

# Methodology for ablation investigations in the VKI Plasmatron facility: Preliminary results with a carbon fiber preform

5<sup>th</sup> Ablation Workshop, Lexington (KY), February 27<sup>th</sup> - March 01<sup>st</sup> 2012

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# Outline

- 1 Introduction
- 2 Ground testing in Plasmatron facility
- 3 Numerical tools development
- 4 Results with a carbon fiber preform (preliminary)
- 5 Conclusions and perspective



# Outline

- 1 Introduction
  - Background
  - Overview of ablation test campaigns at VKI
  - Research goals
- 2 Ground testing in Plasmatron facility
- 3 Numerical tools development
- 4
- 5 Conclusions and perspective





# Overview of ablation test campaigns at VKI

|           |   |   |
|-----------|---|---|
| 2002-2009 | <b>Graphite</b>                           | Material used for basic research (Vancrayenest, Fletcher)           |
| 2009-2011 | <b>Cork composites (P50, AMORIM)</b>      | Advanced TPM (cork/phenol compound) (Norcoat-Liege, ARD back-shell) |
| 2009-2010 | <b>Monolythic carbon composite (MonA)</b> | More advanced, low density TPM (AMOD program)                       |
| 2011-2012 | <b>Carbon fiber preform</b>               | Similar to precursor for European ablator (Mersen Scotland, Ltd.)   |
| ⇒ 2012    | <b>PICA-like materials</b>                | ASTERM tests scheduled for 2012 (EADS Astrium)                      |



## Research goals

Definition of a methodology to characterize  
material response and gas-gas / gas-surface interaction  
of innovative ablators in the VKI Plasmatron facility

Ablation modelling framework for development of  
material response models for carbon / resin composites  
and coupling with in-house codes

Model validation

Flight extrapolation



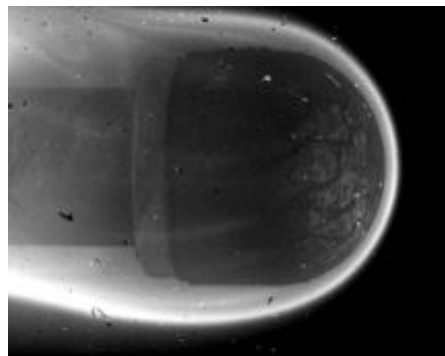
# Outline

- 1 Introduction
- 2 Ground testing in Plasmatron facility
  - Plasmatron facility
  - Recession Analysis
  - Emission Spectroscopy
- 3 Numerical tools development
- 4
- 5 Conclusions and perspective

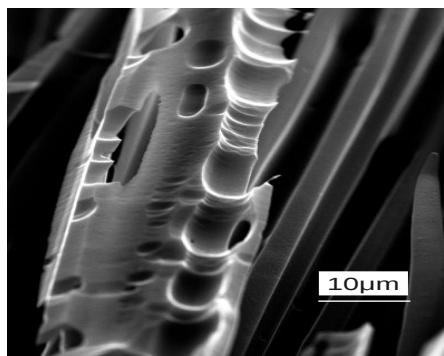


# High-enthalpy tests combined with multiscale characterization

von Karman Institute:  
Analysis in High-Enthalpy Plasma  
Flows



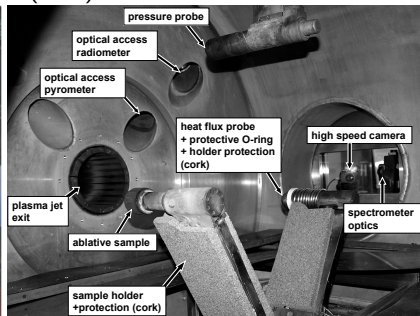
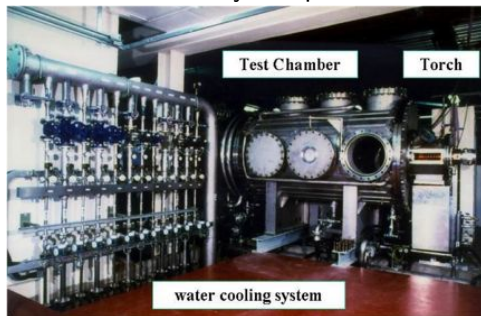
Vrije Universiteit Brussel:  
Multiscale Characterization  
(SEM, EDX, XPS, AES)





