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Bioinspired Design and Fabrication of Green-Environmental Dry Adhesive with Robust Wide-Tip Shape

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In this communication, we have introduced bioinspired polymeric dry adhesive structures made by using low-expertise yet robust over-etching fabrication process and chemical treatment method. The structure of gecko-foot in nature has been adopted into various applications for its remarkable smart adhesion performance. Although a lot of methods have been proposed to fabricate the gecko-inspired dry adhesion systems, they have faced on problem of structural failure of wide-tip on the top of the micro structures. To solve the problem, we have focused on developing proper wide-tip shape of the polymeric dry adhesive structures. Through the controlled over-etching process and C_4F_8 deposition method, we have made wide-tip microstructures without any structural failure. As a result, we develop the robust dry adhesive structures with highly reproducible and accurate wide-tip arrays for strong potential applications such as in green-environmental industries.

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NOMENCLATURE

MEMS = Microelectromechanical systems
SOI = Silicon on insulator
C_4F_8 = Octafluorocyclobutane
PDMS = Polydimethylsiloxane

1. Introduction

Since the first discovery of the principle of hierarchical gecko foot hairs, a number of groups have developed the properties of the bioinspired dry adhesive systems.[1,2] Recent experimental results have demonstrated that the gecko-inspired mushroom-like micropillar arrays is useful in a wide range of applications such as medical skin patch, wall-climbing robot (as shown in movie “Mission Impossible 4”) and green manufacturing transportation devices.[3-9]

In order to make these fascinating dry adhesive structures, a lot of experimental studies about the various multiscale fabrication methods have been proposed over the last decade.[4,7,10-12] For example, conventional MEMS etch process and photolithography are used to fabricate semi-conductor silicon master and soft-micro/nanoimprint lithography are performed to make hierarchical polymer structures. Also, capillary force lithography is developed to produce uniform and controllable multiscale structures for gecko-inspired dry adhesives. Although these methods for manufacturing polymeric dry adhesive structures are useful, it is still difficult to fabricate precise mushroom-like micropillar arrays with a wide-tip on the top of the micro structures. It is well known that the wide-tip of polymeric micro structures is critical for stable and strong normal-adhesion.[2,5,10,13-15] Therefore, it is important to design appropriate condition for fabrication of bioinspired dry adhesives with the wide-tip in order to avoid the structural failure.

Herein we demonstrate a smart and facile method to fabricate bioinspired dry adhesive structures without any structural failure of wide-tip arrays based on controlled over-etching process and chemical treatment method. Using the conventional deep reactive ion etching process with silicon on insulator (SOI) wafer, we make a wide-tip microstructure owing to over-etching step at the etch-stop layer (SiO_2). After C_4F_8 deposition on the fabricated SOI wafer, we replicate it on flexible and transparent polymer surfaces. As a result, we develop the

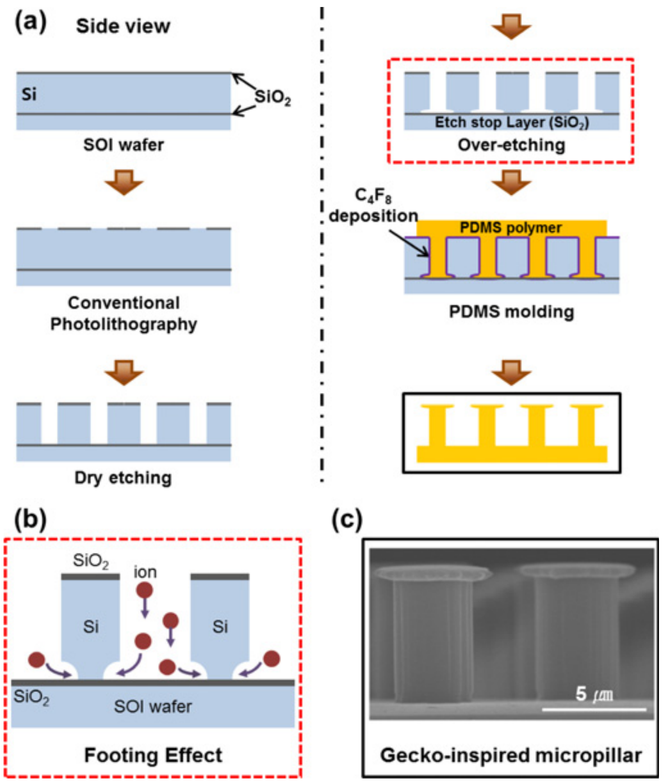

Fig. 1 (a) A schematic illustration for the detailed MEMS over-etching process and (b) Footing effect at the etch-stop layer (SiO_2 layer) with SOI wafer. (c) Representative SEM images of fabricated mushroom-like micropillar using footing effect

robust dry adhesive structures with highly reproducible and accurate wide-tip arrays for strong potential applications such as in green-environmental industries.

