A LIGHT-WEIGHT ABLATIVE MATERIAL FOR RESEARCH PURPOSES

Th. Rothermel, Ch. Zuber
Institute of Structures and Design, German Aerospace Center (DLR)

G. Herdrich
Institute of Space Systems (IRS), University of Stuttgart

L.M.G.F.M. Walpot,
ESA-ESTEC

Objectives and project partners

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

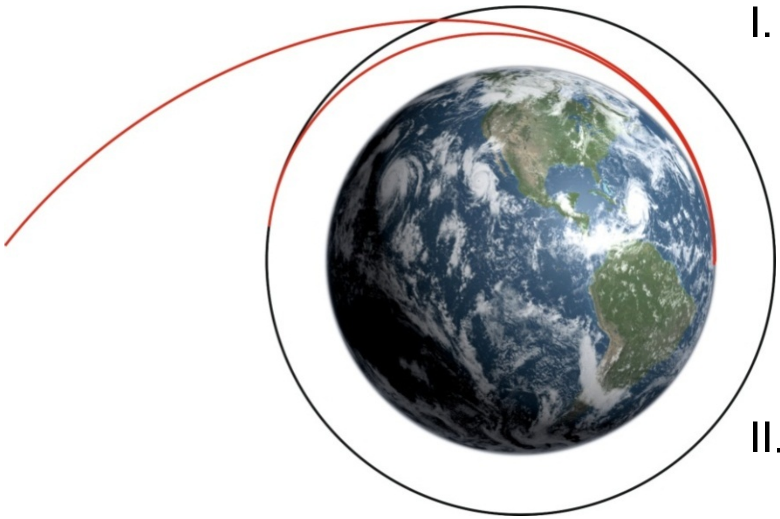
- Understand the key factors to design ablative materials
- Better understanding of underlying physics

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



Low Earth Orbit (LEO):
200 – 2000 km

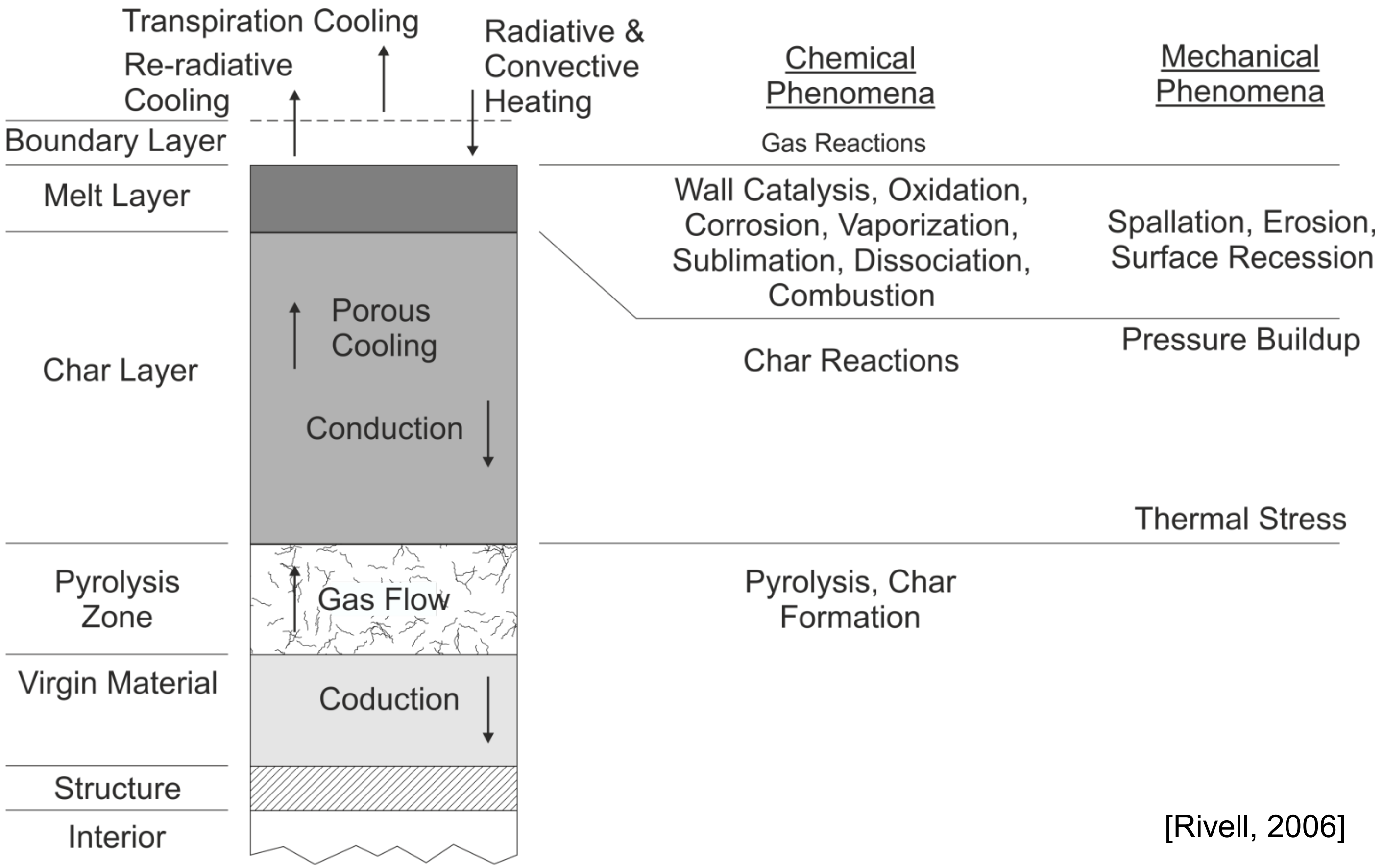
I. Lifting re-entry (e.g. Space Shuttle, SHEFEX)
   - Heat flux $q_{Space\ Shuttle}$ = 0,75 MW/m²

