# Quantitative assessment of real contact area on complex topographies and its role in attachment-detachment mechanisms

C. Kumar<sup>1,2,3,4 a</sup>, T. Speck<sup>2,3</sup> and V. Le Houérou<sup>1,4</sup>

 <sup>1</sup>Institut Charles Sadron, CNRS UPR022, University of Strasbourg, Strasbourg, France
<sup>2</sup>Plant Biomechanics Group and Botanic Garden, University of Freiburg, Freiburg, Germany
<sup>3</sup>Excellence Cluster livMatS @ FIT Freiburg Center for Interactive Materials and Bio-Inspired Technologies, Germany

<sup>4</sup>Laboratoire ICube, CNRS UMR7357, University of Strasbourg, Illkirch, France

<sup>a</sup>email: <u>Charchit.Kumar@glasgow.ac.uk</u>

**Abstract.** Real contact area plays a crucial role in adhesive characteristics of interacting surfaces. This contacting surface is largely affected by surface texturing. This work presents an original approach to quantitatively assess the formation of the real contact area while performing high-precision adhesion measurements on different polymeric surfaces decorated with complex hierarchical patterns. These complex patterns were fabricated on an optically transparent and soft elastomeric material, using a two-step bioreplication technique. A dynamic pull-off tester (modified nanoindenter system) was used to perform adhesion force measurements along with the visualization of real contact areas. Adhesion tests were performed to examine the effect of pre-load on adhesion force characteristics and to analyse topography dependent adhesion mechanisms.

## Introduction

The interfacial adhesive phenomena are met in numerous engineering systems as well as in natural ones [1]. Adhesive characteristics of interacting surfaces have a significant influence on the durability and efficiency of engineering systems, particularly of micro-contact systems with high surface to volume ratio [2]. Over the recent years, several research papers have been published for tuning the adhesive characteristics either by altering the surface physico-chemistry or by introducing micro- and/or nano-textures on the interacting surfaces. If we look into the nature, adhesion also plays an important role in the interaction of biological systems [3]. Inspiringly, the surface of plant leaves are decorated with species-specific surface texturing including textures with different size, shape and various levels of hierarchy [4]. Apart from diverse surface texturing, plant leaves take benefit from complex surface chemistry [5]. This combination leads to offer various noteworthy surface functionalities such as surface wettability, anti-adhesive properties, slippery surface for insect attachment, just to name a few [1,5]. Over the past decade, plant leaf surfaces gained a great interest in the mechanics community to develop bio-inspired functional interfaces [3]. To gain a detailed understanding of adhesion mechanics, attachment-detachment modes and distribution of real contact zones, in-situ imaging appears a promising approach. However, very high complexity of biological surface structures, small contact size and low force range (corresponding to insect-plant interactions) make it technically complicated. Altogether, this work utilises the classical adhesion mechanics approach to systematically explore the adhesion phenomena on highly complex biological topographies, which are forming contact with a model adhesive tip.

## Experimental

After an extensive morphological scanning of various plant leaves, three model plant leaves jewel orchid (*Ludisia discolor*), rubber tree (*Hevea brasiliensis*) and lychee (*Litchi chinensis*) were selected. These surfaces were chosen by considering their micro-structures size range (0.5  $\mu$ m to 100  $\mu$ m), complexity and hierarchical levels [4]. Using a two-step bio-replication technique [6], the surface textures of fresh plant leaves were replicated onto the polydimethylsiloxane (PDMS).



**Figure 1** (Left) Schematic of a typical pull-off test for adhesion force investigation. (Right) Simplified sketch exhibiting how the in-situ real contact visualization was achieved.  $F_L$  = Normal pre-load,  $F_{ad}$  = Pull-off adhesion force.

