Executive Summary Report

Gecko Material for Space Applications

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1. Introduction

This document gives an executive summary of the tasks conducted in the acitivity “Gecko Materials for Space Applciations”.

2. Testing and optimization of gecko adhesives

2.1. Fabrication procedure and conditioning

Gecko-inspired, micropatterned dry adhesives were made from silicone (PDMS) or polyurethane (PU) materials, namely, Sylgard 184 (Dow Corning) and Poly-Optic 14-70 (Polytek Development Corp), respectively. As template for replica molding, milled aluminum molds were used (**Figure 1a**). The bottom of the mold was closed by smooth polyethylene terephthalate (PET) films (Melinex 401 CW, DuPont). The PDMS and the PU are two-component systems consisting of a base and a curing agent. The PDMS and PU were mixed in a 10:1 and 4:5 ratio, respectively. For both materials, the components were mixed with 2350 rpm and degassed at 1 mbar for 3 min using a SpeedMixer (DAC600.2 VAC-P, Hauschild Engineering). The prepolymer mixture was then filled into the mold and degassed for 5 min at 1 mbar. Finally, the prepolymer mixture was cured in an oven – at 65°C for two hours for PU and at 95 °C for two hours for PDMS. Micropatterned arrays consisted of mushroom-shaped pillars with heights of 1.6 mm, diameter of the stalks of 0.4 mm and wider (mushroom-shaped) tips with a diameter of 0.71 mm (**Figure 1b,c**). The backing layer thickness was 5 mm.

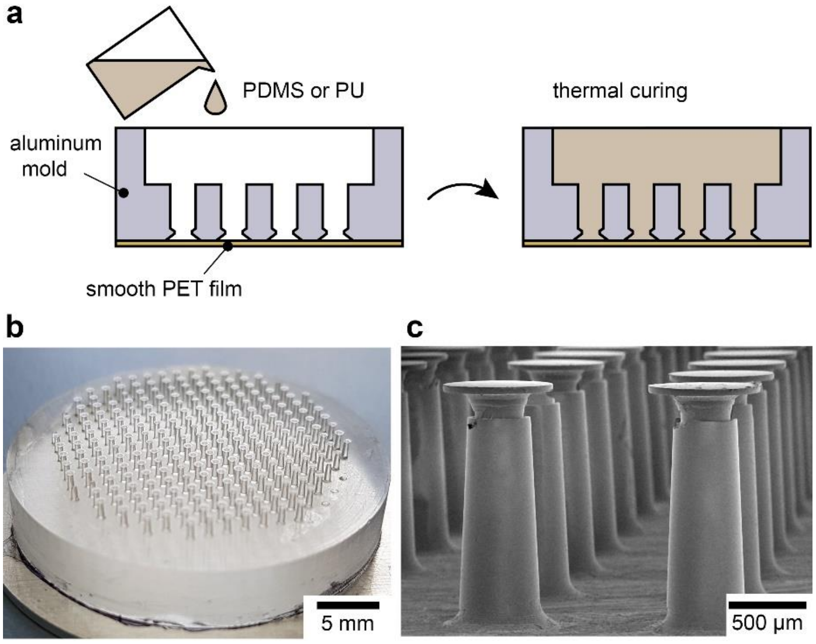


Figure 1: Micropatterned dry adhesives. (a) Schematic illustration of the molding process: Prepolymer (PDMS – Sylgard 184 or PU – PolyOptic 14-70) is filled into a aluminium mold closed at the bottom with smooth PET film. Upon thermal curing of the prepolymer, the specimen is gently demolded. **(b)** Optical image showing the dimensions of the micropatternd specimens. **(c)** Electron micrograph showing a side view of the mushroom shaped microstructures.

It is well known that Sylgard 184, as a standard silicone material for micropatterned adhesives, is a technical grade material, which contains non-crosslinked, free silicone oils in the polymer network.(Kroner, Maboudian, & Arzt, 2010) Therefore, Sylgard 184 was expected to fail thermal vacuum outgassing tests according to ECSS-Q-70-02. To solve this problem, free oils and unreacted prepolymers can be (partially) extracted from the crosslinked silicones by washing in solvents such as heptane, tetrahydrofuran, or acetone. For this purpose, crosslinked materials were immersed into different solvents for 24 h and then dried in vacuum (approx. 1 mbar) for at least 24 h. The specimens treated with this procedure were used for outgassing tests. As a result, the treatment with heptane and acetone was positively evaluated to approve the silicone material for space applications.

Adhesion was measured before and after solvent treatment of Sylgard 184 specimens. The tests were performed using a tensile tester (inspekt 5 table blue, Hegewald&Peschke, Nossen, Germany) equipped with a 50 N load cell to record normal forces (**Figure 2**). The tensile tester was modified to perform adhesion tests on a smooth and nominal flat glass substrate. Below the transparent glass substrate, a mirror and a camera were mounted to optically align the specimen with the substrate mounted to a θ-ϕ-goniometer (MOGO, Owis, Staufen im Breisgau, Germany). In the adhesion measurements, specimen and substrate were brought together with a compressive preload of 1 N (specimen was pressed to the substrate). Upon contact formation, specimens were immediately withdrawn until detachment. The maximum tensile force is defined as pull-off force. The test velocity was 1 mm/min for attachment and retraction. Measurements were repeated 3 times on different positions of the substrate.