# Plasmatron facility

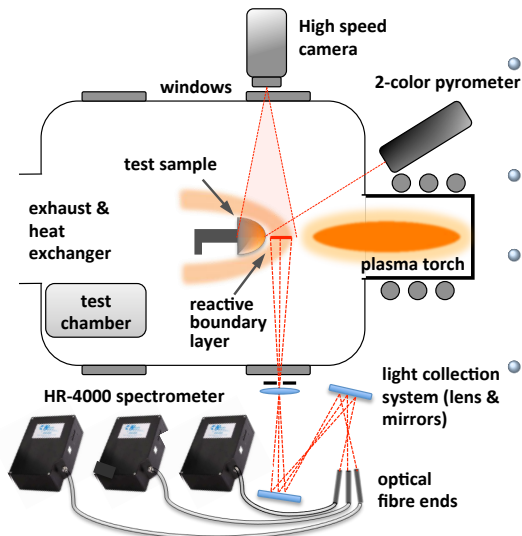
## 1.2 MW Inductively Coupled Plasmatron (ICP)



Plasmatron test chamber showing experimental setup and torch exit

- Gas: Air, N<sub>2</sub>, CO<sub>2</sub>, Ar
- Power: 1.2 MW
- Heat-flux: 90 kW/m<sup>2</sup> - 10 MW/m<sup>2</sup>
- Pressure: 10 mbar - 600 mbar

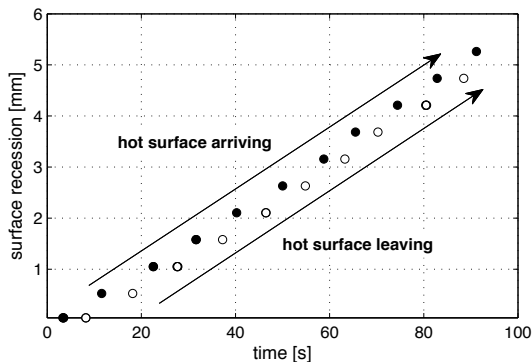
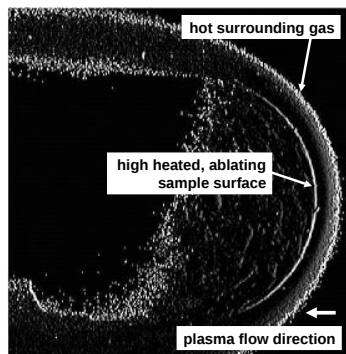
# Measurement techniques development



- **Radiometry**  
surface temperatures & emissivity
- **Thermocouples**  
Internal temperature histories
- **High-speed-camera**  
in-situ recession and gas-phase analysis
- **Optical emission spectroscopy**  
temporally and spatially resolved boundary layer chemistry
- **(future: recession sensors)**

# MT: High Speed Camera Imaging

## In-situ recession and visual gas phase studies (AIAA 2011-2302)

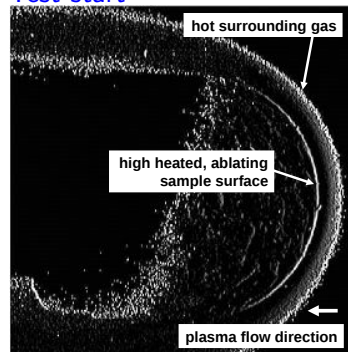


- In-situ recession analysis (linearity, dependence on outgassing)  
 ⇒ Crucial data for model validation (large uncertainties)

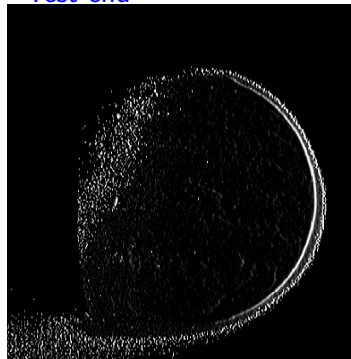
# MT: High Speed Camera Imaging

## In-situ recession and visual gas phase studies (*AIAA 2011-2302*)

### Test start



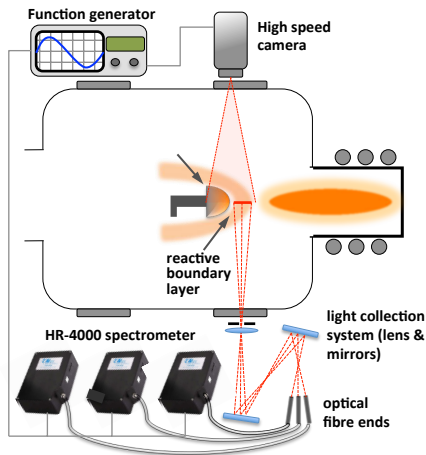
### Test end



- In-situ recession analysis (linearity, dependence on outgassing)
  - ⇒ Crucial data for model validation (large uncertainties)
- Gas phase observation (off-set of surrounding flame)
  - ⇒ Outgassing effects

