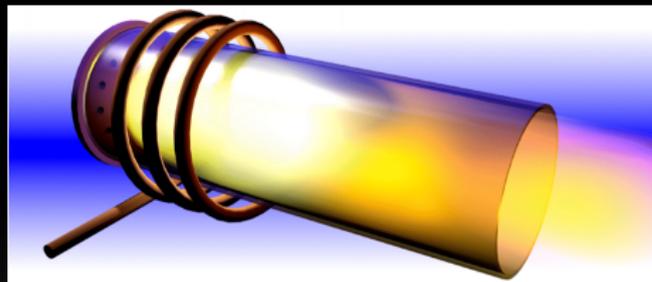
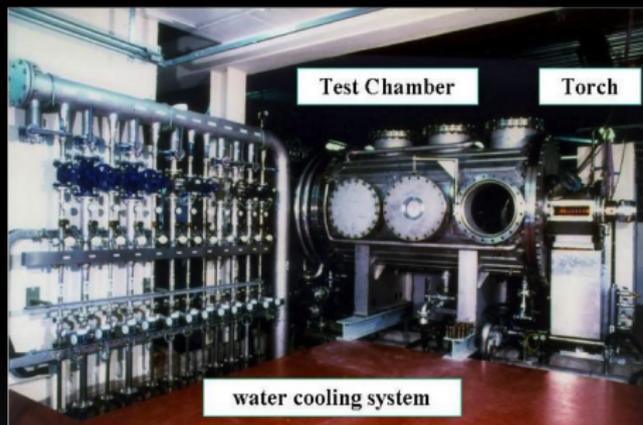
Stgn.pt. heat flux similarity[7]:

$$H_f = H_{exp},$$

$$p_f = p_{exp},$$

$$\beta_f = \beta_{exp}, \beta = (dU/dx)_e$$

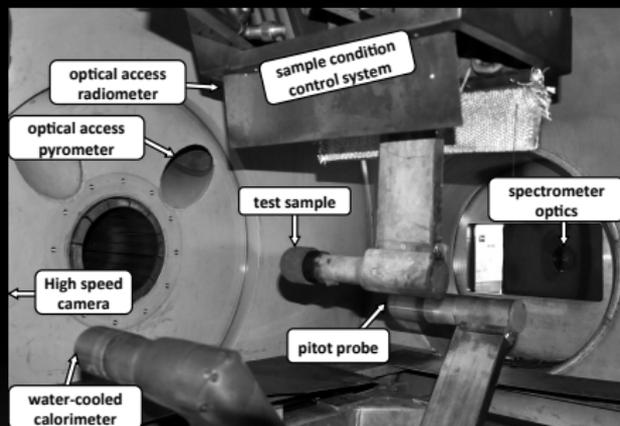
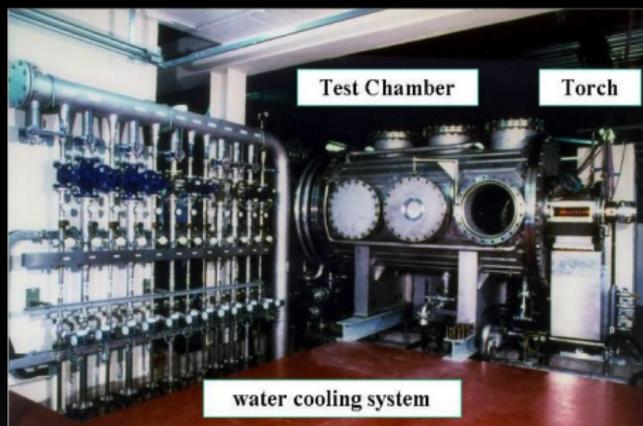
1.2 MW Inductively Coupled Plasmatron



Plasmatron facility and artistic impression of plasma torch

- Originally designed for Hermes project (Ceramic Matrix Composites (CMC) → ablation)
- Gas: Air, N₂, CO₂, Ar
- Power: 1.2 MW (most powerful ICP in the world)
- Heat-flux: up to 10 MW/m² (superorbital re-entry)
- Pressure: 10 mbar - 1 atm

1.2 MW Inductively Coupled Plasmatron



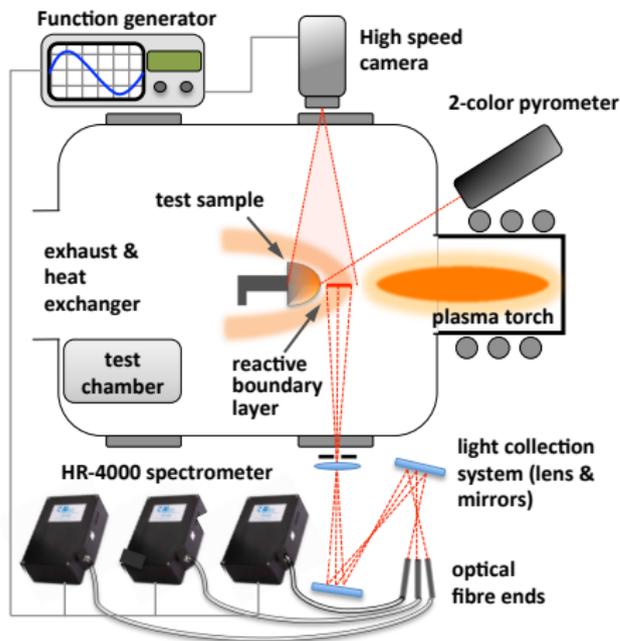
Plasmatron test chamber showing experimental setup and torch exit

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1.2 MW Inductively Coupled Plasmatron