→ *Reusable thermal protection materials suited e.g. C/C-SiC*
   - *Heat flux* $q_{C/C\text{-}SIC} \leq 1$ *MW/m²*
   - $T_{max} \leq 1700$ *ºC*

II. Steep re-entry (e.g. Stardust capsule hyperbolic v = 12.9 km/s)
   - Heat flux $q_{Stardust}$ = 12 MW/m²

→ *Ablator*

Charring ablation

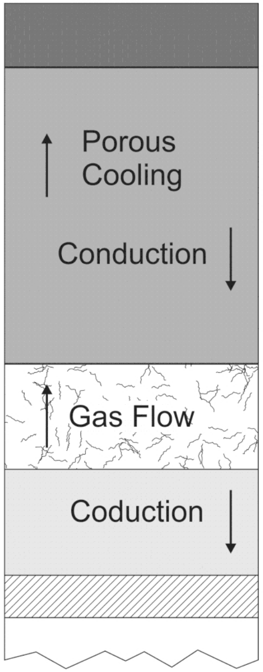


[Rivell, 2006]

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

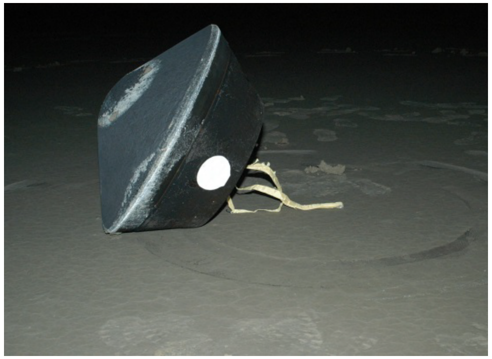
1. Thermal isolation
   - Protection of the substructure (→ avoidance of high temperatures)
   - Causing high surface temperatures (→ beneficial for an effective heat emission by reflection)

   $$M_{e,s} = \varepsilon \cdot \sigma \cdot T^4$$ (Stefan-Boltzmann equation)

