

Main S&T results/foregrounds

Work package 1: Mission profile & TPS specifications.

This work package (led by Airbus DS SAS) addressed the identification of typical mission for which hybrid TPS might be appropriate.

It consisted of two main steps:

- Review the different profiles of various entry missions
- Selection of the most appropriate mission for hybrid TPS

The second aim was to specify the TPS properties and requirements based on the previously selected mission, in terms of:

- thermo-mechanical properties
- ablation rate
- insulation performance

Then these requirements were reviewed in terms of the results achieved in WP5, WP6 and WP7

Work package 1.1: Mission selection

- **Review of various entry mission**

Several potential planetary atmospheric missions were reviewed. They are presented in Table 1, with the targeted planet corresponding atmosphere.

Mission	Type of mission	Planet	Atmosphere
CSTS or similar	Crew Space Transportation System	Earth	N2 ; O2
ARV first missions	Cargo download capability from ISS	Earth	N2 ; O2
Mars Sample Return (MSR) or MarcoPolo	Sample Return	Earth	N2 ; O2
ExoMars, MPL, MSR, Network Science Mission	Exploration	Mars	CO2, N2, Ar
AEROFAST	Exploration	Mars	CO2, N2, Ar
TANDEM / TSSM	Exploration	Titan	N2, CH4, Ar
		and/or Saturn	H2
Exploration missions EVE	Exploration	Venus	CO2, N2

Table 1 –: Considered Entry missions

For each mission, a key parameter is the entry velocity, which directly impacts the energies associated to the entry (heat flux and integrated heat loads) since the kinetic energy is mostly dissipated as heat.

Table 2 summarizes and compares different specific potential energy link to the type of trajectory and depending on the targeted planet.

Mission	Arrival from orbit (*) (MJ kg ⁻¹)	Interplanetary arrival (**) (MJ kg ⁻¹)
Earth	20.8	63
Mars	4.2	13
Titan	1.2	3.5
Saturn	210	630
Venus	17.9	54

Table 2: Energy per unit mass associated with arrival at a planet's surface

The specification of entry conditions for so different potential missions is a very difficult task since too many parameters are involved in the calculation of the heat flux and integrated heat loads.

Therefore it was necessary to limit the analysis to the definition of envelope ranges for only a few key parameters such as heat flux, energy and pressure.

For earth entry mission, Table 3 presents the maximum (along profile) peak (versus time) total heat flux values estimated on the fore body.

Mission		Maximum heat flux (MW/m ²)	Comment
Earth	CSTS	4.5 to 7	Low Lunar Orbit
	CTV / ARV	0.7 to 1.7	LEO / ISS Return
	IXV	0.6 to 0.7 (nose)	suborbital
Earth	Sample Return	8 to 15	tbc : (depends on velocity & FPA)
	Marco Polo	11-15 [RD 5]	

Table 3: Typical maximum heat fluxes on fore body for various planetary Earth Entry Missions

The heat flux on the front part of the entry vehicles is usually considered with higher priority because of higher values, but assessment of the aft-body heat flux is important as well since it can have large impact on the centring of the vehicle.

Even if the mean value is quite lower (between 5 to 15%) of the fore body, one must be aware that associated uncertainties are very much higher up to 100%. In addition, the thermal balance is such that the fraction of heat flux driven in-depth the TPS is significantly higher than on the front. The combination of these 2 aspects generally results in penalty on TPS mass for the rear side.

The Table 4 presents the maximum peak total heat flux values estimated on the aft body for the different Earth missions.

Mission		Maximum heat flux (kW/m ²)	Comment
Earth	CSTS	500 to 800	Low Lunar Orbit
	CTV / ARV	100 to 250	LEO / ISS Return
	IXV	85	suborbital
Earth (Sample Return)	ESA missions Stardust	No published data 300 kW/m ² [RD 6]	tbc : (depends on velocity & FPA)

Table 4: Typical maximum heat fluxes on aft body for various planetary Earth Entry Missions

- **Trajectory selection**

In order to identify the most appropriate application for hybrid TPS, some missions were compared the different criteria as summarized in the Table 5:

Concept	Surface Ablator	SPA type
	ablator hot structure	hot structure ablator
CSTS (LLO)	Range of heat flux is beyond the limit of the hot structure alone : interest to evaluate a hybrid concept	Range of heat flux is beyond the limit of the hot structure alone : interest to evaluate a hybrid concept
	Long term perspective (not in current roadmaps)	
CTV / ARV	Range of heat flux is locally beyond the limit of the hot structure alone : interest to evaluate a hybrid concept	Range of heat flux is locally beyond the limit of the hot structure alone : interest to evaluate a hybrid concept
	Long term perspective (not in current roadmaps)	
Sample Return	In this case : more a concept of hot substructure	Questionable because heat flux is likely too high
IXV	In this case : more a concept of hot substructure	No interest because of moderate heat flux
	Application to IXV itself is not relevant because the project is already advanced. More generally, an other eventual demonstrator could provide a flight opportunity	

Table 5: Assessment of various Earth entry mission

Based on these considerations the main conclusion was that hybrid TPS are not suitable for planetary entries, since the aero-thermal environment is either too severe or too favourable. As a result, the SPA type solution was finally discarded over the surface ablator solution. The SPA type is less complex for a small exploration capsule, but on the other hand, for a large vehicle, the fixation of the CMC hot-structure to the vehicle substructure is extremely challenging to allow at the same time the fixations of the underneath ablator (the CMC/ablator interface is lost once the re-entry is progressing)

The most suitable application is thus Earth re-entry, with LEO or LLO return.

The ESA phase A studies enable a preliminary design and sizing of the front heat-shield and will serve as a reference mission:

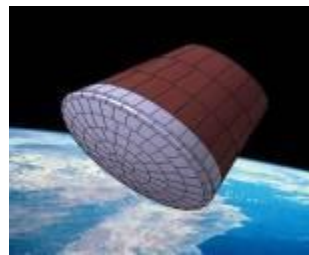


Figure 1: ARV re-entry vehicle preliminary design

Different scenarios (nominal or abort trajectory) are considered leading to a maximum heat flux trajectory and a maximum integrated heat load trajectory.

The predicted aero-thermal conditions are plotted on:

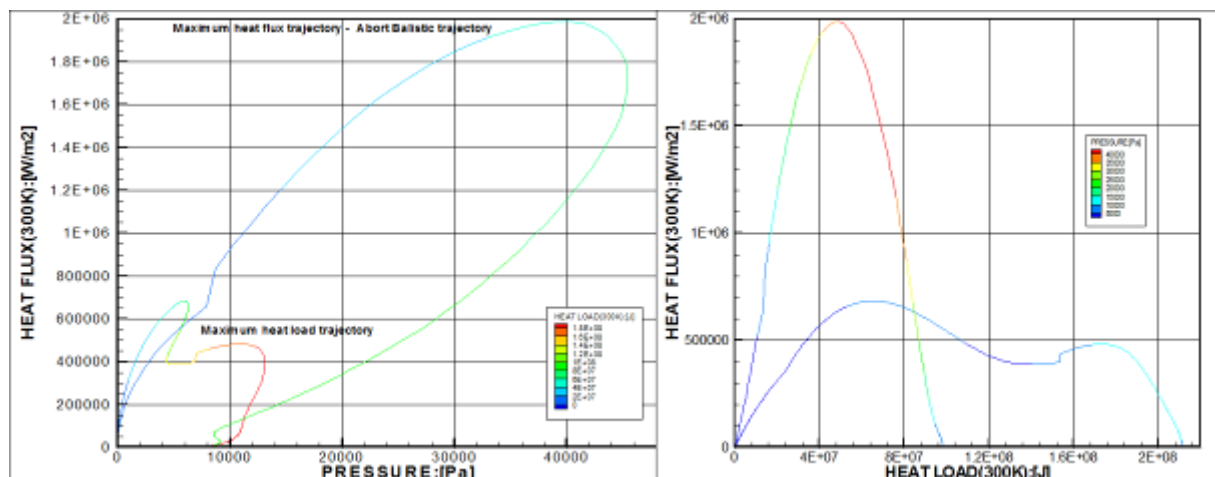


Figure 2: Referenced heat flux and integrated heat load on selected mission

The aborted ballistic trajectory presents a peak heat flux of 2 MW/m² and a maximum stagnation pressure of 0.45 bar, while the maximum heat load trajectory demonstrate a lower heat flux of 600 kW/m² and a lower pic pressure of 0.13 bar. The integrated heat load ratio between the 2 trajectories is around 2.6 with a maximum of 215 MJ/m² for the nominal return trajectory.

