A LIGHT-WEIGHT ABLATIVE MATERIAL FOR RESEARCH PURPOSES

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Objectives and project partners

Development of a light-weight ablative material for research purposes:

- Understand the key factors to design ablative materials
- Better understanding of underlying physics

Participating institutes:

- Institute of Structures and Design, German Aerospace Center
- Institute of Space Systems, University of Stuttgart,
- Institute of Aerospace Thermodynamics, University of Stuttgart
Loads during atmospheric re-entry

Steep re-entry

I. Lifting re-entry (e.g. Space Shuttle, SHEFEX)
   - Heat flux $q_{\text{Space Shuttle}} = 0.75 \text{ MW/m}^2$
   \[ \rightarrow \text{Reusable thermal protection materials suited e.g. C/C-SiC} \]
   - Heat flux $q_{\text{C/C-SiC}} \leq 1 \text{ MW/m}^2$
   - $T_{\text{max}} \leq 1700 \degree \text{C}$

II. Steep re-entry (e.g. Stardust capsule hyperbolic $v = 12.9 \text{ km/s}$)
   - Heat flux $q_{\text{Stardust}} = 12 \text{ MW/m}^2$
   \[ \rightarrow \text{Ablator} \]
Charring ablation

- Transpiration Cooling
- Re-radiative Cooling
- Radiative & Convective Heating

Boundary Layer

Melt Layer

Char Layer

Pyrolysis Zone

Virgin Material

Structure Interior

Chemical Phenomena

Gas Reactions

Wall Catalysis, Oxidation, Corrosion, Vaporization, Sublimation, Dissociation, Combustion

Mechanical Phenomena

Spallation, Erosion, Surface Recession

Char Reactions

Pressure Buildup

Pyrolysis, Char Formation

Thermal Stress

[Rivell, 2006]
Mechanisms of action of charring ablator

Ablative mechanisms and derived requirements:

1. Energy conversion by endothermic reactions
   - Thermal decomposition of the resin

2. Reduction of the convective heat transfer
   - Emission of pyrolysis gases, lifting of a boundary layer

3. Reduction of the heat transfer by radiation
   - Emission of carbon particles

4. Heat dissipation by re-radiation
   - High emissivity
   - Temperature stability up to the radiative equilibrium temperature

5. Conversion of energy by phase change
   - Smelting or preferential sublimation processes
Additional requirements

1. Thermal isolation
   - Protection of the substructure (→ avoidance of high temperatures)
   - Causing high surface temperatures (→ beneficial for an effective heat emission by reflection)
     \[ M_{e,s} = \varepsilon \cdot \sigma \cdot T^4 \] (Stefan-Boltzmann equation)

2. Low specific system mass

3. Mechanically stable virgin ablator and char layer (→ aerodynamic loads)
Reference → Stardust

Stardust capsule [NASA]

Plasma wind tunnel PWK1 (IRS)

Test conditions:

<table>
<thead>
<tr>
<th></th>
<th>Gas</th>
<th>Air</th>
<th>12</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat flux [MW/m²]</td>
<td>2</td>
<td>6</td>
<td>12</td>
</tr>
<tr>
<td>Total pressure [hPa]</td>
<td>33,6</td>
<td>38,7</td>
<td>44,6</td>
</tr>
<tr>
<td>Test duration [s]</td>
<td>60</td>
<td>30</td>
<td>15</td>
</tr>
</tbody>
</table>

[Herdrich et al., 2009]
Material Screening tests

Variation of:

- Precursor resin  \(\rightarrow\) phenolic, epoxy, silicone, polyaromatic resin
- Fiber type  \(\rightarrow\) carbon fibers, mullite fibers
- Fiber length
- Fiber orientation  \(\rightarrow\) short fibers, fabric, felt

Objective:

- Investigation of influence of the variations onto the ablative material properties
Manufacturing processes

- Autoclave process
- Resin transfer molding
- Hot pressing process
Ablation sample for plasma wind tunnel tests

- Manufacturing of more than 72 samples
- Sample geometry: Ø 40 mm x 40 mm
- 5 thermocouples in a depth of 3, 5, 8, 15 and 40 mm related to the ablator front
Measurands

Before test:
- Specific gravity
- Open porosity
- Sample thickness
- Weight

During test:
- Temperature distribution

Post test:
- Pyrolysis zone
- Sample thickness
- Weight
Results of material screening tests
Ablative performance of precursor resin

Delaminated sample HP683#1 after test in plasma wind tunnel:
- 2D fabric reinforcement
- Phenolic precursor
- Test conditions: 6 MW/m², 30 s

→ Due to the massive delaminations an evaluation of the precursor with respect to ablation was not possible