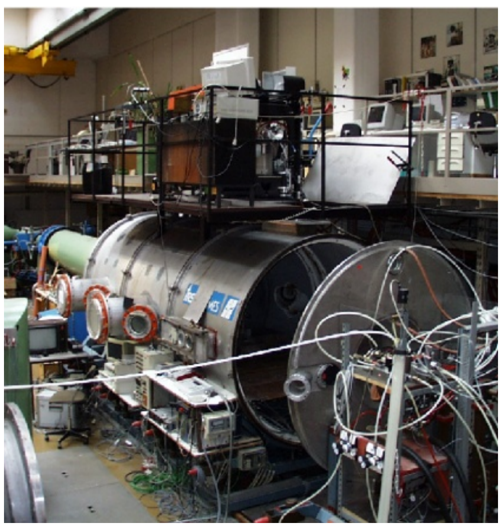
2. Low specific system mass

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

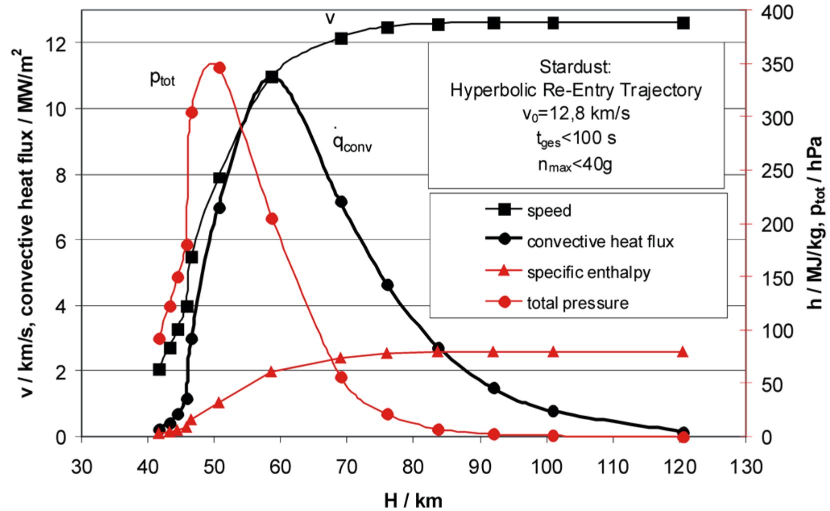
Reference → Stardust



Stardust capsule [NASA]



Plasma wind tunnel PWK1 (IRS)



[Herdrich et al., 2009]

Test conditions:

Gas	Air		
Heat flux [MW/m²]	2	6	12
Total pressure [hPa]	33,6	38,7	44,6
Test duration [s]	60	30	15

Material Screening tests

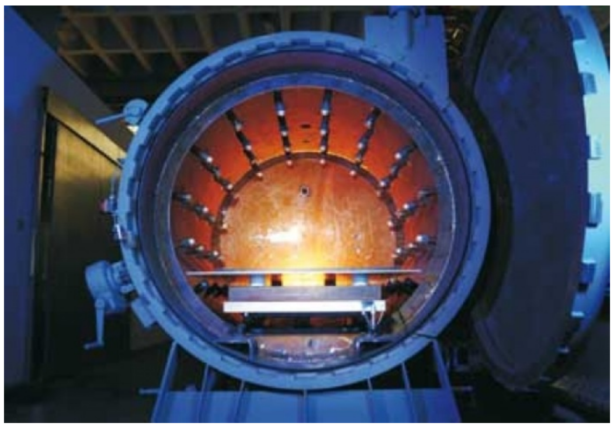
Variation of:

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

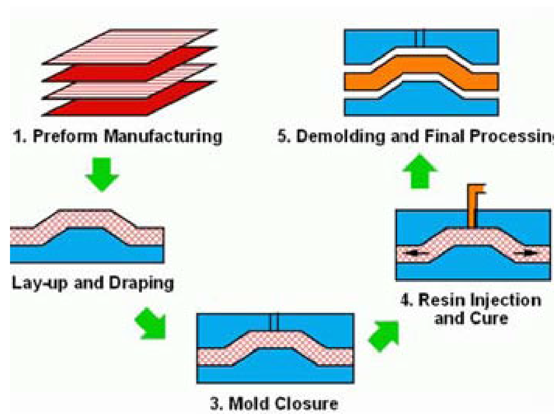
Objective:

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

Manufacturing processes



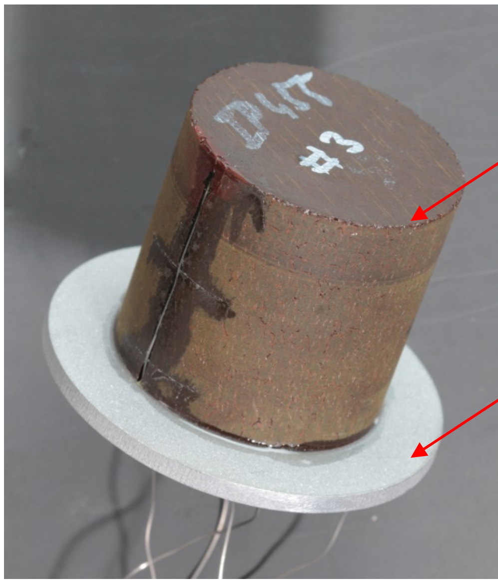
Autoclave process

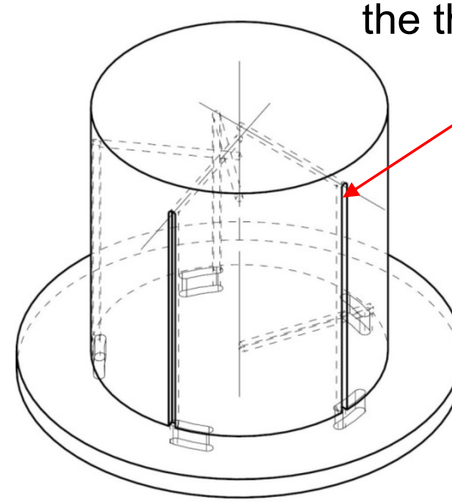


Resin transfer molding

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:

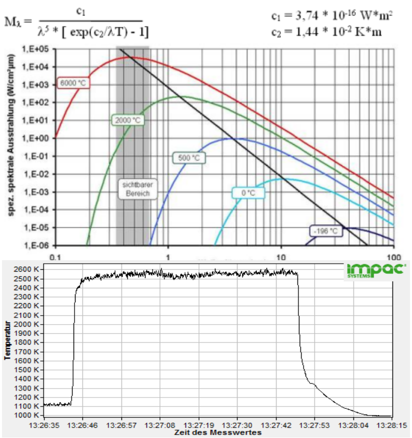
- Specific gravity
- Open porosity
- Sample thickness
- Weight

During test:

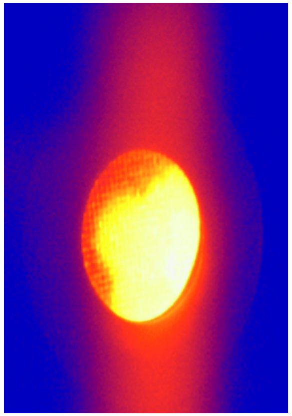
- Temperature distribution

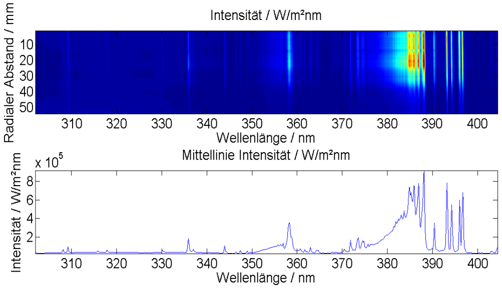
Post test:

- Pyrolysis zone
- Sample thickness
- Weight



Pyrometry


Thermography



Spectroscopy

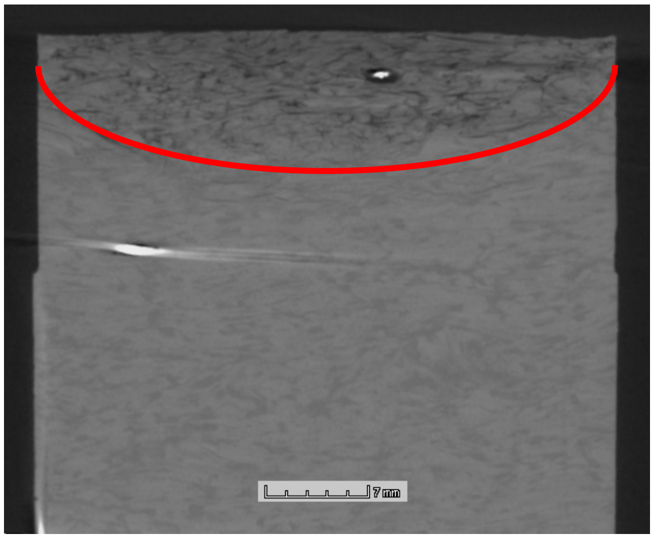
Results of material screening tests
Ablative performance of precursor resin