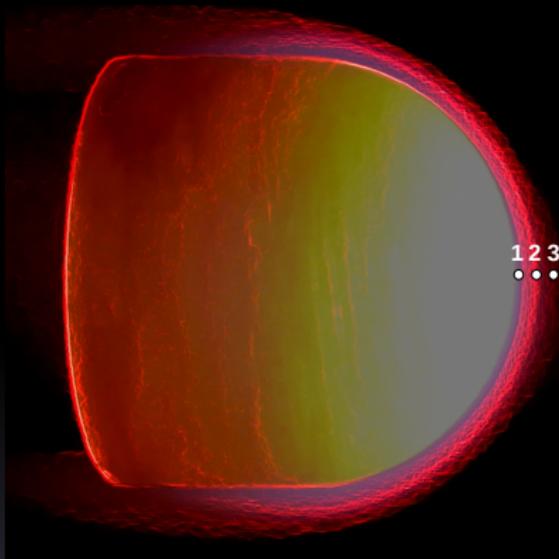
movie loading...

Experimental Techniques for Ablation Characterization



- Radiometry
surface temperatures & emissivity
- Thermocouples
internal temperature histories
- High-speed-camera
 - in-situ recession analysis
 - in-situ determination of spectrometer probing locations
- Optical emission spectroscopy
temporally and spatially resolved radiation profiles in the boundary layer
 - chemical composition
 - temperature estimation

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Materials of Investigation



Carbon fiber preform (Mersen Scotland Holytown Ltd.)

- chopped carbon fibers, fully carbonized no phenol content
- density: 180-210 kg/m³, porosity: 90%



AQ61 (EADS Astrium ST)

- low density carbon-phenolic
- made of short carbon fibers impregnated with phenolic resin
→ compacted & pyrolysed
- low resin content



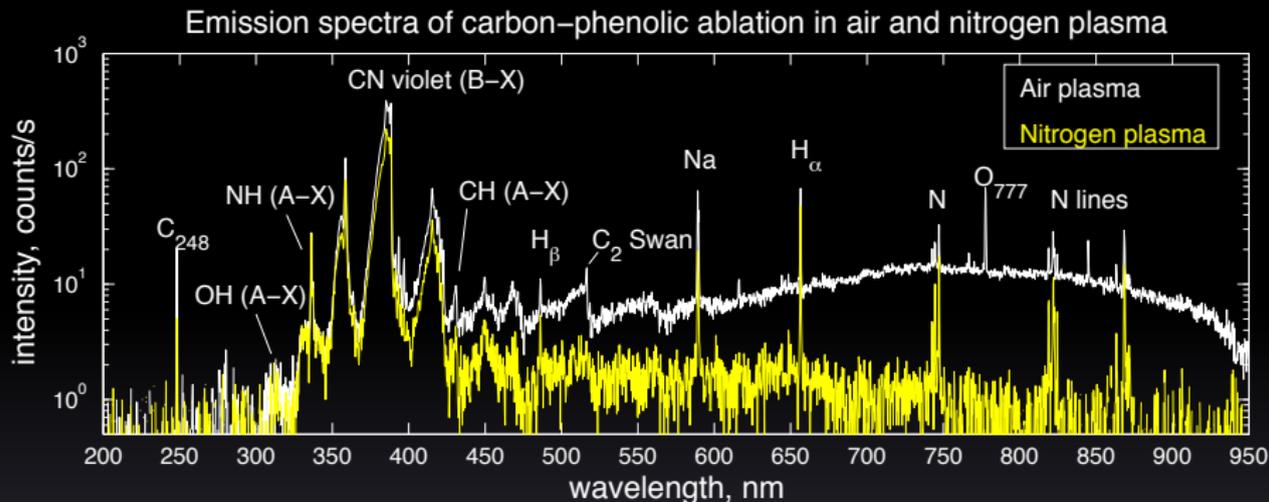
ASTERM (EADS Astrium ST)

- low density carbon-phenolic
- rigid graphite felt impregnated with phenolic resin
→ polymerization
- precursor similar to carbon fiber reform

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 - Results Flow Field: Boundary Layer Chemistry
 - Results Material Field: Surface & Char Examination
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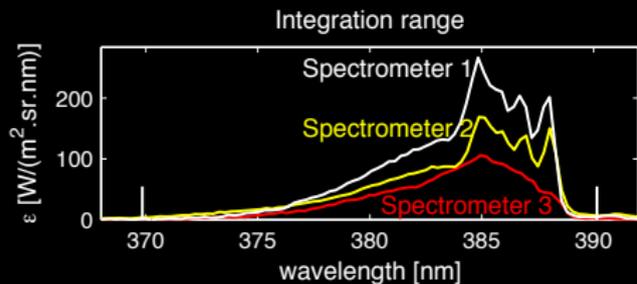
Contaminated Boundary Layer



Detection of contamination products originating from phenol
(C_6H_5-OH)

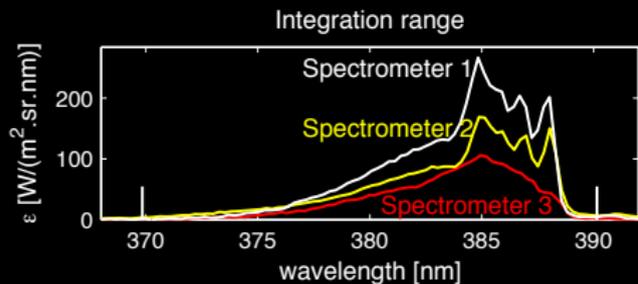
- ① pyrolysis \Rightarrow C, C₂, CH, NH, OH
- ② ablation \Rightarrow C, C₂, CN

