MULTI-SCALE COMPLIANT STRUCTURES FOR USE AS A CHIP-SCALE DRY ADHESIVE

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ABSTRACT

Many insects and lizards display the amazing ability to climb and stick to just about any surface. Recent research has honed in on these systems to better understand how they work\textsuperscript{[1-4]}, particularly on how fine sub-micron hairs enhance van der Waals, or other interactions. Provided enough intimate surface contact these “weak” forces can add up to produce significant amounts of adhesion. Additionally the attractive interaction, adhesion, must be much larger than repulsive forces, due to elastic deformation, for the adhesive to be effective. To achieve this, nature has created a hierarchical structure to conform over a range of size scales\textsuperscript{[5]}. In this work, microfabrication techniques are used to create a synthetic dry adhesive modeled after the fine hair adhesive motif found in nature. The artificial structure consists of a silicon dioxide platform, covered with polymeric nanorods, and supported by a single single-crystal silicon pillar. The Multiscale Integrated Compliant Structures (MICS) offer three levels of surface compliance: (1) the nanorods on the surface (also necessary for enhancing surface adhesion), (2) the fingers of the platforms, and (3) the flexibility of slender silicon pillar supporting the platform. For the first time large arrays, 1cm x 1cm, have been batch fabricated across an entire 10 cm wafer. A new testing technique was developed using a nanoindenter to measure adhesion between the structures and a 5 mm aluminum flat punch. Results indicate improved adhesion with the integration of the nano- and micro-structures. The multi-scale structures also demonstrated improved wear characteristics over isolated nanorods.

Keywords: dry adhesive, nanorod, nanoindenter, 3-D MEMS

INTRODUCTION

In the emerging field of biomimetics, the ever growing knowledge base of biology is brought together with the rapidly developing ability to measure and manipulate properties at very small length scales. Since the time of Aristotle, scientists have been fascinated by the gecko’s ability to scale virtually any surface, and under completely different environmental conditions\textsuperscript{[1, 5]}. In the last hundred years scientists have speculated that the adhesion force in the gecko pad is a result of suction, secretions or capillary forces. Recently Autumn et al. performed a series of experiments giving convincing evidence that van der Waals interactions are the dominant interaction force\textsuperscript{[1]}. An excellent example of convergent evolution, the gecko has developed highly refined 200 nm protrusions to maximize van der Waals interactions\textsuperscript{[1, 2, 5]}. However, for this surface contact to be effective there needs to be a minimum amount of repulsive force from the surface. To achieve this, the fine hair adhesive motif has a multi-scale compliant structure designed to conform to varying levels of surface roughness.

The largest scale of conformation, in the case of the gecko, is the gecko itself with a body and legs able to move in and around tens of centimeter size objects. Moving down in size scale are the toes with the ability to wrap around curved surfaces. Within these toes there are blood sinuses acting as a hydraulic suspension, deforming with little elastic response to millimeter scale roughness. These sinuses support rows of imbricated lamellae composed of rows of keratinous setae 30-130 \textmu m in length and approximately 20 \textmu m in diameter\textsuperscript{[5]}. These slender setae can deform to micrometer scale roughness, and offer the densely packed array of fibers necessary to maintain large amounts of surface contact. The terminus of the setae subdivide into 200 nm diameter spatulae, capable of achieving the last level of surface intimacy necessary for van der Waals forces to become significant.

Prior work has succeeded in replicating the final terminal structure of the spatulae, but centimeter scale testing resulted in negligible adhesion\textsuperscript{[6]}. The low values of adhesion were attributed to reduced surface conformation across multiple length scales, and to the inability of the surface interface to absorb energy and arrest interfacial crack growth. Additionally, it was seen that the hydrophilic nanorods would adhere to each other, and the surface to which they were stuck, reducing wear characteristics.

