Gas/Surface Interaction Study
of Low-Density Ablators in
Sub- and Supersonic Plasmas

NASA Applied Modeling & Simulation (AMS) Seminar Series
June 26, 2014

based on AIAA 2014-2122
11th AIAA/ASME Joint Thermophysics and Heat Transfer Conference

B. Helber, A. Turchi, O. Chazot, A. Hubin, T. E. Magin

1 Aeronautics and Aerospace Department, von Karman Institute for Fluid Dynamics, Belgium
2 Research Group Electrochemical and Surface Engineering, Vrije Universiteit Brussel
TPM for Atmospheric Reentry

Future Missions: Ablative TPM

- Sample return, ISS serving (Dragon, ARV, ...), MPCV
- Atm. reentry speeds > 10km/s
- Mass loss and surface recession
- Prediction of material response required
- High margins decrease payload

New materials (1990’s)

- Phenolic impregnated carbon fiber preform
- Very porous low density ablators

NASA Stardust probe, reentry: Jan. 15, 2006 (12.9 km/s) (AIAA 2008-1202)
TPM for Atmospheric Reentry
Complex Multiphysics - Multiscale Problem

Gas-Surface Interaction

Material

Radiation

Air plasma
Nitrogen plasma

C248
NH (A−X)
OH (A−X)
CN violet (B−X)
CH (A−X)
C2 Swan
Hα
Na
N
N lines
O777
50 µm
We aim at improvement of

Experimental Methods (VKI) ↔ TPS Design / Material (VUB, Airbus DS, ESA)

Material Response Modeling & Validation (VKI, collaborations)
In the following
In the following

Reactive Surface
- surface temperature & emissivity
- centerline recession rate
- post-ablation topology (SEM)
In the following

(2) Surface $\Rightarrow$ Temperatures / Recession
In the following

(2) Surface \[\Rightarrow\] Temperatures / Recession

**Gas-phase**

- Ablation species injection
- Strong radiators and boundary layer absorption
In the following

(2) Surface $\Leftrightarrow$ Temperatures / Recession

(3) Gas-phase $\Leftrightarrow$ BL emission & temp
In the following

(2) Surface ➞ Temperatures / Recession
(3) Gas-phase ➞ BL emission & temp
(4) Material ➞ Pyrolysis outgassing
In the following

1. Materials and Methods for Ablation Characterization
2. Surface $\Rightarrow$ Temperatures / Recession
3. Gas-phase $\Rightarrow$ BL emission & temp
4. Material $\Rightarrow$ Pyrolysis outgassing
Materials of Investigation

Carbon fiber preform (Mersen Scotland Holytown Ltd.)
- chopped carbon fibers, fully carbonised
- density: 180-210 kg/m³, porosity: 90%

AQ61 (Airbus DS)
- low density carbon-phenolic
- made of short carbon fibers impregnated
  → compacted & pyrolyzed
- low resin content

Asterm (Airbus DS)
- low density carbon-phenolic
- rigid graphite felt impregnated
  → polymerization
- precursor similar to carbon fiber reform
Materials of Investigation

Carbon fiber preform, non-pyrolyzing (Mersen Scotland Holytown Ltd.)

AQ61, carbon-phenolic (AIRBUS DS)
Stagnation point similarity:

\[
H_f = H_{\text{exp}}
\]
\[
p_f = p_{\text{exp}}
\]
\[
\beta_f = \beta_{\text{exp}}, \quad \beta = \frac{dU}{dx}\]

**Real flight situation**

Relaxation zone

Shock

Aerospace vehicle nose

\( M >> 1 \)

\( M << 1 \)

TPS sample

**Ground test**

Plasma jet

\( \delta \)

\( V_e \)

\( (dU/dx)_e \)
1.2-MW Inductively Coupled Plasmatron

Gas: Air, N₂, CO₂, Ar
Power: 1.2-MW
Heat Flux: > 12 MW/m²
Pressure: 10 mbar - 1 atm
1.2-MW Inductively Coupled Plasmatron

