Leibniz Gemeinschaft

Abschließender Sachstandsbericht
Leibniz-Wettbewerb

Titel: Gecomer®-Technologie – Transfer des Gecko-Effekts in die industrielle Anwendung
Antragsnummer: 493

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Projektnummer: T56/2015

Federführendes Leibniz-Institut: Leibniz-Institut für Neue Materialien gGmbH

Projektleiter/in: Prof. Dr. Eduard Arzt, Dr. Peter W. de Oliveira

Inhalt

1. Zielerreichung und Umsetzung der Meilensteine

	Deliverables	Remark
AP1a	> 10 base materials screened under consideration of adhesive properties and process suitability	✓
AP1b	Numerical optimization of structure geometry with respect to realistic fabrication methods	✓
AP1c	1. R2R system tested; Coating methods optimized and established 2. > more than 5 mold materials screened for being applied as flexible mold 3. Flexible mold prepared and mounted on R2R line 4. Trial tests done for fabricating microstructures	✓
AP1d	1. Material characterization established for base material screening in terms of mechanical properties (modulus, elasticity), curing rate, viscosity, thermal properties, shrinkage and adhesion 2. Test structures examined in terms of morphology and adhesive properties	✓
1st milestone	1. Test structures obtained from R2R line in a continuous manner. The microstructures were able to demold and cured with UV LED 2. Modelling results allowed fabrication of master templates	✓
AP2a	Structures with 3 different aspect ratio examined; 2 of them have been used in R2R trials	✓
AP2b	1. Curing condition optimized 2. Material properties tuned in term of viscosity, curing rate, surface energy and so on 3. Housing for vacuum and N_2 supply established 4. Vacuum accomplished	✓ Well replicated microstructures obtained even without vacuum
AP2c	Empirical optimization of processing, as vacuum assisted processing was not necessary due to drastical improvements in materials properties (rheology)	✓
2nd milestone	1. Microstructures made from different formulations fabricated on R2R 2. Obtained micropatterned adhesive film demonstrated good demolding behavior, high adhesion as well as reusability	✓
AP3a	1. Prototypes with 2 different microstructures obtained 2. Instead of vacuum, contact pressure was modulated to control microstructure morphology and resulting adhesion	✓
AP3b	Cyclic adhesion tests	✓
AP3c	Stability and vacuum test covered	✓
AP4	Paper manuscripts written, submitted and published	✓
3rd milestone	1. Upscaling with inline control established 2. Aging test 3. Cyclic adhesion tests	✓

2. Aktivitäten und Hindernisse

The project was conducted as an internally collaborative work in INM between two groups “Functional Microstructures (FM)” and “Innovation Center (IC)”, which mainly engaged on methodology & investigation (for FM) and process optimization & validation (for IC). The both groups worked closely during the project all the way from conceptualization to paper publication. During the project, INM showed the Gecomer®-Technology on the following trade fairs:

- Hannover Messe (2016-2018), Hannover, Germany
- Motek (2016), Stuttgart, Germany

Members of the project team participated and presented on the following conferences, symposia and workshops:

- Danube Vltava Sava Polymer Meeting (DVSPM), Vienna, September 5-8, 2017
- Advanced 3D Patterning (ad3pa), Dresden, October 4-5, 2018
- Coatema, Dormagen, October 17-18, 2018

Besides that, contact was made with more than 50 companies during the project, to figure out the most promising markets and to also start direct bilateral cooperation (industry projects).

An alteration was made in project plan by reducing the number of microstructure master mold types from 3 to 2. Instead, we more focused on the long term testing and “real application testing” of our bioinspired adhesives. The reason for this is the following: due to technical challenges, there was significant delay of the supply of master molds by a third party (company temicon). In order to ensure the scheduled progression, the project was carried out on microstructures with two geometries (25 and 50 µm in pillar diameter) instead of three geometries (15, 25 and 50 µm in pillar diameter) as planned, but 15 µm structures could finally be tested on the R2R machine on smaller scale.