Work package 1.2: TPS specifications

Based on the most appropriate selected mission, a set of requirements aiming at supporting the development of a hybrid TPS is presented hereunder.

These functional and performance requirements can be divided in:

- Aero-thermodynamic parameters.

The hybrid TPS solution developed within this activity shall be able to withstand the following loads:

- Peak heat flux levels in the range of 1.7, 5.7 MW/m²
- Integrated heat loads in the range 270, 416 MJ/m²
- Stagnation pressure in the range 180, 650 mbar
- Mass and thermal performance:
 - The specific area mass shall be < 22 kg/m²
 - The final thickness shall be < 80 mm
 - A substructure limit temperature of 180°C
- Ablation performance:
 - Ablation and erosion shall be predictable and reproducible
 - Ablation behaviour shall be homogenous
 - Total ablation should not exceed 20 mm

Other requirements such as mechanical, interface, environment, resource, products assurance, manufacturing, programmatic were also specified but not fully developed in this report.

Work package 2: TPS State-of-the-art and Materials trade-off)

The aim of this WP (led by HPS Portugal) was targeted at getting an overview of the global situation of ablative materials for heat shields of return mission and perform a trade-of for the selection of the materials.

Work package 2.1: State-of-the-art

The first task was to make an overview of the ablative materials available worldwide (with an emphasis in US and specifically in Europe). The materials composition and key performance parameters as well as non-technical parameters such as ITAR restrictions and availability were assessed.

A total of 31 ablative materials are listed and both a compilation table and specific table was filled for each with the key characteristics (density, tested heat-flux, mechanical properties, thermal properties, ITAR issues, supplier, etc...). 13 of the ablators come from European sources, divided by:

- 6 from AIRBUS Defence and Space (ASTERM, AQ60, AQ61, NL, ALEASTRASIL, PICSIL, PROSIAL)
- 3 Thales Alenia Space - Italy (Impregnated Sigratherm and Grafoam, ATLAS)
- 1 from the Institute of Space Systems of the University of Stuttgart (RICA)
- 1 from DLR Stuttgart (ZURAM)
- 1 from AVIO (SV2A)

Eight of those ablatives are available in the market.

#	Name	Composition	Producer	Country	References
1	ASTERM	Carbon Phenolic	Astrium or EADS Transportation	France	[RD8], [RD18]
4	AQ60	Phenolic resin impregnated silica felt	EADS Transportation	France	[RD4],[RD13], [RD16],[RD17], [RD18], [RD29]
5	AQ61	Carbon fiber felt impregnated with a low fraction of phenolic resin	EADS Transportation	France	[RD16]
7	Norcoat/Liege	Cork powder and phenolic resin	Astrium or EADS Transportation	France	[RD4], [RD10], [RD22]; [RD29]
9	RICA	Phenolic resin impregnated carbon	IRS - Institute of Space Systems of the University of Stuttgart in cooperation with NASA Goddard	Germany	[RD7],[RD8], [RD25]
15	Impregnated Sigratherm	Graphite fibers with phenolic resin	Graphite fibers from SGL Carbon. Ablative from University of Roma and TASI.	Italy	[RD3]
16	Impregnated Grafoam	Graphitic foam with phenolic resin	Foam from Graphtech International, USA. Ablative from University of Roma and TASI	Italy	[RD3]
19	Aleastrasil	Silica fibres impregnated with phenolic resin	EADS – SPACE Transportation	France	[RD11],[RD16], [RD26], [RD29]
20	Picsil	Silicon based or silica compounds densified with a phenolic resin.	EADS – SPACE Transportation	France	[RD11], [RD16], [RD29]
21	ATLAS	Carbon fibres impregnated with phenolic resin	TAS	Italy	[RD32]
29	PROSIAL	silicone elastomer with silica hollow spheres	EADS – SPACE Transportation	France	[RD13]
30	ZURAM	Carbon Phenolic	DLR -Stuttgart	Germany	Personnal communication
31	SV2A		AVIO	Italy	Personnal communication

Table 6: List of European ablative materials reviewed organized by producer

Also a review of the historical HYBRID solution was carried-out with the main characteristics of the SEPCORE™ (by HERAKLES group) and SPA™ (by AIRBUS group) solutions, both developed in the 90s:

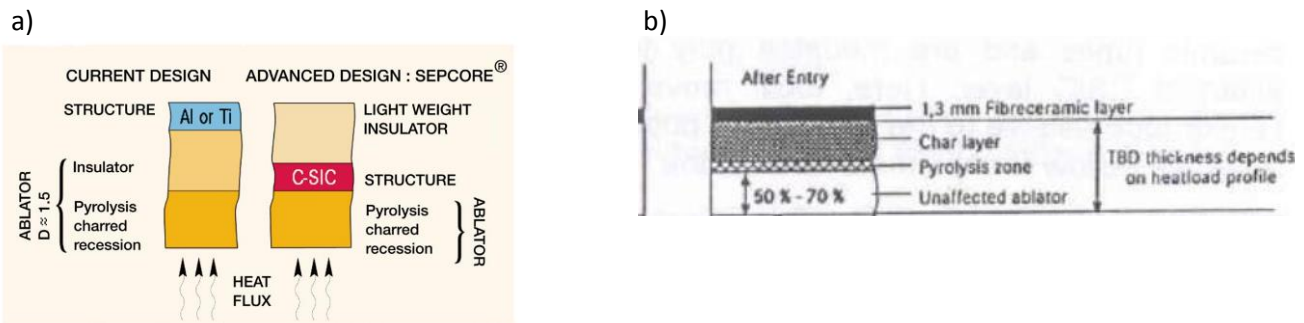


Figure 3: Past historical HYBRID solutions: a) SEPCORE™ and b) SPA™

The main difference with HYDRA is that SEPCORE™ employed a high density ablator (Raycarb C2™), while the SPA™ had the opposite configuration, being the CMC on top the ablator. This last configuration, through and iterative analysis in WP1 and WP2, resulted out of interest for a large capsule (like CTV/ARV and CSTS) as the fixation of the CMC hot-structure to the vehicle substructure is extremely challenging to enable at the same time the fixations of the underneath ablator (the CMC/ablator interface is lost once the re-entry is progressing).

Work package 2.2: Materials trade-off

Based on the State of the Art and on previous experience, a trade-off matrix was made in order to help the selection of the best candidate for the HYDRA mission. However, the choice of the HYDRA ablator was already conditioned to either ASTERM or NORCOAT. Nevertheless, the driver for the materials selection was to perform an up-to-date state-of-the-art of ablators available worldwide and afterwards carry-on with a materials ranking (specifically in view of the mission requirements) and locate the materials from the partners in such ranking. In case one of the materials is not available anymore, the ranking will provide a fast identification of alternative material on the procurement chain.

