Microfabricated dry adhesive displaying frictional adhesion

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Gecko adhesion has become a paradigmatic example of bio-inspired engineering, yet among the many geckolike synthetic adhesives (GSAs), truly geckolike performance has been elusive. We have developed a new GSA, MicroWedges, which for the first time demonstrates the gecko's property of 'dynamic adhesion'– MicroWedge fibers independently detach and reattach during sliding, providing high levels of shear and normal adhesion during drag. This behavior provides a non-catastrophic failure mechanism crucial for high risk applications like climbing robots and devices for human climbing. MicroWedges also exhibit the geckolike properties of directionality, zero force at detachment, a high μ', and massive reusability. Samples were fabricated and tested at two characteristic fiber diameters, 50 μm and 20 μm, and from three silicone based elastomers of varying Shore hardness. Large patches of 1-2 cm^2 showed adhesive pressures greater than 5.0 kPa while simultaneously supporting more than 17 kPa of shear. Lifetime tests performed on MicroWedges exceed all previously reported results by more than two orders of magnitude. After 30,000 trials and over 300 m of drag, MicroWedges retained more than 56% of their initial adhesive pressure and 85% of their shear. No deterioration of dynamic adhesion properties was observed over this period. MicroWedges were manufactured using a molding process where molds were fabricated using a dual-side, dual-angle lithography process on quartz wafers with SU-8 photoresist as the mold material.

dry adhesion | fibrillar adhesive | gecko

Introduction

Biomimetic materials has emerged as a major research field in recent years, where one of the most heavily investigated areas has been gecko-like adhesives. The gecko inspires this degree of attention because of its incredible ability to use a very stiff material, β-keratin (E$\sim$1.6GPA)[3], to create something soft and tacky. Seven major functional properties of the gecko adhesive were identified in 2005. The gecko adhesive is 1) directional 2) attaches strongly with minimal preload 3) detaches quickly and easily, 4) sticks to nearly every material, 5) does not stay dirty or 6) self-adhere, and 7) is nonsticky by default [2]. We believe another critical attribute of the gecko's adhesive is missing from this list: the ability to engage and sustain high levels of adhesion while sliding. Because gecko setae and spatulae are able to independently engage and disengage, a consistent level of friction and adhesion is maintained as samples are dragged in their preferred direction across surfaces[11]. The gecko employs this 'dynamic adhesion' to help engage and load the many terminal spatulae evenly. Dynamic adhesion also provides a non-catastrophic failure mechanism during climbing.

Synthetic gecko adhesives have many potential applications in biomedical devices, manufacturing, consumer products, and, of course, for climbing applications and have therefore garnered the attention of many researchers. The first efforts used polyimide pillars created by electron beam lithography[13]. Carbon nanotubes have also been used as terminal elements[17, 18, 7, 1]. These approaches exploit the contact splitting aspect of the gecko adhesive, and while these two approaches demonstrate impressive amounts of adhesion, they are not directional and have not been scaled up to large areas. Another approach has created a micron scale silicon structure with nanometer scale features made of photoreseist on top[19]. This adhesive demonstrated the hierarchy feature of the gecko adhesive but was also not directional. The group also created structures capable of switching to replicate the gecko's ability to control its adhesive[20]. Up to this point, all structures had been fabricated at right angles to the substrate. We were inspired by work unrelated to adhesion that used multiple angle exposures of SU-8 to create interesting three dimensional structures[24, 22, 23]. A recent published result addressed directionality by fabricating fibers at an oblique angle to the substrate[4]. While these angled pillars lowered the effective stiffness of the sample, no attention was given to the tip shape of the fibers, which at length scales on the order of μm can have a dramatic effect on the amount of adhesion generated[5]. Other non-directional synthetics that have more carefully designed tip shapes have achieved much better performance[25, 21]. One adhesive combines both directionality and attention to tip shape[26, 6] and performed very well on a climbing robot[16], however this result was fabricated at a length scale (d = 380 μm) much larger than the gecko adhesive. Other results suggest that smaller features and features with a lower modulus of elasticity should increase adhesion[8, 9]. Recent work has also shown increased adhesion on both wet and dry substrates through the use of surface coatings[15]. Although many of these synthetics attain some of the attributes of the gecko adhesive system, a structure that replicates all of the gecko system's properties and its subsequent utility as an adhesive remains a holy grail of sorts in the field.

Here, we present a significant step towards that goal in MicroWedges, a new microstructured adhesive that demonstrates dynamic adhesion in a synthetic gecko adhesive for the first time. MicroWedges retain nearly 100% of their adhesion during sliding. Additionally, MicroWedges follow the directional adhesion model observed in the gecko[11], which means that adhesion increases as shear force is induced in the sample along a preferred direction. Another result of this, is that when the shear loading is removed, MicroWedges can be detached with virtually zero force. MicroWedges sharp tip shape helps engage the adhesive at low attachment forces and generates normal adhesive pressures greater than 5.0 kPa. MicroWedges also set a new standard for reusability, retaining 56% of their initial adhesion after a battery of 30,000 test cycles.

Large arrays of one to two cm^2 of MicroWedges were fabricated and tested at two characteristic stalk diameters, 20 μm and 50 μm. MicroWedges were also fabricated out of three different materials of varying bulk properties in order to begin elucidating the relationships between stiffness, adhesion, clumping, and self cleaning.

