

Self-Cleaning and Controlled Adhesion of Gecko Feet and Their Bioinspired Micromanipulators

Yiyang Wan, Zhenhai Xia*

Department of Materials Science and Engineering, Department of Chemistry, University of North Texas, Denton, TX 76203, USA.

*email: Zhenhai.xia@unt.edu

ABSTRACT

Bioinspired micromanipulators have been made based on gecko dynamic self-cleaning mechanism. Various particles such as spherical SiO₂/polystyrene, and short fibrous glass can be captured, transmitted and dropped on glass substrate with precisely predesigned patterns, by using the micromanipulator with the help of atomic force microscope (AFM). It has been demonstrated that particle-pad interface and particle-substrate interface exhibit diverse adhesion behaviors under different z-piezo retracting speed. The particle-substrate adhesion increases faster than the particle-pad adhesion with increasing the detaching velocity, which makes it possible to manipulate the particles by adjusting the retreating speed only. Probability tests was performed to better choose suitable parameters for picking and dropping operations. This work provides a potential solution to manipulation of micro/nano particles for precise assembly.

INTRODUCTION

Geckos have special ability to walk on various kinds of surfaces, indicating that their feet are of high adhesion. It was reported that gecko generate adhesion making use of van der Waals force.^{1,2} What makes it more interesting is the self-cleaning properties of gecko setae,³ which means gecko seta adhesion is well controllable: they generate high adhesion but do not adhere to dirty particles. Inspired by the remarkable ability of gecko, some groups fabricated seta-like structures for adhesive surface.^{4,5} This surface is able to keep sticky on dusty substrates. In our previous work, we have discovered a unique mechanism for gecko self-cleaning: the adhesion is significantly affected by retracting velocity.⁶ When retracting speed increases, the adhesive force between particle and substrate is enhanced dramatically, providing us potential way to manipulate by changing pull-off velocity. A micro-manipulator was made with polyester fiber and graphene gluing at the end. Due to high adhesive force observed on the graphene⁷, similar adjustable stickiness was achieved using the graphene material. However, the detailed mechanism is still unclear for the self-cleaning and manipulation.

In this paper, the effect on the manipulator, including dynamic, shear movement and geometry of fiber/cantilever, will be further investigated. More parameters were considered to better control the adhesive force, to achieve more accurate manipulation of microparticles. SiO₂, Polystyrene microsphere and microfiber segments were tested on glass and sapphire substrate, in normal dry atmosphere. Pull-off velocity was acting as a primary factor to control adhesive force, while shearing was used to enhance both pick-up and drop-off processes. Probability tests under different parameters performed to access the ability of our micro-manipulator. Direct force measurement explains the relationship between particle-substrate interface and particle-graphene

interface. We found that unlike the gecko seta, which holds almost stable adhesive force under different retracting speed,⁶ graphene layers are affected by normal speed, leading to an increasing adhesive force when the speed is growing. Graphene is durable in the manipulation.

EXPERIMENT

Polystyrene (PS) and SiO₂ microspheres (C-PS-10.0 and C-SIO-10.0 Microsphere-Nanosphere Inc.) with diameter of about 10 μm were cleaned in DI water with ultrasonic cleaner for 15 min, at room temperature. Fused silica (FS) substrate, sapphire substrate (Bruker Co., Inc.) and glass slides with fine and course surfaces (12-544-1, Fisher Scientific Co., Inc.) were cleaned with DI water and Ethanol (459844, Sigma-aldrich, Co.). Polystyrene(PS) substrate was cleaned with DI water only and dried for 15 min as well. Then they were dried for 24 hours in room temperature. Epoxy glue (30 min Slow-Cure Epoxy, Kite Studio Co., Inc.) was dispersed onto a glass slide, and PS and SiO₂ microspheres were glue on the glass slide.

Tipless cantilevers with length 125 μm (ACTA-TL, AppNano) were attached to atomic force microscope (Scan-Icon AFM, Bruker Co., Inc.). Polyester fibers (diameter: 8-10 μm) were cut with scissors. There will be a naturally formed pad structure at the end of fiber segments due to the plastic deformation during cutting process. Then the fiber segments (length 120-140 μm) were selected and glued to AFM cantilevers. Monolayer graphene synthesized by chemical vapor deposition (CVD)⁸ was cut into small pieces in microscope. Suitable pieces (10-20 μm in diameter) were selected and glued to the pad of the microfibers using the epoxy. The synthetical fiber with graphene are shown in Figure 1.