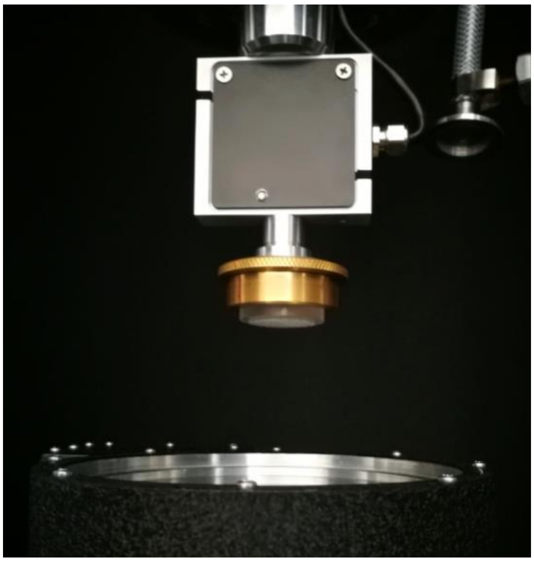


Figure 2: Adhesion tests: Photograph of the adhesion test setup containing glass substrate (bottom), specimen and load cell to record forces.

The results are shown in **Figure 3a**. The adhesion increased by factor 1.1 to 1.2 upon acetone and heptane treatment. These results demonstrate better adhesion performance of silicone based materials upon solvent treatment in addition to the improved outgassing performance. However, we observed a practical limitation of the solvent treatment due to swelling of the specimens by more than 100%. The swelling is further inhomogeneous due to the complex structure of the specimens, leading to inhomogeneous deformations and most likely internal stresses. These stresses partially resulted into defects; particularly, the mushroom tips of the structures partially ruptured, because of the larger deformation of the stalks compared to the thin flaps of the mushroom tips (**Figure 3b**). Interestingly, the adhesion improved despite the fact that such defects occurred.

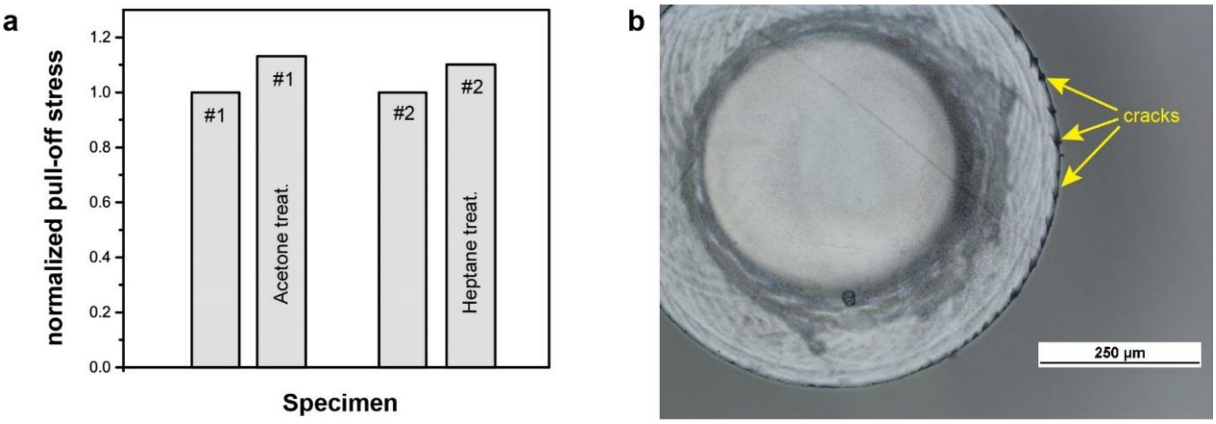


Figure 3: Adhesion test results. (a) The normalized pull-off stress as a function of solvent treatment upon sample fabrication. Left columns without and right with solvent treatment. **(b)** Optical micrograph showing defects at the rim of a mushroom tip upon solvent treatment.

2.2. Testing

2.2.1. Standard outgassing according to ECSS-Q-ST-70-02

Outgassing tests according to ESA-standard ECSS-Q-ST-70-02 were conducted to qualify raw materials for their suitability for gecko adhesives for space applications. The outgassing behaviour was assessed by measuring the weight of the samples before and after thermal treatment. Furthermore, the mass gain of cooled collectors due to condensed matter was measured. By measuring the condensed mass it was possible to evaluate the possible contamination of sensible components (such as mirrors) by outgassed material. As for many applications the water-loss is not relevant, samples were also weighted after a post-conditioning (24 hours at 22°C and 55%rH) allowing recovery of lost moisture. The following parameters were determined as result of an outgassing test:

- **TML** (**T**otal **M**ass **L**oss): Total mass loss relative to the initial sample mass
- **RML** (**R**ecovered **M**ass **L**oss): Mass loss after water absorption during post-conditioning (relative to initial sample mass)
- **CVCM** (**C**ollected **V**olatile **C**ondensable **M**aterial): mass gain of collectors relative to initial sample mass.

The following table states the outgassing test facts.

Table 1: Outgassing facts

Test	Outgassing test according to ECSS-Q-ST-70-02	
Sample dimensions	3 samples per material required by ECSS for statistical evaluation Each sample weight 100 – 300 mg Max. size per sample 8x8x10 mm	
Materials per test run	For each test run 4 materials plus 1 empty sample cup (reference) is measured (i.e. 15 samples)	
Vacuum	P < 10^{-5} mbar	
Temperatures	22°C	24 hours (pre-conditioning at 55%rH)
	125 °C	24 hours (thermal vacuum test)
	(up to 300°C	on request (non-ECSS))

	22°C 24 hours (post-conditioning at 55%rH)
Results & ECSS requirements	TML: mean value < 1.0% standard deviation < 0.1 * mean value RML: mean value < 1.0% standard deviation < 0.1 * mean value CVCM: mean value < 0.1% standard deviation < 0.2 * mean value

Eight Materials have been tested according to above procedure. The results are shown in Table 2 and Figure 4.

