

Development and Testing of Spray-on Thermal Protection System Materials based on Cork and Aerogels

Thomas Reimer Christian Zuber*Helge Seiler*Barbara Milow**Ali Gülhan***Burkard Esser****

**DLR Institute of Structures and Design*

Pfaffenwaldring 38-40, 70569 Stuttgart, Germany

Thomas.Reimer@dlr.de

***DLR Institute of Material Research*

Linder Höhe, 51147 Cologne, Germany

Barbara.Milow@dlr.de

****DLR Institute of Aerodynamics and Flow Technology*

Linder Höhe, 51147 Cologne, Germany

Ali.Gülhan@dlr.de

Abstract

During launch vehicle ascent there is a need for a TPS. It is of advantage to have a material which can be applied in a flexible way via spraying. Materials based on cork and aerogels and resin mixtures were investigated. Materials were developed with a density as low as 195 kg/m^3 and a thermal conductivity of 0.059 W/mK and a mass-specific heat capacity of 1517 J/kgK . Tests in the arc-heated wind tunnel L2K were conducted with good results and corresponding recession values of 2.2 mm after 120 s exposure at 200 kW/m^2 .

1. Introduction

The application for a proposed spray-on TPS can be quite diverse. One possible application is the use on launchers on external surfaces where the thermal load is too high to go without protection. Another issue is the structural material which is used for a launcher. In the case of aluminum, it might be feasible to launch without protection, however, as a result of possible upcoming composite structures, there is probably an unavoidable need for thermal protection during launch. Cork materials have been in use as TPS material for quite some time. There is a broad experience base [1]. However, it has been applied mostly in the form of thin pressed sheets or as shaped parts machined from thick plates, in both cases via bonding to the structure. So there are cost disadvantages to these applications, especially when machining is involved and a complex bonding process.



Figure 1: ExoMars Schiaparelli spacecraft with cork TPS. credit: ESA

There is actually an industrial base of spray-cork available. Spray-on cork layers have found their way into the building industry. One company that offers such solutions for thermal insulation of houses is the Spanish company Vipeq [2]. Aerogels offer very interesting thermal properties and were also taken into consideration for the activity.

2. Theoretical Investigations

At the start of the activity, material data of typical cork insulations and of comparable state-of-the-art-systems were collected as shown in Table 1. In addition the corresponding data of aerogels was contributed by DLR-WF. With this data and load data of a typical launch vehicle, numerical simulations were carried out to assess the potential benefit of a spray-on cork TPS. The simulations were 1-D thermal simulations. It was assumed that the material is applied on a CFRP composite structure, so a requirement of a maximum temperature of 100° C on that structure was put up. The transient heat load on the surface was taken from the trajectory of a current medium-sized launch vehicle ascent.

Table 1: Material data of cork, aerogels and reference system.

	Density	Thermal Conductivity	spec. Heat Capacity
	kg/m ³	W/mK	J/kgK
Vipeq F-08	600	0,068	1848
PROSIAL 2000 [3]	475	0,15	1300
NORCOAT HPK [3]	470	0,09	1800
DLR-Cork	300	0,06	1550
ZURAM	370	0,12	1150
DLR superflex Aerogel	100	0,03	1440
Aerogel Techn. X100 [4]	100	0,024	1605

The simulation results presented in Table 2 showed potential for improvement over the currently used materials. With regard to mass and wall thickness, there is potential for reduction. It has to be said, that the resulting reductions are based on properties that are valid for cork materials in the form of pressed plates, and for aerogels as the basic material property. Hence, the assumed properties are not yet the ones of a sprayable material system, therefore the reductions have to be seen as an indication.

Table 2: Results of 1-D simulations for wall thickness and mass of potential thermal protection materials. Results based on cylinder of 3.44 m diameter and 10.4 m length.

Material	Thickness	Mass	mass delta	mass delta
	mm	kg	kg	%
Airloy X100-100	1,5	16,9	-298,6	-94,6
DLR Superflexible Aerogel Fleece	1,9	21,4	-294,1	-93,2
Active Aerogels Flexible Blanket	2	22,5	-292,9	-92,9
Euro-Cork 200	2,9	62,0	-253,4	-80,3
DLR Kork HP	3,3	111,4	-204,0	-64,7
Airloy X100-550	2,2	136,2	-179,2	-56,8
Vipeq Spray Cork	2,9	195,8	-119,6	-37,9
NORCOAT HPK	3,8	201,0	-114,4	-36,3
ZURAM	5,5	229,1	-86,4	-27,4
PROSIAL 2000	5,9	315,4	0,0	0,0
ELASTOSIL	5,2	641,5	326,0	103,4

