Surface effects and phenomena on ablative materials

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Introduction
MSL predictions

- MSL TPS was a success: performed remarkably well
- The heat shield was instrumented for TPS performance
- Predictions do not match arc-jet test (under-prediction)
- Predictions do not match flight data (over-prediction)

Charring ablators are made by impregnation of phenolic resin on preform matrix.

The material has two “phase”, consisting of a network of carbon fibers and a very porous phenolic matrix dispersed on and in between the fibers.

The highly porous structure makes it a low-density material (ablator).

Micrographs of PICA at different magnifications. (Agrawal et al., 2013)

Charring ablators

- At first, when the material is subjected to heat, the phenolic pyrolyzes, while the carbon fibers stay (essentially) intact

- After a while, the surface starts to recess, as the matrix ablates

- The charring phenomenon enhanced the effectiveness of the TPS in two distinct ways:
  
  - **Mitigates the problem**, by absorbing the heat inside the TPS (endothermic pyrolysis reactions, char cooling)
  
  - **Circumvents the problem**, by attenuating the heat flux before it reaches the TPS (blowing gas in the outer flow, transport property in the boundary layer)

KATS Modeling Framework

- **General**
  - written in C++
  - reads 3D Unstructured grid in CGNS format

- **Parallelization**
  - ParMETIS for domain decomposition
  - MPI for inter-processors communications
  - PETSC Krylov subspace method as linear solver for iteration

- **Spatial discretization**
  - Cell-centered finite volume method
  - Second-order central differencing

- **Time integration**
  - Fully implicit
  - First-order backward Euler time integration

- **Inviscid fluxes scheme**
  - Steger–Warming flux-vector splitting, AUSM+up, Roe, etc.

- **Numerical flux Jacobian and analytical source Jacobian**
3D Conservation equations

\[ \frac{\partial Q}{\partial t} + \nabla \cdot (F - F_d) = S \]

Weak form after integrating over a finite volume \( V \)

\[ \int_V \frac{\partial Q}{\partial t} \, dV = \int_V \nabla \cdot (F_d - F) \, dV + \int_V S \, dV. \]

Apply Gauss theorem

\[ \int_V \frac{\partial Q}{\partial t} = \int_F (F_d - F) \cdot n + \int_V S \]

Implicit backward Euler time integration; Primitive variables is introduced

\[ V_c l \frac{\partial Q}{\partial t} = \sum_{face} (F_d - F)_j \cdot n_j A_j + S V_c l \equiv R^{n+1} = R^n + \frac{\partial R^n}{\partial P} \Delta P \]

Update of primitive variables comes from

\[ \left[ \frac{V}{\Delta t} \frac{\partial Q}{\partial P} - \frac{\partial R}{\partial P} \right] \Delta P = R, \]
Fluid Governing Equation (low speed)

\[
Q = \begin{pmatrix}
\rho_1 \\
\vdots \\
\rho_{ngs} \\
\rho_g u \\
\rho_g v \\
\rho_g w \\
E
\end{pmatrix}, \quad
P = \begin{pmatrix}
p_1 \\
\vdots \\
p_{ngs} \\
u \\
v \\
w \\
T
\end{pmatrix}, \quad
S = \begin{pmatrix}
\dot{\omega}_1 \\
\vdots \\
\dot{\omega}_{ngs} \\
0 \\
0 \\
0 \\
0
\end{pmatrix},
\]

\[
F = \begin{pmatrix}
\rho_1 u & \rho_1 v & \rho_1 w \\
\vdots & \vdots & \vdots \\
\rho_{ngs} u & \rho_{ngs} v & \rho_{ngs} w \\
\rho_g u^2 + p & \rho_g vu & \rho_g wu \\
\rho_g uv & \rho_g v^2 + p & \rho_g wv \\
\rho_g vw & \rho_g vw & \rho_g w^2 + p \\
(E + p)u & (E + p)v & (E + p)w
\end{pmatrix},
\quad
F'_d = \begin{pmatrix}
-J_1 \\
\vdots \\
-J_{ngs}
\end{pmatrix},
\quad
\begin{pmatrix}
\tau_{xx} & \tau_{xy} & \tau_{xz} \\
\tau_{yx} & \tau_{yy} & \tau_{yz} \\
\tau_{zx} & \tau_{zy} & \tau_{zz}
\end{pmatrix},
\quad
\left(\tau u - q - \sum_{i=1}^{ngs} (J_i h_i)\right)
\]
Material Response Governing Equation