Conflict of interest footnote placeholder

Insert 'This paper was submitted directly to the PNAS office.' when applicable.

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Design and Fabrication

Microwedges were designed to exhibit strong adhesion while under shear loading in a preferred direction and negligible adhesion under zero shear load, like the gecko's adhesive system[11]. This capability makes an adhesive particularly well suited to climbing vertical surfaces, where the body weight of an animal or robot induces a large downward shear loading on the contacts and where feet must be detached without disturbing the overall purchase of the remaining legs. Taking inspiration from Directional Polymer Stalks [26, 6], the wedge shape was chosen as a viable anisotropic geometry with a sharp tip shape that would promote high levels of adhesion. A potential fabrication process was also designed based on innovative lithography and molding techniques. Using thick films of SU-8, arrays of wedge shaped cavities were created as a reusable mold into which soft, elastomeric polymers were poured and released.

To create the SU-8 mold, a 2 mask angled exposure technique was developed where an angled backside exposure was sequentially followed by a vertical topside exposure before development to create a 3D structure in the film. After several early failures, care was taken in the final mask designs to reduce volumes in the film that would be unmasked during both exposures. A custom built tilting stage was used to orient the wafer at a repeatable and precise angle under a collimated UV lamp for the angled exposure. This angle was subject to a correction factor to account for the refraction effect due to SU-8's index of refraction (n=1.61) [27]. For the structures presented here, the target angle was 14.8° which required an exposure angle of 23.5°. After the molds were created, various silicone based elastomers were cast under vacuum, spun to a desired backing layer thickness, heat cured, and pulled out of the mold by hand. Molds typically exhibited reusability for over 10 cast/peel cycles. The fabrication sequence is shown in Figure 1 and SEM images of the final structures can be seen in Figure 2.

This process yielded very good results for MicroWedges at both 50 μm and 20 μm diameters. A 4:1 aspect ratio was maintained across molds and three separate polymers (SiliconesInc P-70, Dow Corning Sylgard 170, SiliconesInc P20) were used extensively at both sizes to create a total of 6 sample types for testing.

Results

Data from a single load-drag-pull (LDP) test of a 1 cm^2 patch of 50 μm stalks made from Sylgard 170 is shown in Figure 3 in a standard force vs time representation. In this test, the preload consisted of a 45° angled trajectory to a depth of 80 μm. The most striking phenomenon observed is the continuous adhesion during the 1 mm drag phase between points 2 and 3. This data indicates that stalks are independently detaching and reattaching, a property previously unreported for any synthetic fibrillar adhesive. Previous results would, in contrast, pass through point 2 and immediately drop down to near zero adhesion. The gecko also exhibits adhesion while sliding, which we term 'dynamic adhesion'. This can be seen in Figure 4 which shows LDP data for a gecko setae. Dynamic adhesion is a very significant result for MicroWedges not only because it more accurately mimics the gecko, but because the prominent failure mode is no longer a catastrophic detachment event, but a slow and predictable slip. The implications of this are discussed further in the following section, Discussion. Figure 3 is representative of data seen for all 6 sample types tested, although overall adhesion and shear values varied somewhat between sizes and materials as reported below.

Similar to data from harvested gecko setae and whole gecko toes, MicroWedges follow the frictional adhesion model[11] by demonstrating more adhesion when loaded in a preferred shear direction. Data was collected for the MicroWedges by running a series of load-pulloff (LP) tests where a preload of 45° was performed at different depths ranging from 0% to 80% of the stalks height. This was followed immediately by a pulloff trajectory at a specified angle that ranged from 0° to 180°. The shear and normal forces were recorded at the failure event, which was determined to be the point that the adhesive either popped off or began sliding. These shear and normal forces are plotted against one another in Figure 5 to create a limit surface inside of which contact will be maintained. The shape of this plot shows the directional behavior of MicroWedges; maximum adhesion is reached only when a large value of shear is present. Additionally, because the limit surface intersects the origin, when no shear is applied to a sample, MicroWedges detach without any pulloff forces. This property allows Microwedges to act as a switchable adhesive, effectively controlled by the amount of shear loading imposed upon a given patch. The Microwedge data presented shows some bidirectional behavior. This is attributable to the thin tip at the end of the wedge, which is capable of flopping over in the anti-preferred direction and creating a significant area of contact when loaded in such a way. However, the desirable functions of directionality are attained, 1) adhesion increases with shear loading and 2) the limit surface passes through the origin. It is these two properties that the gecko relies on to activate and release the adhesion of its feet while climbing.

For contrast, flat control patches were also tested with the same trajectories and are shown in Figure 6. This data fits imbedded friction cone behavior, which is isotropic and exhibits the strongest adhesion under no shear loading.

The independent control displacement axes on the Stanford stage allowed for easy optimization of the preload and pulloff angles. The strongest adhesion was generated for preload angles near 30° off horizontal and pulloff angles between 120° and 150°. This data is useful for designing robot gaits and trajectories during MicroWedges arrays in useful systems.