3. Material Development

Cork is a natural material with properties that make it an excellent candidate for thermal protection. Since cork is a natural material, there are considerable variations in its appearance and properties. Cork is harvested from cork oak trees by peeling the bark away from the tree trunk. There are different types of cork oaks which grow in a limited number of areas around the world. The highest quality cork is usually found on trees in Portugal and Spain. Most notably, the density of cork shows a large range of variation. This is due to the species of tree and due to the fact that the bark is a natural product with a layered composition. To the outside of the bark, the density increases; also to the inside, next to the hard wood, the cork is of a lower quality. Cork is a porous material with individual cells. On a macroscopic level, cork is anisotropic due to the radial growth direction of the tree trunk and the bark. On a microscopic level, the cork cells are polyhedrons with typically four to nine faces. Average sizes of the cells are between 10 – 70 μm . The cells can be considered closed if mixing with resins of a typical viscosity is concerned [5].

3.1 Particle Mixtures

Cork is available as an industrial product in a lot of forms. Typically it can be purchased as a granulate, with different particles sizes and properties in terms of density.

An investigation was made into suitable particle mixtures of different size fractions. It is evident that between any mix of particles there is a void volume between the particles. If the particles have the same size initially, the void can be filled with smaller particles. In theory, it is possible to fill the voids with ever smaller particles until the macroscopic volume of the mix is practically filled completely. With regard to these considerations, also the shape of the particles is important, whether it is perfectly spherical or less so, more like an irregular rounded or cornered shape.

There is an analytical approach [6] that allows to determine the share of a specific size fraction if its shape coefficient and the sizes of the particles are known, given as

$$A(d) = \left(\frac{d}{d_{\max}}\right)^q \quad (1)$$

Where d_{\max} is the largest size of the particles in the considered mixture and d is the size of a particle below d_{\max} . The particle exponent q describes the shape of the particles. The shape of typical cork granulates that were available was determined microscopically to be quite similar to that of broken natural stone, the corresponding value for q is 0.3. The resulting A gives the volume fraction of particles with size smaller than d , or, vice versa, $1-A$ gives the share of the volume of the particle fraction of size between d and d_{\max} .

An initial distribution of particles of four different size fractions was desired, based on the program of one supplier of the desired cork quality. Since it was not possible to purchase the indicated sizes from this supplier it was then decided to go with a mix of just two fractions of particle sizes, the coarse particles ranging from 0.5 to 1.0 mm and the fine particles from 0.05 to 0.5 mm. The relative shares of the two fractions and the share of the remaining open volume is given in Table 3.

Table 3: Shares of the particle size fractions in the overall mixture volume.

		Open volume	Fine particles	Coarse particles
size	mm		0.05-0.5	0.5-1.0
relative share		0,407	0.405	0.188

3.2 Process Requirements and Development

The considerations for a suitable cork-resin mixture were focused on the two issues of

- Particle size distribution
- Resin composition
- Relative share of particles and resin

In terms of the resin composition the following objectives were targeted:

- Viscosity suitable for spraying
- Curing time short enough
- Mechanical properties of cured resin/composite
- Thermal stability at thermal exposure
- High char yield of the cured resin at thermal exposure

Based on experience and the results of preliminary trials, a choice was made for a resin system consisting of two different resins mixed together. For one, the phenolic resin MF43 was chosen for tests, plus the silicone resin MSE-100. The motivation for the phenolic resin was that it is quite well known for its properties in other applications and has a high char yield in ablator materials. On the other hand it is of high viscosity and cures only slowly. The slow curing can partially be remedied by using a catalyst. The second choice, the silicone resin, was selected because the viscosity is low and it cures quite fast if a catalyst is used. Furthermore it reduces brittleness in the cured mix. With this approach, a first set of material fabrication tests were carried out, first with the very basic method of application on substrates by spreading and also by spraying with the air gun.

Table 4: Initial material composition.

Composition	Rel. Share (Vol.-%)
Cork	70
Phenolic resin	21
Silicone resin	9

Samples could be produced, however, the quality was quite poor and there were lots of problems in the spraying process with intermittent blocking of the gun nozzle. Anyway, data could be collected with regard to density, thermal conductivity and heat capacity to get a first assessment of the material.

Table 5: Measured properties of the initial material trial results.

		VIPEQ SOCI-6	Prototype	Prozial
Thermal Conductivity	[W/mK]	0,14	0,12	0,1
Heat Capacity	[kJ/kgK]	0,87	1,13	1,42
Density	[g/cm ³]	0,6	0,62	0,5
Tensile strength(RT)	[MPa]	0,59	1,38	1,42

The conclusion from these initial trials was that improvements had to be made on the raw materials, the hardware and also the spraying process.