Table 2: Testresults of the ECSS-Q-ST-70-02 on the eight gecko materials

Material	TML	RML	WVR	CVCM	comment
Nusil 2590/65°C	0,04	0,04	0,01	0,00	"low outgassing"
Sylgard 184/95°C Heptane	0,06	0,05	0,01	0,02	Standard from INM
Sylgard 184/95°C Acetone	0,12	0,10	0,01	0,04	Standard from INM
PU 1470/60°C	1,53	0,24	1,29	0,01	Standard from INM
ESSAR Stretch	1,02	0,97	0,05	0,14	"low outgassing"
Sylgard 184/95°C	1,05	1,05	0,01	0,48	Standard from INM
Sylgard 184/95°C THF	0,68	0,67	0,00	0,25	Standard from INM
Sylgard 184/75°C	1,17	1,17	0,01	0,55	Standard from INM

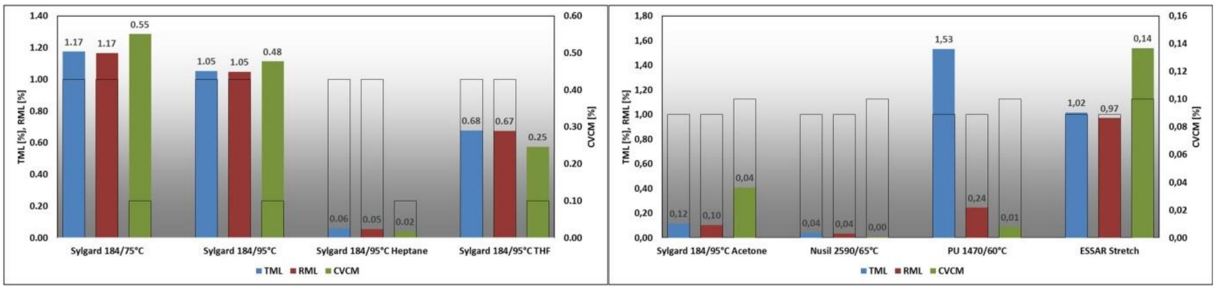


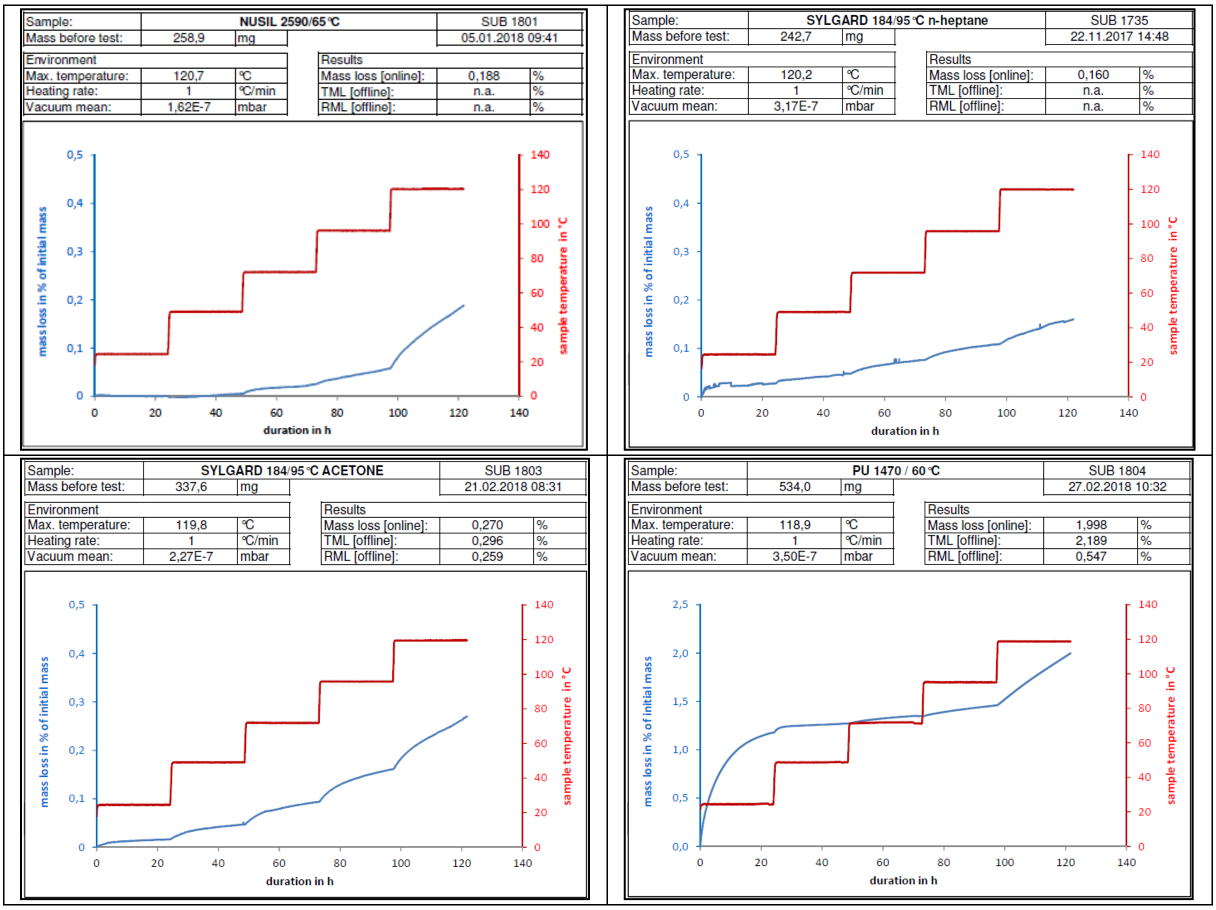
Figure 4: Testresults of the ECSS-Q-ST-70-02 on the eight gecko materials