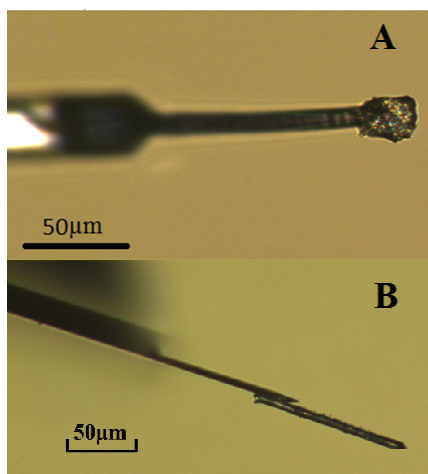


Figure 1. Microscope Images of 140 μm polyester fiber stick on 125 μm cantilever (A)Front view, and (B) Side View

Mimicking gecko toe movement, forward-shear-retract was performed using AFM with adjustable parameters, including retraction speed, shearing distance and speed, contact time and preload. The AFM chamber atmosphere was maintained humidity $\sim 30\%$ and temperature $\sim 25^\circ\text{C}$. Both motor and piezo were used to control the pull-off speed. The z-piezo has a height limit of about 13.4 μm . Contact time was set as 5 seconds except in contact time probability test. A shear movement, 2 μm in length with shearing speed 4 $\mu\text{m/s}$, was applied in probability tests unless specified. Microparticles were transferred to clean substrates before tests in order to eliminate

effect of wetting bond. In force measurement each data point represented an average of at least 8 loads. There are three important forces: (i) graphene-substrate adhesion force; (ii) sphere-substrate adhesion force; and (iii) graphene-sphere adhesion force, tested in this experiment as shown in Fig.2. During probability tests, SiO₂ microspheres were selected randomly and 30 trials pick-up on glass for each set of parameters were tested; if microspheres could be picked up, drop-off tests would be performed at various pull-off speed/preload until the particle was removed from graphene.

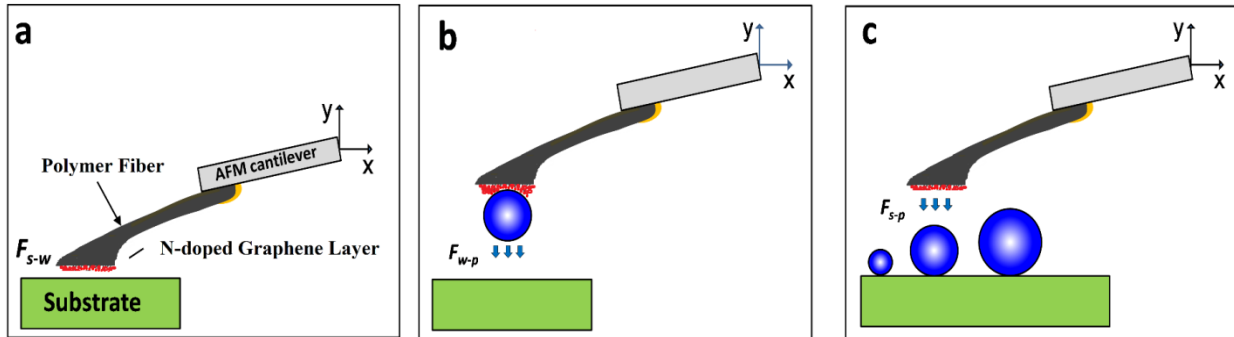


Figure 2. Illustration of force measurement in this experiment (A) graphene-substrate adhesion; (B) sphere-substrate adhesion; (C) graphene-sphere adhesion

RESULTS

We examined the dynamic adhesion on different substrates. The graphene pad bonded at the end of microfiber, which was pre-glued on AFM cantilever, was engaged on the surface of substrates, and kept for a contact time of 5 seconds, followed by a normal retraction with normal velocity of V_n . The cantilever deformation was recorded by AFM. By changing V_n , we could observe the dynamic effect on graphene adhesion. As shown in Figure 3A, the adhesive force F_{s-w} between substrates and graphene pad is obviously speed-dependent. Unlike gecko seta array performance, which shows almost speed-independent adhesion,⁶ the adhesion F_{s-w} increased with increasing retracting velocity V_n .