Regarding the material, the largest drawback had been the availability of just one size fraction. A second fraction of the size of 0.05 – 0.5 mm could be purchased in a good quality of low density. Since there had been a lot of blocking of the gun nozzle during the trials, a check was made if the particle sizes were really as they were advertised by the suppliers. It was found that this was not the case. There was a significant amount of particles exceeding the specified maximum size. As a result the cork granulate was then sieved before every spray test to eliminate this problem with much improved results and practically no more nozzle blockings. A smaller nozzle of 4 mm diameter instead of 6 mm could be used as a result.

3.3 Pump System

Initially a spray gun with only a bucket on top was used as the material feed system. Quickly this was considered not optimal and an industrial-grade pressure fed material conveying system was purchased. In this system, the cork-resin mix was in a pressure vessel with the material exit on the bottom and air under pressure above the material. Pressures ranged between 2 and 6 bar. Although the system was an improvement, the cork-resin mix had to be of relatively low viscosity to result in good material flow through the feed tube to the gun which meant that the resin share had to be higher than desired. If this was not the case, usually pressure mounted and the liquid was pressed out leaving the dry cork in the tube without flow. Another pump system was acquired. It was an auger pump which uses a rotating auger

shaft to push the material through the stator into the feed tube and to the pump. This proved to be effective finally but also with this type of pump, there are limits to the cork-resin-mixes which can be pumped. The pumps are displayed in Table 6.

Table 6: Investigated material feed systems.



System	Open bucket	Pressure vessel	Auger Pump
Feed type	Gravity fed	Pneumatic	Hydraulic / mechanical
Max. Pressure		8 bar	40 bar

The spraying trials were conducted into a ventilated cabin. This is essential since there is always some amount of the resin/dilutant vaporized into the air which will pose a threat to health in the case of e.g. phenolic resin. As target substrate plates, aluminium and CFRP plates of 200 x 200 mm dimension were used as shown in Figure 2.



Figure 2: Ventilated spraying cabin with substrate plates ready to be coated.



Figure 3: Coated substrate plates after spraying.

3.4 Spraying Results

The spraying tests were organized in sessions, for which a certain amount of cork-resin material was prepared using certain parameters. The number of coated plates varied typically between 6 – 8. The usual post-spray curing process was to dry the fresh plates at 60° C for a few hours, typically overnight, and then to apply a 150° C curing cycle for another couple of hours. In Table 7 data is given on the most important parameters of different mixtures that were prepared for spraying sessions. The session numbers listed here are representative of the most important development steps. Much more parameter data was recorded, but it would be impractical to list this.

Table 7: Composition data of different mixes used during spraying sessions.

Session	Cork fine	Cork coarse	Phenolic	Silicone	Isopropanol	Resin/cork mass ratio	Resin/cork vol. ratio	Liquid/dry volume ratio
	g	g	g	g	g			
1	350.4	105.4	2016	820.8	-	6.22	0.37	0.366
5	450.5	135.3	3024	1231.1	390	7.26	0.43	0.486
9a	228.4	52.8	325.2	132.4	42.3	1.627	0.096	0.109
9d	228.4	52.8	975.7	397.3	127	4.88	0.29	0.329
12	228.4	58.8	58.54	23.84	974.1	0.29	0.017	0.317
14	540.18	124.82	67.5	27.5	2500	0.14	0.008	0.342
15	843.6	0	175.6	71.5	2922	0.293	0.017	0.334
16	0	843.6	175.6	71.5	2922	0.293	0.017	0.334

Session 1 was the starting point using a mix as it had been used at the end of the preliminary investigations. No diluting isopropanol had been added, the result was a very compact layer, with obvious room for improvement. In sessions 2 – 4 the issue of the nozzle size was investigated since there was clogging and isopropanol was added. In general, the ratio of liquid to cork was increased. Sieving of the cork was introduced after session 3 and session 4. This had a major impact. The nozzle size could be reduced to 4 mm and spraying was much more consistent. Spraying session 5 worked well in terms of the spraying process and gave good results in terms of the coating. The surface was much smoother than before. However, there were still a few cracks in the coating and the resin content was considered still high. In Figure 4 the plate #24 of session 5 is depicted.



Figure 4: Plate #24 from spray session 5 after curing.

Sessions 6 – 8 dealt with aerogel mixtures which are described after the pure cork experiments. The objective of sessions 9a – 9d was to reduce the share of the resin in relation to the cork. Session 9a started out with very little combined mass of the phenolic and silicone resin and also just a little of isopropanol. The parameter liquid-to-dry-volume-ratio was 0.109. No flow at all could be realized with this mix. The amount of resin and isopropanol was then increased stepwise, maintaining a constant relation among the three different constituents phenolic, silicone and isopropanol. In session 9c, a slow flow could be achieved with a liquid-to-dry-volume-ratio of 0.274 but it was not satisfying. Session 9d produced good results as indicated in Table 7. The share of the resin mass in relation to the cork mass was brought down from 7.26 to 4.88. One example plate #38 from session 9d is presented in Figure 5.