3. Ergebnisse und Erfolge

In this project, a continuous and scalable roll-to-roll process was established for fabricating dry adhesives with mushroom-shaped microstructures. Through the utilization of a large-scale roll-to-roll system which features a total web length of 24 m and a steel imprinting roll of 50 cm in diameter, urethane acrylate oligomer was micro-imprinted and UV-cured on PET foil in a continuous manner, generating adhesive films of 50 cm in width and several tens of meters in length, suitable for wide range of applications. The integrity of the microstructures and demolding quality were ensured by the utilization of the flexible template. The morphology and adhesion were controlled by the compression on the flexible template (**Figure 1**).

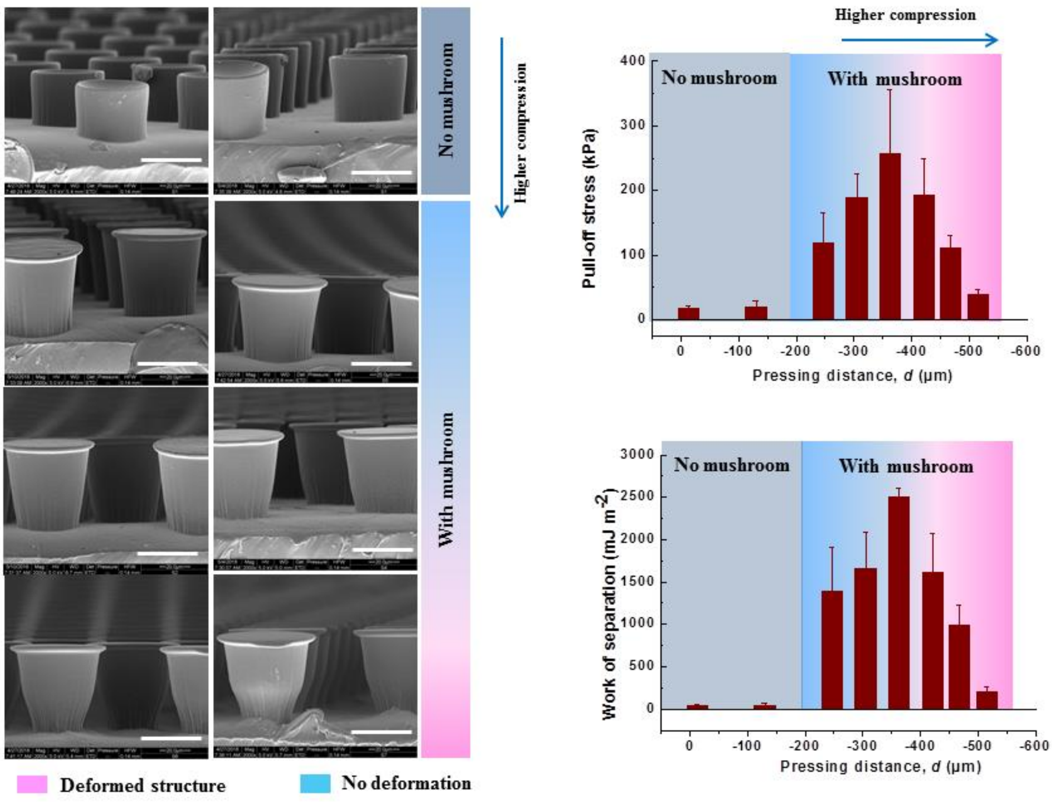

Figure 1. Scanning electron micrographs of UA16 micropillars fabricated with different compression (left). Scale bars are 40 µm. The dependence of pull-off stress and work of separation on compression (right).

The replication quality, demolding readiness and adhesive performance of the obtained dry adhesives highly depend on the adhesive material compositions and process conditions. A double layer fabrication method was adopted. The prepolymers were tuned in composition by modulating the components with different properties; and the process parameters were adjusted in system speed, UV power, pressing distance, coating thickness and type of PDMS for flexible templates.