A material trade-off criteria matrix was elaborated and the available materials were trade-off against these criteria. These criteria were selected among the most relevant from the specifications (D1.2) and proper weighing factors were applied. In fact two different trade-off were considered for materials below and above 5.7 MW/m^2 , in order to fulfil the selection of materials for the two envisaged mission: CTV/ARV and CSTS:

Once the trade-off was performed the materials ranking was carried-out, and the materials envisaged in the project were finally located in such ranking (finally in the first positions):

Lunar Return Mission (CSTS-like)

Ablator	Rate	TRL	Country	Density
ASTERM	119	5	EADS/ST	270-500
PICA	97	9	NASA Ames	270
MONA	94	3	LM	350
AQ60	102	9	EADS/ST	300
AQ61	96	3	EADS/ST	300
PhenCarb	79		ARA	320-580
RICA	99	4	IRS / NASA GODDARD	1360
ATLAS	107		TAS	250
ZURAM	104	4	DLR	360

Table 7: CSTS ablative materials ranking

Lunar Return Mission (CTV/ARV-like) 1.7MW/m2

Ablator	Rate	TRL	Manufacturer	Density
SLA-561	82	9	LM	550
Norcoat/Liége	101	8	EADS/ST	470
ACUSIL I	73		Aerotherm/ Exelis	480
SIRCA	94	8	NASA Ames	240
HYPERLITE A	90	3	ARA	210
PROSIAL	89	9	EADS/ST	600
SRAM 17/20	84	5	ARA	224-384
PCA-28	70		ARA	448-518

Table 8: CTVS/ARV ablative materials ranking

As a conclusion ASTERM and NORCOAT-Liége show in the main key criteria and for the tailored mission:

- Excellent technical performance of the ablator in the performed tests.
- No identified material failure during high enthalpy tests within the specified heat flux/ pressure envelope
- The producer and developer are situated in a certified ESA member country. Also the supply sources for raw material are European.

Work package 3: Ablative protection shield

This work package (led by Astrium SAS) refers to the selection and manufacture of ablative materials, where also HPK Liéges has participated.

Work package 3.1: Ablators based on resins (carbon fibres)

Airbus DS France has provided the standard version of ASTERM (since ASTERM is available for a wide range of density) with an average density of 280 kg/m³ and therefore highly similar to the US PICA, which has gained heritage on several different types of missions (Stardust, MSL Orion...). The standard material is under development and should reach TRL6 at the end of 2014.

The material is based on European standardized raw material and the whole manufacturing process is controlled by Airbus DS in its in-house workshop.

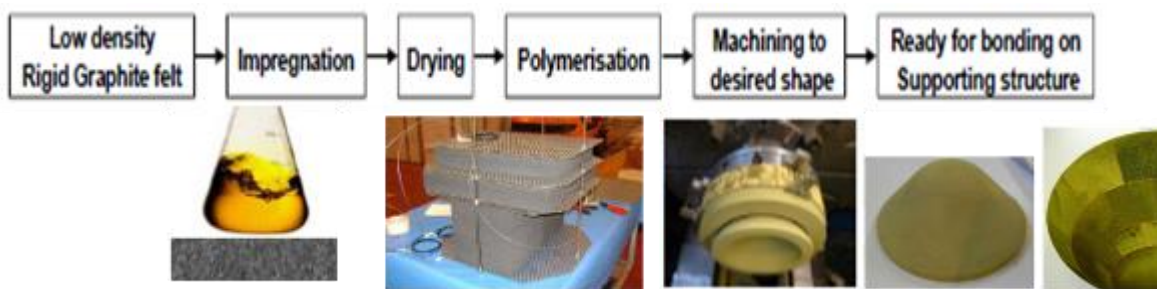


Figure 4: Simplified manufacturing process

It has been successfully tested in the most severe conditions compliant with the ones under which it has been designed.

The material performances have been validated through a successful coverage of the flight domain by more than 40 plasma wind tunnel tests. The next repeatability tests aim at extending this domain to find the material limits and assess the statistical distribution of the thermo-mechanical properties.

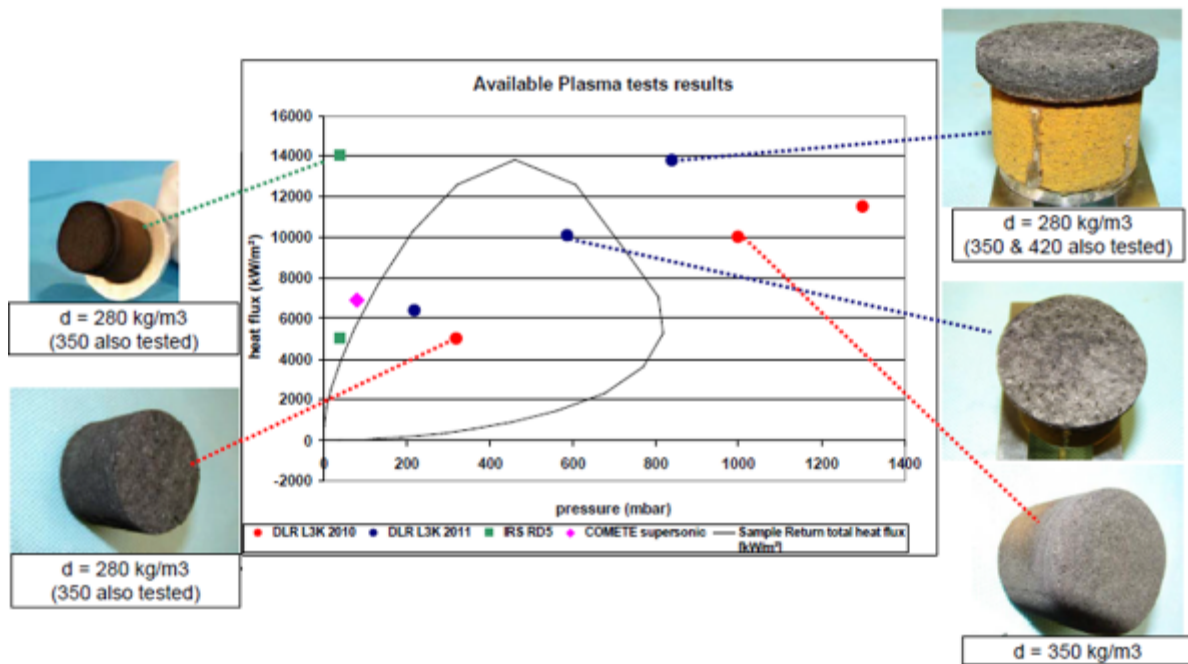


Figure 5: Successful ground plasma test campaign coverage of the flight domain

Work package 3.2: Ablators based on cork

The advanced cork materials are delivered by HPK. The most suitable material finishing is taken into account (outgassing, machining, ...). Samples were delivered for bonding testing. Among the activities carried out, HPK has:

- Compiled knowledge in cork material itself.
- Completed the gluing processing.
- Applied complex coat systems.
- Carried out tooling or modelling cork formulations.
- Executed prototype activities.

Based on the results from the material trade-off (WP2) and the simulation & design (WP6) the most promising ablative materials have been selected: ASTERM and NORCOAT. HPK has been in charge of the manufacturing of the NORCOAT. The dimensions of the required samples were 550x550x70 mm.

The maturity level of NORCOAT is high, being the development of the material started in the 1990's. 10 panels were manufactured and delivered to TECNALIA at the end of April 2013, as seen in Figure 6.



Figure 6 Norcoat panels: a) 1 panel 550 x 550 x 70 mm and b) Microstructural inspection at arrival

In the frame of this work package “in-situ” joints have been performed by HPK. The process consisted of the direct manufacture of the NORCOAT FI on top of the C/C-SiC samples (on the rough/desiliconized face). For this approach three samples of $100 \times 100 \times 10 \text{ mm}^3$ samples were fabricated (see Figure 7a). The 10 mm thickness corresponded to 5 mm of CMC and 5 of NORCOAT (carefully calculated to be the result after the fabrication and final pressing step).

This led to a very smooth interface, as shown in Figure 7b and c, and it was observe that even the resin polymerized inside CMC fibres. Three additional samples were prepared with perforations with the aim to improving the shear strength of the interface.