Figure 5: Plate #38 from spray session 9d.

The liquid-to-dry-volume-ratio 0.32 was kept in the following as it proved beneficial for the spraying process. In a subsequent step the objective was to reduce the amount of resin in terms of its mass share even more. To this aim, the resin was replaced by more isopropanol in the spraying process. Session 12 was the result of this. The liquid-to-dry-volume-ratio was indeed 0.317 and the spraying worked well, but the total mass of the resin was reduced to a mere 82 g in comparison to 1373 g as it was in session 9d. The resin-to-cork-mass-ratio could be reduced from 4.88 to 0.29.

An effort was made to reduce the resin share to once again lower levels. In session 14 the resin-to-cork-mass-ratio was reduced by another 50% to 0.14. The spraying process worked fine. However, during drying and curing the plates showed cracking and debonding from the substrate plates and were quite fragile. This was an indication of the amount of resin being too low, so that there was not sufficient binding between the individual cork particles.

In two more sessions it was investigated what happens if just one size fraction of the particles is used with otherwise unchanged parameters for the liquid binder items. In session 15 just the fine cork particles were used and in session 16 just the coarse particles. The spraying in session 15 was good but ..., whereas in session 16 basically no flow could be realized.

Concluding the processing investigations, the composition of session 12 is considered the optimum.

3.5 Aerogel Spray Results

In addition the the spraying trials with cork, also aerogels were investigated. Aerogels have very good thermal insulating properties which make them a natural candidate [7]. In sessions 6 a mixture of aerogel particles with a binder system of sodium silicate were used. The choice of the binder was due to previous experience of DLR-WF. In session 8 it was investigated if a mix of fine cork particles with the coarse aerogel particles gives good results. The aerogel used was Cabot P300. The particle size is 1.2 – 4 mm. A sieved fraction was created with the size range from 0.5 – 1 mm to have better control about the size of the mixture. In Table 8 the relevant parameters of the spraying trials are given.

Table 8: Parameters of the aerogel spraying sessions.

Session	Cork fine	Aerog. fine	Aerog. coarse	Aerog. mass	Phen. mass	Silic. mass	Sodium silicate	Water	Binder/aeg mass ratio	Binder/aeg vol. ratio	Liquid/dry volume ratio
	g	l	l	g	g	g	l	l			
6		3	3	465			1		2.989	0.166	0.166
8	233.4		3.25	243.75			1.15	1.15	6.558	0.354	0.708
11	228.4		0.784	58.8	1451.5	591			9.2	0.436	0.497

The spray results were very mixed but quite interesting. In session 6 with the pure aerogel particle mix, the flow from the pump to the spray gun was slow with high pressure build-up. The particle-binder mix had the appearance of relatively dry powdery snow. However, the spraying as such worked really well. Also the as-sprayed plates looked very good as indicated in Figure 6. After drying, however, the plates showed a lot of cracking due to binder shrinkage.



Figure 6: Aerogel-binder mix in the pump material container (left) and one of the as-sprayed plates (right).

In another test to use the aerogel, a mix of fine cork and coarse aerogel particles was used in session 8, again with the sodium-silicate binder. The spraying process worked well with the amounts of particles and binder, also the as-sprayed plates looked well. However, due to binder shrinkage there were also cracks in the dried plates, Figure 7.