# MT: Temporally & spatially resolved emission spectroscopy

## Thermo-chemistry in reactive boundary layer



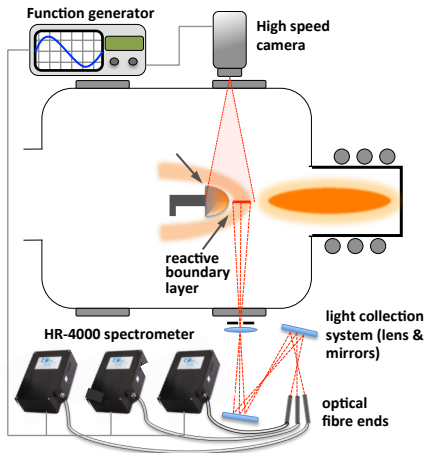
**3 spectrometers with broad wavelength range** per acquisition (200 – 1000 nm)  
 ⇒ temporal resolution (up to 5 ms)

imaging mode with **optical magnification** factor of  $\sim 2$   
 ⇒ narrow boundary layer screening (all spectrometers within  $\sim 3\text{mm}$ )

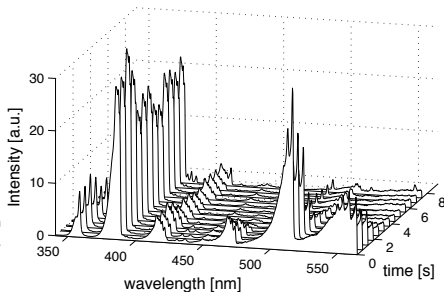
Function generator to **trigger acquisition with high-speed-camera**  
 ⇒ temporally resolved distance of probing volumes from ablating surface

# MT: Temporally & spatially resolved emission spectroscopy

Thermo-chemistry in reactive boundary layer (*AIAA 2011-2302*)



Temporal spectra of ablation of MonA in nitrogen plasma



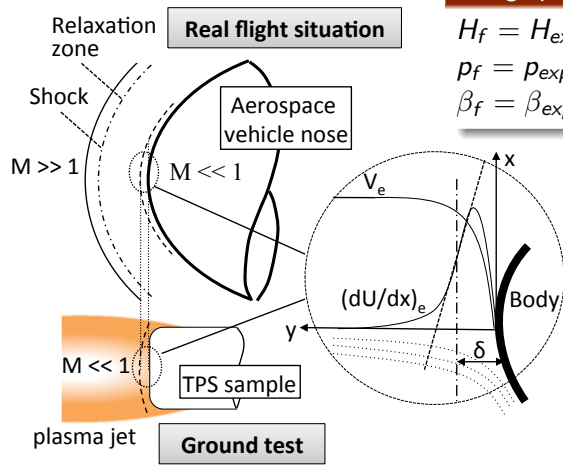
→ CN violet, CN red,  $C_2$  Swan, CH, Ca, K, Na, N,  $N_2$ ,  $N_2^+$ , O

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# Local Heat Transfer Simulation (LHTS)



Rebuilding of freestream properties  
& stgn.pt. heat flux similarity:

$$H_f = H_{exp},$$

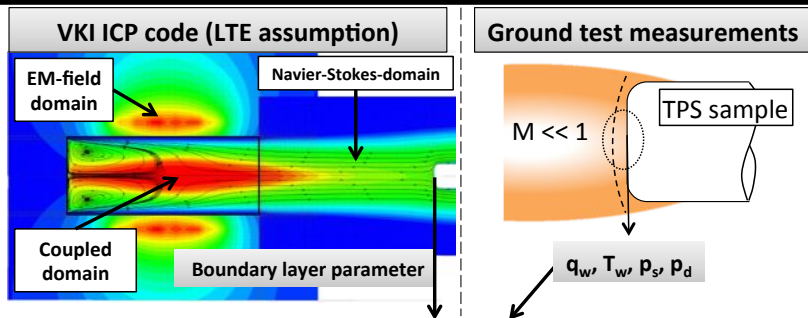
$$p_f = p_{exp},$$

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

Kolesnikov, *Fluid Dynamics*, 28(1):131-137, 1993.