Microwedges were fabricated at two characteristic stalk diameters, 50 μm and 20 μm, both at a 25% fill factor in a square pattern. Assuming a similar loading trajectory scaled to the sample size, the array of 20 μm features have the same real area of contact, but with a 2.5× increase in the summed perimeter of contacting elements. Results for similar patches fabricated from Sylgard 170 at these two characteristic size scales with identical backing layers are plotted in a force space representation below in Figure 7. It has been reasoned that contact splitting will increase overall adhesion in a vertical preload/vertical pulloff experiment[8], and although similar behavior is observed in Figure 7 for directional tests, this was not the case across all materials tested and results varied based on the backing thickness of samples as well. Further research on a broader variety of samples is needed for conclusive comparisons on feature size, backing thickness, and overall levels of adhesion. However, the general directional behavior of the stalks, their ability to adhere dynamically, and the shape of the limit surface did remain consistent at both characteristic sizes across all trials.

In total, six sample types were fabricated and tested extensively, 50 μm and 20 μm diameter wedges of 3 material types: Silicones Inc. P-20, Dow Corning Sylgard 170, and Silicones Inc. P-70. Bulk material properties for these elastomers were measured using flat control samples and macroscale cubes of material. The Young's Moduli for the materials were measured as 340kPa, 760kPa, and 930kPa for P-20, Sylgard 170, and P-70 respectively. It was expected that the softest material, P-20, would show the strongest levels of adhesion, but would be susceptible to dirt and fouling. Although samples were successfully fabricated and tested using this soft polymer, the material proved to be too sticky and widespread clumping of wedges was observed in both the 50 μm and 20 μm arrays of stalks, resulting in significantly reduced performance. Arrays of Sylgard 170 and P-70 cast from the same mold did not clump extensively, but performed similarly to one another at both size scales. This led to the recognition of a need to test more drastically different materials if a quantitative empirical relationship between effective stiffness and adhesion is to be achieved.

There has been little data presented on the longevity of synthetic fibrillar adhesives in the literature to this point, and where it is present, it is almost always seen that performance has dropped significantly after only a few tests because structures have broken or become dirty[13, 17, 18, 19, **?**]. The exception to this, Directional Polymer Stalks[26, 6] and The Max Planck Institute's mushroom shaped adhesive[25], claim reusability over 10s of attachment/detachment cycles before performance degraded and cleaning was required. After this cleaning, the samples regained their initial performance. Figure 8 shows data collected for a 1 cm^2 patch of Sylgard 170 over a battery of 30,000 Load-Drag-Pull cycles. The drag phase of these tests was extended to 10 mm to increase the rate of wear on the stalks. A preload depth of 100 μm was used and the average shear and adhesion during the drag phase was taken as representative data points for each trial and plotted. It is clearly seen that MicroWedges are massively reusable without frequent cleanings, likely due to the stiffness of the material from which they are fabricated and their ability to detach and reattach without damaging the structure. The gecko's adhesive system is also massively reusable, as it must climb for long periods of time before new growth replaces the setae and terminal spatular elements of its toe pads.

Discussion

A major result of MicroWedges is their ability to dynamically adhere to surfaces while sliding. In climbing applications where the loss of adhesion could result in catastrophic consequences, the ability for a contacting foot to have a controlled, small failure allows an animal/robot time to compensate for the failure by reattaching the foot in another location or by placing more feet on the surface to secure its grip. Additionally, the sliding phenomenon in geckos helps to orient all of the terminal spatulae for good contact while not damaging individual contacts. A similar behavior would be desirable in future versions of synthetic fibrillar adhesives that are hierarchical. Dynamic adhesion would reduce the demands on the accuracy of foot presentation to the climbing surface, allowing stalks to slide into their preferred alignment rather than expecting the initial placement achieve the correct orientation for all of the individual fibers.

Currently, it is very difficult to utilize synthetic gecko adhesives in real world applications because without the highly controlled mounting and loading procedures of the experimental setups on which they are tested, patches fail to make good contact over the useful areas required for macroscopic adhesion. In fact, a common oversight in the literature has been the neglect of issues that arise when trying to scale up adhesion results from small arrays of fibers on the order of a few square mm or less (in some cases much less) to macroscopic areas of several square cm, like a Tokay gecko's toe. For microscopic features to truly imitate the gecko's adhesive system, they must have a mechanism that allows them to self-align and promote widespread intimate contact of their distal ends with a variety of surfaces that may vary in roughness on the order of the stalk size or even greater. If this is to be accomplished through a hierarchy as has been proposed, then it is essential that the terminal features are able to detach and reattach without damaging themselves or losing performance.

Climbing applications also require the ability to control adhesion. There exists many materials that provide adequate amounts of adhesion to support a gecko or a small robot (i.e. duct tape), but of these solutions, only materials that can switch on and off are useful because of the need to attach and detach feet without disturbing the overall motion of the robot. A robot climbing with non-directional adhesives would experience large pull off forces at detachment that could cause it to fall from the climbing surface. A climbing adhesive's limit surface must intersect the origin for rapid, stable climbing to be achieved.

While easy detachment of feet is crucial to climbing quickly and efficiently, the importance of easy attachment of an adhesive patch must not be overlooked. The gecko, utilizing a four-tiered hierarchy, exhibits a μ' of between 8 and 16 depending on conditions[10], which means that it can pull with 8 to 16 times the normal force with which it has preloaded its' feet. μ' values for synthetic fibrillar adhesives in the literature range from 0.02 to 7.5 [18, 25] For MicroWedges, the highest μ' observed was 2.0 when stalks were loaded at an incoming angle of 30°, preventing the buckling of fibers. Adhesion during this trial was 4.9 kPa, very near the maximum adhesive pressure observed. It is interesting to keep in mind that a four legged climbing robot has a theoretical minimum μ' of 0.33 because the attached feet must support the preload of the next step.