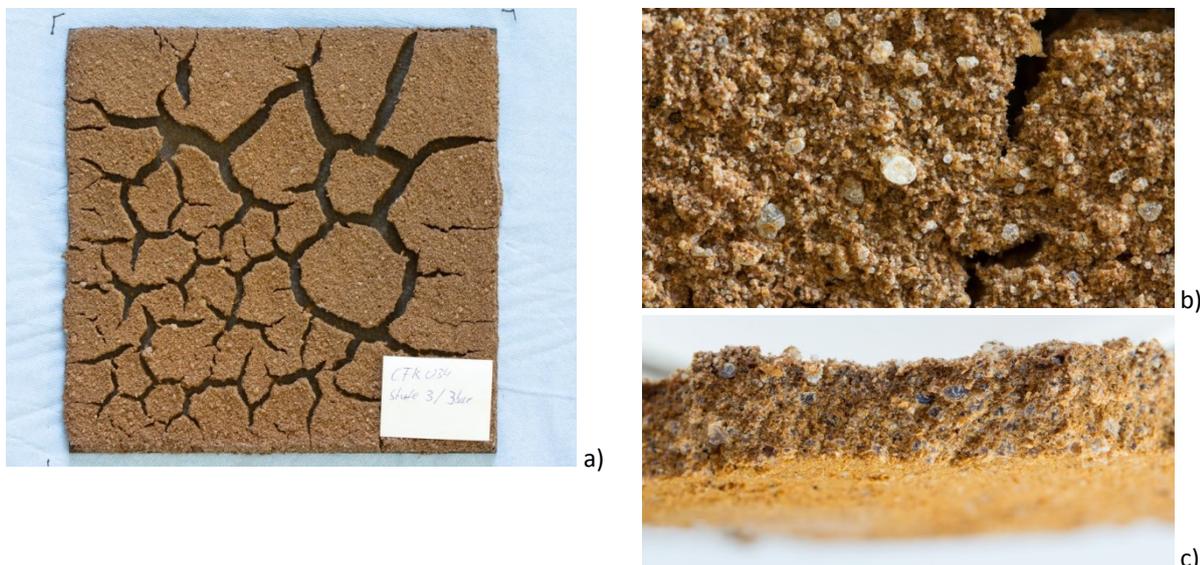


Figure 7: Plate with cork-aerogel-mix (a), surface view (b), cross-section with embedded aerogel particles (c)

It was interesting to see that the larger aerogel particles are well visible embedded in the volume of finer cork particles as shown in the surface view and cross-section view in Figure 7. Considering the fact that the pumping process itself in the auger pump goes along with quite some mechanical loads, there was the question if the aerogel particles would survive the process or if they would be ground into fine particles. As the image indicates, the particles seem to survive the process.

In spray session 11 the coarse aerogel particles were added to the fine cork and the mix was prepared using the phenolic/silicone resins. The resin content was rather high, but the goal was to produce samples for further possible use in characterization and testing. The plates could be sprayed successfully.

3.6 Material properties

Preliminary data was acquired with respect to the thermal properties and to the density of the the as-sprayed and cured coating, presented in Table 9.

Table 9: Measured thermal properties of sprayed materials.

Session	Density Kg/m ³	Thermal conductivity W/mK	Mass-spec. Heat Capacity J/kgK
5	639.2	0.121	1502
8	407.7	0.043	805.9

9d	613.8	0.095	1003.9
12	195.1	0.059	1517.1
11	744.5	0.127	1528.5
14	177.6	0.052	1474.1

The measurements were done with a HotDisk device, the model being the TPS 3500S. The measurement principle is based on the so-called transient-plane-source method [8]. The method can be applied in a very flexible way which is of advantage if the samples are of different shapes and sizes. The only thing which is required, is two samples of the same material type with each of them having a flat surface, as shown as an example in Figure 8.

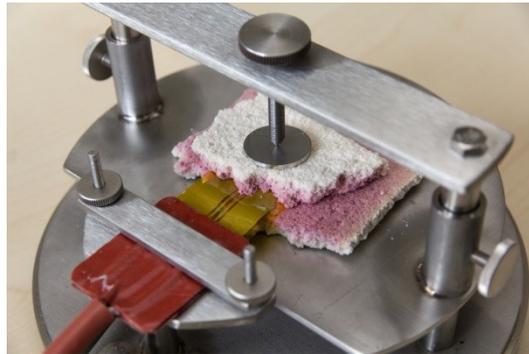


Figure 8: HotDisk conductivity measurement on aerogel samples.

It can be noted that the reduction in density from session 9d to 12 is considerable. Another interesting point is that the conductivity does not go down significantly from session 12 to 14, so there would be no big gain anyway, also the density is not much lower. It is also noteworthy that the use of aerogel particles does indeed have a significant effect on the conductivity which is the lowest of all the samples measured.

4. Wind Tunnel Testing

From the sample plates that were coated in the spraying trials as described above, specimens were cut to be tested in the arc-heated wind tunnel L2K of DLR AS-HYP in Cologne. The test matrix is presented in Table 10. Measured data with regard to mass loss and thickness loss is given in Table 11.

Table 10: Overview about wind tunnel tests.

Test	Run #	Sample	Heat Flux @700K kW/m ²	Test duration s	Remarks
01	291018_01	BT 42	200	120.7	
02	301018_01	BT 18	200	120.9	Sample considerably detached from base plate
03	301018_02	BT 46	200	121.0	
04	301018_03	BT 37	200	120.2	
05	311018_01	BT 43	200	180.0	Extended duration
06	311018_02	BT 50	200	120.4	
07	311018_03	BT 39	200	120.6	
08	081118_01	BT 47	200	180.5	Extended duration
09	091118_01	BT 48	200	121.4	
10	091118_02	BT 41	300	80.3	Higher heat flux

L2K one of the two test legs of DLR's arc heated facilities LBK. The setup of LBK is schematically plotted in Figure 9. L2K uses a Huels-type arc heater with a maximum electrical power of 1.4 MW to energise the working gas to high enthalpy conditions. Hypersonic free stream velocities are generated by a convergent-divergent nozzle. The nozzle's expansion part is conical with a half angle of 12°. Different throat diameters from 14 mm to 29 mm are available and

can be combined with nozzle exit diameters of 50 mm, 100 mm, and 200 mm. So, the facility setup can effectively be adapted to particular necessities of a certain test campaign. For the tests on the cork samples the nozzle had been set up with a throat of 29 mm and an exit diameter of 200 mm. The samples were placed at a distance of 300 mm downstream of the nozzle exit.