For graphene-microsphere interface, we tried to conform if the similar phenomenon would happen. SiO₂ microspheres were pre-glued to glass slides/graphene. The same graphene pad above was firstly loaded randomly to microspheres, contacting for 5 seconds, and then was withdrew with a normal velocity of V_n . The adhesive force F_{s-p} between graphene and particle was measured in the same way. As shown in Figure 3B, the results followed the same trend as mentioned previously: the adhesive force increased with increasing pull-off velocity. As for the adhesive force F_{w-p} between graphene and substrate, it slightly increased but the amplitude is much less than that of F_{s-p} .

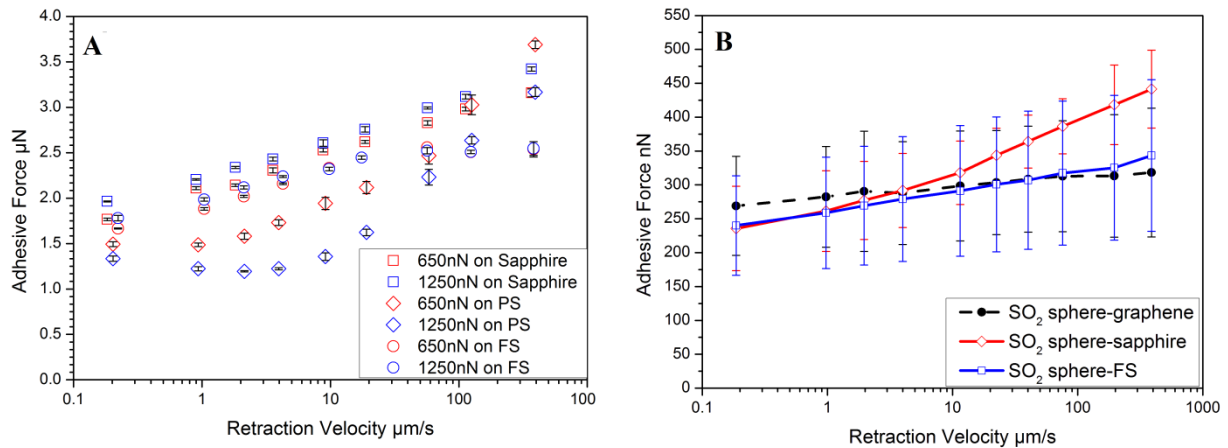


Figure 3. (A) Adhesive force between graphene and substrates with preload 650/1250 nN, and (B) Adhesive force between different samples and microspheres with preload 650/1250 nN

To access the manipulating ability of the bioinspired micromanipulator, probability tests were performed on 30-trial events for SiO_2 microsphere pick/drop. The forward-shear-retract process was performed in the probability tests. Shearing velocity and distance were firstly set $4 \mu\text{m/s}$ and $2 \mu\text{m}$, respectively. Shearing direction was left or right. Because the particles were spherical and the graphene pad was 2D flat, the adhesive force was in little regard to shearing direction. Pulling speed was changed from $\sim 0.2 \mu\text{m/s}$ to $\sim 400 \mu\text{m/s}$. As seen in Figure 4A, the pick-up is more likely to happen in low-speed-high-preload region, while drop-off prefers high-speed-low-preload. Overall, pick-up performance is better than drop-off performance in our speed range. Because higher preloads lead to better surface match between graphene and micro-particles and graphene adhesion is bravely based on contact area, higher pick-up rate was observed in high force region. Nevertheless, too high a preload may introduce another phenomenon: microsphere was extruded from graphene pad area, which could be considered as a new method of dropping or self-cleaning mechanism. With this micromanipulator, we were able to pick, transmit and drop SiO_2 microspheres to make pattern on glass substrates, as shown in Figure 4B.