Gas: Air, N₂, CO₂, Ar
Power: 1.2-MW
Heat Flux: > 12 MW/m²
Pressure: 10 mbar - 1 atm
Techniques for In-Situ Ablation Characterization

Our interest

Surface temperature
Emissivity
Internal Temperature
In-situ recession analysis
Volumetric recession
Chemical composition
Temperature estimation

(AIAA 2013-2770)
Techniques for In-Situ Ablation Characterization

Our interest

Surface temperature
Emissivity
Internal Temperature
In-situ recession analysis
Volumetric recession
Chemical composition
Temperature estimation

(AIAA 2013-2770)
Three Dedicated Test Campaigns
Three Dedicated Test Campaigns

1. **General ablation tests:**
   - air and nitrogen
   - heat fluxes: $1 - 3 \text{ MW/m}^2$, $p_{\text{tot}} = 15 - 200 \text{ hPa}$
Three Dedicated Test Campaigns

1. **General ablation tests:**
   - air and nitrogen
   - heat fluxes: $1 - 3 \text{ MW/m}^2$, $p_{\text{tot}} = 15 - 200 \text{ hPa}$

2. **Grain study:**
   - only air
   - $0.3 - 1 \text{ MW/m}^2$, $p = 15\text{hPa}$
Three Dedicated Test Campaigns

1. General ablation tests:
   - air and nitrogen
   - heat fluxes: 1 - 3 MW/m$^2$, $p_{\text{tot}} = 15 - 200$ hPa

2. Grain study:
   - only air
   - 0.3 - 1 MW/m$^2$, $p = 15$ hPa

3. High-heat flux tests (supersonic)
Volumetric Ablation (shape change)
Non-pyrolyzing carbon-preform

• linear (axial) recession up to r = 20mm
• small shape change ➞ constant boundary conditions

• edge ablation (shoulder) due to peak heating
• shape change ➞ increase of surface temperature

• high centreline ablation
• iso-q shape ➞ caused by shock or nozzle (35mm)
Surface Temperature Distribution by IR-imaging

$q_{cw} = 1\text{MW/m}^2, \ p_s = 15\text{hPa}$

cylindrical

$T_{stag}$ increase

Test start

hemispherical

Test end
Test Sample Shape Affects Temperatures

$q_{cw} = 0.3\text{MW/m}^2$

$q_{cw} = 1.0\text{MW/m}^2$
Test Sample Shape Affects Temperatures

- **Hemispherical**: quasi-steady
- **Cylindrical**: increasing

- For $q_{cw} = 0.3\text{MW/m}^2$:
  - Temperature initially increases then stabilizes.

- For $q_{cw} = 1.0\text{MW/m}^2$:
  - Temperature increases rapidly and then decreases.

*Key Points:*

- Hemispherical shape has a more stable temperature profile.
- Cylindrical shape shows a significant increase in temperature, especially with higher injection rates.

*Notation:* $q_{cw}$ represents the heat flux density per unit area.
Fiber Direction Slightly Affects Temperatures

![Graphs showing temperature changes over time for different fiber directions.](image)
Fiber Direction Slightly Affects Temperatures

with-grain: higher Ts
through-grain: lower Ts

with-grain: higher internal temperature
- unexpected (lower conduction in axis direction)
- but: higher conduction in radial direction
  ⇒ stronger sidewall heating
Emissivity: 0.86 - 0.97

Gray body emissivity (8 – 9µm, T):

\[ \varepsilon_{8-9\mu m, T} = \frac{B(IR)}{B^{bb}(Pyrometer)} \]

Planck's law (spectral radiance):

\[ B_{\lambda}(T) = \frac{2hc^2}{\lambda^5} \frac{1}{e^{\frac{hc}{\lambda k_B T}} - 1} \left[ \frac{W}{m^2 \cdot sr \cdot nm} \right] \]

Measured: \( T = 1724K, p_s = 15\text{mbar} \)
Emissivity: 0.86 - 0.97

Gray body emissivity \((8 - 9\text{µm}, T)\):

\[
\varepsilon_{8-9\text{µm}, T} = \frac{B(IR)}{B^{bb}(Pyrometer)}
\]