Air was used as a working gas. At the L2K facility, there is, however, continuous and substantial experience in facility operation with different other gases. Carbon dioxide, nitrogen, and argon have intensively been used during the recent years. A more detailed description of the facility is given by Gülhan et al. [9, 10].

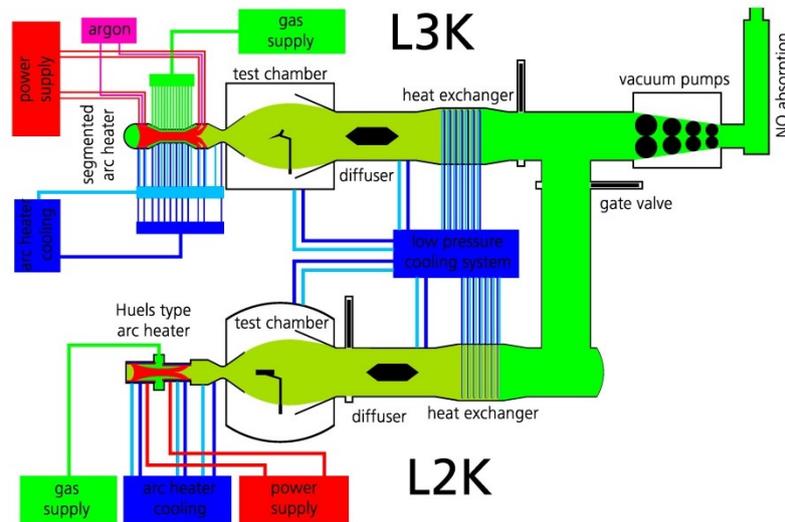


Figure 9: Sketch of the LBK facility.

Table 11: Measured data on mass loss and thickness change.

Test	Sample	Spray Session #	Diameter mm	Duration s	Mass loss g	Thickness change					Aver. recession mm	Aver. Rec. rate mm/s
						A mm	B mm	C mm	D mm	E mm		
1	BT 42	10	100	120,7	10,28	-2,00	-1,44	-1,24	-1,15	-1,46	-1,46	-0,0121
2	BT 18	5	100	120,9	17,37	2,25	0,44	-0,13	2,09	0,14	0,96	0,0079
3	BT 46	11	100	121,0	15,39	-0,11	-0,78	-0,04	-0,97	-0,09	-0,40	-0,0033
4	BT 37	9d	100	120,2	10,59	-0,62	-1,10	-0,59	-0,63	-0,69	-0,73	-0,0060
5	BT 43	10	100	180,0	15,32	-2,90	-1,40	-2,19	-2,11	-1,54	-2,03	-0,0113
6	BT 50	12	100	120,4	12,45	-2,12	-1,08	-2,34	-2,47	-2,40	-2,08	-0,0173
7	BT 39	9d	100	120,6	11,07	-0,42	0,13	-0,17	-0,14	-0,40	-0,20	-0,0017
8	BT 47	11	100	180,5	19,39	0,05	-0,33	-1,68	-0,53	-0,15	-0,53	-0,0029
9	BT 48	12	100	121,4	10,06	-2,24	-2,25	-2,25	-2,37	-2,23	-2,27	-0,0187
10	BT 41	10	100	80,3	10,55	-1,16	-0,60	-1,44	-1,78	-0,55	-1,11	-0,0138

As a typical example of the testing, Test 6 conducted on sample BT 50 is presented in more detail. The sample was cut from a plate that was coated in spray session 12, so the data can be related to the material processing described above.

As the coating was applied on substrate plates, the samples were cut out of these plates with a diameter of 100 mm. The substrate plate acted as the base plate which was mounted on the wind tunnel holder. Temperatures were recorded with thermocouples as well as with pyrometers. One thermocouple was embedded in the material at a depth of 1.5 mm from the back plate. A second thermocouple was directly at the substrate plate. The pyrometers were focused on the center of the sample surface, one spectral pyrometer was used and one two-colour pyrometer. Figure

10 shows the sample before and after the test. During testing all of the samples developed a crack pattern that is quite typical of cork materials.



Figure 10: Sample BT 50 (from session 12) from Test 6 before and after the test.