Four of the eight materials passed the outgassing test:

- Nusil 2590/65°C – which is commercially offered as low outgassing material
- Sylgard 184/95°C Heptane
- Sylgard 184/95°C Acetone
- PU1470/60°C (the TML of 1.53% exceeds 1%, but this is not relevant according to last version of ECSS-Q-ST-70-02)

2.2.2. Advanced outgassing according to ECSS-Q-TM-70-52A

The four candidate materials that passed the standard outgassing tests were tested according to ECSS-Q-TM-70-52A. Target is to gain data of outgassing behaviour and a long term prediction for a refernce temperature of 25°C. The mass loss (in % of m_0 – blue line) and temperature (red line) over time is shown in Figure 5. The temperature is increased in 25°C steps up to 125°C every 24 hours. With the data gained from the temperature isothermals a long term outgassing prediction for the TML is calculated. Figure 6 shows the plot of the longterm TML prediction of the sample for a reference temperature of 25°C. Residual gas analysis was done as well. No noticable molecules were found in any of the four tests.



Sample:	NUSIL 2590/65°C		SUB 1801
Mass before test:	258,9	mg	05.01.2018 09:41

Environment		
Max. temperature:	120,7	°C
Heating rate:	1	°C/min
Vacuum mean:	1,62E-7	mbar

Results		
Mass loss [online]:	0,188	%
TML [offline]:	n.a.	%
RML [offline]:	n.a.	%

Sample:	SYLGARD 184/95°C n-heptane		SUB 1735
Mass before test:	242,7	mg	22.11.2017 14:48

Environment		
Max. temperature:	120,2	°C
Heating rate:	1	°C/min
Vacuum mean:	3,17E-7	mbar

Results		
Mass loss [online]:	0,160	%
TML [offline]:	n.a.	%
RML [offline]:	n.a.	%

Sample:	SYLGARD 184/95°C ACETONE		SUB 1803
Mass before test:	337,6	mg	21.02.2018 08:31

Environment		
Max. temperature:	119,8	°C
Heating rate:	1	°C/min
Vacuum mean:	2,27E-7	mbar

Results		
Mass loss [online]:	0,270	%
TML [offline]:	0,296	%
RML [offline]:	0,259	%

Sample:	PU 1470 / 60°C		SUB 1804
Mass before test:	534,0	mg	27.02.2018 10:32

Environment		
Max. temperature:	118,9	°C
Heating rate:	1	°C/min
Vacuum mean:	3,50E-7	mbar

Results		
Mass loss [online]:	1,998	%
TML [offline]:	2,189	%
RML [offline]:	0,547	%

Figure 5: mass loss in % of initial mass of the samples for Nusil 2590/65°C (top left), Sylgard 184/95°C n-heptane (top right), Sylgard 184/95°C Acetone (bottom left), PU1470/60° (bottom right)