CN Spatial Radiation Profiles in Boundary Layer

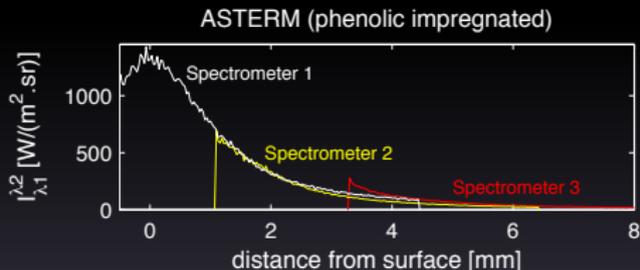
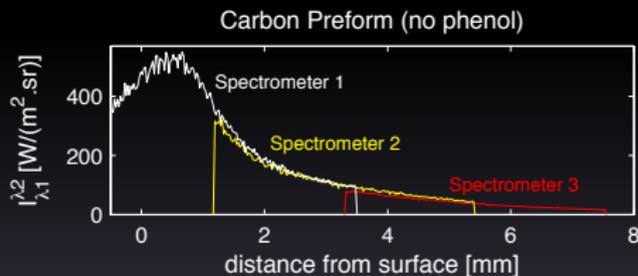


$$I_{CN}^{(t)} = \int_{370\text{nm}}^{390\text{nm}} \epsilon(\lambda) d\lambda \quad (1)$$

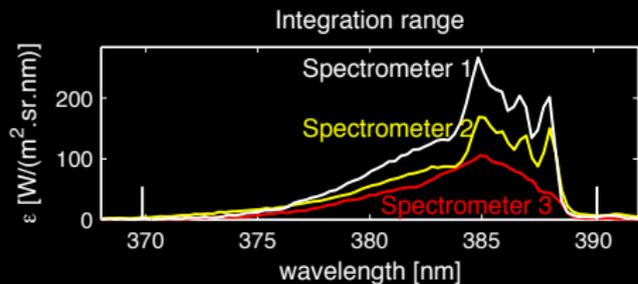
CN Spatial Radiation Profiles in Boundary Layer



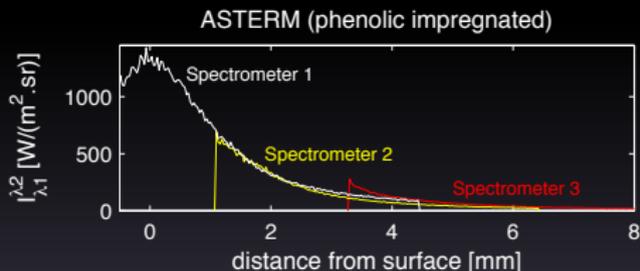
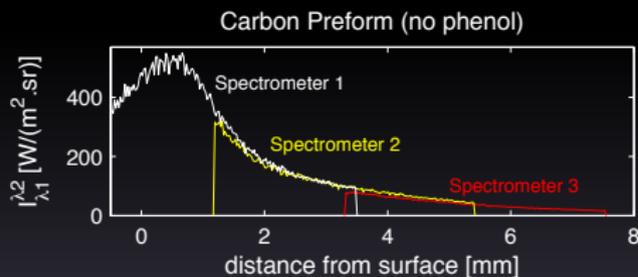
$$I_{CN}^{(t)} = \int_{370nm}^{390nm} \epsilon(\lambda) d\lambda \quad (1)$$



CN Spatial Radiation Profiles in Boundary Layer

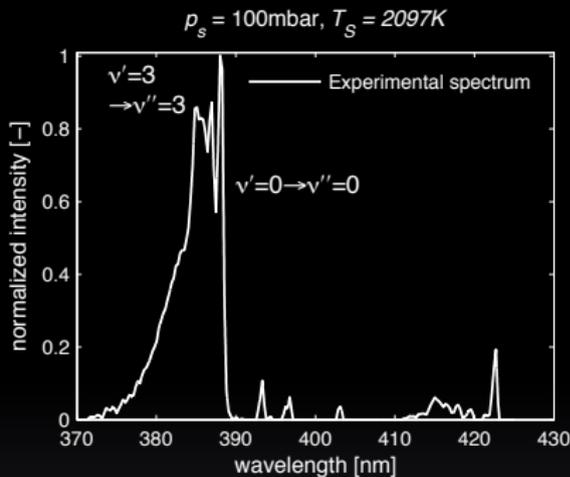
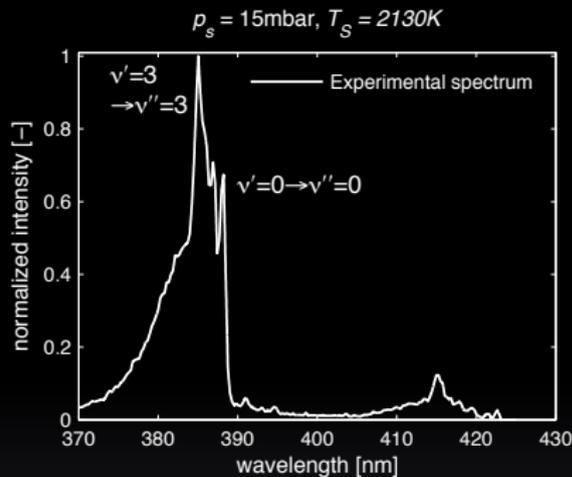