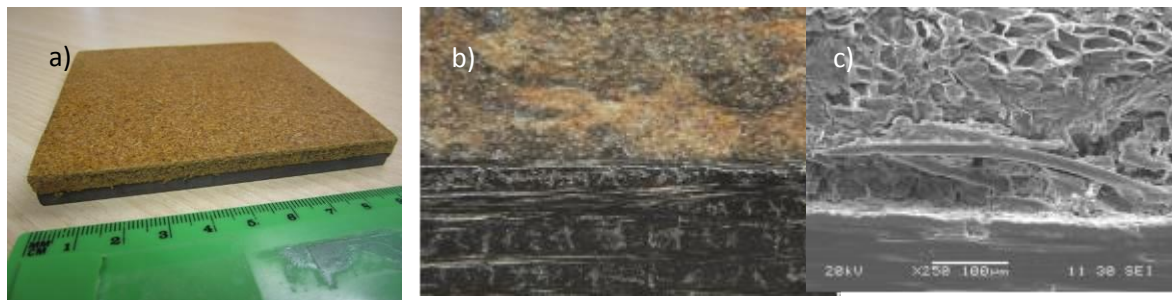


Figure 7 NORCOAT© + C/C-SiC© in-situ joining: a) general view, b) interface view and c) detailed SEM image

Work package 3.3: Manufacture of heat-shield parts

Both Norcoat Liéges FI and ASTERM parts have been machined at Tecnalía (part for single materials characterisations, joints characterisation and samples/breadboard manufacture).

Work package 4: Structural ceramic core

This work package (led by Airbus DS GmbH) refers to the manufacture and procurement of CMC structures to be used as substrate of the TPS hybrid solution.

Work package 4.1: Development, manufacture, characterisation and delivery of CMC samples

- Development & production of CMC material (material samples & proof-of-techno samples) for bonding technology development and support of this task
- Production of CMC material for characterization and characterization of open issues identified for HYDRA.
- Contribution to testing and evaluation of proof-of-technology samples and bonding technology from CMC point of view

Work package 4.2: Development and delivery of CMC demonstrator material

- Collection/provision of CMC material properties for Hydra modelling/simulation
- NDI methods identification and application on proof-of-techno samples
- Manufacturing and provision of CMC demonstrator material
- Contribution to 'use & dissemination plan' from large system integrator point of view
- Manufacturing & delivery of TPS demonstrator (CMC part).

Work package 5: Full protection system assembly

This work package led by Tecnia addressed the screening, selection and developments of proper ablator (CMC) advanced joining technologies that would combine the use of high temperature adhesives and mechanical stand-offs as well as the integrate technologies and parts to assembly samples and breadboards that monitored the performance test of the joints (thermal shock, plasma wind tunnel test and so on).

Work package 5.1: Definition of bonding processes

The work consisted of the **definition of the appropriate bonding process and adhesives for ablative materials** based on resin or cork as defined in WP3 and **thermo-structural ceramic core** as defined in WP4 **taking into account the environmental specifications defined in WP1 and modelling in WP6**. State of the art methods and innovative approaches were tested against the specifications. The most promising ones are incorporated in WP5.2 and WP5.3.

Adhesive Joints

Within previous WP ablator and CMC **materials** were selected:

Ablator materials:

- Cork based materials: NORCOAT FI (back shield)
- Graphite based materials: ASTERM (front shield)

CMC materials:

C/C-SiC (Supplier DLR Stuttgart).

C/SiC SICARBON© (Supplier Airbus Group Innovations)

Based on the specifications established and in order to define the appropriate bonding process, two adhesive joints between the ablator and the CMC were identified (Fig. 5):

Joint 1: ASTERM-SiCarbon (for the front shield of the vehicle, studied by NCSR “Demokritos”)

Joint 2: NORCOAT-C/C-SiC (for the back shield of the vehicle, studied by Tecnalia)

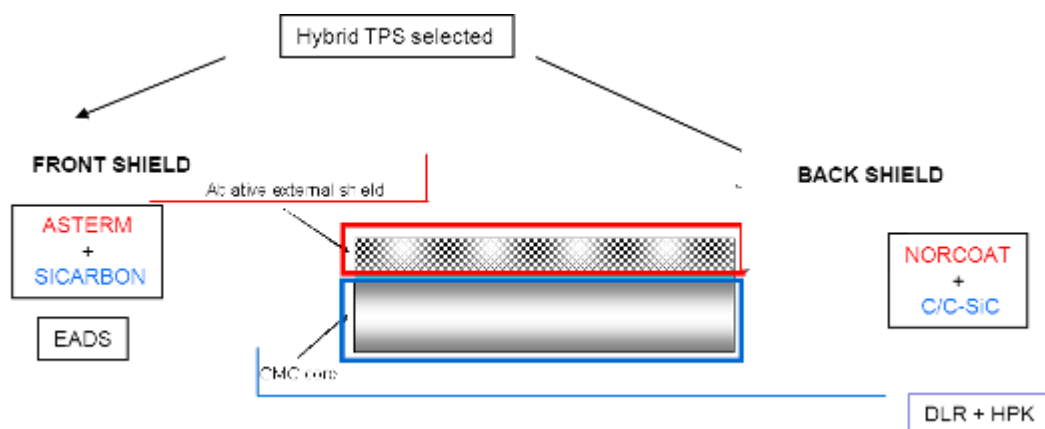


Figure 8: Adhesive joint scheme

Adhesive selection

The selection of the best adhesives for the joints was based on two main aspects:

- Ablator decomposition temperature. This temperature, about 150-200°C, sets the maximum allowable curing temperature. The thickness of the TPS material is directly related to the allowable temperature for the adhesive bond-line.
- During re-entry: the adhesives must withstand very high temperatures (strong temperature dependence of ablator thickness) for relatively short time periods.

Thus, the following aspects were considered as critical:

- Maximum temperature required
- Thermal expansion between materials to be bonded
- Bond strength requirements
- Low outgassing/ REACH
- Viscosity and handling
- Substrates surface porosity. The porosity of the ablator could be useful to mechanically interlock a low viscosity adhesive that could serve as a primer for the second and more viscous adhesive to bond the CMC.

The high temperatures reached during the re-entry strongly imposed the selection of inorganic adhesive. The presence of inorganic fillers in the composition allows the adhesive to withstand temperatures in the range 760-3000°C depending on the nature of the filler (Zirconia, Alumina, Graphite). Table 9 summarizes the selected adhesives for the screening. It can be noticed that for each of the filler, two viscosity levels were chosen to assess the penetration capability of the adhesive on the ablative surface.

Adhesive	Ceramabond 670	Ceramabond 569	Ceramabond 685-N	Ceramabond 835	Ceramabond 669	Cotronics Resbond 931
FILLER	Al ₂ O ₃	Al ₂ O ₃	ZrO ₂ - ZrSiO ₄	ZrO ₂ - ZrSiO ₄	Graphite	Graphite
Viscosity, cP	2,500- 5,000 Low	Paste HIgh	5,000-20,000 Low	20,000-40,000 HIgh	20,000 - 40,000 Low	Paste HIgh
Temperature Limit, (°C)	1650	1650	1371	1371	760	2980

Table 9: Adhesives selected

Adhesives characterization

The thermal stability of the adhesives was examined using Thermo-Gravimetric Analysis (TGA) and pull-off tests. The pull off tests showed that for Ceramabond™ 670, 835 and Graphibond 669 there is no detachment between the base materials.

In addition, in order to assess the adhesion of the various adhesives on the base materials, drops of the adhesive were left to drip on both SICARBON and ASTERM ablative material and subsequently force was applied to detach the adhesive from the material. The performance of the adhesives was similar to that of the pull-tests.

Consequently, the adhesives that presented the best performance and were selected to be further characterized as part of an adhesive joint in terms of Thermal shock on bonded joints, Shear strength at room temperature and Cryogenic temperatures are:

Alumina	Zirconia	Graphite
Low viscosity	High viscosity	Low viscosity
Ceramabond 670	Ceramabond 835	Craphibond 669

Table 10: Adhesives preliminary selected.