# Combined numerical/experimental rebuilding procedure



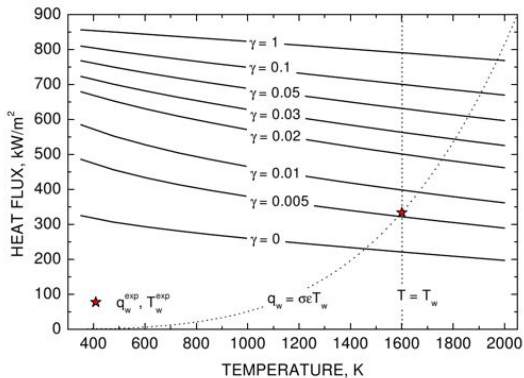
## Boundary layer solver

- **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)

# Stagnation point chemical environment

## State-of-the-art (VKI)

### Flight extrapolation & catalycity rebuilding for reusable heat shields



[Panerai, F., *Aerothermochemistry Testing of Thermal Protection Systems, Ph.D. thesis, VKI / University of Perugia*]

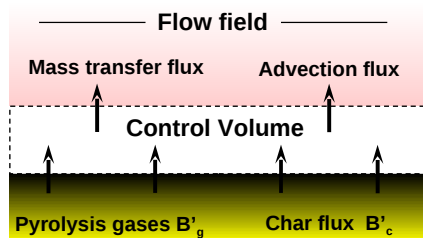


# Stagnation point chemical environment

## Extension to ablation

GSI models for CFD codes:

**Control volume approach** for B'-table generation (MUTATION)



### Assumptions:

- Chemically active surface:  
→ carbon char reacts with oxygen
- Chemically active species from  
→ pyrolysis of decomposing material  
→ edge of boundary layer (equilibrium chemistry)

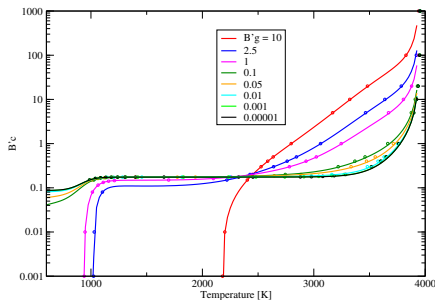
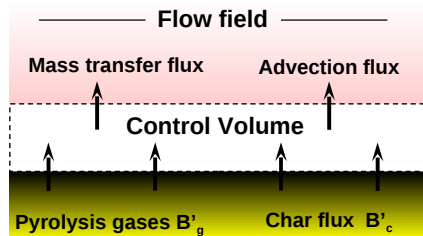
Collaboration J. de Muelenaere (VKI),  
J. Lachaud & N.N. Mansour (NASA Ames) (AIAA 2011-3616)

# Stagnation point chemical environment

## Extension to ablation

GSI models for CFD codes:

**Control volume approach** for B'-table generation (MUTATION)



⇒ Coupling with stagnation line formulation

⇒ Development of a new approach to build B'-tables

Collaboration J. de Muelenaere (VKI),  
J. Lachaud & N.N. Mansour (NASA Ames) (AIAA 2011-3616)

# Stagnation point chemical environment

## High-fidelity material response models

Boundary layer chemistry:

Damköhler number:  $\tau_f / \tau_{\text{chem}}$

Required for ablation:

Gas-surface interaction and char layer chemistry:

⇒ **Char-Damköhler number:**

Reaction vs. diffusion competition in porous media

⇒ **Thiele number:**

$$\Phi = \frac{L}{\sqrt{D_i / s_f k_f}} \quad (1)$$

Boundary layer edge

LTE,  $H_e$ ,  $P_e$ ,  $\beta$ , BC

Gas chemistry

Char layer

Pyrolysis zone

Virgin material

Ablation zone



# Stagnation point chemical environment

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Boundary layer edge

LTE,  $H_e$ ,  $P_e$ ,  $\beta$ , BC

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Pyrolysis zone

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Ablation zone

estimation of the effective reactive surface area not available

→ **Experiments with microscale characterization**



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  - Plasma tests of carbon fiber preform
  - Scanning Electron Microscopy
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# Plasma tests of carbon fiber preform