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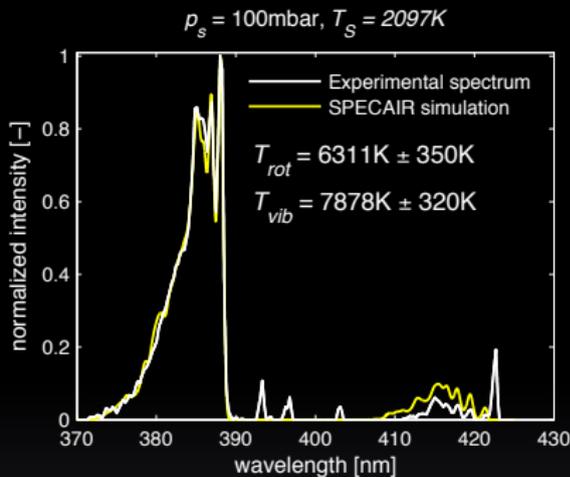
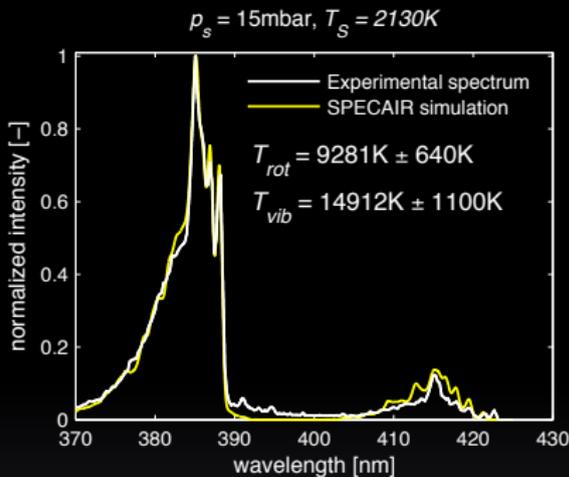
We are interested in temperature and concentration profiles in the boundary layer
 → molecular radiative signature of CN violet system

CN Radiation Simulation for Temperature Estimation



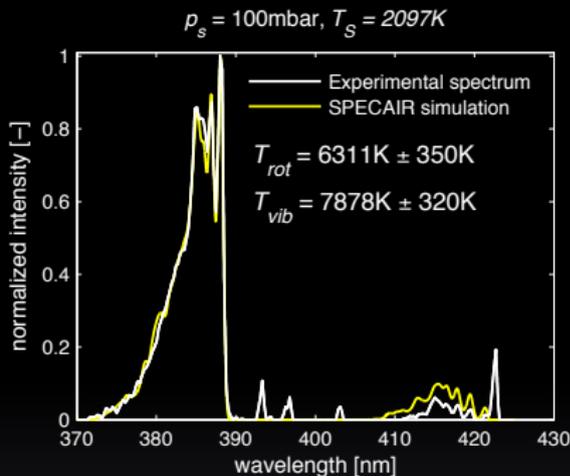
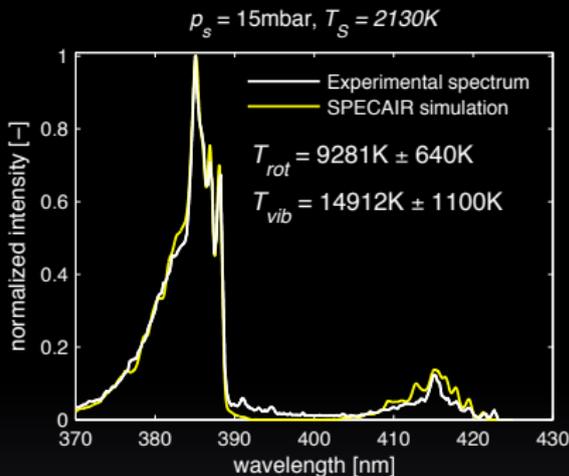
- Vibrational levels variations for different chamber pressures (close to wall)
→ temperature estimation using simulation tool SPECAIR [8]

CN Radiation Simulation for Temperature Estimation



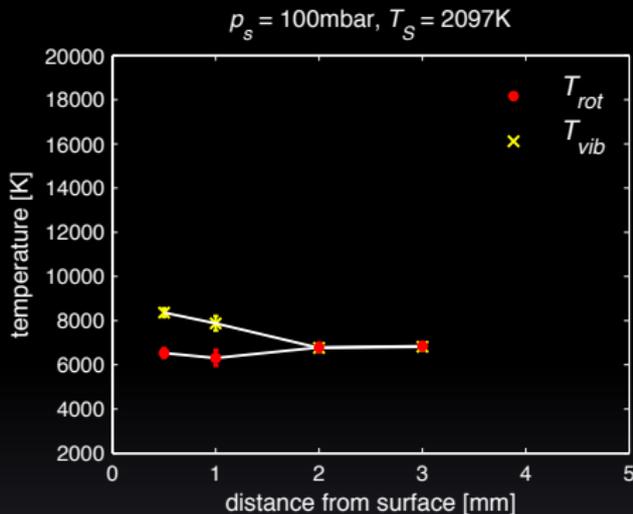
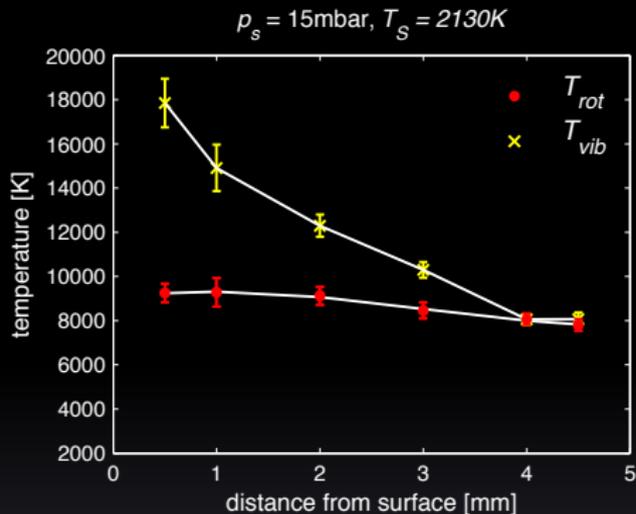
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- Deviation from thermal EQ w.r.t. T_{rot} and T_{vib} (Boltzmann distribution!)
- Evident for all three materials (Preform, AQ61, ASTERM)
- Only electrically excited states are probed (CN B-X)