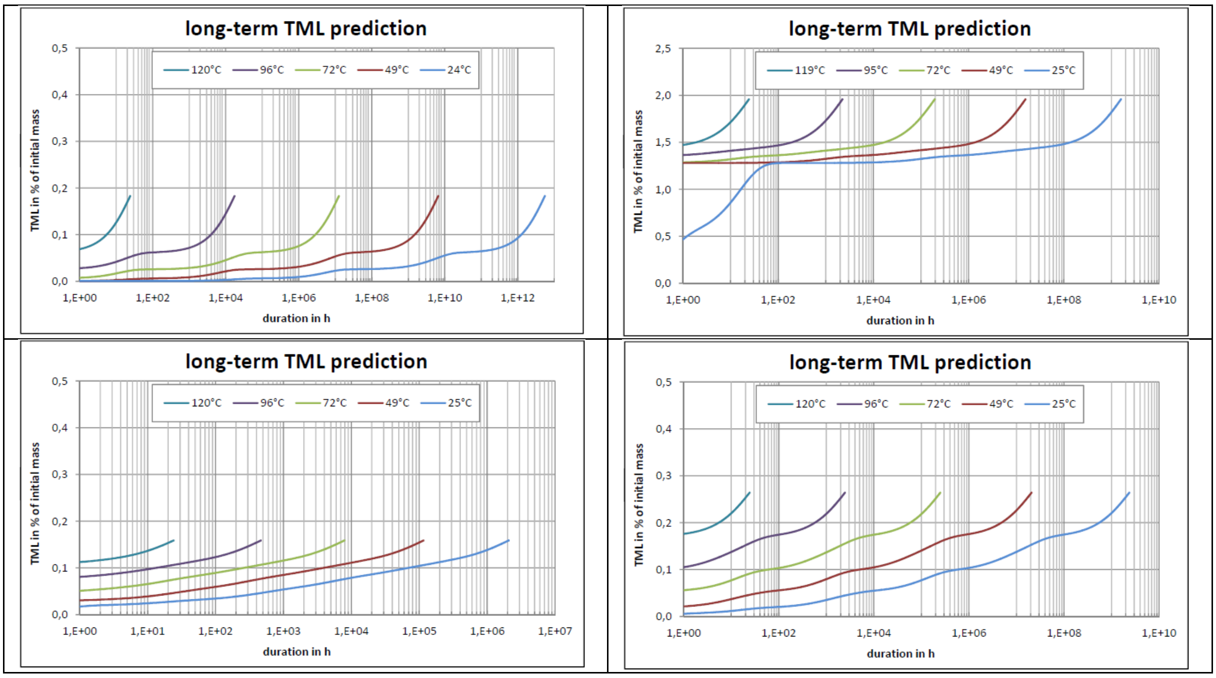


Figure 6: longterm TML prediction for Nusil 2590/65°C (top left), Sylgard 184/95°C n-heptane (top right), Sylgard 184/95°C Acetone (bottom left), PU1470/60° (bottom right)

2.2.3. Adhesion testing

Three materials have been exposed to thermal cycles in vacuum (Nusil has been excluded due to cost). The cycling was done between -125°C and +125°C for 10 times. The influence of the thermal cycling on adhesion was afterwards investigated in adhesion tests. For each of the three gecko-inspired materials, tests were performed for different preloads. Each material was tested in virgin state and in thermally cycled state to compare if thermal cycling has influence on the performance. Each material was tested in air and in vacuum. Thus, for every material four different sets of data are available:

- material virgin state, tested in air
- material virgin state, tested in vacuum
- material thermal cycled, tested in air
- material thermal cycled, tested in vacuum

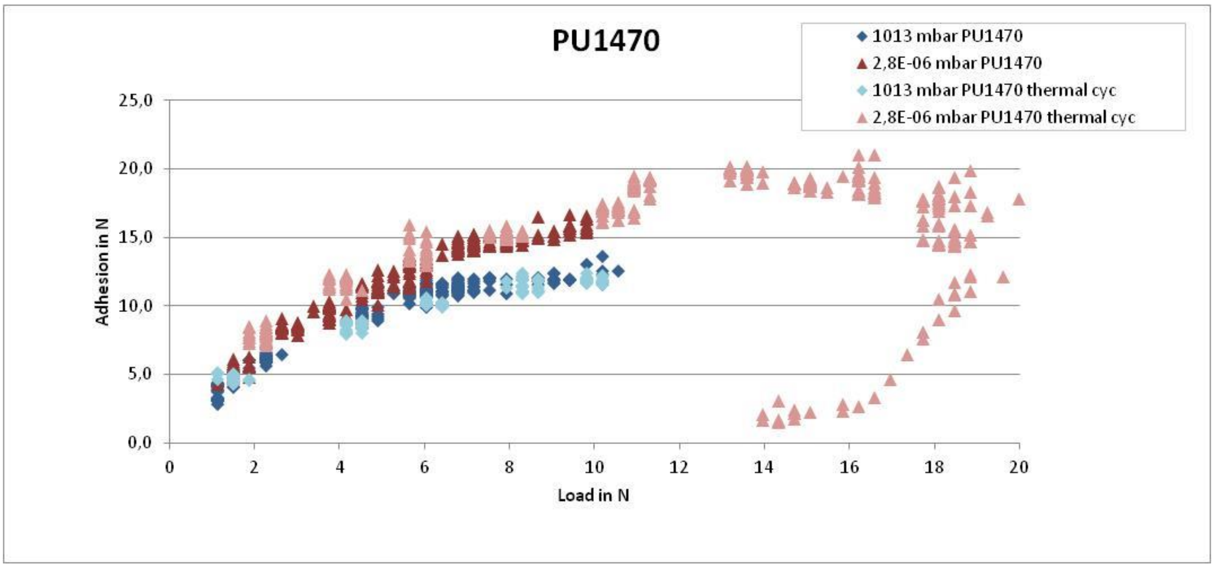


Figure 7: adhesion forces of PU1470

- PU1470 showed highest adhesion forces of all tested materials.
- Adhesion forces in vacuum are slightly higher than in air for loads higher than 6N
- At loads higher than 18N, adhesion might breakdown – these high loads were only tested in vacuum at the thermal cycled material. The geckomaterial might be damaged at those high loads (visual difference to unused area)
- No difference of virgin geckomaterial and thermal cycled geckomaterial is seen