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

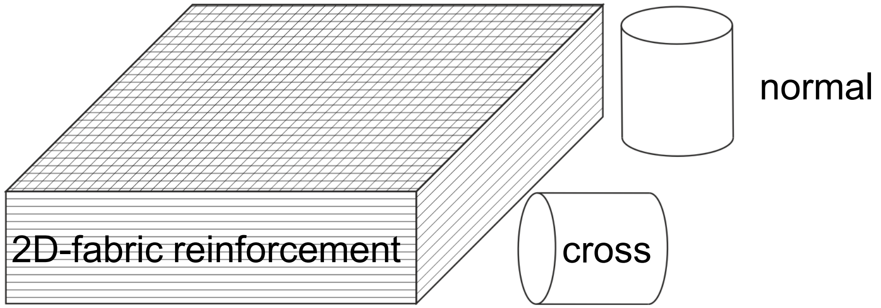
Test conditions: 2 MW/m², 60 s

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

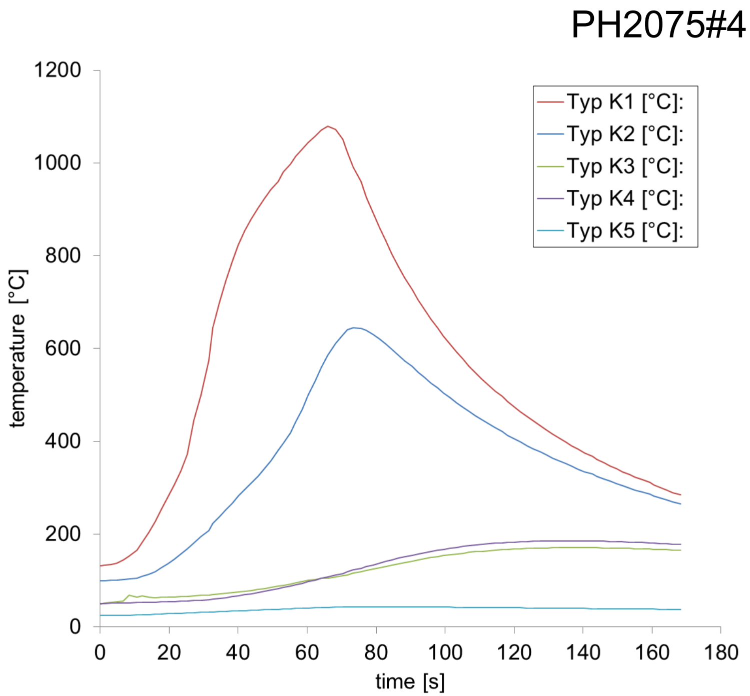


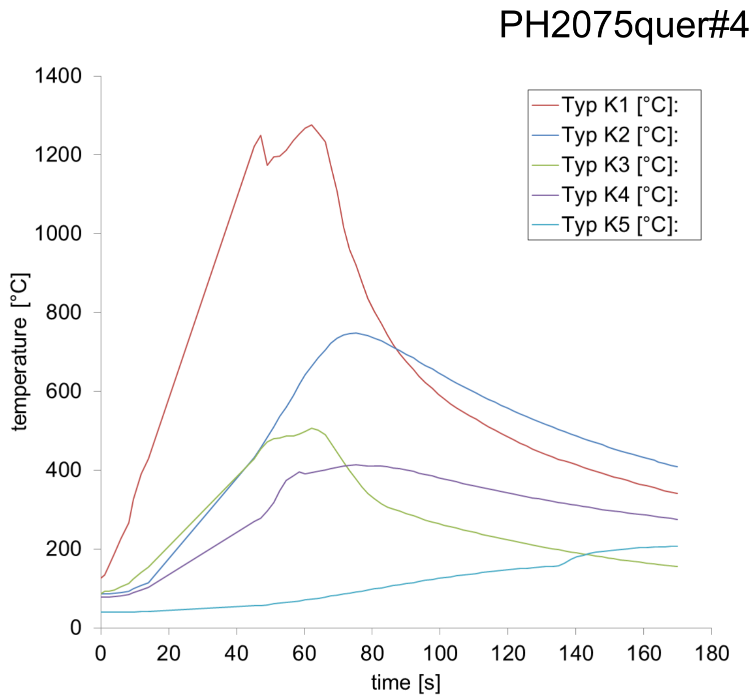
PH2075#4

PH2075quer#4

Results of material screening tests

Temperature distribution & fiber orientation





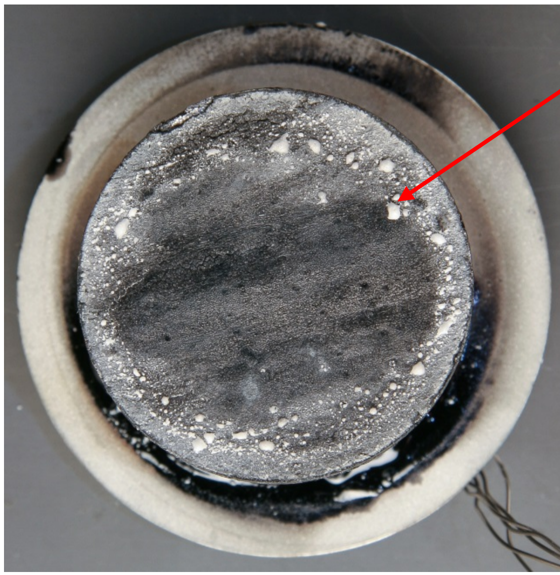
Temperature distribution:

	T [ºC] (3 mm depth)	T [ºC] (5 mm depth)	T [ºC] (8 mm depth)
PH2075#4	1025	475	100
PH2075quer#4	1250	650	500