CN Radiation Simulation for Temperature Estimation



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 - Deviation from thermal EQ w.r.t. T_{rot} and T_{vib} (Boltzmann distribution!)
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 - Only electrically excited states are probed (CN B-X)
- Check for various distances off the surface

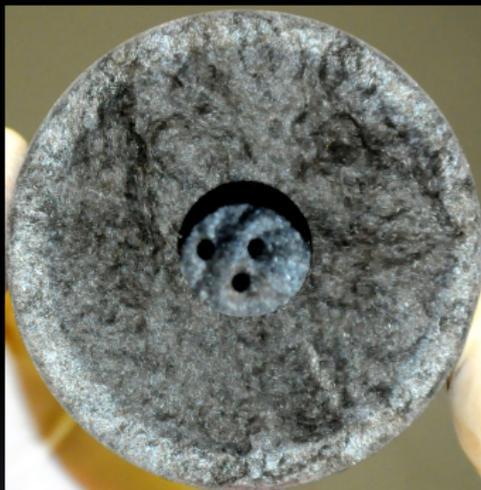
Boundary Layer Temperature Profile



- Deviation from thermal EQ close to the wall (low pressures)
- Equilibrating effect throughout BL
- Mainly equilibrium condition at high pressure (right)

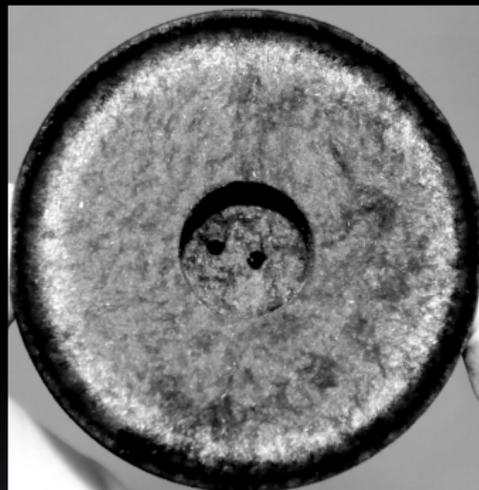
⇒ AIAA-2013-2770

Post-Test Visual Inspection



after ablation in air

- Macroscopic char identification
- Symmetric charring of AQ61

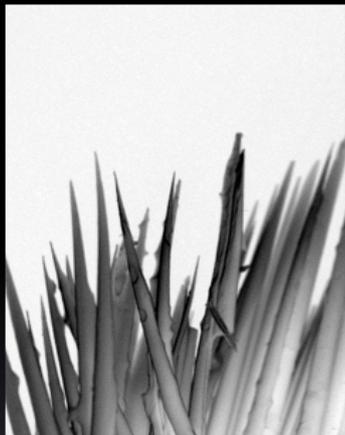


after ablation in N_2

- Black char over whole surface
- Symmetric charring of AQ61

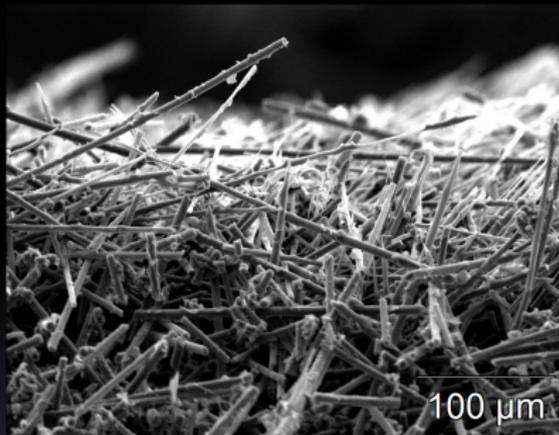
SEM Inspection: Stagnation Point (Air Ablation)

Carbon Preform



- icicle shaped fibers after ablation in air
- icicle angle and depth of ablation depend on oxygen diffusion [9]

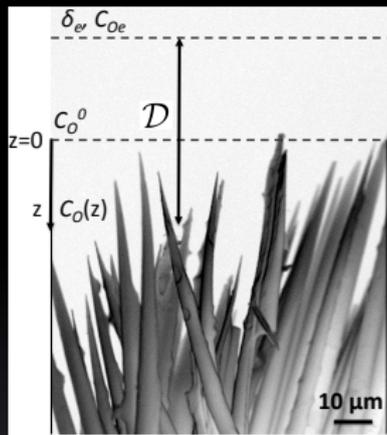
AQ61



- icicle shaped fibers & high porosity (charred resin sparsely identified)