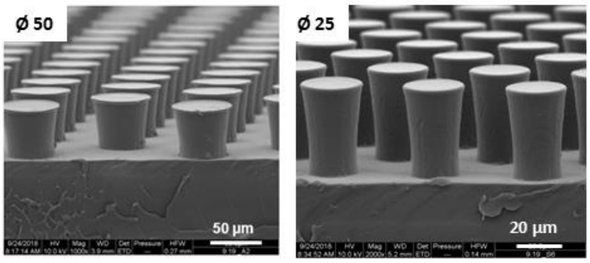

Figure 2. Scanning electron micrographs of the microstructures with pillar diameter of 50 µm and 25 µm.

The modified prepolymer and optimized process parameters enabled the integrity of the complex microstructures (**Figure 2**) and good adhesion results (**Table 1 and Figure 3**). The optimized results for micropatterned adhesives with pillar diameter of 50 µm exhibited 116 kPa in pull-off strength, 1.4 N cm^{-1} in peel strength and 71 kPa in shear strength. Furthermore, repeated attachment-detachment tests over 100,000 cycles demonstrated good reusability. The adhesive with pillar diameter of 25 µm showed pull-off strength of 196 kPa, shear stress of 60 kPa, peel strength of 1.2 kPa and reusability of 1,340,923 attachment-detachment cycles. The main results are published.

Table 1. Summary of adhesion results

Microstructure	Pull-off (kPa)	Shear (kPa)	Peel (N cm^{-1})	Durability (number of cycles)
50 µm	116	71	1.4	100,000
25 µm	196	60	1.2	1,340,923

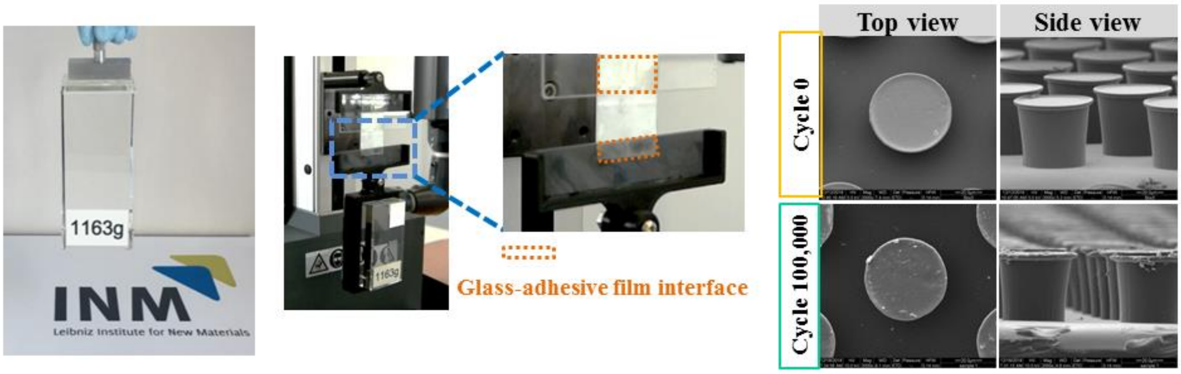

Figure 3. Photographs of handling a glass prism with mass 1.163 kg demonstrates normal adhesion (left) and shear adhesion (middle) of the adhesive film of U6L7-U6E4. Scanning electron images before and after 100,000 attachment-detachment cycles show good endurance of the adhesive film U6L7-U6E4 (right).

Publications:

- Yu D, Beckelmann D, Opsölder M, Schäfer B, Moh K, Hensel R, Oliveira PWd, Arzt E (2019) Roll-to-roll manufacturing of micropatterned adhesives by template compression. Materials 12(1), 97 *(Open Access)*
- Yu D, Hensel R, Beckelmann D, Opsölder M, Schäfer B, Moh K, Oliveira PWd, Arzt E (2019) Tailored polyurethane acrylate blend for large-scale and high-performance micropatterned dry adhesives. Journal of Material Science, 54(19), pp 12925-12937 *(Open Access)*

Conferences:

- Poster "Gecko-inspired dry adhesives fabricated by roll-to-roll process with modulated UV-curable polymers" at DVSPM in Vienna, September 5-8, 2017.
- Poster "Roll-to-roll fabrication of mushroom-shaped micropatterned dry adhesives using flexible templates" at ad3pa in Dresden, October 4-5, 2018.