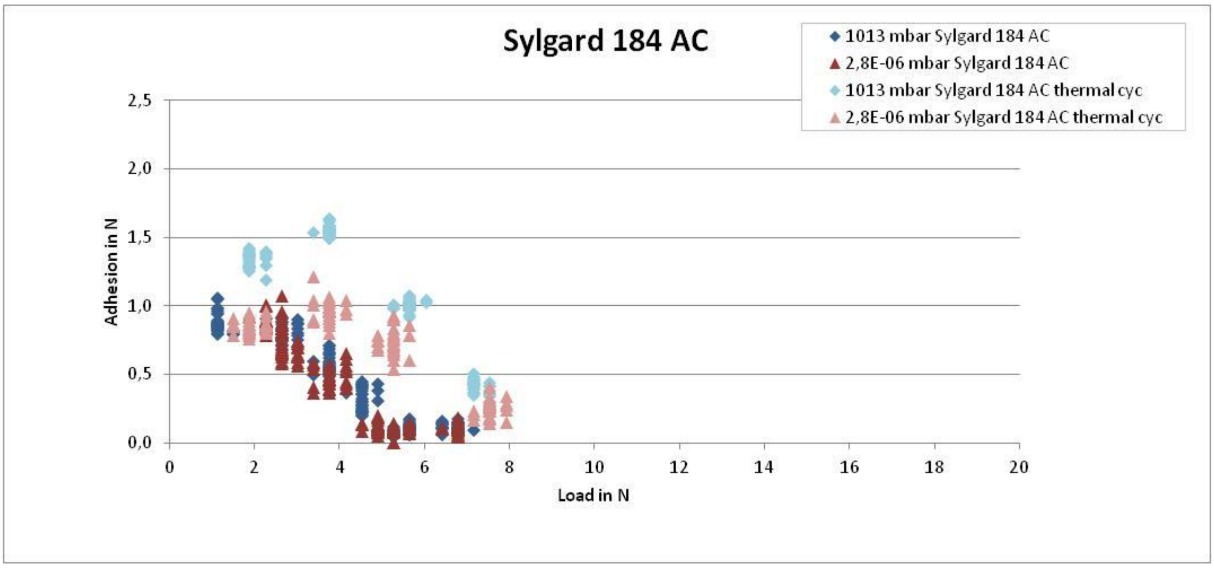


Figure 8: adhesion forces of Sylgard 184 AC

- Sylgard 184 AC shows very low adhesion forces compared to PU1470
- At loads higher than 4N, no adhesion force is measured
- The thermal cycled material shows slightly higher loads, where adhesion breaks down (~6N)

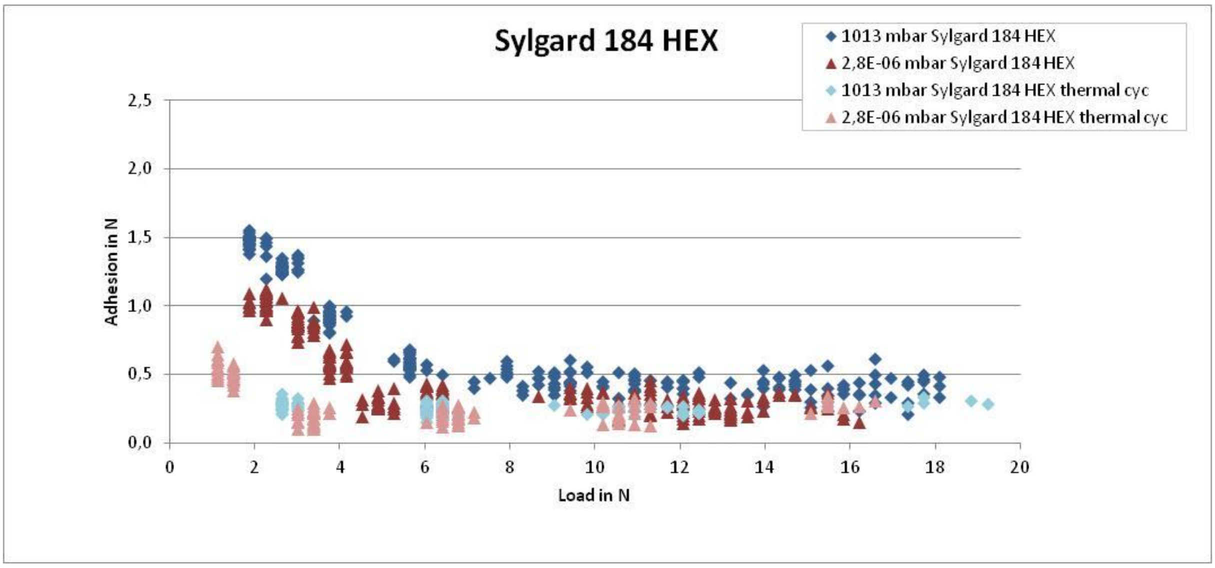


Figure 9: adhesion forces of Sylgard 184 n-heptane

- Sylgard 184 heptane shows very low adhesion forces compared to PU1470
- At loads higher than 4N, no adhesion force is measured

The thermal cycled material shows lower adhesion forces

2.2.4. Operational parameters testing

The influence of the following operational parameters was analyzed:

- Use cycles
- Target surface

The influence of number of use cycles was analyzed by repeating load cycles with different preloads. Results are shown in Figure 10 (left). The influence was assessed to be negligible. To analyze a potential influence of the target surface, titantium and painted metal were used as substrates in addition to acrylic glass. Results are shown in Figure 10 (right) The influence was assessed to be negligible.

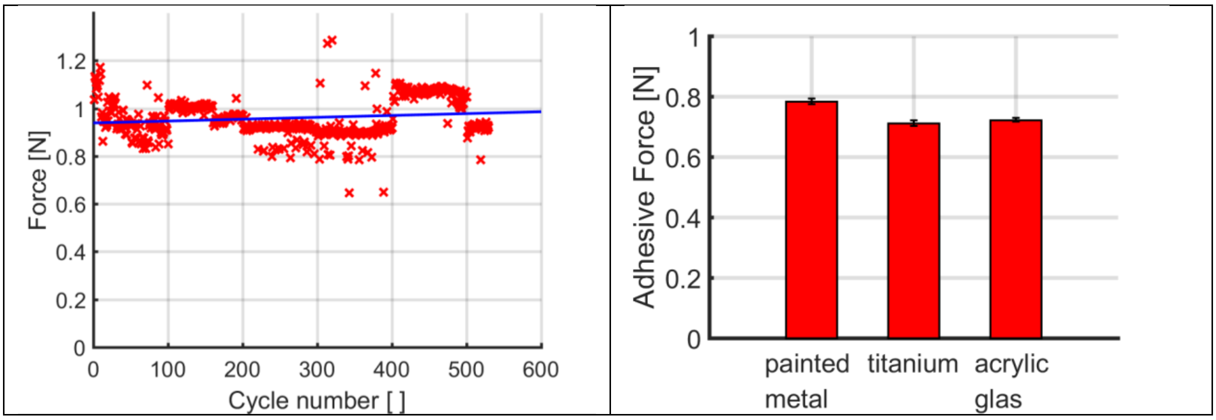


Figure 10: Influence of use cycles (left) and target material (right)