Results of material screening tests

Influence of reinforcement fiber type

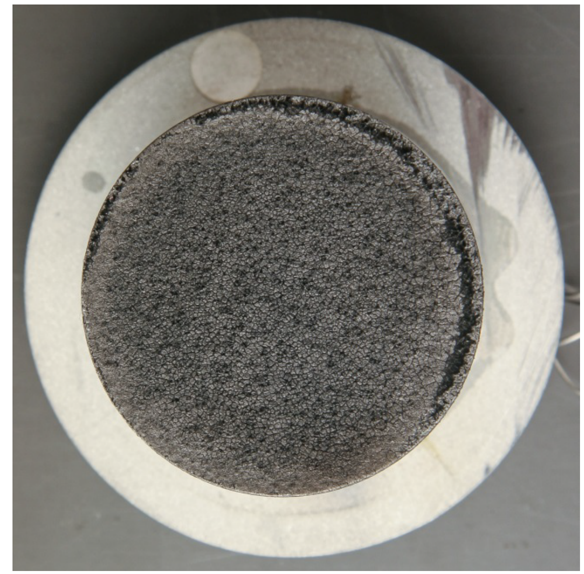
PWT sample IP438 #4



Molten mullite fibers ($28\ \%\ SiO_2$ + $72\ \%\ Al_2O_3$)

Reinforcement fibers: Mullite fibers
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)

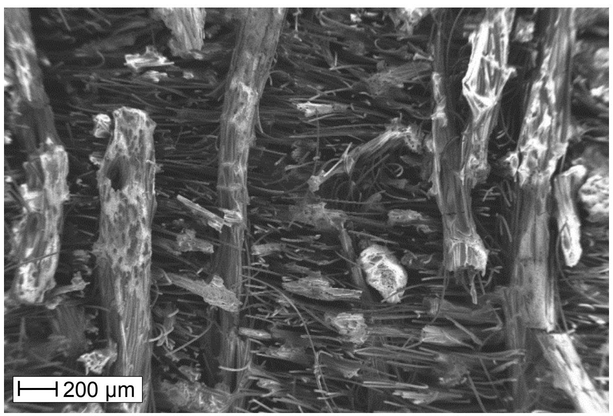
→ Mullite fibers exhibit melting (undesirable), carbon fibers sublimate