Work package 5.2: Ablative/ceramic frames joining

In this WP, joints of CMC/Ablative material were fabricated in order to be mechanically tested at ambient and liquid nitrogen conditions. Two families of joints were fabricated:

- SICARBON/ASTERM (fabricated by NCSR “Demokritos”)
- C-C/SiC/NORCOAT (fabricated by Tecnalia)

For each family, the three adhesives CeramabondTM 670 (Al_2O_3), CeramabondTM 835 ($\text{ZrO}_2\text{-ZrSiO}_4$) and GraphibondTM 669 (graphite) were employed. The bonded area of the sandwich specimens was $10 \times 10 \text{ mm}^2$.

The shear tests that were carried out for SICARBON/ASTERM joints give the following conclusions:

Ambient conditions: For all the adhesives tested without thinner application on the ASTERM, the shear strength of the joints is similar (average value of 0.70 MPa) and reflects the strength of the ablative material since in all tests the failure takes place inside the ASTERM material. This indicates a good compatibility between the adhesive and both the CMC (SICARBON) and the ablative (ASTERM) material, showing that the defined and applied surface treatment is the correct procedure leading to the base material failure. The joints with $\text{ZrO}_2\text{-ZrSiO}_4$ based adhesive present the highest ultimate shear. For the $\text{ZrO}_2\text{-ZrSiO}_4$ and graphite based adhesives, the application of thinner on the ASTERM surface prior to bonding strengthens the ablative material and this results in an increase of both the shear strength and the ultimate shear strain.

Liquid nitrogen: For both $\text{ZrO}_2\text{-ZrSiO}_4$ and graphite based adhesives, the ultimate shear strength is enhanced compared to that at ambient conditions, due to the stiffening of the ASTERM ablative

material at the liquid nitrogen temperature. In addition, the use of the thinner increases drastically the shear strength in liquid nitrogen whereas it reduces the ultimate shear strain.

The predominant mechanism of fracture during shear testing of the sandwich specimens appears to be gradual tearing of the ASTERM adjacent to the adhesive. The apparent beneficial influence of prior treatment with thinner is therefore due to the added load-bearing capacity provided by the thinner penetrating a small distance into the ASTERM. Fractured surfaces of SICARBON / ASTERM joints were studied using SEM-EDX analysis (Figure 9).

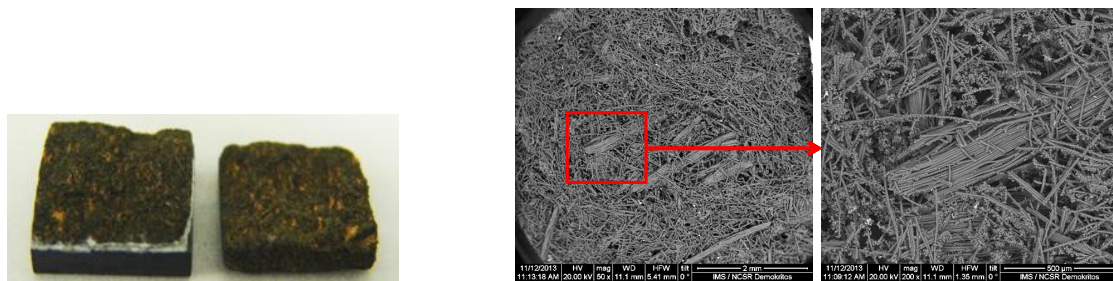


Figure 9: ASTERM fractured surface of Al_2O_3 -based adhesive (Ceramabond™ 670) SICARBON / ASTERM joint without thinner application on ASTERM, mechanically tested at ambient conditions.

The shear tests that were carried out for C/C-SiC/Norcoat joints give the following conclusions:

Ambient conditions: According to the Ultimate Shear Strength results, there are not big differences in the adhesive behavior. It is necessary to take into account that the majority of fractures take place in the Norcoat material, and not in the adhesive layer. Therefore, in general, the obtained values are not valid to characterize the adhesive strength. The differences in the ultimate strength values could be attributed to the Norcoat inherent anisotropy. Analyzing the Ultimate Shear Strain (%), the results are similar, independently on the adhesive tested. That is, the determined strain registered corresponds to the Norcoat and not to that of the bond.

Liquid nitrogen: At liquid Nitrogen, the ultimate shear strength increases while shear strain decreases sharply compared to that at ambient conditions. Possibly these variations are due to the stiffening of the Norcoat substrate, enhancing the strength but reducing the deformability. However, analysing graphite based adhesive, where failure mode was through adhesive layer, shear strength and shear strain present lower values.

As a whole, C/C-SiC /NORCOAT joints do not present a unique failure mode, but the most repetitive one is crack propagation through Norcoat material. The adhesive-substrate compatibility is considered good enough and suitable for this application.



Figure 10: Norcoat fractured surface of Al_2O_3 -based adhesive (Ceramabond™ 670) C/C-SiC/Norcoat joint, mechanically tested at ambient conditions.

Some tests at room temperature were also performed using a second geometry comprised of CMC/ablator/CMC sandwiches of $60 \times 60 \text{ mm}^2$ bonded area (see WP5.4). The latter geometry was used for the mechanical tests at elevated temperatures.

Work package 5.3: Fabrication of TPS breadboard

On this task specimens and breadboards have been prepared for the Thermal shock, Plasma Wind Tunnel, Infrared and vibrations tests, carried out in WP7.

- THERMAL SHOCK

In order to characterize the mechanical behaviour of samples after exposure to high temperature, adhesive joints CMC and ablative material (CALCARB) were constructed using the three selected adhesives.

INCAS performed Thermal Shock test at 1100°C with 2 min holding. During cooling a 3 bar pressure level was used on the ablative/CMC samples: 9 samples of CALCARB/ C/C-SiC and 9 CALCARB/SICARBON.

After the test, the samples weight was measured and the mass loss during the testing was calculated. In conclusion, the gravimetric analysis showed that the average ablative material weight loss after testing was 0.72g for the CALCARB/adhesive/ C/C-SiC set of samples and 0.42g for the CALCARB/adhesive/SICARBON set.

Microstructural characterization after these tests was done by Optical Microscopy or SEM with the aim of analysing the cross section bond-line and the adhesive integrity.

CALCARB/ C/C-SiC joints show good adhesion after the Thermal shock tests. CALCARB/SICARBON samples integrity was not suitable for the cross section analysis in Zirconia and Alumina adhesives. Consequently the post analyses (by SEM) were performed just with the graphite based adhesive samples. In general the adhesion is good. It is possible to observe some Calcarb fibres attached on the top of adhesive layer.

- PLASMA WIND TUNNEL

Samples using both of Hydra ablative materials (ASTERM and NORCOAT) as a representative lightweight ablator and both CMC (SICARBON and C/C-SiC) as hot structure material have been manufactured and tested in the PWK1 and PWK2 test facility, respectively for the two envisaged combinations (ASTERM + SICARBON and NORCOAT + C/C-SiC) using graphite-based Ceramabond 669 and Ceramabond 835

zirconia-based adhesive. These have been driven to their respective working-limit temperatures of 1000 and 1600 K.

Three test Campaigns were defined: a pre-campaign (ASTERM + SICARBON with graphite adhesive), a first campaign, increasing the heat flux with respect to the pre-campaign (ASTERM + SICARBON with both zirconia and graphite based adhesive) and second campaign with NORCOAT + C/C-SiC. In addition a scaled representative circular tile (from stagnation region of the front shield) has been manufactured for large ($\varnothing$ 160mm) plasma test article. The breadboard is composed of an aluminium plate (representative of the cold structure), an insulation layer (Kerfom), and the dual layer ASTERM/SICARBON joined by Zirconia adhesive. As stand-offs, three U shape Ti6Al4V have been shaped and brazed on the CMC.

INFRARED AND VIBRATION

Four specimens based on SICARBON and ASTERM as ablative material were manufactured using two adhesives, Zirconia and Graphite adhesives (2 samples per each) in order to test sample with a representative large size with the aim of reproducing as close as possible the temperature profile of the interface. Two extra specimens for vibration test were manufactured using CALCARB), having the IR design as baseline.