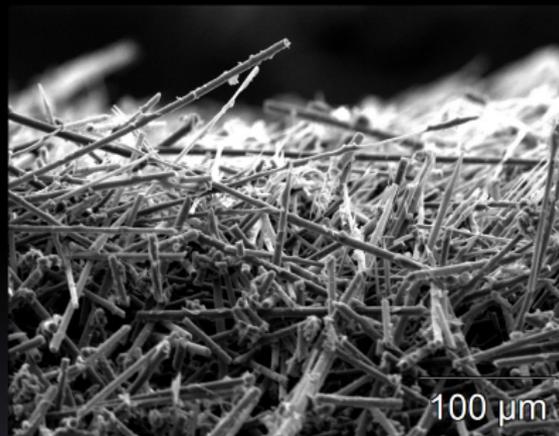
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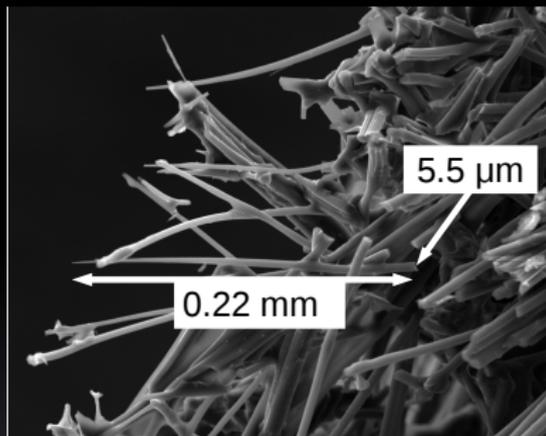
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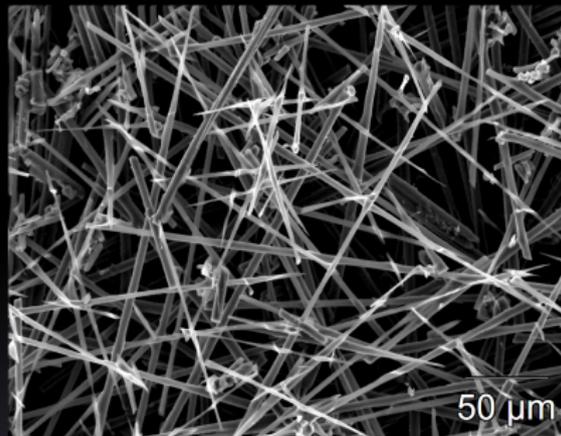
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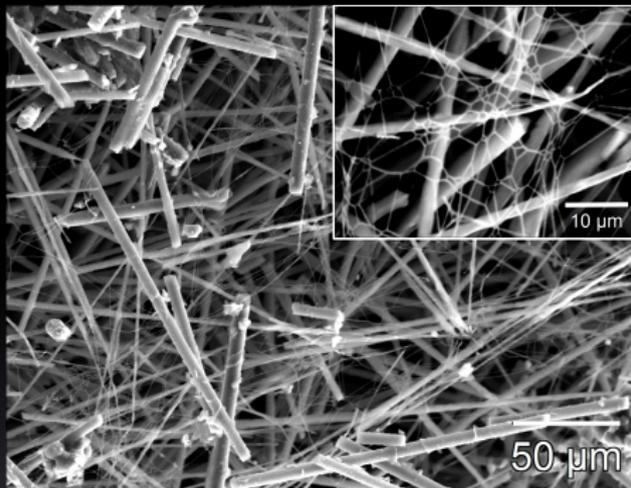
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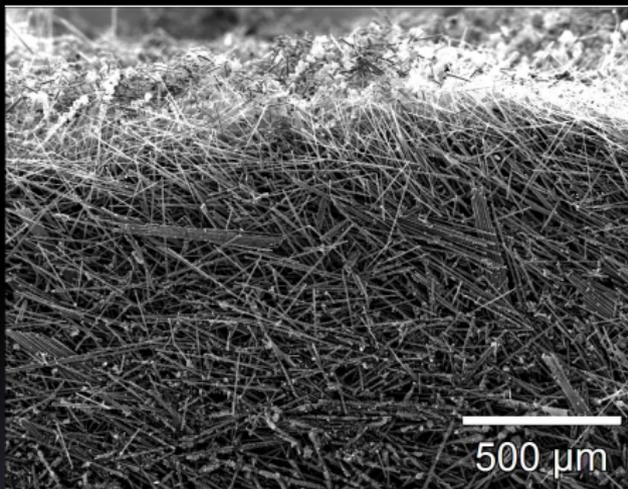
SEM Inspection: Ablation in Nitrogen

Stagnation Point



- 'cross filaments' found on the surface after ablation in N
- production of strong & stable C-C bonds (catenation?)