## Carbon fiber preform, Mersen Scotland Ltd.

- Chopped carbon fibers in phenol-resin slurry, fully carbonized (1200K)
- density:  $180 - 210 \text{ kg/m}^3$
- specific surface area:  $18 \text{ m}^2/\text{g}$   
(fiber volume surface:  $3.24\text{E}6 \text{ m}^2/\text{m}^3$ )
- 2D randomly oriented carbon fibers, through-the-thickness (TTT) direction parallel to flow field in this study
- similar to precursor for European ablator ASTERM





# Plasma tests of carbon fiber preform

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# Plasma tests of carbon fiber preform

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Table: Test conditions ( $t=90s$ )

|  | (#1) | (#2) | (#3) |
|--|------|------|------|
| $p_s$<br>[kPa]                         | 1.5  | 10   | 20   |
| $\dot{q}_{CW}$<br>[kW/m <sup>2</sup> ] | 1021 | 946  | 1026 |



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| $\dot{q}_{CW}$<br>[kW/m <sup>2</sup> ] | 1021 | 946  | 1026 |
| $\dot{m}$<br>[g]                       | 6.88 | 5.49 | 5.11 |
| $r$<br>[mm]                            | 7.0  | 5.0  | 5.5  |



# Plasma tests of carbon fiber preform

Table: Test conditions ( $t=90s$ )

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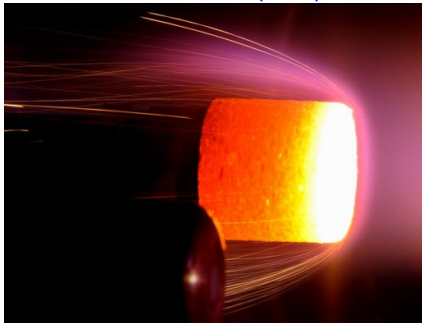
- strongest degradation at lowest static pressure
- ⇒ diffusion mechanisms in diffusion limited regime ( $T_S > 2000K$ )?
- ⇒ other failure modes (Spallation?)



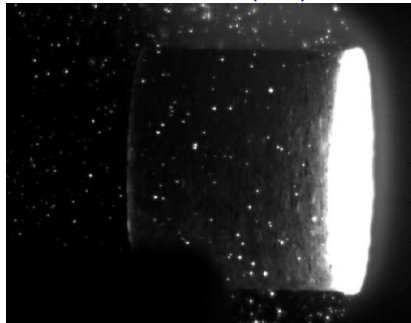
# Plasma tests of carbon fiber preform

## Spalling of particles

Conventional image (5ms)



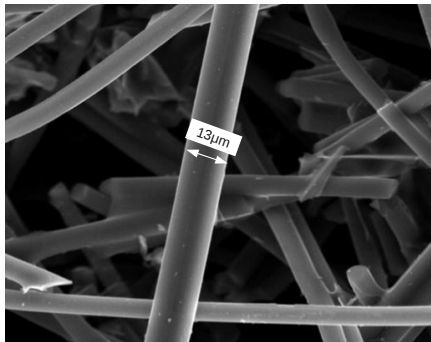
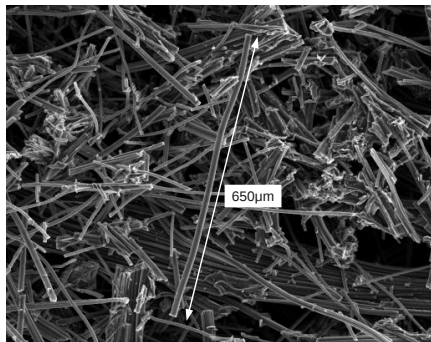
High-speed-camera (5 $\mu$ s)



⇒ Continual release of particles into the flow field, burn-off in plasma flow

# SEM of carbon fiber preform

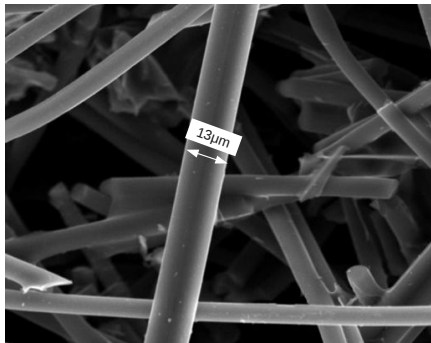
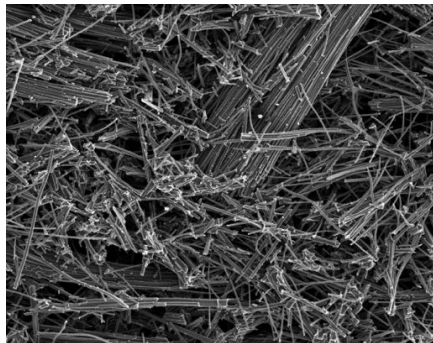
## Carbon preform surface before test