3. Docking mechanism development

Based on the desired properties and the accompanied solutions, a detailed design of the mechanism was created. The mechanism's dimensions are similar to the dimensions of a 1U CubeSat, with a mass of approximately 1.33 kg. The mechanism adheres through four gecko adhesive pads that resemble the four feet of the gecko. The adhesive surface area accumulates to approximately 15 cm². Each adhesive can passively adapt to the target, while load can actively be shared between the adhesives. Both properties mimic the gecko's feet properties. Additionally, the mechanism has a rigidization functionality, which allows to change the connection from a flexible state to a stiff state. This functionality can also be used for detachment of the mechanism. The next section gives an overview of the mechanism design, and the subsequent sections describe the different functionalities in detail.

The mechanism is divided into an upper layer and a lower layer. As part of the upper layer, the mechanism features an electro-mechanical interface. The upper layer is composed of the rigidization unit, the on board computer, the main electronics PCB and the electro-mechanical interface. The lower layer includes the load sharing units, the load measurement electronics, the four adhesive units, and a sensor unit. In order to have a generic attachment capability, the mechanical interface is based on a 3x3 matrix of M5 screws.The electrical interface houses a voltage regulator connected to a 15 pin female D-Sub plug connector for power supply as well as a number of general-purpose input/output pins (GPIO) for data and command exchange.

After production and procurement of all mechanical parts, the mechanism has been assembled and visually inspected. Figure 11 shows the assembled mechanism.

Figure 11: Visual inspection of the mechanism

4. Performance Characterisation

In order to test the adhesion performance of the prototype, a simple adhesion test facility was used. This facility consists mainly of the test substrate, a mechanical aparatus to be able to change curvature of the test substrate as well as load angle, and a 1D force sensor for adhesion measurements.

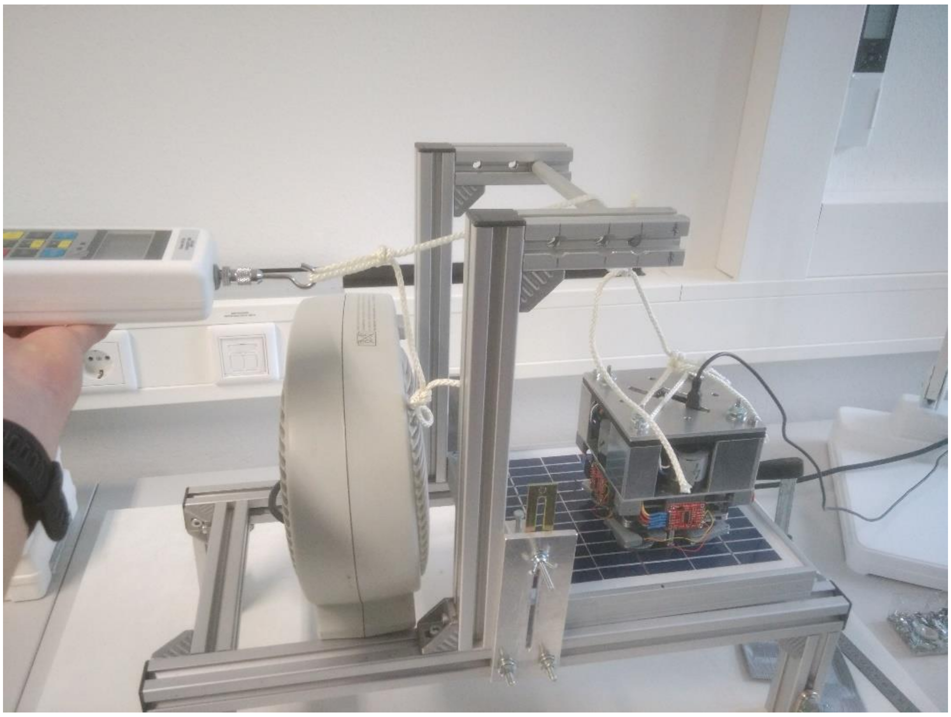


Figure 12: Adhesion measurement facility

For dynamic free-floaitng tests, the ”Experimental Laboratory for Proximity operations and Space Situational Awareness” (ELISSA) free-floating laboratory was used that is based on air-bearing technology and allows for three DOF free-floating motion.