→ 3D-reinforcement is necessary
Results of material screening tests
Pyrolysis zone on 3D-reinforced samples

CT-picture PWT sample HP691#4 after testing

Test conditions: 2 MW/m², 60 s

Cut view of PWT sample PH2075quer#1 after testing

Test conditions: 6 MW/m², 30 s
Results of material screening tests
Temperature distribution & fiber orientation

2D-fabric reinforcement

normal

PH2075#4

cross

PH2075quer#4
Results of material screening tests
Temperature distribution & fiber orientation

Temperature distribution:

<table>
<thead>
<tr>
<th></th>
<th>T [°C] (3 mm depth)</th>
<th>T [°C] (5 mm depth)</th>
<th>T [°C] (8 mm depth)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PH2075#4</td>
<td>1025</td>
<td>475</td>
<td>100</td>
</tr>
<tr>
<td>PH2075quer#4</td>
<td>1250</td>
<td>650</td>
<td>500</td>
</tr>
</tbody>
</table>
Results of material screening tests
Influence of reinforcement fiber type

PWT sample IP438 #4

Reinforcement fibers: Mullite fibers
Test conditions: 2 MW/m², 60 s
Damages: Molten mullite fibers (28 % SiO₂ + 72 % Al₂O₃)

→ Low heat conduction causes local heat peaks (critical at edges and narrow radius)

→ Mullite fibers exhibit melting (undesirable), carbon fibers sublimate

PWT sample IP455 #1

Reinforcement fibers: carbon felt
Test conditions: 2 MW/m², 60 s
New Manufacturing Process

Lessons learned from screening tests:
- 3D-reinforcement is necessary
- Avoid local heat peaks
- Use phenolic resin to generate high amount of residual carbon to reduce the radiative heat transfer (from literature research)

Carbon felt (Schunk K73) + phenolic resin

Modified process

Carbon fibers embedded into micro porous phenolic resin foam

ρ = 0.3 g/cm³

Carbon felt (Schunk K73) + phenolic resin + addition agent

ρ = 0.3 g/cm³
A new material
Zuram R

Carbon preform + phenolic resin + addition agent
A new material
Plasma wind tunnel tests

Test conditions: 12 MW/m², 15 s
Averaged recession: 1.80 mm
Mass loss: 1.92 g

Temperature distribution within ZURAM PWT sample
Characterization
DSC

Heat capacity of ZURAM R
Characterization

LFA

Heat conductivity in plane

Heat conductivity perpendicular to plane

→ Anisotropic behavior due to pre-form
Characterization
Mechanical

Compressive strength
Characterization
Properties of interest

- virgin and char density
- virgin and char thermal conductivity
- virgin and char heat capacity
- emissivity/ absorptivity
- thermal decomposition data
- elemental composition
- porosity/ permeability
- flow characteristics
- mechanical characteristics
- recession rates
Conclusions

- **Goal:**
  - Better understanding of behavior and underlying physics of ablative materials

- **Status and knowledge gained:**
  - A new material “ZURAM R” was developed
  - A new manufacturing process was developed
  - Tests, including PWT tests, were performed for characterization
  - From the material screening tests:
    - 3D reinforcement is necessary
    - Foam-like closed porous microstructure is desirable
    - Carbon fiber preform seems advantageous over aluminum oxide preform

- **Ongoing and prospective:**
  - Further material development, variation of material composition
  - Further characterizations with different load cases, in states other than virgin material and PWT shear tests are foreseen
Future Steps: An invitation to participate

Main interest:

- Research the important parameters on how to manufacture a better ablator
- Aim at a broad range of future scientific planetary and sample return missions
- Perform fundamental research on ZURAM; vary material properties to better understand its behavior at various conditions

- DLR has the capability to manufacture a reproducible ablative material (will be further confirmed by PWT test at DLR facilities in Cologne)

- Material composition could be modified to necessity or liking.
Future steps: An invitation to participate
TPS facility inter-calibration test

Providing common test material to facilities would allow for:
- Repeatability of test conditions in a facility
- Comparison of results gained in different facilities

- We would deliver 4 ISO-Q samples (e.g. ø 50 mm x 40 mm) for free, keep track of the samples and collect the results

- Measurands 1st round:
  - Temperature @ 5 locations inside the specimen
  - Total recession and mass loss
  - Flow characterization
  - + whatever you like to measure

Please regard as invitation for discussion.
Future steps: An invitation to participate
TPS facility inter-calibration test

**Additional result:** exhaustive and consistent set of material data

- Supplement or substitute synthetic model like TACOT (mid term)
- allow not only for verification but also validation of models
Questions? Comments?

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Thank you for your attention