A high value of μ' can help a fibrillar adhesive patch's durability by preventing the damage of stalks during the loading procedure. In the case of MicroWedges, durability is also a result of the elastomeric materials used. While a gecko's adhesive system is massively reusable with a very stiff material, β-keratin (E $\sim$ 1.6GPa [3]), it only achieves this because of the multi-level hierarchy that distributes the load to individual spatulae. Spatulae also avoid overload damage by detaching and resticking as needed, a property also observed in MicroWedges. Without these attributes, it is not surprising that single level, stiff, non-sliding adhesives only show good levels of adhesion for a few tests before suffering irreparable damage. Based on the durability data for MicroWedges and assuming a robot stride length of 5 cm, a 1500 meter building could be scaled using MicroWedges before performance dropped below 50%. Current research is focussing on the development of a hierarchical system for MicroWedges to make this scenario a reality and move from the tightly controlled parameters of an experimental stage to climbing robots and mechanisms for human climbing. Work is also ongoing to manufacture MicroWedges at even smaller sizes and with more complicated geometries that could enhance their performance.

Testing

In order to investigate the directional and dynamic adhesion properties of our samples, we need to test our samples in both the normal and tangential direction. We collected data using two different experimental test setups. Each setup is capable of moving a flat glass substrate and sample into and out of contact along trajectories in both the normal and tangential direction simultaneously. One setup is the local test stand for the Biomimetics Dexterous Manipulation Laboratory (BDML) at Stanford University, Stanford, CA. The BDML setup consists of a 3-axis linear stage, force transducer and 2-axis manual tilt stage. The motion stage (Velmex, MAXY4009W2-S4 and MA2506B-S2.5) has a $\pm 10\mu$m positioning resolution in the tangential direction and a $\pm 1\mu$m positioning resolution in the normal direction. The flat glass substrate is attached to the motion stage while the sample is placed upon a 6-axis force/torque sensor (ATI Industrial Automation, Gamma Transducer SI-32-2.5). The force transducer is mounted onto a manual 2-axis tilt stage (Newport, 30 Series Tilt Platform) which is used for manual alignment of the sample relative to the substrate. The second setup is located at Lewis and Clark College, Portland, OR. The Lewis and Clark setup consists of a 2-axis linear stage, force transducer and 2-axis tilt stage. The motion stage is formed by two Aerotech ANT-50L (Aerotech, Pittsburgh, PA) linear actuators, which provide positioning control on the nanoscale, and the flat glass substrate is attached rigidly to the motion stage. The sample is mounted onto a Kistler9328A three-axis piezoelectric force sensor (Kistler, Winterthur, Switzerland). Two Newport goniometers, GON65L and TR120BL, (Newport, Irvine, CA) are used to align the sample and substrate.

A variety of different experiments were run on the test apparatuses. One such experiment is a "load-drag-pull" (LDP) [11]. A LDP experiment consists of a loading phase, then a drag phase and finally a pulloff phase. In both the loading and pulloff phase, the substrate moves along a trajectory at a predefined angle relative to the sample, and during drag phase the substrate moves a specified distance in the tangential direction. LDP experiments are used to explore directionality and dynamic characteristics of our adhesives, and the adhesion reported is the average adhesion of the dynamic adhesion region. The second experiment is a "load-pull" (LP). The LP experiment is analogous to the LDP experiment except there is no drag phase and adhesion is reported differently. LP experiments explore both the directionality and adhesion limit surface, as defined in [26]. The pulloff adhesion for LP experiments is measured as the maximum adhesion at the time of contact failure by either separation or sliding of the sample relative to the substrate. The third experiment was for longevity, a sample undergoes many cycles of the same LDP experiment and adhesion is reported for each cycle as the adhesion from the individual LDP experiment.

