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Meso-scale adhesion testing of integrated micro- and nano-scale structures

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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, particularly on how fine sub-micron hairs enhance van der Waals, or other short-range interactions. Provided enough intimate surface contact these "weak" forces can add up to produce significant amounts of adhesion. Additionally, the attractive interaction must be much larger than repulsive forces, due to elastic deformation, for the adhesive to be effective. To achieve this, nature has created a hierarchal structure to conform over a range of size scales. In previous work, micro-fabrication techniques were 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 organic looking polymeric nanorods ("organorods"), and supported by a single single-crystal silicon pillar. The multiscale integrated compliant structures (MICS) offer three levels of surface compliance: (1) the organorods 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. In this work large arrays, 1 cm × 1 cm, have been batch fabricated across an entire 10 cm wafer. Additionally, to characterize meso-scale adhesion, a nanoindentation adhesion test technique was extended to measure the adhesion between the micro-fabricated samples and a rough 5 mm diameter 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 solid supported organorods.

Keywords: Adhesion; Nanorod; Nanoindenter; 3D; MEMS; Gecko; Dry-adhesive

1. 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 [1,5]. In the last 100 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 [1].

An excellent example of convergent evolution, the gecko has developed highly refined 200 nm protrusions to maximize van der Waals interactions [1–5]. 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 μm in length and approximately 20 μm in diameter [5]. These slender setae can deform to micro-meter scale roughness, and offer the densely packed array of fibers necessary to maintain large amounts of surface contact. The terminus of the setae subdivided into 200 nm diameter spatulae, capable of achieving the last level of surface intimacy necessary for van der Waals forces to become significant.

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Prior work has succeeded in replicating the final terminal structure of the spatulae, but centimeter scale testing resulted in negligible adhesion [7]. 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 reducing wear characteristics.

The emergence of the field micro-electromechanical systems (MEMS) over the last three decades has brought with it a variety of microsensors and transducers [8–16]. One of the many challenges still remaining in the microsensors field is the deployment and placement of microdevices. The development of a technique to micro-fabricate an adhesive, capable of sticking to virtually any surface, could greatly enhance the potential for "fly on the wall" distributed sensing.

In previous work the authors have used micro-processing techniques to create novel micro- and nano-structures mimicking the hierarchal structure of the gecko [6]. Here, this fabrication technique has been extended to create larger 1 cm × 1 cm arrays and a new testing methodology has been implemented to measure meso-scale adhesion characteristics of the system. The processing technique is fully compatible with standard micro-processing 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 organorods (Fig. 1). The silicon pillars can have high aspect ratios 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 organorods composed of positive photoresist (Fig. 2). 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 nm diameter organorods.

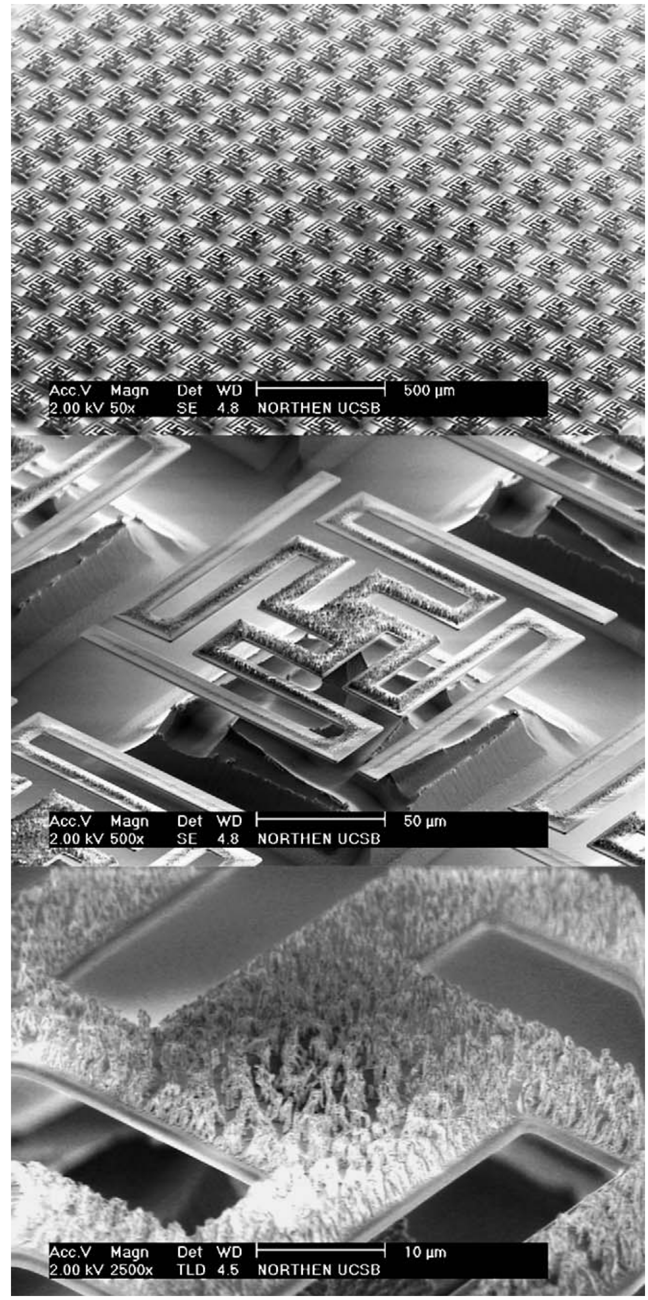

Fig. 1. Multiscale integrated compliant structures (MICS). Portion of a 2500 array of MICS (top), scale bar 500 μm. Individual MICS (middle), scale bar 50 μm. Central portion of a platform showing organorod integration (bottom), scale bar 10 μm.

2. Fabrication

The MICS structures were fabricated using a single lithographic step and multiple etch process [6,17]. Single crystal silicon wafers in the (1 0 0) orientation were used for all fabrication. Wafers were first coated with 1 μm of thermal oxide using a wet oxidation process at 1150 °C. Positive photoresist (Shipley™ SPR220-7) was spun on the wafers to a thickness of 7 μm. The top platforms were then patterned in the resist using projection lithography. This pattern was then transferred into the underlying oxide using an inductively coupled plasma (ICP) etch with CHF_3 chemistry defining the platforms in the silicon dioxide. 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 SF_6 etch is run, undercutting the silicon dioxide, and creating oxide platforms supported by a single silicon pillar (Fig. 1).

To create the polymer organorods, 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