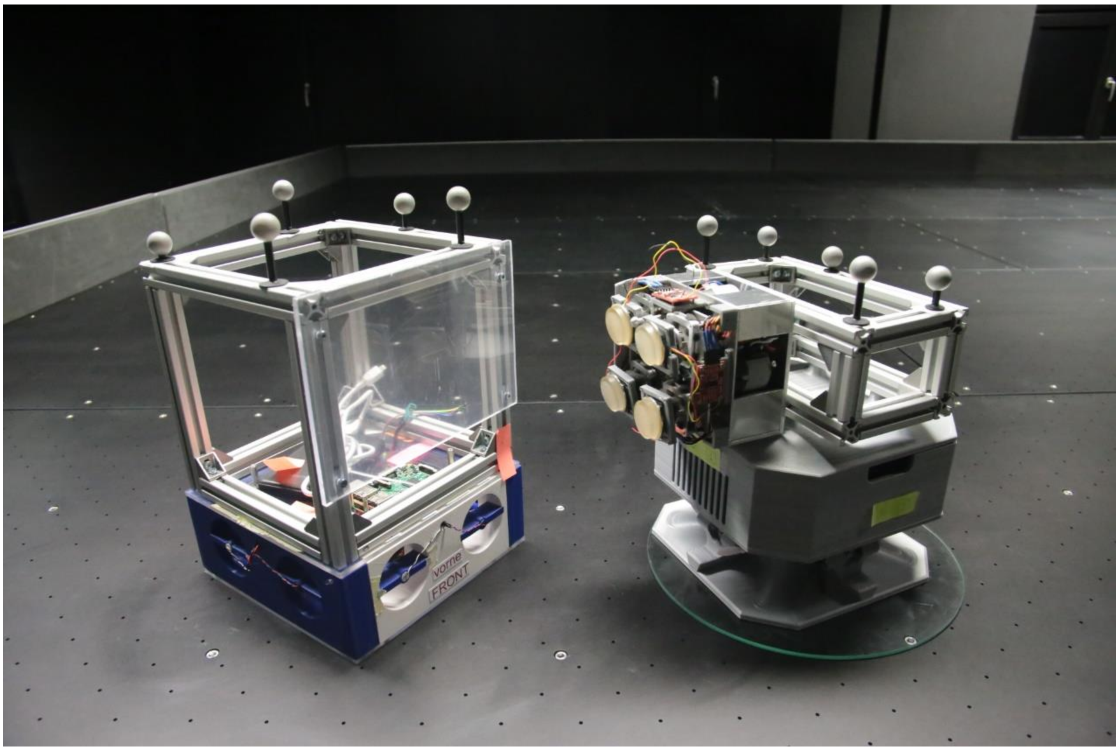


Figure 13: Test setup in the ELISSA laboratory

Summarizing the tests, it was was not possible to verify all the anticipated functionalities. The prototype's rigidization subsystem seems to increase robustness in terms of load angle, but has no influence on overall maximum adhesion. The load sharing subsystem could not be successfully tested, thus no information could be gathered regarding the performance of this subsystem. The release functionality could be successfully shown to work. The overall adhesion is very low and can currently not be considered successful. This might be already overcome by improved gecko-inspired adhesives, as well as the use of more sophisticated actuators. In the dynamic free-floating environment, the prototype could not successfully dock to a target object.

Concluding, some but not all of the core functionalities could be shown with the current prototype. However, further work is required in maturing the prototype. As a proof of concept, the prototype can still be considered successful.

5. Suitability assessment

We demonstrated that selected materials can be utilized for space applications, but require special treatment or specific conditions for their use. The amount of low molecular weight components was successfully reduced by solvent treatments.

There exist silicones in higher quality with space approval, however, the price for the raw material is usually two orders of magnitude higher compared to silicones with technical grade as used in our work. Polyurethanes often show a glass transition temperature between -100 and +100 °C, and often exhibit more than one glass transition. Thus, adhesion, which is linked to mechanical properties, drastically varies when temperature changes. We suggest that for reliable application of PU in space applications, the adhesive device has to be used at a constant temperature, for instance, set by a heating unit.

The hardware prototype’s general functionality was tested successfully. When assessing the performance of the subsystems, it was observed that the load sharing concept could successully be employed due to shortcomings in the components. For the rigidization subsytem, it was observed that performance was generally as expected.

6. Development plan

In order to prepare a docking mechanism bsaed on gecko-inspried adhesives for space applications, the following required improvements have been identified:

- Improvements to the materials
- Improvements to the fabrication process
- Additional environmental tests
- Study of operational limits of the mechanism
- Influence of surface degradation
- Influence of atomic oxygen and radiation
- Improvements of technical maturity of components
- Preparation for ISS experiments

7. Summary & conclusion

This activity contained three main technical objectives. The first objective was to analyse how gecko-inspired adhesives perform under the influence of the space environment. For this purpose, the state of the art in terrestrial applications was assessed regarding both the adhesives and mechanisms that use the adhesives, before analysing which environmental and operational parameters of space-based reference missions could impact the adhesion performance. Subsequently, standard and advanced outgassing tests were performed according to ECSS standards in order to find suitable raw materials for gecko-inspired adhesives. The second objective was the optimisation of gecko-inspired adhesives for space applications. Based on the raw materials evaluated in the first opbjective, gecko-inspired adhesives were produced and subjected to thermal-vacuum adhesion tests in order to assess their adhesion performance. In parallel, the third objective was addressed, developing and testing a docking mechanism prototype bsaed on the adhesives that performed best in the adhesion performance tests. The prototype was then tested, equipped with the gecko-inspired adhesives that had performed best. Based on the test results, the suitability of the concept of gecko-inspired docking was assessed, a development plan derived for application of the concept in space, and lessons learnt documented for follow-up projects.

Although not all of the core functionalities of the gecko-inspired mechanism prototype could be shown, as a proof of concept, the prototype is still considered successful.

Future work should be targeted towards improving the performance of the mechanism prototype to address some of the issues encountered during the characterisation testing (low overall adhesion, no influence of the rigidization system on overall maximum adhesion, unsuccessful load sharing subsystem). The use of improved gecko-inspired adhesives, as well as the use of more sophisticated actuators is expected to already provide substantial improvement in those performance aspects.