2. Principle and Fabrication of Bioinspired Wide-Tip Micropillar Arrays

The fabrication process of polymer surfaces was shown in Fig. 1(a) (more details are described in elsewhere).[16] A prepared four-inch SOI wafer was patterned in shape of 5 μm hole over an area of 30 mm × 30 mm by using conventional photolithography. After anisotropic deep reactive ion etching the upper silicon of the SOI wafer to the normal direction until facing SiO_2 etch-stop layer, additional etching process (over-etching process) was carried out for several times in order to develop gecko-inspired (or mushroom-like) wide-tip micropillars. A detailed schematic description of the principle of the over-etching process is shown in Fig. 1(b). Conventional MEMS dry etching system, which is consist of BOSCH process, has methodological advantages that it could perform anisotropic, directional etching with high resolution and cleanliness. Performing the repetitive dry etch process on silicon wafer containing etch-stop layer (SiO_2), the SF_6 and Ar plasma ion etch the silicon layer to the lateral direction upon facing silicon oxide (SiO_2) layer. Sequentially, it could be footing effect due to the different etch selectivity between silicon and SiO_2 etch-stop layer as shown in Fig. 1(b). Using SiO_2 etch-stop layer of SOI wafer, we carried out over-etching process and obtained flat bottom area which mimics gecko-

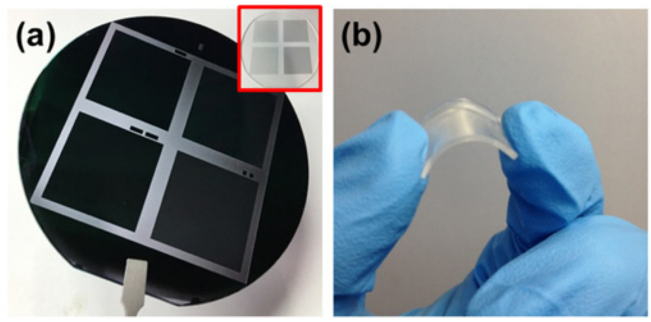

Fig. 2 (a) Digital camera images of fabricated SOI master with over-etch process in large area (over -3 cm × 3 cm) and (b) replicated flexible PDMS mold. The inset shows the replicated PDMS mold in SOI master area

foot-shape. Then, C_4F_8 gas was deposited on the surface of SOI wafer in order to lower surface energy of the substrate. To obtain robust polymeric dry adhesive surfaces, prepared PDMS mixture was poured on the fabricated SOI master and placed in an oven at 80°C for 40 min. After peeling off PDMS replica from the SOI master, we demonstrated the bioinspired polymeric wide-tip micropillar structures as shown in Fig. 1(c).

The finally fabricated SOI master is shown using digital camera images in Fig. 2(a). The inset image is the replicated PDMS surface containing gecko-inspired micropillar arrays from the SOI master. It is worthwhile that the bioinspired wide-tip micropillar structures could be fabricated on a large silicon wafer area over -3 cm × 3 cm. Also, the flexibility of fabricated polymeric PDMS surface (Fig. 2(b)) would promise the wide range of application in green-environmental industries.

3. Results and Discussions

3.1 Obtaining Robust Wide-Tip Shape

The wide and flat tip of the fabricated gecko-inspired micropillar arrays is a key structure for a dry adhesive. The presence of wide-tip maximizes adhesion force due to its large and thin spatulate area. Also, it is relatively more stable and durable as compared with other multiscale polymeric structures.

In order to realize this functional robust wide-tip shape, we consider two main designing factors; (1) over-etching process time and (2) C_4F_8 chemical deposition. First, the wide-tip size can be controlled by modulating over-etching time as shown in Fig. 3. The tip size of gecko-inspired micropillar structures becomes wider with increasing over-etching time. However, it is stuck by PDMS polymer in replicating process because of too large flat bottom area of SOI wafer due to the excessive over-etch time (Figs. 3(e) and 3(f)). In other words, a proper over-etching time is necessary to avoid structural failure of wide-tip during PDMS replication process from SOI master.