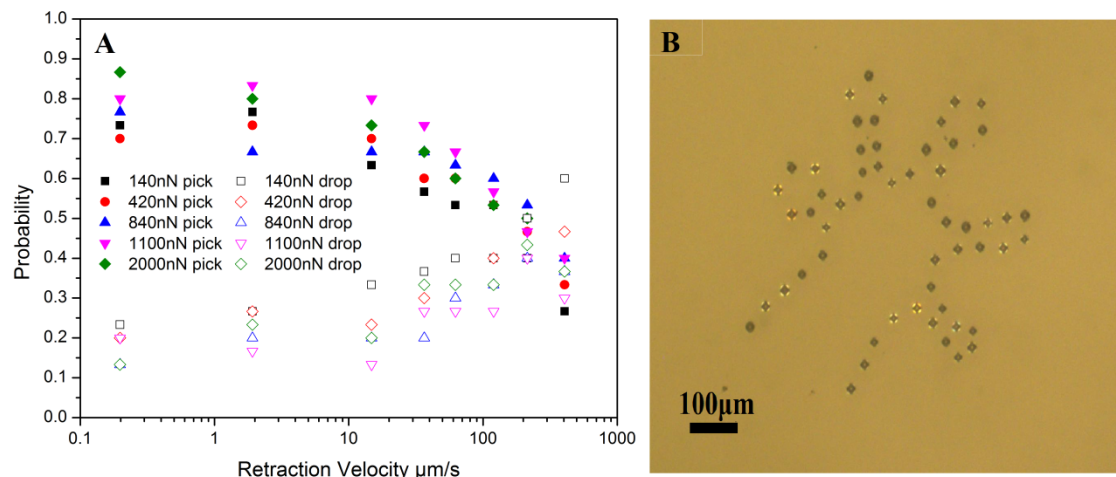


Figure 4. (A) Functional probability v.s. retraction velocity on picking/dropping SiO₂ microspheres at various preload force, and (B) Artistic pattern made with SiO₂ beams micromanipulation

DISCUSSION

This manipulator was designed to prove dynamic effect on gecko-inspired self-cleaning⁶. They have similar adhesion mechanism based on van der Waals force. The gecko seta holds stable adhesive force while graphene cannot get rid of pull-off speed effect. We believe this difference derives nanostructure of contact interface: gecko seta branch into 3D nanofibers⁹ while graphene has soft winkle 2D surface. On the other hand, 2D structure has an advantage that adhesive force doesn't change with lateral directions which makes it easier to operate micro-particles with irregular shapes, such as fibrous micro segments, because we can freely change the shear direction. The primary thought of this manipulator is to tune the adhesive forces in two interfaces: particle-graphene F_{w-p} and particle-substrate F_{s-p} . When $F_{w-p} > F_{s-p}$ achieved at small normal velocity V_n , particles are picked up from the substrate; On the contrary when $F_{w-p} < F_{s-p}$ at high normal velocity, particles are dropped off from graphene. In this paper we have tested three different manipulator tips, all of which shared the same trend when pull-off speed was changing. Probability tests on picking/dropping SiO₂ particles support our conclusion: pick-up succeeds more at low velocity than at high velocity while drop-off stands on the contrast. The reason why we could not achieve 100% picking/dropping is that all adhesive forces vary in a wide range. For example, the fiber sample was still able to make 40% picking on fused silica at $\sim 400 \mu\text{m/s}$, because F_{w-p} and F_{s-p} overlapped in a huge degree. Even if F_{s-p} is much larger on sapphire substrate, we still had a little chance to pick SiO₂ with graphene.