Planck’s law (spectral radiance):

\[
B_\lambda(T) = \frac{2hc^2}{\lambda^5} \cdot \frac{1}{e^{\frac{hc}{\lambda k_B T}} - 1} \left[ \frac{W}{m^2\cdot sr\cdot nm} \right]
\]

Measured: \(T = 1724K, p_s = 15\text{mbar}\)
In-situ Recession Analysis (HSC)

Preform (15mbar)

AQ61 (15mbar)

diffusion limited at 15mbar between 2000K and 3000K
**In-situ Recession Analysis (HSC)**

**Preform (15mbar)**

- Recession, mm vs. Time, s
- Data points and lines for temperatures: 2848K, 2180K, 1724K, >3100K
- Note: diffusion limited at 15mbar between 2000K and 3000K

**AQ61 (15mbar)**

- Recession, mm vs. Time, s
- Data points and lines for temperatures: 2884K, 2167K, >3100K, 2167K
- Note: high T: carbon-phenolic

---

**diffusion limited at 15mbar between 2000K and 3000K**
In-situ Recession Analysis (HSC)

Preform (15mbar)

AQ61 (15mbar)

diffusion limited at 15mbar between 2000K and 3000K

Preform (15mbar)

T_{\text{surface}}

t-g

w-g

>3100K

2180K

1724K

2848K

1724K

>3100K

2167K

2884K

2307K

2347K

1686K

1724K
In-situ Recession Analysis (HSC)

Preform (15mbar)

AQ61 (15mbar)

diffusion limited at 15mbar between 2000K and 3000K

higher recession
Ablation Regimes of Preform and AQ61

![Graph showing recession rate vs. temperature for different pressures of Preform and AQ61](image)

- Preform 15hPa
- Preform 80hPa
- Preform 200hPa
- AQ61 15hPa
- AQ61 80hPa
- AQ61 200hPa
Ablation Regimes of Preform and AQ61

- diffusion limited ablation and sublimation regime
- recession not much influenced by pressure! (AIAA 2012-2876)
Ablation Regimes of Preform and AQ61

- Diffusion limited ablation and sublimation regime
- Recession not much influenced by pressure! (*AIAA 2012-2876*)
- Strong recession in N\textsubscript{2} for T>3000K ➔ sublimation

Comparison num. model: talk A. Turchi
Post-Oxidation Micrographs

Carbon preform oxidation

AQ61 oxidation
Detection of contamination products originating from phenol
1. pyrolysis $\Rightarrow$ C$_2$, CH, H, NH, OH
2. ablation $\Rightarrow$ C, CN
Boundary Layer Radiation Profiles
Experimental: Spatial CN violet emission (AIAA 2013-2770)

CN Production:
- gas phase: $\text{CO} + \text{N} \rightleftharpoons \text{CN} + \text{O}$
- wall: $\text{C}_w + \text{N}_w \rightarrow \text{CN}$
Boundary Layer Radiation Profiles

Experimental: Spatial CN violet emission (*AIAA 2013-2770*)

CN Production:
- gas phase: $\text{CO} + \text{N} \rightleftharpoons \text{CN} + \text{O}$
- wall: $\text{C}_\text{w} + \text{N}_\text{w} \rightarrow \text{CN}$

![Graph showing CN production rates and spatial distribution](image)

$T = 2180\text{K}, p_s = 15\text{mbar}$

**Spectrometers:**
- Spectrometer 1
- Spectrometer 2
- Spectrometer 3
Boundary Layer Radiation Profiles
Experimental: Spatial CN violet emission (AIAA 2013-2770)

CN Production:
- gas phase: \( CO + N \rightleftharpoons CN + O \)
- wall: \( C_w + N_w \rightarrow CN \)

\[ T = 2180K, p_s = 15mbar \]
Approach for ablation modelling (Kendall et al.[1])

VKI: 1D Stagnation line description w/ surface ablation
(A. Turchi AIAA 2014-2125)