Second, C_4F_8 chemical treatment on the fabricated SOI wafer to lower surface energy of the substrate is also important to obtain defect-free wide-tip shape. As shown in Fig. 4(b), the wide-tip is stuck in the SOI master without C_4F_8 gas deposition process. The inset optical microscope images in Figs. 4(a) and 4(d) show the specific structural

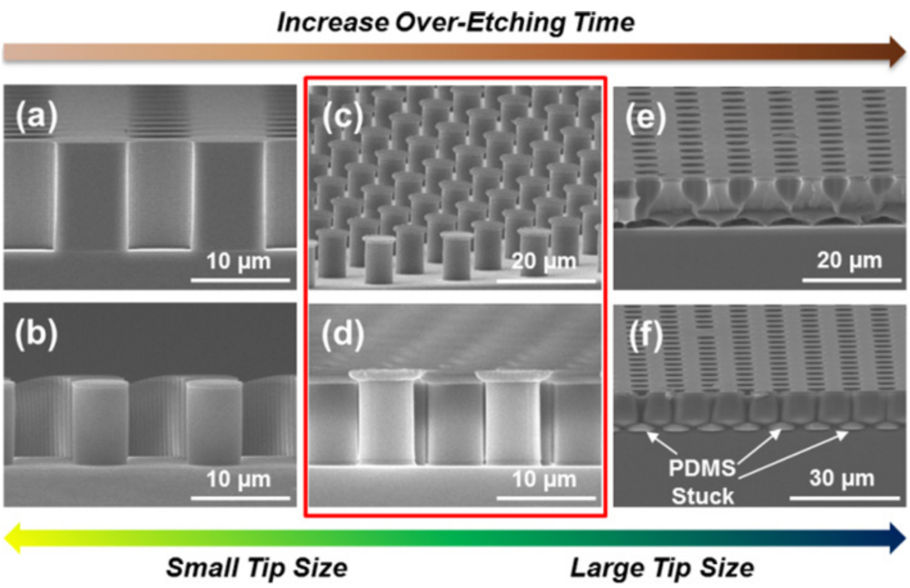

Fig. 3 Wide-Tip size variation with increasing over-etching time. The red squared SEM images (c) and (d) are selected as appropriate gecko-inspired dry adhesive structure in this experiment

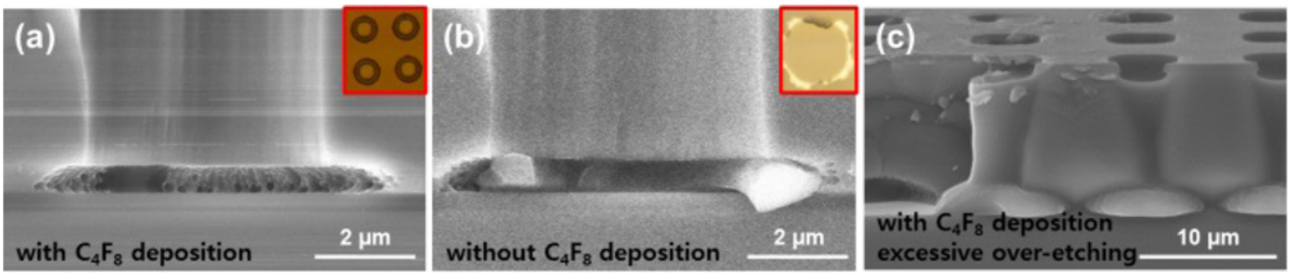

Fig. 4 The effect of C_4F_8 chemical deposition on development of wide-tip shape. The structural failure of wide-tip occurred during PDMS replication process from SOI master in case of (b) and (c)

difference between the results of final wide-tip both with and without C_4F_8 deposition. It is noted that the wide-tip is also stuck in the SOI mater made by performing excessive over-etching time even with C_4F_8 deposition (Fig. 4(c)).