\[ Q = \begin{pmatrix} \phi \rho_{g1} \\ \vdots \\ \phi \rho_{gns} \\ \rho_{s1} \\ \vdots \\ \rho_{s_{nss}} \\ \phi \rho_{g} u \\ \phi \rho_{g} v \\ \phi \rho_{g} w \\ \phi E_g + E_{s} \end{pmatrix}, \quad P = \begin{pmatrix} \phi \rho_{g1} \\ \vdots \\ \phi \rho_{gns} \\ \rho_{s1} \\ \vdots \\ \rho_{s_{nss}} \\ u \\ v \\ w \\ T \end{pmatrix}, \quad S = \begin{pmatrix} \dot{\omega}_{g1} \\ \vdots \\ \dot{\omega}_{gns} \\ \dot{\omega}_{s1} \\ \vdots \\ \dot{\omega}_{s_{nss}} \\ D_x \\ D_y \\ D_z \\ S_D \end{pmatrix} \]

- **Mass flux**

- **Momentum fluxes**

- **Energy flux**

- Convective fluxes

- Diffusive fluxes
Material Response Governing Equation

- Porous medium flow model
  - Gas momentum is solved as distinct momentum equations
  - Diffusive effect of porous media is treated as source term,
    \[ D_x, D_y, \text{ and } D_z \]
    \[
    \begin{pmatrix}
    K_{xx} & K_{xy} & K_{xz} \\
    K_{yx} & K_{yy} & K_{yz} \\
    K_{zx} & K_{zy} & K_{zz}
    \end{pmatrix}
    \begin{pmatrix}
    D_x \\
    D_y \\
    D_z
    \end{pmatrix}
    = -\phi \mu
    \begin{pmatrix}
    u \\
    v \\
    w
    \end{pmatrix}
    \]
- If transverse isotropy is assumed
  \[
  \begin{pmatrix}
  K_{xx} \\
  K_{yy} \\
  K_{zz}
  \end{pmatrix}
  \begin{pmatrix}
  D_x \\
  D_y \\
  D_z
  \end{pmatrix}
  = -\phi \mu
  \begin{pmatrix}
  u \\
  v \\
  w
  \end{pmatrix}
  \]
  \[ D_x = -\frac{\phi \mu}{K_{xx}} u, \quad D_y = -\frac{\phi \mu}{K_{yy}} v, \quad D_z = -\frac{\phi \mu}{K_{zz}} w. \]
- Conductive heat transfer model
  - Similarly, if the material is transverse isotropic
    \[
    F_{\text{cond},x} = k_{xx} \frac{\partial T}{\partial x}, \quad F_{\text{cond},y} = k_{yy} \frac{\partial T}{\partial y}, \quad F_{\text{cond},z} = k_{zz} \frac{\partial T}{\partial z}.
    \]
Coupling strategy

• Two meshes carefully aligned at the interface

• Resulting interface values are taken as boundary conditions for both solver

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6th Ablation Workshop, April 10-11 2014, Champaign IL
Test-case: Flow tube experiment

- Flow in a tube, then in a porous sample
- Laminar flow (Re = 2.3)
- “Free” flow using KATS-CFD, porous media using KATS-MR
- Both code integrated using flux coupling

\[
\begin{align*}
\text{sample} & \quad \text{inlet} & \quad \text{outlet} \\
\end{align*}
\]
Filled cylinder ablator

Figure 3. Photograph of virgin (left) and oxidized (right) FiberForm samples.

In Fig. 5 the surface temperature measured with the pyrometer is plotted as a function of time for sample a7, tested at a furnace temperature of 1300 K. During the heating phase, with He flow, the temperature of the sample gradually increases to the target value. Enough time is given for the temperature to stabilize to the set value. As the gas flow is switched from He to air and the oxidation phase is started, a jump in surface temperature is observed (a magnified view is shown on the right plot of Fig. 5). As predicted, the jump is due to the exothermic nature of oxidation reactions, causing the surface temperature to increase. This is also confirmed by the numerical predictions presented later in the paper. Values of $T = T_w$, where $T_w$ is the temperature of the surface and $T$ the target furnace temperature, are found to be between 9 and 13 K.

Figure 4. Scanning electron micrographs of virgin (left) and oxidized (right) FiberForm R.