Surface Mass Balance (SMB)

Approach for ablation modelling (Kendall et al.\cite{1})

VKI: 1D Stagnation line description w/ surface ablation
(A. Turchi AIAA 2014-2125)

\textbf{Surface Energy Balance (SEB)}

- convective flux
- enthalpy by diffusion
- \textcolor{red}{HOT GAS} radiation
- re-radiation
- convected enthalpy

\textbf{Surface Mass Balance (SMB)}

- mass blowing
- \textcolor{red}{HOT GAS} conduction
- \textcolor{red}{chem. active surface}
- species diffusion

Boundary condition from experiments & plasma free-stream

Experimental data for validation

GOAL: Coupling 1-D SL-code & material code

(PhD Candidate P. Schrooyen)
Boundary Layer Radiation Profiles
Numerical: Simplified approach using Specair slab

Simulate line-of-sight measurement

locations $x_i$

stagn. line solution $\chi_i, T_i, p_i$,

slab width $\Delta y_i$ at $x_i$

boundary layer

SPECAIR $I_i(\lambda)$
Boundary Layer Radiation Profiles
Numerical: Simplified approach using Specair slab

Simulate line-of-sight measurement

locations $x_i$

stagn. line solution $\chi_i, T_i, p_i,$

slab width $\Delta y_i$ at $x_i$

$\text{SPECAIR}$

$I_i(\lambda)$
Boundary Layer Radiation Profiles
Numerical: Simplified approach using Specair slab

Simulate line-of-sight measurement

locations $x_i$

stagn. line solution $\chi_i, T_i, p_i$

slab width $\Delta y_i$ at $x_i$

SPECAIR

$I_i(\lambda)$

Perspective:
Radiation Coupling
(PhD Candidate J.B. Scoggins)
Comparison of Boundary Layer Radiation Profiles
Experimental - Numerical

T = 2020K, $p_s = 200$ mbar

$I_{\lambda_1}^2$ [W/(m$^2$.sr)] vs. Distance from surface, mm

- Exp. data
- Polynomial fit
- 95% Conf. bnd
- Num. data
Comparison of Boundary Layer Radiation Profiles
Experimental - Numerical

T = 2020K, $p_s = 200$mbar

$T = 2783K, p_s = 200$mbar
Comparison of Boundary Layer Radiation Profiles
Experimental - Numerical

T = 2020K, $p_s = 200$ mbar

T = 2783K, $p_s = 200$ mbar
Comparison of Boundary Layer Radiation Profiles
Experimental - Numerical

- Locations of maxima
- BL thickness
- Order of magnitude
- no nitridation in model
- strong assumption of constant properties over slab

\[ I_{\lambda,1}^2 \text{ [W/(m}^2\text{.sr)]} \]

\( T = 2020\text{K, } p_s = 200\text{mbar} \)

\( T = 2783\text{K, } p_s = 200\text{mbar} \)
Pyrolysis-Gas Blowing Rate Determination
Non-pyrolyzing carbon-preform

Subsonic, 0.3MW/m², 15hPa

\[ m_{pg} + m_c = (\rho V)_w \]

\[ m_{pg} = m_{tot} - (V_{abl} \cdot \rho_c) \]

Carbon Preform (non-pyro) :

\[ m_c = m_{tot} = V_{abl} \cdot \rho_c \]
Mass Loss Carbon Preform: Weighed vs HSC

discrepancy:
- water
- initial density
- damage by deinstallation
AQ61 Pyrolysis-Gas Blowing Rates

STA (Simultaneous Thermal Analysis, VKI):
charred AQ61: $\rho_c = 80-85\% \rho_v$

Subsonic, $0.3\text{MW/m}^2$, 15hPa

<table>
<thead>
<tr>
<th></th>
<th>T</th>
<th>p</th>
<th>m</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>AQ1</td>
<td>2167</td>
<td>15</td>
<td>1.12</td>
<td>23</td>
</tr>
<tr>
<td>AQ6</td>
<td>2884</td>
<td>15</td>
<td>1.77</td>
<td>44</td>
</tr>
<tr>
<td>AQ7</td>
<td>2906</td>
<td>200</td>
<td>2.26</td>
<td>60</td>
</tr>
</tbody>
</table>