– text of acknowledgments here, including grant info –

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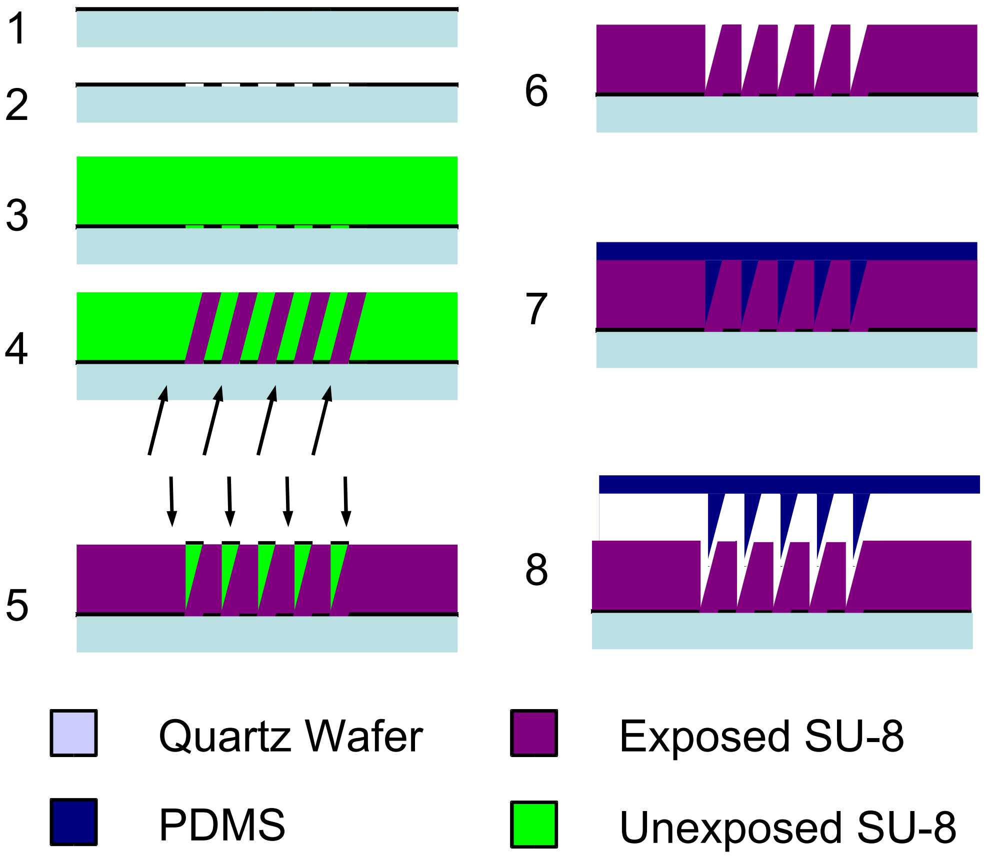

Fig. 1. Fabrication Sequence 1) Deposit aluminum on quartz wafer 2) Pattern aluminum to create self-aligned mask 3) Deposit SU-8 on top of aluminum 4) Angled self-aligned UV exposure from backside 5) Align mask to topside and UV expose 6) Develop 7) Cast and spin PDMS 8) Peel out cast microwedges and backing layer

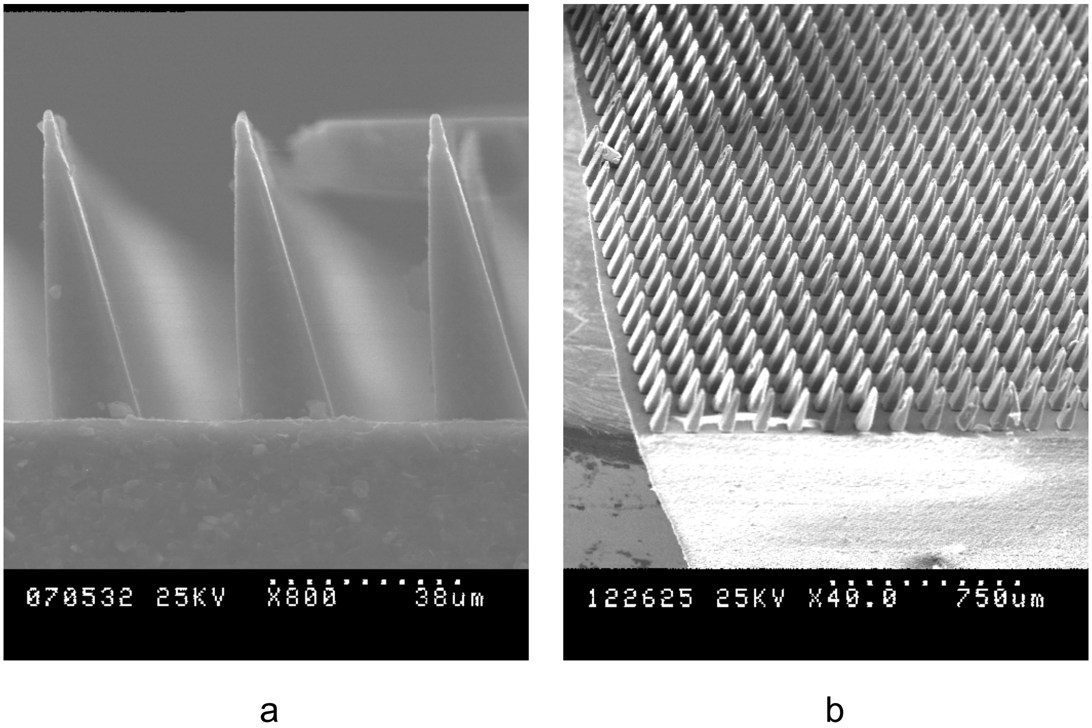

Fig. 2. SEM images of MicroWedges a) side view of 20 μm diameter MicroWedges b) diagonal view of large array of 50 μm diameter MicroWedges