The fiber glued sample had better performance than non-fiber samples during probability tests, which gives us the thought why gecko spatula grows at the end of fibrous seta instead of at toes directly. In geometry fiber-graphene structure has another advantage that it can operate smaller targets with little interface from nearby particles. Preload was tuned to improve both picking and dropping rates. High preload makes it possible to extrude microspheres during lateral movement. Shearing is complicated but essential for overall performance. For gecko seta, shearing only contribute to self-cleaning or dropping off dusts⁹, which is not the case for graphene-based manipulators. Lateral movement is a favorable behavior in both picking and dropping microspheres. The reason why shearing matters is that it changes surface structure of graphene and thus changes contact area. At the same time, wetting effect which may lead to strong adhesion at high humidity is dismissed. What's more, high-speed-long-distance shearing provides another method to drop particle back, which also proves shearing mechanism in gecko self-cleaning, as a byproduct. Though shear direction does not show significant effect on spherical targets, it was not the same case to fibers or complicated structures. Shearing in radial direction was in connection with rolling friction while shearing in longitudinal direction referred to sliding friction. They made different contribution during adhesion crack propagation. Due to outstanding mechanical properties of graphene, durability of the manipulator is confirmed. In case it is damaged, fast repairing is achievable by gluing a new layer graphene. It should be noticed that preload can be as low as $\sim 140 \text{ nN}$, meaning ignorable hurt on target. Collaborated with other technology, it is a potential solution to manipulate soft materials, including bio-materials.

CONCLUSION

Bioinspired micromanipulators have been made using synthetic fiber and graphene. The adhesive forces of the micromanipulator were measured to understand the adhesive and manipulating mechanism. It was found that there was a dynamic effect of this manipulator. The adhesive forces increase with increasing the retreating speed of the manipulator, but the change rate between particle and manipulator and that between particle and substrate is different. The particles can be picked at low speed and dropped off at high speed on demand. This work provides a novel approach and a potential solution to manipulation of micro/nano particles for precise assembly.

ACKNOWLEDGMENTS

We thank National Science Foundation (1662288) and Air Forces MURI program (#FA9550-12-1-0037) for the support of this research.

REFERENCES

- [1] Hiller, U. Form und Funktion der Hautsinnesorgane Bei Gekkoniden. *Form. Funct.* **1971** *4*, 240–253.
- [2] Autumn, K.; Sitti, M.; Liang, Y. A.; Peattie, A. M.; Hansen, W. R.; Sponberg, S.; Kenny, T. W.; Fearing, R.; Israelachvili, J. N.; Full, R. J. Evidence For Van Der Waals Adhesion in Gecko Setae. *Proc. Natl. Acad. Sci. USA.* **2002**, *99*, 12252–12256.
- [3] Hansen, W. R.; Autumn, K. Evidence For Self-Cleaning in Gecko Setae. *Proc. Natl. Acad. Sci. USA.* **2005**, *102*, 385–389.
- [4] Sethi, S.; Ge, L.; Ci, L.; Ajayan, P. M.; Dhinojwala, A. Gecko-Inspired Carbon Nanotube-Based Self-Cleaning Adhesives. *Nano Lett.* **2008**, *8*, 822–825.
- [5] Geim, A. K.; Dubonos, S. V.; Grigorieva, I. V.; Novoselov, K. S.; Zhukov, A. A.; Shapoval, S. Y. Microfabricated Adhesive Mimicking Gecko Foot-Hair. *Nat Mater.* **2003**, *2*, 461–463.
- [6] Xu, Q.; Wan, Y.; Hu, T. S.; Liu, T. X.; Tao, D.; Niewiarowski, P. H.; Tian, Y.; Liu, Y.; Dai, L.; Yang, Y.; Xia, Z. Robust Self-Cleaning and Micromanipulation Capabilities of Gecko Spatulae and Their Bio-Mimics. *Nat Comms.* **2015**, *6*, 8949.
- [7] Koenig, S. P.; Boddeti, N. G.; Dunn, M. L.; Bunch, J. S. Ultrastrong Adhesion of Graphene Membranes. *Nature Nanotech.* **2011**, *6*, 543–546.
- [8] Xue, Y.; Yu, D.; Dai, L.; Wang, R.; Li, D.; Roy, A.; Lu, F.; Chen, H.; Liu, Y.; Qu, J. Three-Dimensional B,N-Doped Graphene Foam as a Metal-Free Catalyst for Oxygen Reduction Reaction. *Phys. Chem. Chem. Phys.* **2013**, *15*, 12220.
- [9] Autumn, K.; Liang, Y. A.; Hsieh, S. T.; Zesch, W.; Chan, W. P.; Kenny, T. W.; Fearing, R. Adhesive Force of a Single Gecko Foot-Hair. *Nature.* **2000**, *405*, 681–685.