- $l_f = 650 \mu\text{m}$
- doubled fiber diameter ( $d_f = 6.5 \mu\text{m}$ )

# SEM of carbon fiber preform

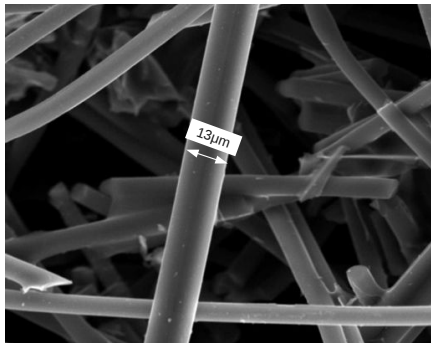
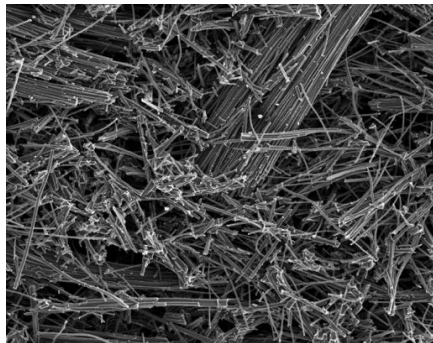
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- glued fibers → bundles

# SEM of carbon fiber preform

## Carbon preform surface before test



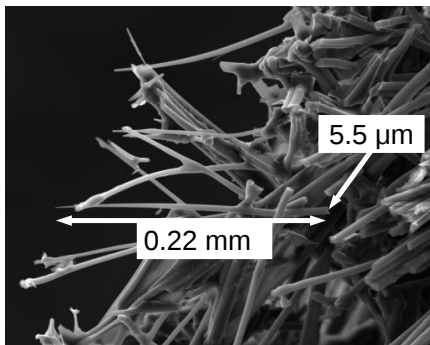
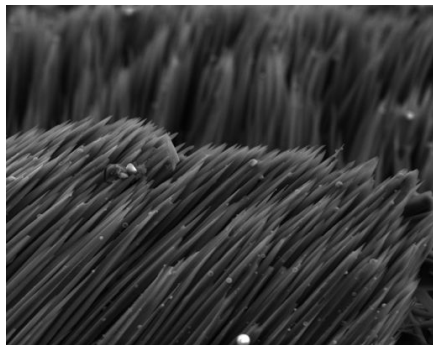
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- glued fibers → bundles

Does ablation of individual fibers lead to detachment of fiber bundles?



# SEM of carbon fiber preform

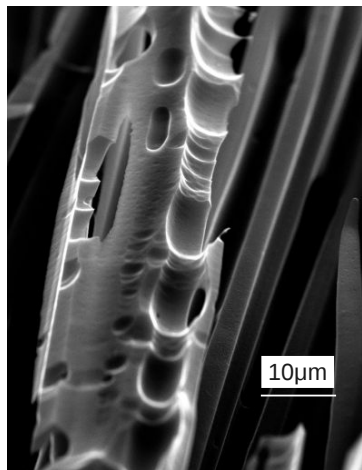
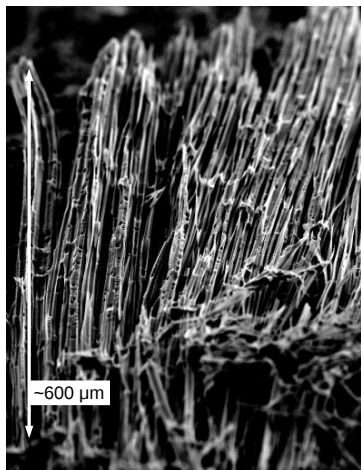
Surface of ablated sample ( $p_s = 10\text{kPa}$ ), stagnation region



- fibers shortened to a length of  $\sim 220\ \mu\text{m}$
- oxidation leads to icicle shape
- diameter increasing to original fiber diameter of  $d_f = 6.5\ \mu\text{m}$   
→ oxidation zone of  $\sim 250\ \mu\text{m}$ ?