Work package 5.4: High temperature and oxidation conditions testing and thermo-mechanical characterization

The WP5.4 includes the fabrication of joints and the results from their thermo-mechanical tests from room temperature up to 900 °C. The geometry of the joints comprised of CMC/ablator/CMC sandwiches of 60x60 mm² bonded area. Two families of joints were fabricated:

- SICARBON/ASTERM/SICARBON (fabricated by NCSR “Demokritos”)
- C-C/SiC/NORCOAT/C-C/SiC (fabricated by TECNALIA)

For each family, the two adhesives Graphibond™ 669 and Ceramabond™ 835 were used. For the SICARBON/ASTERM/SICARBON joints, thinner was applied on ASTERM’s surface prior to bonding.

The investigations that were carried out give the following conclusions regarding the SICARBON / ASTERM / SICARBON joints:

The ultimate shear strength of the graphite-based joints presents its highest value (0.41MPa) at room temperature; it decreases by 52% at 150 and further 25% (totally 78%) at 700 °C. At room temperature and 150 °C the values are higher than the required 0.1MPa (≈ 0.41 and 0.20) and just below the limit of acceptance (0.09 MPa) at 700 °C. ZrO₂-ZrSiO₄ adhesive joints at 700 °C exhibit higher ultimate shear strength as well as ultimate shear strain than graphite adhesive joints. Specifically, the average shear strength value of ZrO₂-ZrSiO₄ joints is 0.13 ± 0.04 MPa, exceeding the required 0.1 MPa value. Also, at 700°C for both adhesives fracture takes place through the adhesive layer, but parts of ASTERM remain bonded to the adhesive in the case of ZrO₂-ZrSiO₄ joints. At 900°C, the ultimate shear strength and % strain values of ZrO₂-ZrSiO₄ joints decrease to 0.09 ± 0.025 MPa and $0.41 \pm 0.19\%$, respectively. This strength value is equal to the strength of

graphite joints tested at 700°C. Also, the % strain value of ZrO₂-ZrSiO₄ joints at 900°C is higher than that of graphite joints at 700°C.

Overall, the ZrO₂-ZrSiO₄ adhesive shows the best thermo-mechanical performance at high temperatures and fulfils the requirements concerning the shear strength for the case of the SICARBON/ASTERM/SICARBON joints.

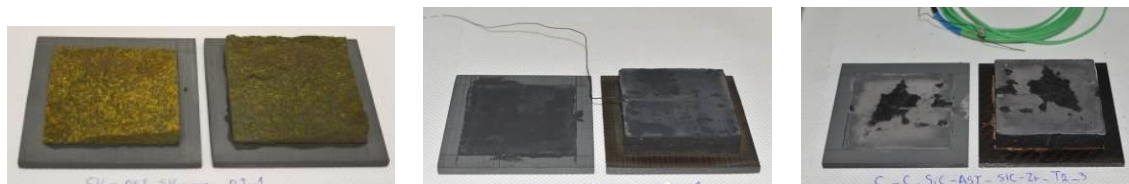


Figure 11: Fractured surfaces of SICARBON/ASTERM/SICARBON joints: with Graphibond 669 (graphite) tested at room temperature-LEFT, with Graphibond 669 (graphite) tested at 700°C- CENTER- and with Ceramabond 835 (ZrO₂-ZrSiO₄) tested at 700°C-RIGHT.

Regarding the C-C-SiC/NORCOAT/C-C-SiC joints the results can be summarized as follows: The joints based on the ZrO₂-ZrSiO₄ adhesive show an increase for USS of 40% for joints tested at RT and 22% for joints tested at 150°C when compared to graphite based joints.

The two type of adhesives present Ultimate Shear Strength values at RT and at 150°C well above the minimum USS value specified for the application, the latter being 0.1MPa. At higher temperatures (700 °C for graphite based joints and 900°C for ZrO₂-ZrSiO₄ joints the Ultimate Shear Strength is similar for both adhesives and much lower than the required value of 0.1 MPa.

The poor performance of both graphite and ZrO₂-ZrSiO₄ based C-C-SiC/NORCOAT/C-C-SiC joints at 700°C/900°C (Figure 12), compared to that of the SICARBON / ASTERM / SICARBON could be attributed to the non-usage of thinner on the NORCOAT surface as it was done for the case of ASTERM.



Figure 12: Fractured surfaces of C/C-SiC/NORCOAT/C-C-SiC joints: with Graphibond 669 (graphite) tested at 700°C LEFT, with Ceramabond 835 (ZrO₂-ZrSiO₄) tested at 700°C-RIGHT.

Work package 6: Modelling, simulation & TPS design

This work package (led by ICMCB-CNRS, with a strong participation of Airbus DS France) addressed the problem of predicting numerically the behaviour of the hybrid TPS during atmospheric entry, and also analysed the thermal performance and aerial mass optimization of the hybrid solution.

Work package 6.1: Simulation of the oxidation in porous materials

Selection of the local zones to be simulated in 3D

The 3D simulation at the microscopic scale of the complete ablative thickness is not conceivable, at least for the moment, because of the required computing power. A change of scale approach, aiming in understanding the relation between microscopic phenomenon and macroscopic behaviour, is then necessary. Two zones seemed appropriate for this kind of modelling approach: The pyrolysis zone (zone 1) where a large part of the heat flowing through the TPS is absorbed, and the porous charred zone (zone 2) consisting of fully reacted material where a high gas flow takes place simultaneously to heat transfer by conduction, convection, and, eventually radiation. Considering the complexity and the critical importance of the pyrolysis zone for the global performances of an ablative material, the project focuses on this zone where a better understanding of the material behaviour presents a high potential for macroscopic model improvement.

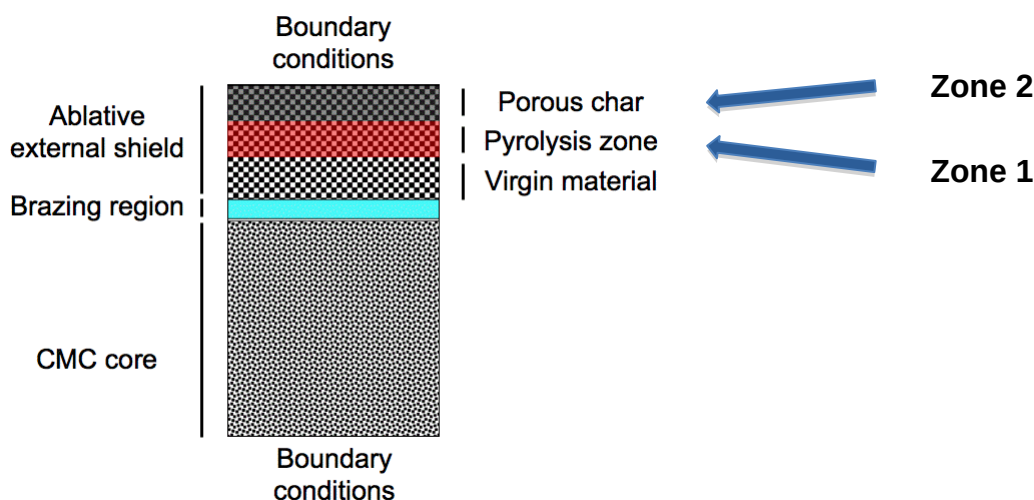


Figure 13: Localisation of the zones considered for 3D simulation

The upper boundary, where ablation occurs, is not taken into account in this simulation.

Selection of the change of scale approach

Upscaling consists in passing information from the pore scale to the macroscopic scale. Since the level of complexity of the continuum equivalent model is significantly lower than the pore-scale physics (the entire geometrical information is lost), upscaling can be interpreted as a lossy information compression method. Upscaling methods handle well linear phenomena, such as Fickian

diffusion or dispersion. However, nonlinear phenomena very rarely pass through the upscaling procedure without requiring strong approximations. The objective of the pore scale modelling being to improve the relevance of the macroscopic modelling, simplification of the local non-linearities seems inappropriate. In order to detect the local phenomenon requiring a precise handling, we decided to explore the possibilities offered by the downscaling approach.