4. Chancengleichheit

The Leibniz-Institute for New Materials is in accordance with the Equality Implementing Agreement, the Saarland State Equality Law, as well as the Research-Oriented Standards on Gender-Equality of the German Research Foundation. It also follows the Leibniz Equality Standards, added in 2016. The project team is constituted by all genders with a female scientist as technical lead and also the "senior" technician being female. Equal opportunities were given to each team member regardless of e.g. gender, nationality, age, or religion. Details on how INM cares about Gender Equality, Promotion of Young Researchers, Audit Certificate "Beruf und Familie" and more can be found on https://www.leibniz-inm.de/en/institute/equality/

5. Qualitätssicherung

The INM is eager to assure the best quality of research possible. Therefore, it not only published the most important results of this project exclusively as Open Access Articles (DOI: 10.3390/ma12010097 and DOI: 10.1007/s10853-019-03735-x)
and thus follows the Open Access Policy of the Leibniz-Association, but it is also committed to the Good Scientific Practice, according to the Guidelines of the Leibniz-Association. Details on how INM cares about Good Scientific Practice and about the "Ombudsperson" of INM can be found on https://www.leibniz-inm.de/en/institute/good-scientific-practice/

6. Zusätzliche eigene Ressourcen

In order to being able to operate the project, the Leibniz-Institute for New Materials contributed a number of own resources, which were mainly infrastructure, machines and devices, but also personnel. In more detail, this has been a large (industrial scale) roll-to-roll machine which is completely encased by a 200 m² clean-room and which was blocked exclusively for the whole project duration. Six long-term testing devices have been designed and produced by the workshop of INM. For the analysis of the materials properties developed and used within the project, the following devices were used, around 200 hours each by the project team: Digital Microscope, Scanning Electron Microscope (SEM), Tensile Tester, Dynamic Mechanical Thermal Analysis (DMTA), Photo-Differential Scanning Calorimetry (Photo-DSC), and Contact-Angle Goniometry. For investigating the adhesive properties like pull-off strength of the produced microstructures, several home built adhesion tester were used for hundreds of hours. Additional analyses like Gas-Chromatography coupled with Mass-Spectrometry (GCMS) were conducted by the service group "Chemical Analytics". Despite that, both program divisions involved (FM and IC) contributed with additional personnel and laboratory space. The administration of INM took over everything related to purchasing, accounting, controlling and Human Resources.

7. Strukturen und Kooperation

This project was a single institution project with no further cooperation partners from other institutions. Within the INM, the project was conducted by the Functional Microstructures Group in close collaboration with the InnovationCenter of INM. During the project, no change in any kind of cooperation was made.

8. Ausblick

The project has been very succesful, especially in improving and scaling-up the production of bioinspired adhesives by a Roll-To-Roll Process. Additionally, the endurance of the bioinspired adhesives was drastically increased from envisaged 100,000 cycles to more than 1,300,000 cycles at the projects end, making this adhesives suitable for industrial use. As a direct outcome, a company named INNOCISE GmbH was founded, which has the aim to especially commercialize bioinspired adhesives for robotics, for use in vacuum, and for pick & place of micron-sized objects.
Besides that, during the project it turned out, that functionalized bioinspired adhesives (e.g. electrical or thermal conductivity, optical properties, …) could have a huge impact in many different fields. This can vary from sensing applications to improvement of production processes by enabling thermal management in vacuum. Therefore, modelling of those functionalized bioinspired adhesives, developing the necessary new materials and composites, and finally producing and testing those structures will now be in focus.