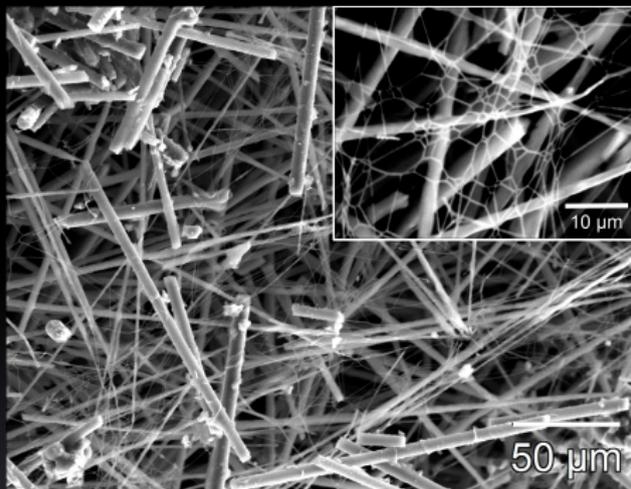
Side- & Backface



- black carbon (similar to soot) deposited at surface

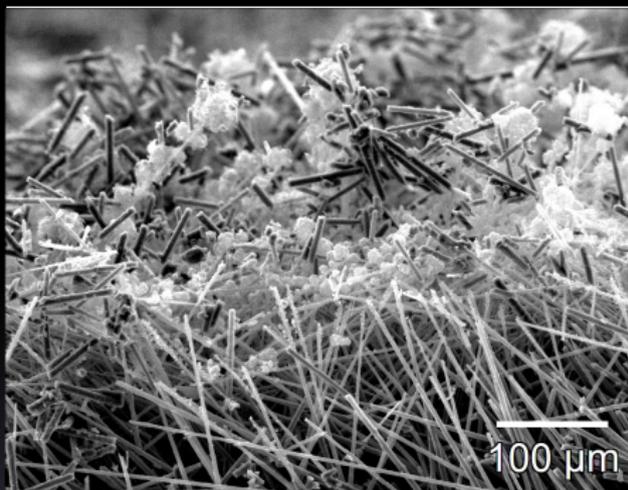
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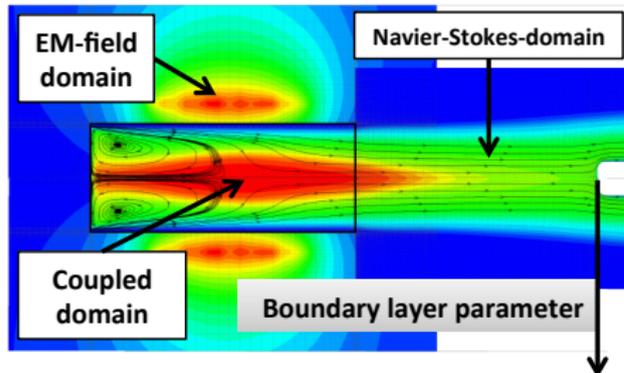
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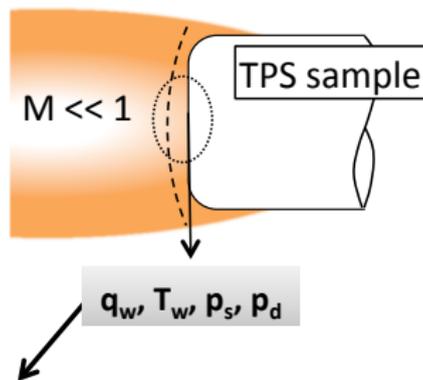
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 - Combined Numerical/Experimental Rebuilding Procedure
 - State of the Art Ablation Modeling
 - 1D Stagnation Line Description with Surface Ablation (Poster: A. Turchi)
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Combined Numerical/Experimental rebuilding Procedure

CFD simulation (VKI ICP code)



Experiments

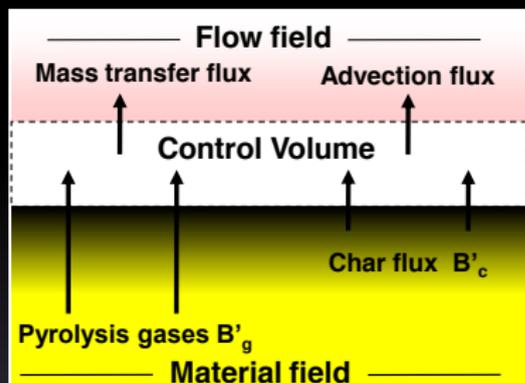


Boundary layer solver

- **Input:** Boundary layer parameter (LTE CFD computation) & measurements from experiments
- **Procedure:** Iteration on boundary layer edge temperature T_e :
 $\Rightarrow q_w^n = q_w^{(exp)} = q_w(\gamma, T_w, p_e, h_e, \beta, \dots)$
- **Output:** Edge enthalpy H_e , boundary layer chemistry, (catalycity)

State of the Art Ablation Modeling

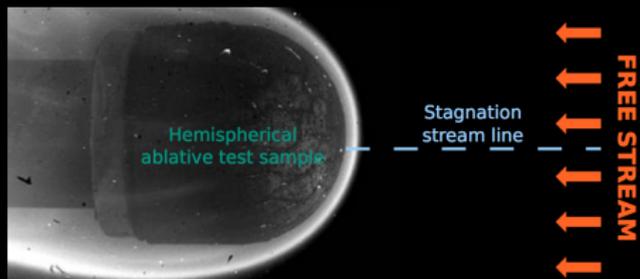
Common strategy (Kendall et al.[4]):



Assumptions:

- Material and flow decoupled
→ **Control volume approach**
- Chemically active surface
→ carbon char reacts with oxygen
- Chemically active species from
→ pyrolysis of decomposing material
→ edge of boundary layer (equilibrium chemistry)

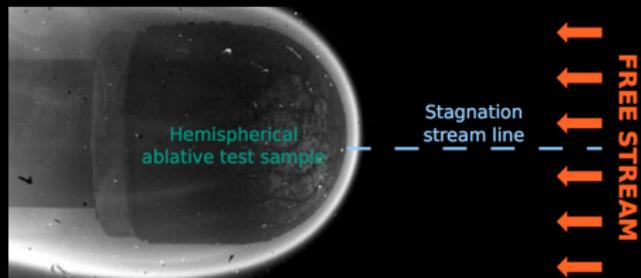
1D Stagnation Line Description w/ Surface Ablation (Poster: A. Turchi)



Approach:

- SPECIES SURFACE MASS BALANCE (SMB)
- SURFACE ENERGY BALANCE (SEB)

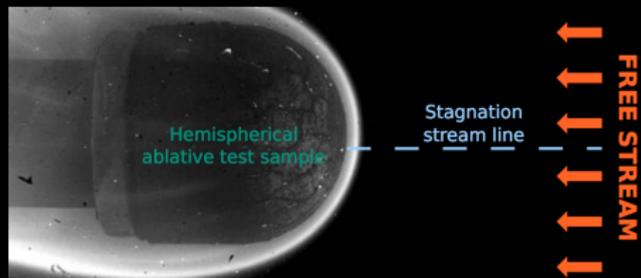
1D Stagnation Line Description w/ Surface Ablation (Poster: A. Turchi)