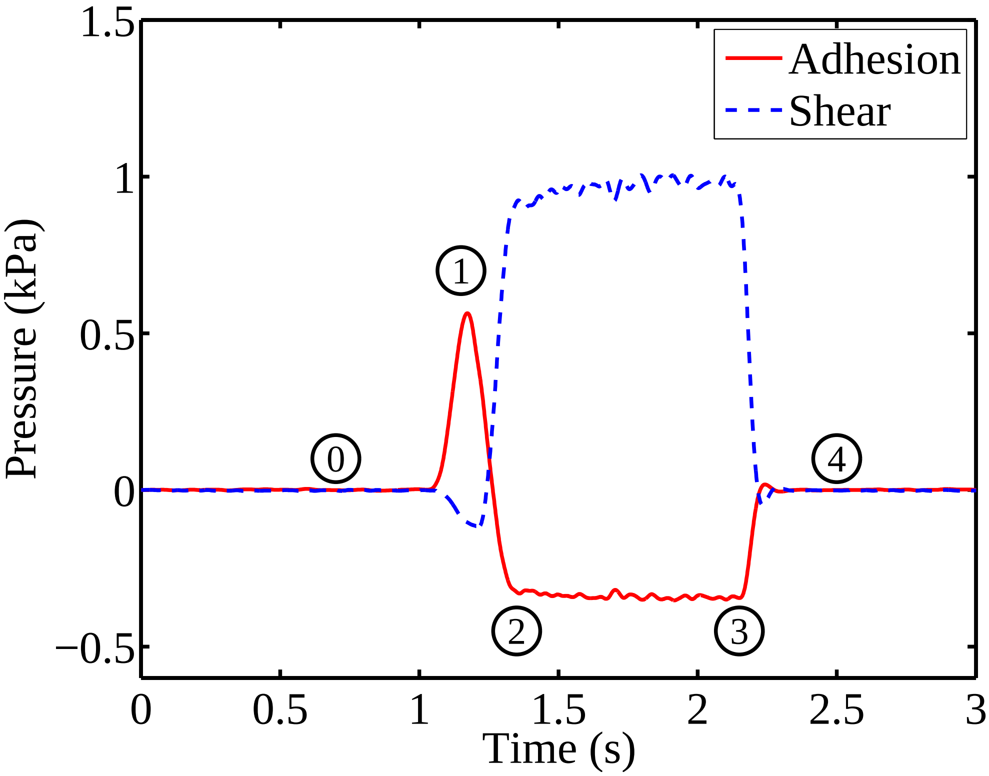

Fig. 3. MicroWedge load-drag-pull data from a single trial. Preload occurs from point 0 to 1 consisting of a 45 degree approach angle to a depth of 80 μm. This was followed by a 1 mm drag (points 2-3) and a vertical pulloff (points 3-4). The substrate moved at a constant 500 μm/s over the course of the trial. Notice the sustained dynamic adhesion and shear between points 2 and 3, indicating independent detachment and reattachment of single wedges.

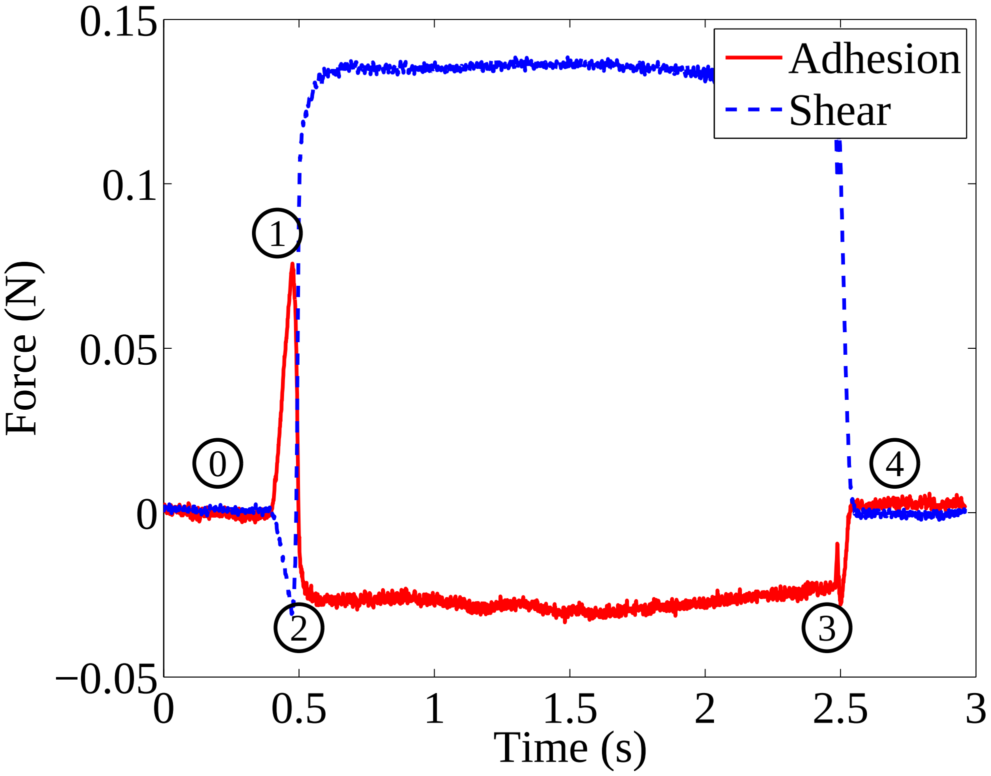

Fig. 4. Gecko setae load-drag-pull data from a single trial. Comparison with figure 3 shows the strong behavioral similarity between MicroWedges and gecko setae, especially the dynamic adhesion observed between points 2 and 3.