-30000
-20000
-10000
0
10000
20000
30000
40000
50000
60000
70000
80000
90000
100000
110000

(a) Centerline pressure
(b) Centerline temperature
(c) Centerline Density
(d) Centerline velocity

No overall surface recession of the sample is observed for temperatures ﾁ900 K. At these conditions, oxidation is purely a volumetric process. The time scale of reactions is longer than that of diffusional transport in the porous medium (reaction-limited regime, small Thiele numbers). Higher temperatures result in faster reactions and the in-depth oxidation processes slow until oxidation becomes a purely surface phenomenon.
Hollow cylinder ablator

The plot, proposed in [9], presents Knudsen number (mean free path) transport conditions within the porous material, as shown in Fig. 4. Below the 220 mm of the upstream extension tube.

The test gas supply caused the pressure to rise to the target value, where the regulation is taken over by the PID controller to have a duration of 5 min. For the present setup, entrance length \( L = 25.4 \text{ mm} \) and pressure are maintained constant throughout the test time. The transient phase to the target pressure is monitored by adjusting the suction rate of the mechanical pump downstream of 100 Pa and a restoring of the He flow, during the cooling phase of the system.

Oxidation time during each run. Gas properties are calculated using the Chemical Equilibrium with Applications code [16] from the NASA Glenn Research Center. The Reynolds number \( \text{Re} \) confirms that the flow is fully developed at the sample location. For a laminar flow, the entrance length \( L \approx D_0.06 \text{Re} \mu / \rho u \text{D} \), well documented in Sec. III. The characteristic scale of the ablation is typically continuum during the portion of reentry where ablation is relevant [17]. The characteristic scale of the fibrous ablator is much larger than that of the characteristic scale of the oxidizer [18]. The characteristic scale of the oxidizer is much smaller than that of the characteristic scale of the reentry [19].

The ratio between the mass measured with the balance and the volume calculated from caliper measurements. Initial densities of the specimens are found to easily crumble into pieces in any attempt of manual handling, especially for those resulting from oxidation processes. Therefore, the specimen/holder assembly is impregnated in epoxy resin, by means of an encapsulation process with Struers A/S, Ballerup, Denmark) is poured into a plastic mold hosting the assembly and oven-cured for 18 h. A resin/hardener mixture (CaldoFix, LL7100, Struers A/S, Ballerup, Denmark) is poured into a plastic mold hosting the assembly and oven-cured for 18 h. A resin/hardener mixture (CaldoFix, LL7100, Struers A/S, Ballerup, Denmark) is poured into a plastic mold hosting the assembly and oven-cured for 18 h.

Prior to and post flow-tube oxidation, mass measurements are documented in Sec. III. The encapsulated samples are cut into specimens by means of a diamond blade, by means of a diamond blade. Mass measurements are performed using an analytical balance (AB104S, Mettler-Toledo, Columbus, Ohio) with an uncertainty of \( 0.1 \text{ mg} \). The mass of the specimens is determined from the mass difference between the solvent sample and the solvent/holder assembly.

The thermal properties of the samples are determined following the method described in [20]. The bulk density of the material is estimated as \( \rho_A = 165 \text{ kg m}^{-3} \), with an uncertainty of \( 0.0006 \text{ kg m}^{-3} \). Density changes for all specimens are easily measured with a precision of \( 0.1 \text{ mg} \).

Different techniques are applied to analyze the carbon preform specimen brittleness of the carbon preform specimens. The oxidized samples are clearly indicating a laminar flow. The calculated Reynolds number \( \text{Re} \) is below 3.7 at all test conditions, as clearly indicated by the flow visualization in Fig. 2. The entrance length \( L \approx D_0.06 \text{Re} \mu / \rho u \text{D} \), well documented in Sec. III. The characteristic scale of the ablation is typically continuum during the portion of reentry where ablation is relevant [17]. The characteristic scale of the fibrous ablator is much larger than that of the characteristic scale of the oxidizer [18]. The characteristic scale of the oxidizer is much smaller than that of the characteristic scale of the reentry [19].