- Reconstruction of experiments
- Gas mixture properties:
Thermo-chemistry library
MUTATION++ (Poster: J.B. Scoggins)
- 1D stagnation-line formulation with
SMB & SEB

1D Stagnation Line Description w/ Surface Ablation

(Poster: A. Turchi)



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Table: Preliminary results on a Carbon Preform (no phenol content)

Experiment	T_w , K	\dot{m}_c , $\text{kg}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$
$(\dot{q}_{cw} = 3 \text{ MW}/\text{m}^2, p_s = 20 \text{ kPa})$	2783	$\begin{matrix} 0.0175 \\ 0.0111 \end{matrix}$
Isothermal ablation w/ nitridation	2783 (imposed)	0.0202
SEB ablation w/ nitridation	2198	0.0201
SEB ablation w/o nitridation	2174	0.0152

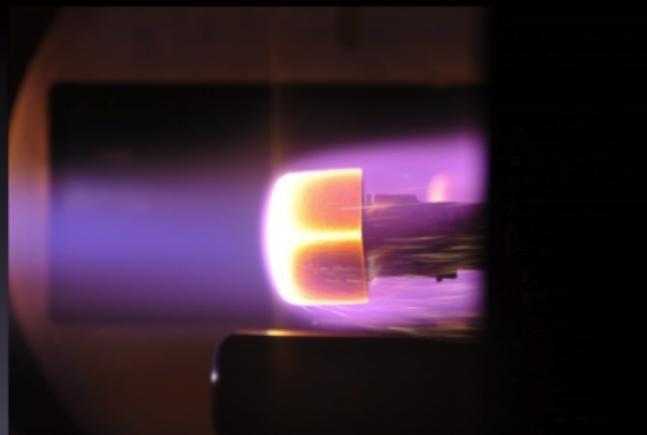
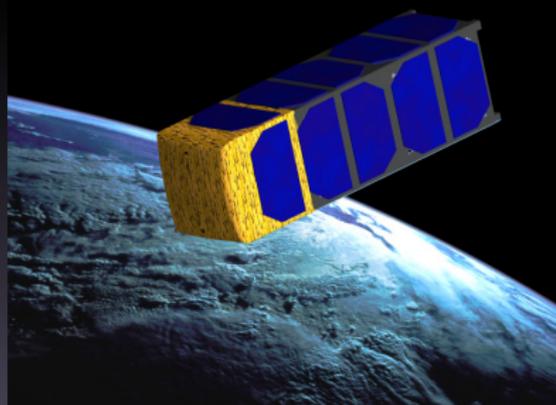
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Application to Mission Design (Presentation: I. Sakraker, G. Baillet)

QARMAN: QubeSat for Aerothermodynamic Research and Measurements on Ablation
(Re-entry cube-sat as part of the VKI QB50 project)

⇒ TPM selection campaign (heat load reproduction)



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Conclusions and Perspectives

In-situ observation: Recession rates, sample temperature response, boundary layer thermo-chemistry (AIAA-2013-2770)

Post-test Analysis Char layer examination at carbon fiber length scale:
Mechanisms of fiber oxidation in diffusion-limited regime
Carbon deposition on surface in N_2 environment

1D-Code Comparison: Stagnation line description matches experimental results within uncertainty

- More conditions for additional comparison / validation
- Extend to carbon-phenolic ablator (loose coupling with material code)
- Goal: Comparison of BL chemistry and spectroscopy data (profiles, mole fractions) for code validation

References

- [1] M. Stackpoole, S. Sepka, I. Cozmuta, Post-flight evaluation of stardust sample return capsule forebody heatshield material, in: AIAA 2008-1202, Reno, NV, USA, 2008.
- [2] H. Tran, C. Johnson, D. Rasky, F. Hui, M.-T. Hsu, T. Chen, Y. K. Chen, D. Paragas, L. Kobayashi, Phenolic Impregnated Carbon Ablators (PICA) as Thermal Protection Systems for Discovery Missions, in: NASA, TM 110440, 1997.
- [3] H. Ritter, O. Bayle, Y. Mignot, E. Boulrier, P. Portela, J.-M. Bouilly, R. Sharda, Ongoing european developments on entry heatshields and tps materials, in: 8th International planetary probe workshop, Portsmouth, Virginia, 6.-8. June, 2011.
- [4] R. Kendall, E. Bartlett, R. Rindal, C. Moyer, An analysis of the coupled chemically reacting boundary layer and charring ablator: Part i, NASA CR 1060, NASA (1968).
- [5] J. Lachaud, I. Cozmuta, N. Mansour, Multiscale approach to ablation modeling of phenolic impregnated carbon ablators, J. Spacecraft Rock. 47 (6) (2010) 910–921.
- [6] J.-M. Bouilly, L. Dariol, F. Leleu, Ablative Thermal Protections For Atmospheric Entry. An Overview Of Past Missions And Need For Future Programmes, in: Proceedings 5th European Workshop on Thermal Protection Systems and Hot Structures, Noordwijk, The Netherlands, 2006.
- [7] A. F. Kolesnikov, Conditions of simulation of stagnation point heat transfer from a high-enthalpy flow, Fluid Dyn. 28 (1) (1993) 131–137.
- [8] A. Cipullo, B. Helber, F. Panerai, O. Chazot, Experimental Characterization of the Free-stream Plasma Flow Produced by the VKI Plasmatron Facility using Optical Emission Diagnostics, in: RTO-EN-AVT-199, Paper 17, von Karman Institute, Rhode-Saint-Genèse, Belgium, 2012.
- [9] B. Helber, O. Chazot, T. Magin, A. Hubin, Ablation of carbon preform in the VKI Plasmatron, in: AIAA paper 2012-2876, 2012.

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