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



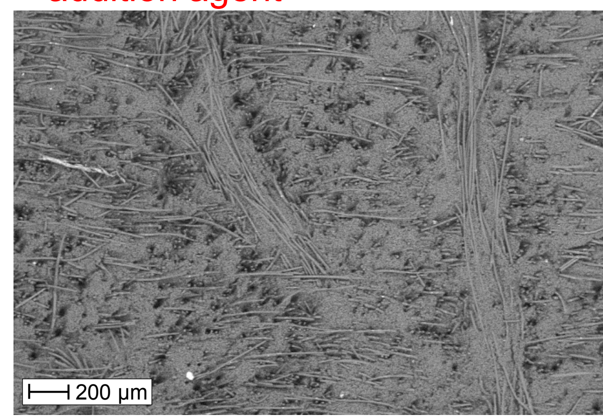
ρ = 0,3 g/cm³

Modified process

→

Carbon fibers embedded into micro porous phenolic resin foam

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



ρ = 0,3 g/cm³

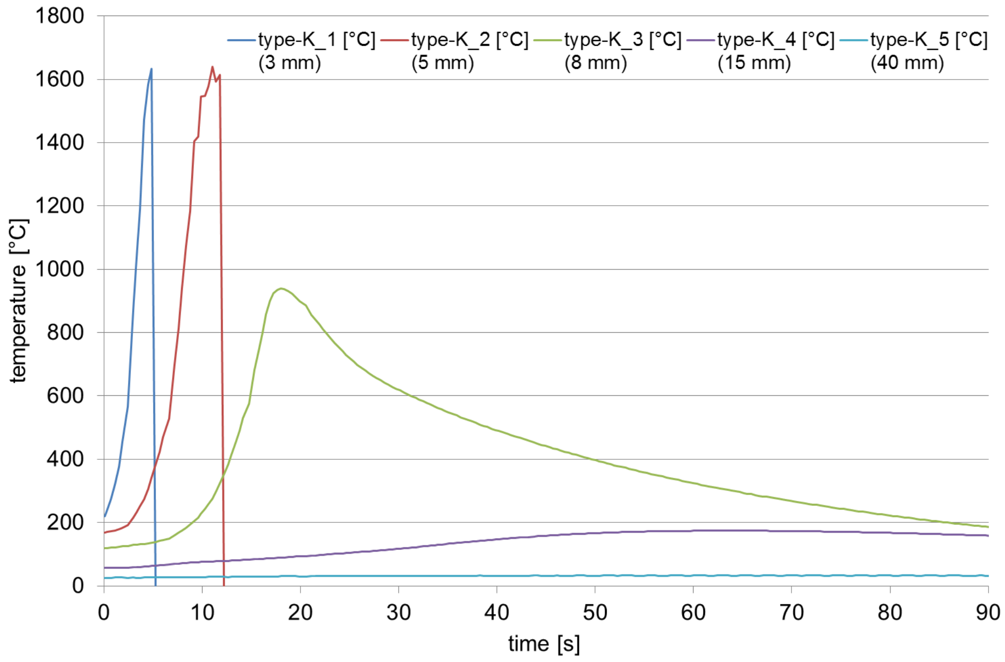
A new material
Zuram R

Carbon preform + phenolic resin + addition agent

A new material
Plasma wind tunnel tests

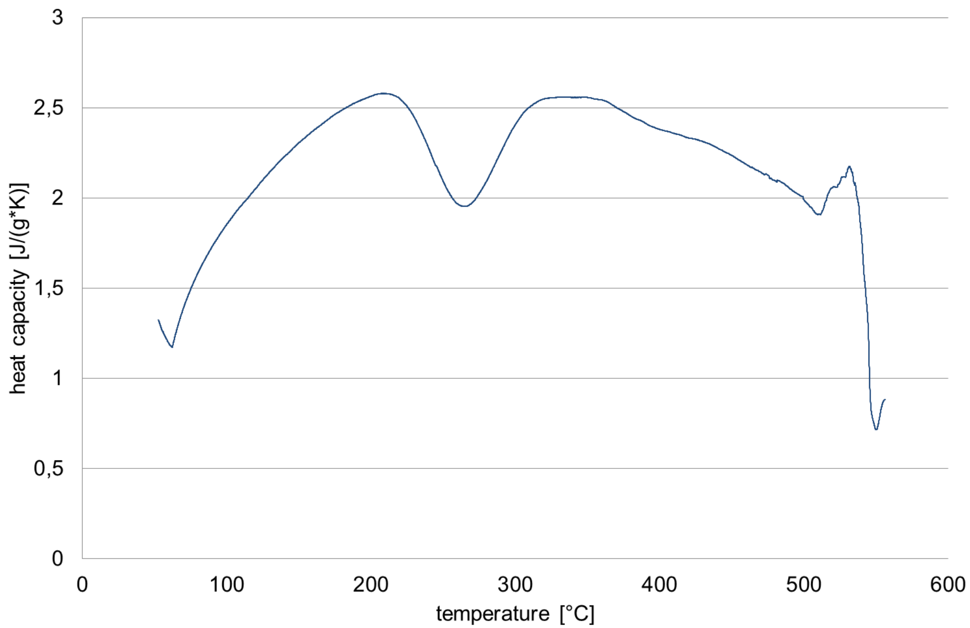
Test conditions: 12 MW/m², 15 s
Averaged recession: 1.80 mm
Mass loss: 1.92 g

ZURAM sample after PWT test



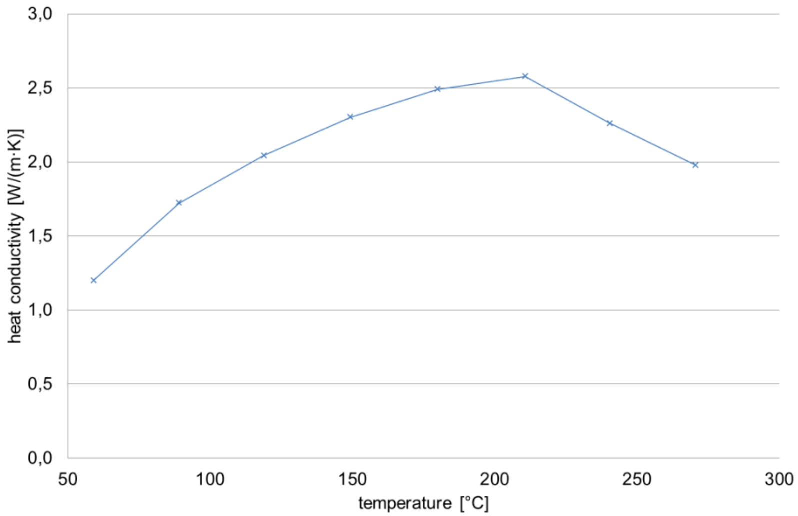
Temperature distribution within ZURAM PWT sample