The emergence of the field microelectromechanical systems (MEMS) over the last three decades has brought with it a variety of microsensors and transducers\textsuperscript{[7, 8]}. In addition, processing techniques from the integrated circuits community have been greatly extended to enhance three dimensional processing capabilities\textsuperscript{[7]}. One of the many challenges still remaining in the microsensors field is the deployment and placement of microdevices. The development of a technique to microfabricate an adhesive, capable of sticking to virtually any surface, could greatly enhance the potential for “fly on the wall” distributed sensing.
In this work microprocessing techniques have been extended to create novel micro- and nano-structures mimicking the principles found in nature. The processing technique is fully compatible with standard microprocessing, requires only a single lithographic step, and uses only dry etch techniques. The structures produced follow a similar motif to the fine hair adhesive in nature by creating multiple levels of compliance. The Multiscale Integrated Compliant Structures (MICS) consist of a single single-crystal silicon pillar supporting a silicon dioxide platform coated by polymeric nanorods (Fig. 1). The silicon pillars can have aspect ratios in excess of 40 and diameters as small as 1 µm. The silicon dioxide platforms are 1 µm thick and consist of four radial meandering fingers extending 50 µm from a central square platform. Atop these platforms are arrays of vertically aligned ~250 nm diameter, ~4 µm tall nanorods composed of positive photoresist. Combining these three structures an analogous system to the fine hair adhesive is created mimicking the multiple levels of compliance on a chip. The first level of compliance, like the toe of a gecko, is the small size of the chips that can be produced, allowing the chip itself to fit within centimeter scale roughness. The next level of compliance is the flexible silicon pillars, allowing the entire platforms to rotate, accommodating sub-millimeter scale roughness. The fingers of the oxide platforms allow for conformation to tens to hundreds of micron size features. And finally to maximize surface area contact, and enhance van der Waals interactions, are 250 nanometer diameter nanorods.

**FABRICATION**

The MICS structures were fabricated using a single lithographic step and multiple etch process[9]. Single crystal silicon wafers in the (100) orientation were used for all fabrication. The wafers were first coated with 1µm of thermal oxide using a wet oxidation process at 1150°C. The photoresist was patterned using an i-line stepper with 7 µm thick positive resist (Shipley™ SPR220-7), defining the platform tops. This pattern was then transferred into the underlying oxide using an inductively coupled plasma (ICP) etch with CHF₃ chemistry. Deep reactive ion etching, using the Bosch process, was then used to extend the oxide pattern vertically ~35 µm into the bulk silicon. Subsequent to the extension etch an extended isotropic SF₆ etch is run, undercutting the silicon dioxide, and creating oxide platforms supported by a single silicon pillar (Fig. 1).

![Fig. 1. Multiscale integrated compliant structures (MICS). Portion of a 2,500 array of MICS (top), scale bar 500 µm. Individual MICS (middle), scale bar 50 µm. Central portion of a platform showing nanorod integration (bottom), scale bar 10 µm.](image)
To create the polymer nanorods, the photoresist coated structures were placed in an inductively coupled oxygen plasma. By controlling the oxygen pressure, RF bias, and time the photoresist surface was transformed into first a roughened morphology and then into a coating of organorods[10] (Fig. 2). Uniform coatings of nanorods have been created over a complete 100 mm wafer, with and without structures. As can be seen in Figure 1, incomplete coverage was seen on the platform fingers as a result of previous degradation of the photoresist.

**Fig. 2.** Scanning electron micrograph of polymeric nanorods on the central portion of a platform, scale bar 5 µm.

Using this method wafers have been created with 10 separate 10 mm x 10 mm arrays of 2,500 platforms, each containing ~50,000 nanorods. The process technique is true batch fabrication allowing for simultaneous creation of 25,000 platforms and a billion nanorods. This massively parallel approach highlights potential of this fabrication technique for future mass production device implementation.

**EXPERIMENTAL**

Previous testing of artificial fine hair dry adhesives has focused on measuring the adhesion of individual nanorods[2, 6, 11]. Scaling of individual nanorod adhesion greatly overestimates the adhesion of larger scale (50µm) adhesion testing [6]. This suggests the need to develop alternate test techniques for measuring the meso-scale adhesion of these systems. Additionally, a rough surface was desired to test the MICS in a simulated application environment, e.g. an aircraft wing. To accomplish these testing requirements a Hysitron Triboindenter™ was retrofitted with 5 mm aluminum flat punch tip. The tip was fabricated using a stock indenter shaft and a 5 mm aluminum puck (RMS roughness of 2.5 µm). To bring the two components into registration the puck was placed on the sample stage, with a drop of glue on the top surface, and the shaft was fitted into the Triboindenter™ transducer. By performing a standard load controlled indent, the tip was brought into contact with the puck and allowed to rest there while the glue dried.

Operating the nanoindenter in displacement control, the flat punch tip was brought into contact with the test surfaces and withdrawn at a constant rate recording the normal force and displacement. Upon unloading an adhesion event would cause a negative normal load. The maximum negative force was taken to be a measurement of adhesion. Repeated tests were performed in the same location to assess wear characteristics of the structures.