3.2 Measurement of Adhesion Force

Using the fabricated PDMS micropillar arrays, the normal adhesion forces (pull-off forces) were evaluated against a flat smooth silicone surface. The measurement was conducted by using custom-built equipment, which consist of a flat stage and load cell (333AL, KTOYO, Korea) under an ambient condition with relative humidity of 40% and temperature of 25°C (Fig. 5(a)). As shown in Fig. 5(b), a circular dry adhesive pad 10 mm in diameter and 1 mm in thickness glued to the load cell was attached to the silicone substrate under a preload of -0.1 N/cm^2. Then, the pad was pulled by motorized load cell until a detachment occurred. All adhesion tests were carried out 30 times for each sample for reliability. Total number of samples is 5 pieces per each end-tip types. Three different end-tip types of the fabricated micropillar arrays were employed to measure the adhesion test: flat, wide-tip, and tip-failure shapes, whose SEM images are represented in Fig. 6. As shown in the graph, the wide-tip type displayed the largest adhesion force (-10.3 N/cm^2) among the sample types. The tip-failure type recorded large adhesion force (-5.1 N/cm^2) compared to flat type (-2.2 N/cm^2), but the value was inferior to that of

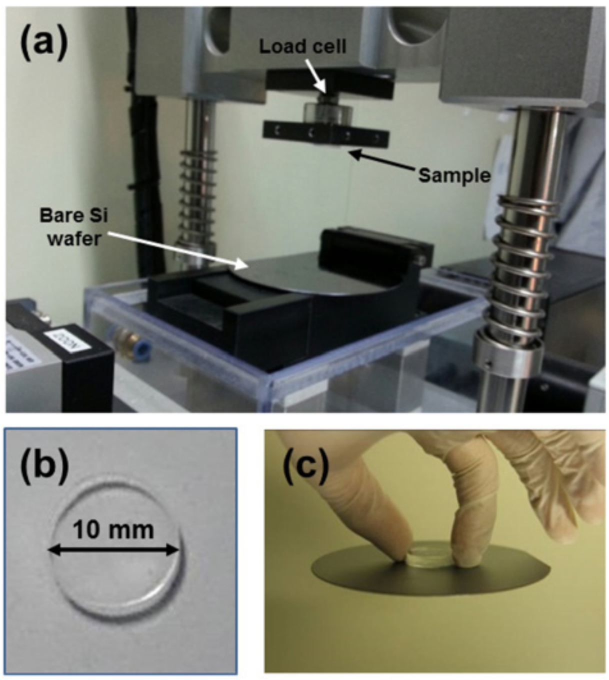

Fig. 5 (a) Optical images of custom-built equipment for adhesion test, (b) The circular dry adhesive pad sample is 1 mm thick with a patterned area of 10 mm in diameter, (c) Manually transport a silicone substrate by using the fabricated dry adhesive pad

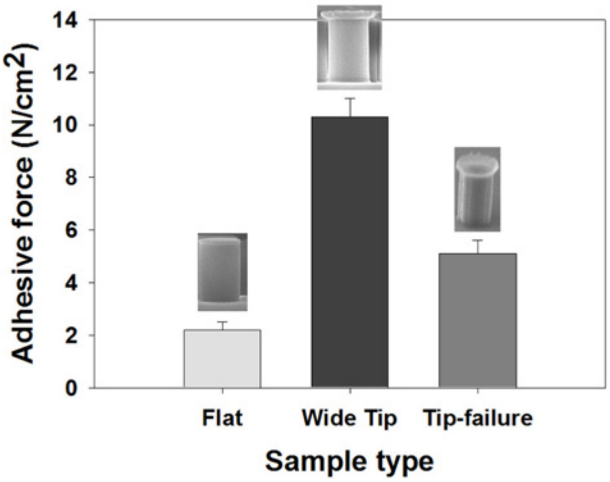

Fig. 6 Measurement of normal adhesion forces with three different end-tip types of the fabricated micropillar arrays

wide-tip type due to the structural defect of the end-tip shape. Notably, the end-tip shape of the micropillar structures is critical to obtain specific adhesion strength of bioinspired dry adhesive, which is highly important for the application of precise green-manufacturing and transportation systems.

4. Conclusions

In conclusion, we have presented a rational design and fabrication method in order to make gecko-inspired mushroom-like micropillar arrays with robust wide-tip shape. By controlling over-etching time and C_4F_8 chemical deposition, the wide and flat tip on the top of the micropillar structures was obtained in a one-step PDMS replication process. The fabricated bioinspired dry adhesive pad has displayed sufficient adhesion force for external force application without any additional requirement. It is envisioned that this low-expertise yet robust fabrication route could present wide insight to precise green-environmental manufacturing and transportation industries.

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