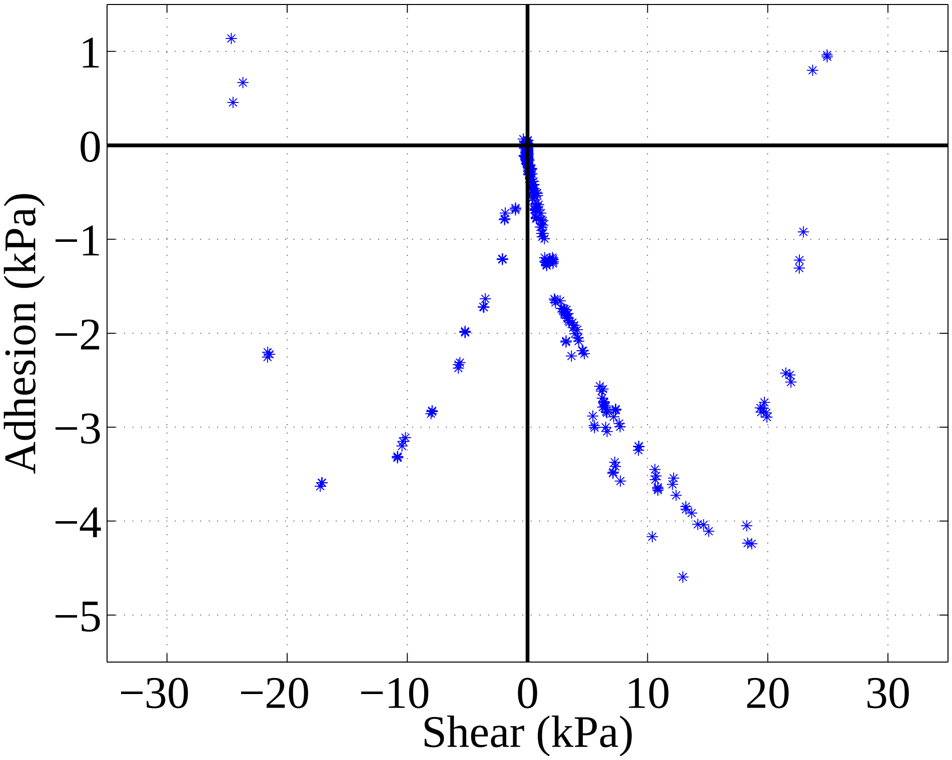

Fig. 5. Limit surface of a Sylgard 170 sample of 50 μm stalks on a 225 μm backing layer. Points indicate contact failures either through slipping or detachment from the surface. Important to note: adhesion is only achieved in the presence of shear loading, following the frictional adhesion model [11]. Also, the limit surface intersects the origin, indicating that when no shear is present, MicroWedges can be detached with zero force.

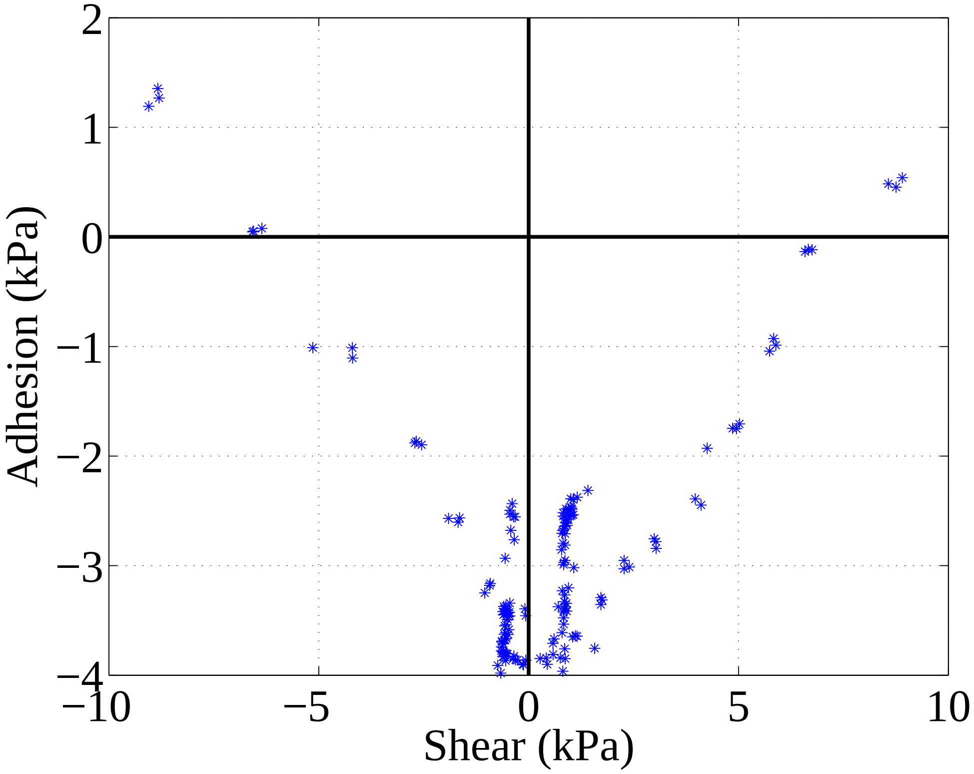

Fig. 6. Limit surface of a flat patch of Sylgard 170. This set of data follows the imbedded cone model [?] exhibiting the strongest adhesion when no shear loads are present.