To determine the exact volume recession due to oxidation for each specimen, the specimen/holder assembly is impregnated in epoxy resin, by means of an encapsulation process with Struers A/S, Ballerup, Denmark) is poured into a plastic mold hosting the assembly and oven-cured for 18 h. A resin/hardener mixture (CaldoFix, LL7100, Struers A/S, Ballerup, Denmark) is poured into a plastic mold hosting the assembly and oven-cured for 18 h. A resin/hardener mixture (CaldoFix, LL7100, Struers A/S, Ballerup, Denmark) is poured into a plastic mold hosting the assembly and oven-cured for 18 h.

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Pyrolysis gas transport
Why we need 3D modeling?

- Side-wall heating on small samples
- Gas transport inside the material
- Anisotropic material properties
Iso-Q geometrical blowing effects

Temperature

Pressure

Strong blowing through the sides

Solid density

Mass flux
Iso-Q geometrical blowing effects

- The TACOT (Theoretical Ablative Composite for Open Testing) is used — similar to PICA
- Only charring ablation is modeled: no surface recession allowed to occur
- Four geometries with different aspect ratio H/R are chosen

![Geometries](image1)

![Heat flux](image2)
Iso-Q geometrical blowing effects

Sample A

Sample B

Sample C

Sample D
2) Cylinder — permeable side wall

Table 2: Geometric specifications for Case 2.1 to 2.4

<table>
<thead>
<tr>
<th>Sample-holder location</th>
<th>Case #</th>
<th>( d ), cm</th>
<th>( h ), cm (( x_1, z_1 )) to (( x_2, z_2 )), cm</th>
</tr>
</thead>
<tbody>
<tr>
<td>A-</td>
<td>2.1</td>
<td>2.5</td>
<td>6 (0.75,-6) to (1.25,-4)</td>
</tr>
<tr>
<td>B-</td>
<td>2.2</td>
<td>5</td>
<td>6 (1.5,-6) to (2.5,-4)</td>
</tr>
<tr>
<td>C-</td>
<td>2.3</td>
<td>10</td>
<td>6 (0,-6) to (3,-5.5)</td>
</tr>
<tr>
<td>D-</td>
<td>2.4</td>
<td>10</td>
<td>3 (0,-3) to (3,-2.5)</td>
</tr>
</tbody>
</table>

Figure 4: Sample geometry for sample A- to D-

The initial and boundary condition of the two sets are identical, and are adopted from the arc-jet experiments of Ref. [13]. These conditions are summarized in Table 3. Note that, along the iso-Q sample surface, the pressure...
Iso-Q geometrical blowing effects

Figure 28: Gas streamlines for Case 2.1, at various time

Figure 29: Gas mass flow rate through the surface of iso-Q sample

Figure 30: Gas mass flow rate through the surface of iso-Q sample

Figure 31: Gas mass flow rate through the surface of iso-Q sample

Figure 32: Gas mass flow rate through the surface of iso-Q sample

Figure 33: Gas mass flow rate through the surface of iso-Q sample

Figure 34: Gas mass flow rate through the surface of iso-Q sample

Figure 35: Gas mass flow rate through the surface of iso-Q sample

Figure 36: Gas mass flow rate through the surface of iso-Q sample

Figure 37: Gas mass flow rate through the surface of iso-Q sample

Figure 38: Gas mass flow rate through the surface of iso-Q sample

Figure 39: Gas mass flow rate through the surface of iso-Q sample

Figure 40: Gas mass flow rate through the surface of iso-Q sample

Figure 41: Gas mass flow rate through the surface of iso-Q sample

Figure 42: Gas mass flow rate through the surface of iso-Q sample

Figure 43: Gas mass flow rate through the surface of iso-Q sample

Figure 44: Gas mass flow rate through the surface of iso-Q sample

Table 4: Gas mass flow rate through the stagnation point (arc length = 0 m)

<table>
<thead>
<tr>
<th>Sample</th>
<th>5 sec</th>
<th>10 sec</th>
<th>20 sec</th>
<th>30 sec</th>
<th>5 sec B-</th>
<th>10 sec B-</th>
<th>20 sec B-</th>
<th>30 sec B-</th>
</tr>
</thead>
<tbody>
<tr>
<td>A-</td>
<td>0.05</td>
<td>0.03</td>
<td>0.02</td>
<td>0.01</td>
<td>0.04</td>
<td>0.02</td>
<td>0.01</td>
<td>0.008</td>
</tr>
<tr>
<td>B-</td>
<td>0.07</td>
<td>0.05</td>
<td>0.03</td>
<td>0.02</td>
<td>0.06</td>
<td>0.04</td>
<td>0.02</td>
<td>0.01</td>
</tr>
<tr>
<td>C-</td>
<td>0.09</td>
<td>0.07</td>
<td>0.05</td>
<td>0.04</td>
<td>0.08</td>
<td>0.06</td>
<td>0.04</td>
<td>0.03</td>
</tr>
<tr>
<td>D-</td>
<td>0.11</td>
<td>0.09</td>
<td>0.07</td>
<td>0.06</td>
<td>0.10</td>
<td>0.08</td>
<td>0.06</td>
<td>0.05</td>
</tr>
</tbody>
</table>
Stagnation point blowing rates for all samples