Characterization
DSC

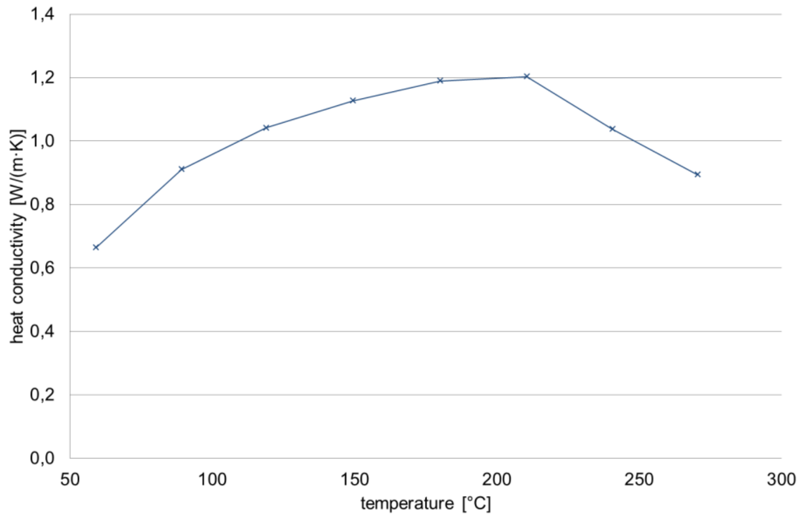


Heat capacity of ZURAM R

Characterization
LFA



Heat conductivity in plane

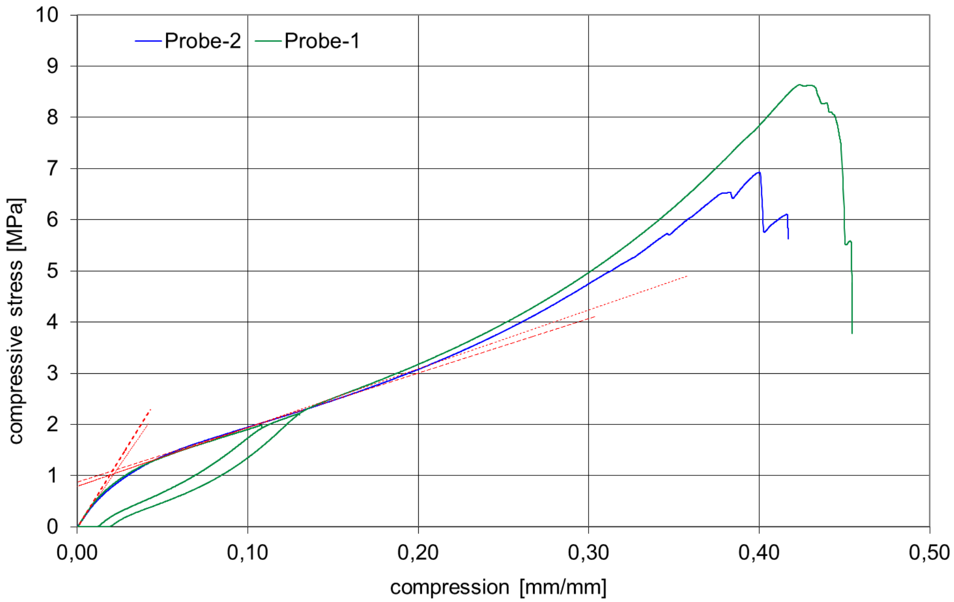


Heat conductivity perpendicular to plane

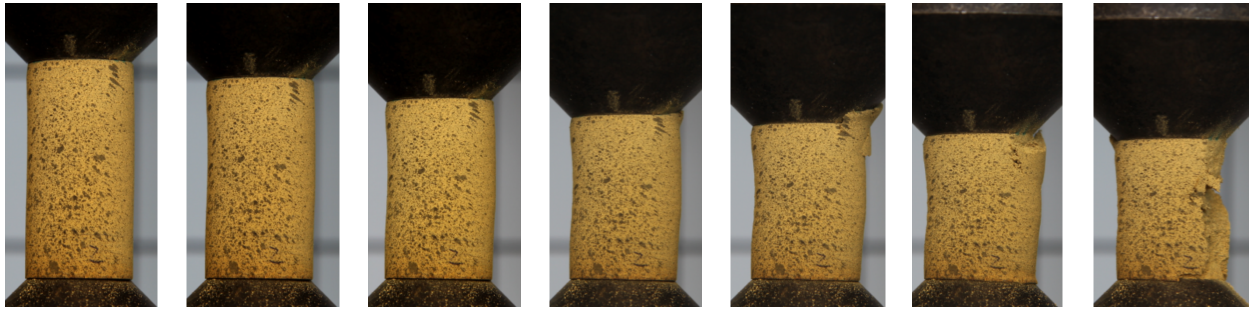
→ Anisotropic behavior due to pre-form

Characterization

Mechanical



Compressive strength



Characterization

Properties of interest

- virgin and char density
- virgin and char thermal conductivity
- virgin an 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?

thomas.rothermel@dlr.de
christian.zuber@dlr.de

Thank you for your attention