# SEM of carbon fiber preform

Surface of ablated sample ( $p_s = 1.5$  kPa)



⇒ Strong degradation along whole fiber length  $\sim 650$   $\mu\text{m}$

# SEM of carbon fiber preform

## Diffusion in a porous medium

### species diffusion coefficients

$$\mathcal{D}_i = \frac{1 - x_i}{\sum_{j \neq i} x_j / \mathcal{D}_{i,j}}$$

### binary diffusion coefficients

$$\mathcal{D}_{i,j} = \frac{3}{16} \sqrt{\frac{2\pi k_B T (m_i + m_j)}{m_i m_j}} \frac{k_B T}{\rho \Omega_{i,j}^{(1,1)}}$$

(Capitelli, M., Gorse, C., Longo, S., and Giordano, D., *JTHT* 14 (2) (2000) 259-268.)

### Thiele number

$$\Phi = \frac{L}{\sqrt{\mathcal{D}_i / s_f k_f}}$$

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### Enhanced diffusion at low pressures

Boundary layer edge

|  | (#1) | (#2) | (#3) |
|--|------|------|------|
| $p_s$<br>[kPa]                             | 1.5  | 10   | 20   |
| $\rho_e \times E4$<br>[kg/m <sup>3</sup> ] | 6    | 38   | 79   |

# SEM of carbon fiber preform

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| $c_O \times E3$<br>[mol/m <sup>3</sup> ]   | 8.7  | 54.4 | 112.1 |
| $\mathcal{D}_O$<br>[m <sup>2</sup> /s]     | 0.52 | 0.09 | 0.04  |

# SEM of carbon fiber preform

## Diffusion in a porous medium

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| $\mathcal{D}_O$<br>[m <sup>2</sup> /s]     | 0.52 | 0.09 | 0.04  |

⇒ valid in continuum flow regime (stagnation region → Knudsen number)

⇒ in porous media defined by mean pore diameter:  $Kn = \frac{\bar{\lambda}}{d_p}$  ( $Kn > 0.1$  possible)

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## Conclusions: Carbon fiber preform

- Enhanced diffusion effects at low pressures may cause stronger ablation in diffusion-limited regime
- Oxidation of fibers shows variation with (static) pressure
  - Conform with theory on species diffusion
- Ablation of individual fibers may lead to detachment of fiber bundles → mechanical failure
  - Enhanced at lower pressures

- ⇒ More experiments necessary to confirm trends
  - Define methodology to estimate reactive surface area
- ⇒ Extend to full ablator (ASTERM, AQ61)
- ⇒ Modelling of carbon preform ablation tests
  - Start with control volume approach suppressing pyrolysis





# Perspectives for ablation studies at the VKI

- Testing condition and boundary layer characterization
  - ⇒ Application of LHTS methodology to ablation testing
  - ⇒ Uncertainty quantification methods
- Microscale analysis of virgin and tested ASTERM / AQ61
  - ⇒ Comparison to preform (no phenolic impregnation)
  - ⇒ Definition of reactive surface needed, char layer depth, porosity
- Very high heat-flux testing
  - ⇒ Appropriate measurement techniques
- Reference test case definition for model validation
- Flight extrapolation methodology



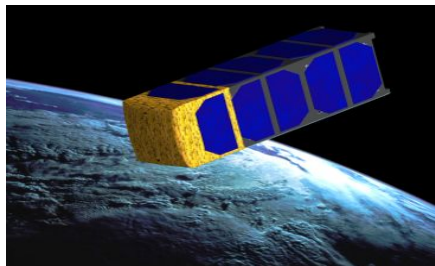
# Perspectives: Extend ablation framework to related studies at VKI

## Converging nozzle



- Supersonic plasma flow studies at VKI
- Extension to high heat flux testing (aim:  $10 \text{ MW/m}^2$ )

## QB50 / flight demonstrator



- 50 double-unit cubesats: in-situ, multipoint & long-duration measurements
- 10 triple-unit cubesats: re-entry flight demonstrator for ablation studies



# Questions?

## Acknowledgements

- AFOSR
- ESA
- Lockheed Martin UK (Amphill)
- Mersen Scotland Holytown Ltd.
- EADS Astrium ST
- Agency for Innovation by Science and Technology (IWT)
- N.N. Mansour, J. Lachaud, J.-M. Bouilly

