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 $q_{\text{C/C-SiC}} \leq 1 \text{ MW/m}^2$
  - $T_{\text{max}} \leq 1700 ^\circ \text{C}$

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
- Chemical Phenomena
  - Gas Reactions
    - Wall Catalysis, Oxidation, Corrosion, Vaporization, Sublimation, Dissociation, Combustion
  - Char Reactions
  - Pressure Buildup
- Mechanical Phenomena
  - Spallation, Erosion, Surface Recession
  - 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>Gas</th>
<th>Air</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat flux [MW/m²]</td>
<td>2</td>
</tr>
<tr>
<td>Total pressure [hPa]</td>
<td>33,6</td>
</tr>
<tr>
<td>Test duration [s]</td>
<td>60</td>
</tr>
</tbody>
</table>

[Herdrich et al., 2009]
Material Screening tests

Variation of:

- Precursor resin
  - phenolic, epoxy, silicone, polyaromatic resin

- Fiber type
  - carbon fibers, mullite fibers

- Fiber length
- Fiber orientation
  - 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

Pyrometry
Thermography
Spectroscopy
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>Material</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:  Molten mullite fibers (28 % SiO₂ + 72 % Al₂O₃)
Test conditions: 2 MW/m², 60 s
Damages:

PWT sample IP455 #1
Reinforcement fibers: carbon felt
Test conditions: 2 MW/m², 60 s

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

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

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