In Figure 11 the surface temperatures are plotted. After the initial rise phase both pyrometers settle around 1100° C with constant value to the end of the test. In Figure 12 the thermocouple delta-temperature (compared to the start of the test) is plotted. At the test end the temperature has only risen by around 30° C, the maximum of 100° C occurs after the test.

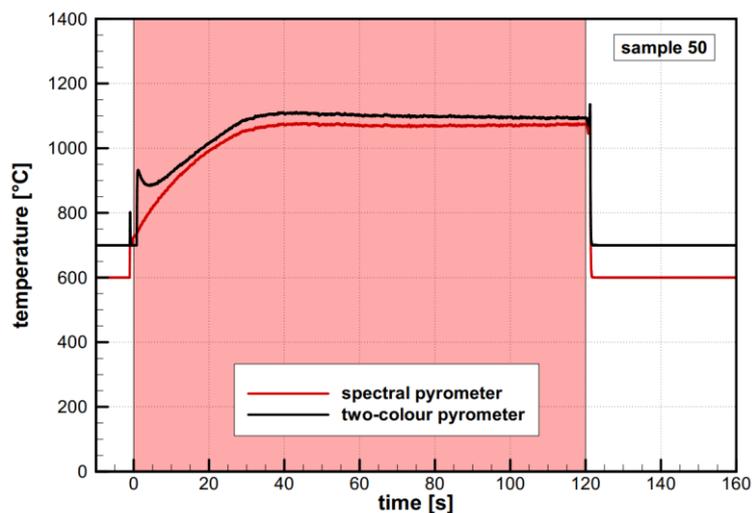


Figure 11: Measured surface temperatures on sample BT 50.

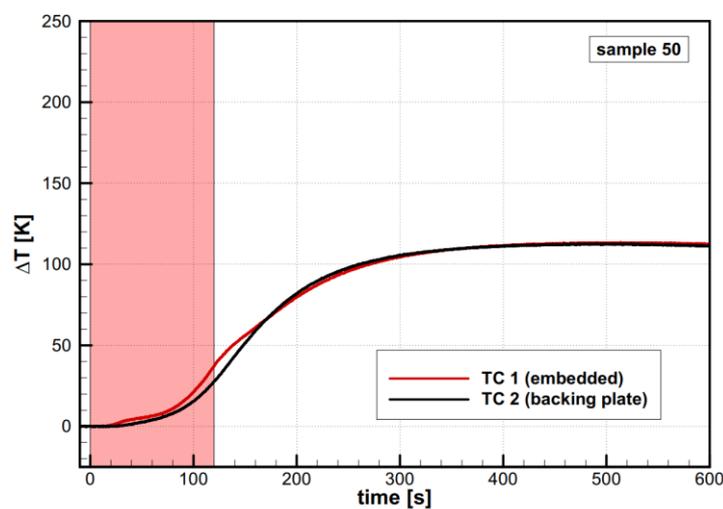


Figure 12: Measured in-depth temperatures of sample BT 50.

Also tests with two samples of the cork-aerogel mixture were tested. The sample before and after the test is shown in Figure 13. The surface appearance of the as-sprayed plate was rather rough. The material showed quite similar behavior during the test as the pure cork materials. The noticeable difference was that the recession was significantly lower. This is a result which calls for further exploration since it could be linked to the use of the aerogel.



Figure 13: Sample BT 46 (session 11) before and after the wind tunnel test.

The surface temperatures were roughly 120° C lower compared to the pure cork materials as is shown in Figure 14. The temperature increase in the material was around 60° C, which is roughly double the one of the cork materials, which can be explained by the higher thermal conductivity.

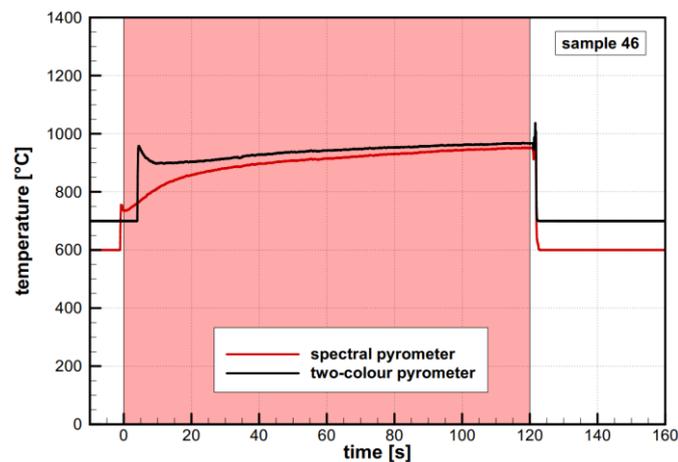


Figure 14: Measured surface temperatures on sample BT 46.