Passing information from the pore scale to the macroscopic scale by upscaling has been worked on for a long time now, the reverse, *downscaling*, was only pioneered recently in the field of porous materials. Downscaling remains a challenging topic and the current study is mainly centred on assessing the possibilities of this method.

Determination of the dimensional constraint for a local 3D simulation

A change of scale approach is possible only if some separation of scale exists: the average variables depend on the macroscopic scale and the local deviation can be evaluated only considering local characteristics (i.e. geometry, physical properties, etc.)

From the geometrical point of view, the constraint is easy to state:

$$l \ll r_0 \ll L$$

where l , r_0 , and L are, respectively, the characteristic length of the solid, of the volume over which the averaging is performed (the computation domain), and of the macroscopic domain. In our case, L has to be defined based on the macroscopic (averaged) temperature variation, as the local boundary conditions will depend on this variable. To do this, the macroscopic temperature gradient has been evaluated using the numerical results produced by the module AMARYLLIS of SAMCEF (cf. WP6.2 below). The characteristic distance L is now defined as:

$$L = \frac{DT}{\left| \frac{dT}{dX} \right|} = \frac{h_1 T}{\left| \frac{dT}{dX} \right|}$$

where the macroscopic temperature variation over the distance L (ΔT) is proportional to the macroscopic temperature divided by the module of the gradient. Introducing $\eta_\square$, the coefficient of proportionality between r_0 and L gives:

$$r_{0,\max} = \frac{h_1 h_2 T}{\left| \frac{dT}{dX} \right|} = h_1 h_2 \cdot 0.018 \text{ (m)}$$

This estimate is based on the temperature values computed by AMARYLLIS. Acceptable computation domains will have a size around 1.8 mm for a product $h_1 h_2$ equal to 0.1 and 0.18 mm for 0.01. This indicates a rather poor separation of scales, but considering the extreme conditions to be simulated, a local approach can be considered as feasible.

Definition of the local problem for downscaling

The local problem derived using the downscaling approach have been developed and solved for a simple transport test problem where a fluid is flowing through an array of cylinders supporting non-linear surface heat sources. The domain of applicability of the downscaling approach has been determined comparing, for this problem, the solution obtained by direct numerical modelling and the solution reconstructed by downscaling. In the pyrolysis zone, the spatial distribution of the reactive material, i.e. the phenolic resin, is not taken into account in the classical macroscopic models (the only parameter is the amount of reactive material). In order to show the importance of this spatial distribution, a parametric study has been done for the test problem to compare the results with different distributions of the surface heat sources. It is clearly demonstrated that this spatial distribution affects the macroscopic results and that downscaling is adapted to estimate this effect. Finally, an example has been treated where downscaling is used as an error detector, exactly the usage that is envisaged for further applications of this approach to complex transport problems like pyrolysis in ablative materials, fire protection, nuclear reactor accident, etc...

Work package 6.2: Full thermal modelling of the hybrid concept

At this macroscopic scale we only used results obtained by ASTRIUM running the module AMARYLLIS of SAMCEF in order to simulate the materials (Ablator, CMC and Insulator) under different environments.

The material thermal, charring and ablation model includes a comprehensive description of physicochemical phenomena.

The main results of the thermal and ablative analyses performed so far were:

- The assessment the thermal, density and pressure macroscopic gradient in order to feed the subscale model.
- The support the plasma campaign logic and to design the IRS plasma probe.

To rebuild the plasma wind tunnel and to finally increase the confidence in the finite element macroscopic model based on a schematic description of the standard IRS plasma probe a 2D axisymmetric thermal charring and ablative finite element model is built (in SAMCEF AMARYLLIS module) taking into account the material specificity, different mode of heat transfer, and the cooling of the holding system.

The real thermocouples will be modelled and temperature measurements at the specified depth will be compared to the simulated ones.

- To define the IR campaign and to predict thermal performance of a representative hybrid breadboard.

The full hybrid system response has been simulated in order to tune the ablator thickness and instrumentation in order to reach target temperature at the bond-line. The full hybrid concept behaviour has been successfully simulated under plasma and IR environment. The experimental data reveal some reasonable discrepancies with the macroscopic model predictions with regard to:

- Temperature prediction at thermocouple location
- Final and instantaneous thermochemical ablation

However, this engineering model could be enhanced based on the better understanding of the pyrolysis phenomena and oxidation at pore scale level. The information transfer to the engineering model from the downscaling methodology has unfortunately not been initiated. However, the very promising downscaling framework highlighted the unknown and missing physicochemical parameters that are needed and pave the way for future technology transfer and progress in the TPS modelling community.

Work package 6.3: TPS final design

The thermo-mechanical charring and ablative material response macroscopic 3D finite element code (SAMCEF AMARYLLIS) is used to design the front-heat shield of an Apollo shape atmospheric re-entry vehicle.

Simulation showed that the hybrid system concept developed and matured during the HYDRA project rely not only on the dual top layer Ablator-adhesive-CMC but also on an insulator that thermally decoupled the substructure from the hot hybrid system.

The main function of the hybrid Ablator-adhesive-CMC being the protection of the substructure from oxidizing and highly reactive plasma environment, the local optimization of their aerial mass can be achieved but at the expense of a pure global thermal performance.

Therefore, the choice of the insulator material and its thickness revealed to be crucial in the overall performance of the full hybrid concept. Then, the numerical analyses focused on the research of an optimal thermal protection system (TPS) hybrid design. It is shown that the total aerial mass of the whole hybrid TPS could be lower than the traditional stand-alone ablator concept even if this latest one is based on the most advanced lightweight ablator.

A very preliminary tile pattern for the front heat-shield of an Apollo shape re-entry body based on the Atmospheric Re-entry Vehicle (ARV) studies was proposed and allowed the assessment of the mass budget of hybrid TPS.

At this maturity phase, this concept demonstrates a nearly 40% mass gain compared to the standalone traditional ablator system. This achieved by de-coupling thermo-chemical ablation performance and the pure thermal insulation.

The manufacturing of a large hybrid breadboard aimed at increasing the TRL of such new TPS solution. By testing the breadboard under plasma representative environment, the maximum size of the breadboard was constrained by the plasma generator electrical power, nozzle exit area and available sample holder of the IRS facility.

The large breadboard was fully representative of a scale 1 tile and embeds a thermo-plug in its centre to further validate the thermal performance and behaviour under plasma environment of the hybrid solution.

Work package 7: Characterisation, re-entry and validation

This work package (led by IRS) was addressed to the characterization of the high temperature materials envisaged within the activity. Moreover the project partly aims for an assessment beyond coupon type characterization such that single parts, assemblies and breadboards, which were investigated as well.

Work package 7.1: Microstructural and thermo-mechanical characterisation

This sub-work package of WP7 contains the characterisation at two levels: single materials and hybrid joints

Firstly it has allowed the full characterisation of the single materials, particularly the ablators, and among the two envisaged NORCOAT and ASTERM, the effort has been addressed towards the second, as it is a new material still under development and qualification phases. This characterisation has included:

- Microstructural analysis
- Thermo-physical properties: Thermal-diffusivity, thermal conductivity, coefficient of thermal expansion, emissivity, in the RT-1600 °C range).
- Mechanical properties: Tensile, compression and flexural (at cryogenic, room and high temperature)
- Pyrolysis analysis.