Main challenges:
Side-wall outgassing, non-1D effects, too-long test times

compare to num. model
Conclusions

(1) Materials and Methods
- hemispherical samples
- HSC imaging → volume recession → (4)
- IR-camera: emissivity

(2) Char blowing rates
- higher recession for fibers through-grain
- diffusion limited ablation & sublimation (P+AQ61)
- Nitrogen: sublimation and nitridation of surface (SEM)

(3) BL emission
- steady ablation process
- num. estimation: order of magn., location of maxima, BL thickness

(4) Pyrolysis outgassing
- Vol. ablation + TGA → \( \dot{m}_{pg} \)
Future Work

Material:
- simultaneous TGA/DSC at VKI of resin, AQ61, Asterm (ongoing)
- additional SEM (fiber direction study)

Modeling:
- rebuild experiments with thermal response model (e.g. SAMCEF Amaryllis, or PATO)
- rebuild boundary layer emission with updated model (nitridation, recombination → talk A. Turchi)
- compare experimental pyrolysis outgassing rates to model
Acknowledgements

Funding and materials supply:

In particular:

> N.N. Mansour (NASA ARC), J. Lachaud (UC Santa Cruz), F. Panerai (University of Kentucky) for informative support

> Jean-Marc Bouilly & Gregory Pinaud (Airbus Defence & Space)

> VKI Plasmatron & Ablation Team

> VUB SURF research team
Oxidation of Preform & AQ61 in air

Oxidation of Preform and AQ61

Carbon preform oxidation

AQ61 oxidation
Preform & AQ61 in nitrogen
Nitridation Preform
Preform & AQ61 in nitrogen

Nitridation Preform
Preform & AQ61 in nitrogen

Nitridation AQ61

AQ61 nitrogen

AQ61 nitrogen
Preform & AQ61 in nitrogen
Nitridation AQ61

AQ61 nitrogen

AQ61 nitrogen
APPENDIX: Combined Rebuilding Procedure

Boundary Layer Solver

- **Input**: Boundary layer parameter (LTE CFD computation) & measurements from experiments

- **Procedure**: Iteration on boundary layer edge temperature $T_e$:
  \[ q_w^n = q_w^{\text{exp}} = q_w(\gamma, T_w, p_e, h_e, \beta) \]

- **Output**: Edge enthalpy $H_e$, boundary layer chemistry, (catalysis)
Complex Multiphysics - Multiscale problem
Coupled phenomena

Gas-phase
- Ablation species injection
- Strong radiators and boundary layer absorption

Gas-surface interaction
- Surface reactions & recombination
- Recession rate

Material
- Pyrolysis gases production and outgassing
CN Radiation Simulation for Temperature Estimation

Non-equilibrium?

ASTERM, $p_s = 15 \text{mbar}, T_S = 2130K$

ASTERM, $p_s = 100 \text{mbar}, T_S = 2097K$

Experimental spectrum

$\nu' = 3 \rightarrow \nu'' = 3$

$\nu' = 0 \rightarrow \nu'' = 0$
CN Radiation Simulation for Temperature Estimation

Non-equilibrium?

ASTERM, $p_s = 15$ mbar, $T_S = 2130K$

$T_{rot} = 9281K \pm 640K$
$T_{vib} = 14912K \pm 1100K$

ASTERM, $p_s = 100$ mbar, $T_S = 2097K$

$T_{rot} = 6311K \pm 350K$
$T_{vib} = 7878K \pm 320K$

Non-thermal vibrational level distribution at low pressure (AIAA 2013-2770)

⇒ Thermal non-equilibrium?
⇒ Deviation from Boltzmann distribution?
Boundary Layer Temperature Profile

Non-equilibrium at the wall?

$p_s = 15\text{mbar}, T_S = 2130\text{K}$

$p_s = 100\text{mbar}, T_S = 2097\text{K}$