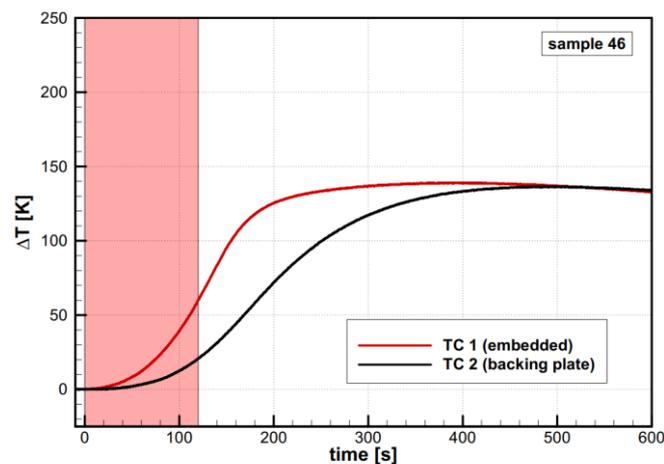


Figure 15: Measured in-depth temperature increase for sample BT 46.

Finally, Figure 16 shows an optical image from the test of sample BT 46 with interesting features of purple gas jets emerging from the cracks in the cork/aerogel material during the wind tunnel test. The surface appearance with the cracking is similar to the cork materials but the purple colour of the jets was only present with the aerogel material.

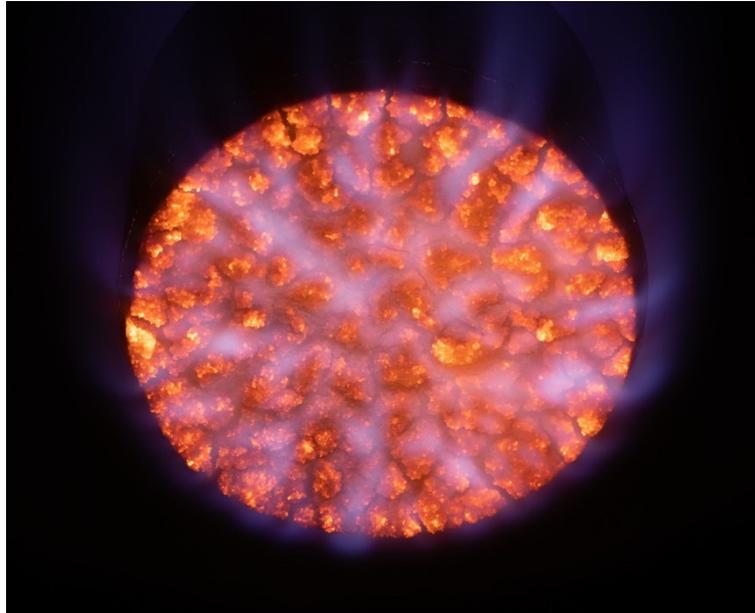


Figure 16: Optical image taken during L2K-test of sample BT 46.

5. Summary and Conclusions

It was investigated if a sprayable thermal protection system material can be developed based on cork and aerogels. Preliminary theoretical calculations had shown that there should be a potential for considerable mass reduction on typical launch vehicles, specifically on the fairings of such vehicles or in other critical areas which are thermally loaded like the base region around the engines.

First trials with cork-resin mixtures were difficult due to unknown factors of the raw materials and the lack of experience with the pumping of highly viscous particle-liquid mixtures. Those problems were related mostly to the quality of the supplied cork raw material but also to the pumping hardware. Concerning the cork, the particle fraction has to be re-sieved to make sure there are no particles of excessive size which can block the spray-gun nozzle. In terms of the pump, an entry-level industrial-grade auger pump was the final choice, but if industrialization on a larger scale was concerned, the larger the pump the better.

After these initial problems had been sorted out, good progress could be achieved in terms of adjusting the mixture parameters so that a lightweight material could be produced with properties as targeted. The density of the coating could be reduced to 195 kg/m^3 which is below the value of typical cork plate materials that are in the range of $300 - 470 \text{ kg/m}^3$. The corresponding thermal conductivity is 0.059 which is exactly on par with the DLR plate material but significantly below the value of Norcoat.

Tests in the arc-heated wind tunnel L2K in DLR Cologne showed that also the performance of the material in a re-entry condition is good. The material behaved as expected without surprises. The test environment was 200 kW/m^2 , the surface temperatures went up to 1100°C and were very stable for the whole test time of 120 s . The average recession values were on the order of 2 mm for the time of 2 minutes .

Also materials with cork-aerogel mixtures could be produced. The processing was more difficult and not successful when the initially chosen sodium-silicate binder was used. A mix with cork particles and phenolic/silicone resin plates could be produced successfully. The thermal properties can certainly be improved, however, the first goal was to test the material in the wind tunnel. Indeed, the recession data of those plates is quite promising with very low values of 0.46 mm on average.

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