**RESULT AND DISCUSSION**

Adhesion testing was performed on three test surfaces including: a silicon wafer coated with photoresist, wafers covered with nanorods, and arrays of MICS. The photoresist surface showed very little adhesion with an average adhesion strength of 0.1 Pa. The nanorod surface demonstrated an improved adhesion strength of 6.5 Pa. However, integrating the pillar supported platforms with the nanorods enhanced adhesion by more than a factor of three to 21.8 Pa. No significant dependence was seen on the maximum positive applied normal load.

**Fig. 3.** Comparison of repeated adhesion between a nanorod surface on solid substrate (bottom) and a nanorod surface on compliant structures (top).

Upon repeated adhesion tests in the same location, the MICS demonstrated no reduction in adhesion by the fifth iteration. Whereas the adhesion to the nanorod surface decreased nearly to zero by the fifth indent (Fig. 3). The diminished adhesion is likely a result of damage to the nanorod surface by the indenting puck. Individual nanorods are either pushed down to the surface where they remain, are plastically deformed moving them out of the contact zone, or condense into clumps of nanorods. The integration of the flexible platforms may improve wear by flexing before a critical load is placed on the nanorods, causing non-recoverable damage. This behavior is not unlike that of the
gecko, where the long shafts of the setae can bend before the nanoscopic spatulae receive an excessive load.

The experimental values of adhesion strength for the MICS of ~20 Pa is much lower than those reported for the gecko, *Gekko gecko*, of ~100 kPa[1, 12]. Although there is much work left to be done to rival nature, there are several significant testing differences that account for the orders of magnitude difference in adhesion strength. The first major difference is that the MICS adhesion was measured in a normal direction to the surface. While in the gecko testing, adhesion was measured in a direction parallel to the surface, likely creating a composite adhesion and friction force. Autumn et al. did show that adhesion in the transverse direction was over a factor of 30 times greater than in the normal direction. By measuring the force of a single 20 µm seta, on an atomically smooth surface, normal adhesion strengths of 3 kPa were measured[1]. Additionally, based on transverse adhesion measurements, a 10 fold reduction in adhesion can be expected for macro-scale testing (or whole gecko pad tests) [1]. Taking this into account, a normal adhesion measurement between a smooth surface and an entire gecko pad may yield adhesion values around 300 Pa. This leaves only an order of magnitude difference in adhesion without taking onto account the reduced adhesion expected on a rough surface.

Diminished performance of the adhesive can be attributed to the incomplete nanorod coverage on the platform fingers. The ends of the fingers offer the highest compliance of the structure, providing the potential for improved surface contact. However, without the nanorod coating van der Waals interactions are greatly reduced, along with the subsequent adhesion strength. Improved adhesion is expected with refined processing to produce complete coverage.

The difficulty comparing values here clearly presents the need for a standardized test technique for adhesion on the millimeter size scale. For now it may be best to perform experiments on standardized samples to determine relative adhesion strengths. In this work, the photoresist surface was used as a baseline and relative improvements were shown with each additional level of compliance.

Future work will focus on enhancing adhesion through modification of the three key design components. These components are the silicon support pillar, the silicon dioxide platform, and the nanorod surface. The aspect ratio of the silicon pillar can be modified to assess an optimum height and shape for adhesion. The pattern of the silicon dioxide platform can be modified to further enhance surface compliance while maintaining structural stability. Also the spacing between individual platforms can be reduced to offer larger surface contact area. Perhaps, most importantly, the height, size, spacing, and material of the nanorods can be modified to optimize adhesion.

**CONCLUSION**

Multi-scale Integrated Compliant Structures (MICS) have been fabricated with aligned vertical photoresist nanorods coating lithographically defined flexible silicon dioxide fingers supported by a single silicon pillar. The MICS have been produced using batch fabrication techniques creating 10 mm x 10 mm arrays across an entire 100 mm wafer.

To measure adhesion a new test technique has been developed. The technique employs nanoindenter instrumentation to measure the pull-off force between a 5 mm diameter flat punch aluminum disk and test surfaces. Appreciable increases in adhesion are observed with the integration of the multiple scales of compliant structures. Additionally, the MICS offer enhanced wear characteristics with sustainable adhesion over repeated testing.

Future work will focus on optimization of the structures for improved adhesion. Concurrent development of nanoindentation adhesion testing will allow for iterative design improvement, leading to a chip-scale dry adhesive modeled after the naturally occurring system.

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**References**