All surface morphological characterization of original leaf samples and replication quality evaluation of replicas were performed using scanning electron microscopy (qualitatively) and confocal laser scanning microscopy (quantitatively) techniques. Prior to adhesion measurements, all samples (replicas and smooth PDMS benchmark) were treated in of n-heptane and 1-dodecanethiol (0.01 %) solution to remove the free unreacted chains. To perform adhesion force investigations, a nano-indenter (UNHT<sup>3</sup>, Anton Paar Tritec, Switzerland) was modified into a JKR (Johnson, Kendall and Roberts) contact mechanics based pull-off force tester [7]. This test device was upgraded with a novel technique to access and record the real-contact visualization, based on the principle of transmitted light microscopy (Figure 1).

#### Results

High-precision PDMS replicas were successfully fabricated directly from the original plant leaves [6]. Pull-off adhesion force tests were performed on each PDMS replica and a smooth PMDS sample, by forming contact with a model adhesive tip. A representative force-time curve obtained for smooth PDMS sample, at a pre-load of 1.5 mN is shown in Figure 2. The pull-off adhesive force (F<sub>ad</sub>) is estimated as the maximum negative force value during the unloading cycle. An originally developed visualization system efficiently recorded real contact zones with a resolution down to sub-micron sized single cuticular fold level [1]. For smooth PDMS sample, pull-off force was recorded 0.77 mN, which is the highest out of all four surfaces investigated. However, *L. discolour* replica sample (coarse sized micro-structures) and *L. chinensis* replica surface (complex hierarchical micro-structures with overhanging sub-structures) significantly lowered the adhesion force.



**Figure 2** Representative force-time (displacement) curve recorded for a typical pull-off adhesion test on a smooth PDMS sample, at a pre-load ( $F_L$ ) of 1.5 mN.

Fine wrinkled micro-textures on *H. brasiliensis* replica showed the highest adhesion force ( $F_{ad} = 0.45 \text{ mN}$ ) of all bio-based samples, but lower than the smooth PDMS surface. Interestingly, *H. brasiliensis* replicas exhibited pre-load sensitive adhesive force behaviour, which could be explained (thanks to in-situ real contact videos) with the filling up of the fine pockets at high-load conditions [1]. Furthermore, recorded in-situ real contact videos confirmed that the distribution of real contact zones highly depends on the size and morphologies of surface textures. Detailed analysis of real-time force-data-point & synchronized contact-image disclosed distinct surface-specific attachment and detachment mechanisms. Results are presented and discussed along with the real contact videos.

#### References

- C. Kumar, D. Favier, T. Speck and V. Le Houérou, In situ investigation of adhesion mechanisms on complex microstructured biological surfaces, Advanced Materials Interfaces. 7 (2020) 2000969.
- [2] B. Bhushan, Springer handbook of nanotechnology, Springer Science & Business Media, 2010.
- [3] K. Koch, B. Bhushan and W. Barthlott, Multifunctional surface structures of plants: an inspiration for biomimetics, Progress in Materials Science. 54 (2009) 137–178.
- [4] C. Kumar, A. Palacios, V.A. Surapaneni, G. Bold, M. Thielen, E. Licht, T.E. Higham, T. Speck and V. Le Houérou, Replicating the complexity of natural surfaces: technique validation and applications for biomimetics, ecology and evolution, Philosophical Transactions of the Royal Society A. 377 (2019) 20180265.
- [5] B. Prüm, H.F. Bohn, R. Seidel, S. Rubach and T. Speck, Plant surfaces with cuticular folds and their replicas: influence of microstructuring and surface chemistry on the attachment of a leaf beetle, Acta Biomaterialia. 9 (2013) 6360–6368.
- [6] C. Kumar, V. Le Houérou, T. Speck and H.F. Bohn, Straightforward and precise approach to replicate complex hierarchical structures from plant surfaces onto soft matter polymer, Royal Society Open Science. 5 (2018) 172132.
- [7] K. Johnson, K. Kendall and A. Roberts, Surface energy and the contact of elastic solids, Proceedings of the Royal Society of London. A. Mathematical and Physical Sciences. 324 (1971) 301–313.