<table>
<thead>
<tr>
<th>$t$, sec</th>
<th>1D</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>A-</th>
<th>B-</th>
<th>C-</th>
<th>D-</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>3.01E-02</td>
<td>4.64E-03</td>
<td>6.76E-03</td>
<td>9.65E-03</td>
<td>1.19E-02</td>
<td>4.37E-03</td>
<td>6.51E-03</td>
<td>9.78E-03</td>
<td>1.30E-02</td>
</tr>
<tr>
<td>10</td>
<td>2.33E-02</td>
<td>1.61E-03</td>
<td>2.25E-03</td>
<td>3.56E-03</td>
<td>4.66E-03</td>
<td>1.60E-03</td>
<td>2.07E-03</td>
<td>3.73E-03</td>
<td>5.63E-03</td>
</tr>
<tr>
<td>20</td>
<td>1.76E-02</td>
<td>4.99E-04</td>
<td>6.31E-04</td>
<td>9.52E-04</td>
<td>1.27E-03</td>
<td>4.30E-04</td>
<td>5.78E-04</td>
<td>1.07E-03</td>
<td>2.49E-03</td>
</tr>
<tr>
<td>30</td>
<td>1.49E-02</td>
<td>2.33E-04</td>
<td>6.46E-04</td>
<td>9.62E-04</td>
<td>1.79E-03</td>
<td>1.05E-04</td>
<td>6.55E-04</td>
<td>1.08E-03</td>
<td>6.01E-03</td>
</tr>
</tbody>
</table>

- Could be several order of magnitude of difference when compared to the 1D model
Why we need 3D modeling?

- Side-wall heating on small samples
- Gas transport inside the material
- Anisotropic material properties

NASA’s MSL entry capsule

Courtesy: NASA Ames Arc-Jet facility
Multi-scale approach
Micro-scale approach

- Light weight charring ablators (such as PICA) are composed of a fiber matrix impregnated by a resin
- Only very recently has the capability to access real microstructure become possible both experimentally and computationally
- To understand the behavior of the material at the macro-scale, the micro-scale effects are taken into account

The artificial material misses several features!
Fiber-scale features are accurately resolved
Orthotropic geometry and thermophysical properties are confirmed for preform composites
Analysis of the tomographs allows accurate computations of important properties for
material response modeling (tortuosity, permeability, porosity) etc.
Reconstruction of porous carbon materials
Spallation
Spallation

- Mechanical erosion of the material
- Accelerates material failure
- Undesirable because hard to predict (and model...)
- Can be caused by
  - Fracture from high pyrolysis gas pressure
  - Soot formation (coking)
  - Volumetric fiber erosion and detachment
  - Fracture from high thermal stress
  - Shear stress on fibers

![Post test image]

Agrawal et al., 2013
Thermo-Mechanical Response

- Most specimens failed between 600 and 900 kPa
- The charred PICA samples made from arc-jet articles showed similar behavior and tensile strength as the furnace char.

- 10 sec of constant heat flux
- High stress region does not correspond to high temperature region
- When combine with volumetric fiber ablation, could results in mechanical erosion

(Parul Agrawal, 2013)
Spalled particles in arc-jet environment

Mach 5 High-enthalpy Argon flow

Radius effect
Angle effect
Velocity effect

- One way coupling of CFD and Lagrangian particle code
- Surface kinetics on particle
- Inverse-problem approach for parameter identification


DLR H2K wind tunnel test, Cologne, Germany

Arc-Jet at NASA Ames Research Center

6th Ablation Workshop, April 10-11 2014, Champaign IL
Concluding remarks

• Development of models to capture
  • multi-scale effects
  • coupling effects
• Detailed experiments aimed at
  • understanding near surface properties
  • calculating micro-scale parameters
Questions?