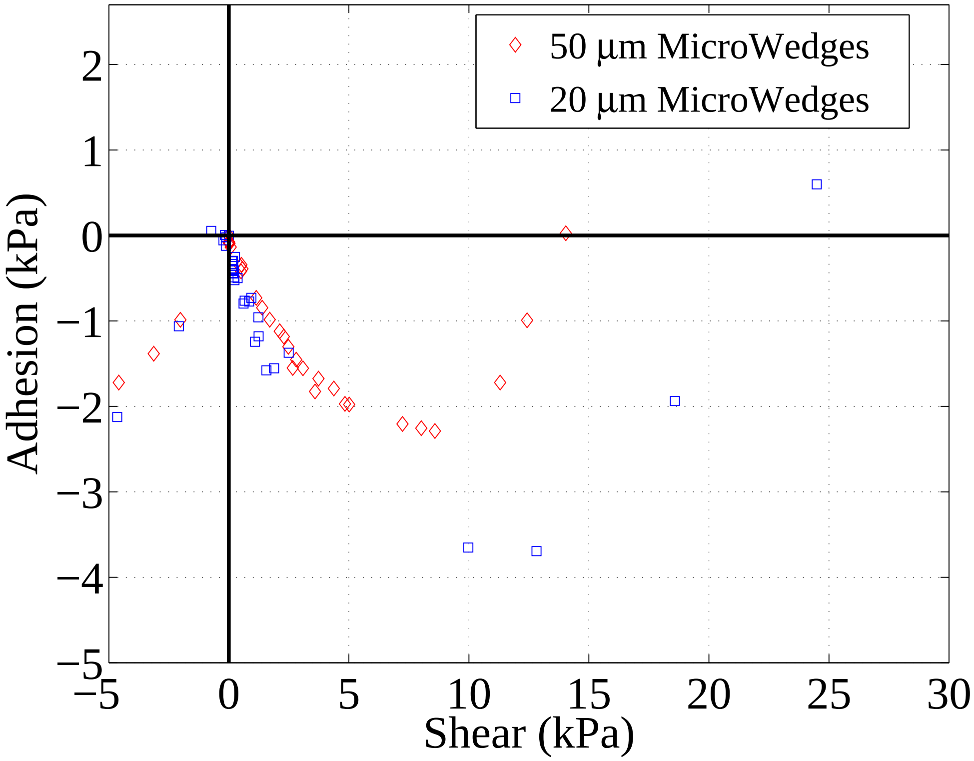

Fig. 7. Size Comparison Adhesion

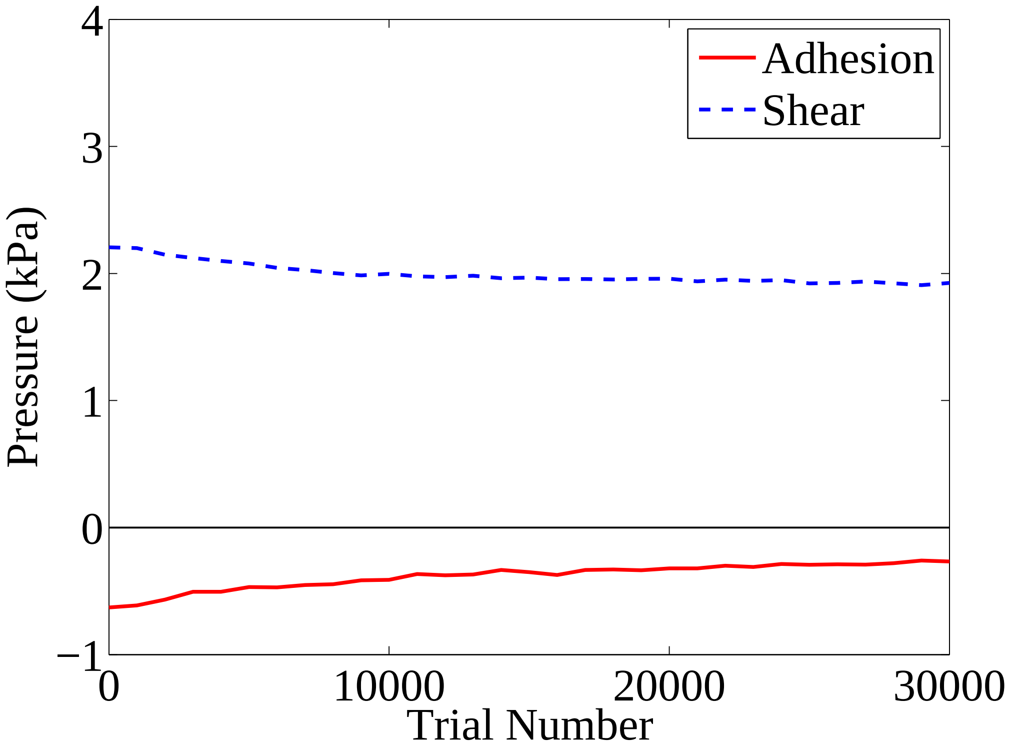

Fig. 8. Lifetime data for a single sample of 50 μm stalks made from Sylgard 170. MicroWedges are massively reusable, retaining over %50 of their adhesion after 30,000 trials. This is more than two orders of magnitude longer than any other published data on GSAs.