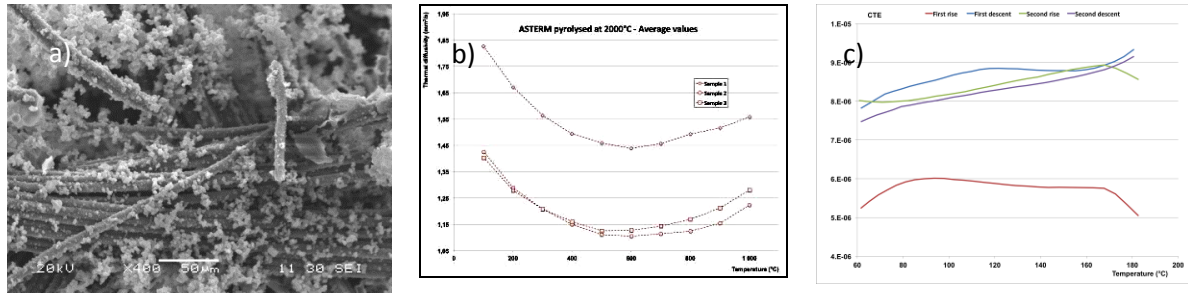


Figure 14 ASTERM Characterization: a) SEM Microstructure, b) Thermal diffusivity by laser flash and c) coefficient of thermal expansion

Secondly, the hybrid joints (both combinations ASTERM + SICARBON and NORCOAT + C/C-SiC) have been programmatically characterised with different techniques and environment, in order to select the most promising adhesive. The techniques included:

- Thermo-mechanical behaviour at INDUTHERM facility (DLR). Room and high temperature (up to 900 °C).
- Thermal shock at QTS-2 facility (INCAS). To prevent the damage of the facility the ablator was limited to CALCARB (preform of ASTERM).
- Vibration test at HALT Rig (CTA, Spain). These test have been subcontracted and ASTERM + SICARBON with zirconia and graphite adhesives has survived the Volna and Ariane 5 specifications.

Main results are collected in D7.1. As a results of this test the Alumina adhesive was discarded and further test (IR, see WP6.2 description) or PWT (see WP7.2 description) were carried-out with the other two adhesives (zirconia and graphite based)

Work package 7.2: Re-entry testing

CSTS and CTV/ARV relevant conditions have been assessed at PWK1 using the MPD generator RD5. The target heat flux, 5.7MW/m^2 , is achieved at 285 mm from the nozzle exit. Total pressure at this position is roughly 38hPa. The flow characterisation was done through axial and radial profiles for enthalpy and total pressure not only at the target position, but also along the plasma plume.

A total of 9 material tests were conducted at PWK1. 7 tests were performed at nominal conditions with success. All tested samples survive the very high heating rates and the outstanding integral heat loads. High temperature ceramic glues were exposed to temperature picks larger than the operational temperature limit with no failure or any observable damage. Thermography, TC distributed in the ablator layer and laser-based recession measurements where assessed in all tests. 1 test was under 5 MW/m² at 10 mm of the target position. One test failed on the evacuation process before plasma ignition due to separation of the ablator layer from the CMC part.

LEO return relevant conditions have been assessed at PWK3 using the inductively heated plasma generator IPG3. The target heat flux, 1.8 MW/m^2 , is achieved at 235 mm from the nozzle exit. Total pressure at this position is roughly 43hPa. The flow characterisation was done through axial and radial profiles for enthalpy, heat flux and total pressure.

A total of 7 tests were conducted at PWK3. All tests were performed at nominal conditions with half success. Tested samples during short exposure times (30s) survive, but obtaining very low temperatures at the bonding due to the low conductivity of Norcoat-Liege. Plasma exposure times were enlarged to 60s in order to increase the bonding temperature, which were still lower than the operational temperature of the ceramic glues. Bonding failure happened in one of the samples using Zirconia-based glue. 2 of the samples with graphite glue were driven to a completely removed ablator layer, which occur at 90 s after the test started. Thermography, TC distributed in the ablator layer and laser-based recession measurements were assessed in all tests. Very large thermal expansion was observed in all the Norcoat-Liege experiments.

The potential impact (including the socio-economic impact and the wider societal implications of the project so far) and the main dissemination activities and exploitation of results

The efforts towards enabling a strong impact of the project where not only addressed to the scientific community (indexed paper and technical conferences and the EU citizen in general (FP7 initiatives, webpage entries, EU tools, etc...), but also to the space industry stake holders (technical workshops, news in Aerospace clusters and so on).

Concerning dissemination activities the following figures are highlighted:

- 2 papers at indexed journals
- 5 lectures at international technical conferences
- 8 dissemination activities to the public in general (news, poster, media, etc..)
- 3 Master/PhD thesis, arising from the results of the project and another 2 on going
- Scientific/technical workshop with relevant international experts and researchers.

Figure 15 shows an example of selected dissemination Items: figure and captions of few of such items, such as the HYDRA samples at the permanent exposition, sited at the hall of the headquarters building of Tecnalia (San Sebastian), a snapshot of the Lecture given in at the Polytechnic University of Madrid in November 2013, the poster for the International Planetary Probe Workshop - IPPW 2014, in Pasadena, CA and the first page of the article published in the Research EU Magazine N° 34.

Regarding the exploitation of the results:

- Elaboration of an exploitation plan for each member of the consortium, also including non-space application.
- Specific use plan for the end user and industrial partners, including roadmaps, analysis of a value chain and technology transfer to other non-space products and applications (industry, energy, scientific infrastructure, etc...)

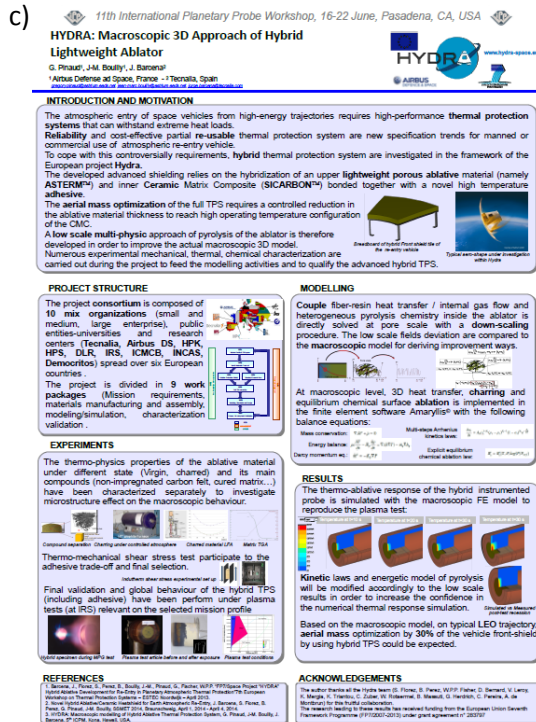
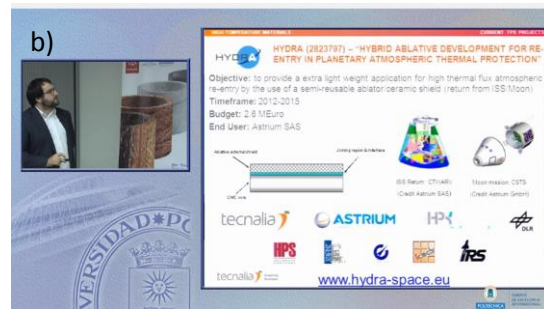


Figure 15: Selection of pictures and sketches of dissemination items: a) HYDRA samples at Tecnalia's permanent exposition b) Lecture given at the Polytechnic University of Madrid in November 2013 , c) Poster at IPPW 2014 and d) Article at Research EU Magazine N° 34

The address of the project public website

A web site has been designed and constructed: www.hydra-space.eu, and keep updated in order to aid the dissemination activities (main publications and presentation are available for downloading). A use plan for each participant, with special emphasis on the industrial partners is released.

The website was divided in two different areas, one for public dissemination of the project and, the second one, only for project members (private area): Major dissemination items are available at the website for open downloading.



Contact points at the website are:

- Dr. Jorge Barcena (Project Coordinator, jorge.barcena@tecnalia.com) and
- Mr. Jesus Marcos (Space and Defence market director, jesus.marcos@tecnalia.com)

In addition new tools such Google analytics, allows to monitor the visit to the webpage. Along the three years the website has received around 4,700 visits with an average duration of 2.5 minutes.