



Adhesion of elastomeric surfaces structured with micro-dimples



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ABSTRACT

Topography has a dominant role in determining the adhesion properties of a surface. In this work we explore how arrays of micron-sized dimples can alter the adhesion performance of elastomeric surfaces. We study the effect of the dimple surface coverage, showing that the dimples act both as passive suction devices, allowing to exceed the adhesion performance of untextured surfaces, and crack-like defects, generating stress concentration at the edge of the contact area between the surface of the sample and a flat surface. Interestingly, our results reveal that the suction effect generated by the negative pressure produced by the dimples can be effectively tuned by adjusting their depth. These findings have significant relevance for the fabrication of adhesive systems in which selective adhesion to objects with small difference in weight is required.

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1. Introduction

Adhesion of solid objects is receiving great attention in the last years because of promising practical applications arising from research. New insight into the adhesion mechanism of biological systems [1–6] showed that, in addition to the chemistry, the morphology is also critical in determining the adhesion properties of a surface. For example, it has been demonstrated both theoretically and experimentally that by splitting a surface into smaller fibrillar micro-contacts can lead to an increase of adhesion [7,8]. The remarkable performance of such fibrillar surfaces derives from the mechanical independence of each topographic feature. In particular, the separation between a fibrillar surface from a substrate can be represented by a crack that has to be initiated for each fibril, whereas for a flat surface a single crack propagates continuously and uninterrupted after its initiation. Inspired by this principle, in recent years much progress has been made in the fabrication of biomimetic structures similar in design and performance to those found in biological systems [9]. Furthermore, following biomimetic designs, surfaces with switchable adherence have been fabricated using responsive materials that modify their topography under the

action of an external stimulus [10–12], resulting in the modification of the final adhesion response. However, such surfaces do not fit those applications where precise control of adhesion is needed, since the detachment always occurs at high loads.

The ability to fine tune the adhesion between two different surfaces [13] is highly desirable in many fields, including micro and nanoelectronics, biotechnology and robotics. For this reason, alternative adhesive systems were developed. For instance, it has been shown how it is possible to regulate the adhesion with a relative easy real-time control by reversibly tuning the topography of polydimethylsiloxane (PDMS) wrinkles from a sinusoidal wavy shape to completely flattened [14]. In another work it has been demonstrated the transfer printing of solid objects by kinetically controlling the adhesion of elastomeric stamps [15]. The transfer printing strategy relies on relatively strong adhesion of rubber to solids at fast peel rates, and significantly weaker adhesion at slower rates. Despite such few remarkable examples, surfaces showing controllable adhesive forces remain poorly implemented.

In this work we explore the adhesion properties of elastomeric surfaces textured with micron-sized dimples with the expectation of providing effective rules for fabricating surfaces with custom-tailored adhesive properties. We prove that specific pull-off forces can be obtained, higher or smaller than that of the respective untextured surface, as a result of the combined effect of crack formation at the edge of the contact area, and suction phenomena generated inside the dimples. In particular, we correlate the dimple surface coverage with the stress concentration generated at the edge of the contact area, and we demonstrate how the pull-off force can be effectively adjusted by varying the dimple depth. This study is

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relevant for the design of adhesive systems based on elastomeric surfaces for selective pick-and-place of objects where high weight sensitivity and selectivity is required.

2. Experimental

2.1. Materials and equipments

Sylgard 184 was purchased by Dow Corning Corporation (Midland, MI, US). SU-8 type 25 and type 100, and SU-8 developer were purchased from Micro Chem Corporation (Newton, MA, US). S1813 resist was obtained by Shipley Europe Ltd. (Coventry, UK). According to the manufacturer, it is possible to fabricate structures in S1813 as high as 2 μm . In order to fabricate structures higher than 2 μm using this material, we let the solvent of S1813 to partially evaporate, obtaining a more viscous resist (v-S1813). A spin-speed versus thickness calibration-curve provided us the information required to select the appropriate spin conditions to achieve the desired film thickness in v-S1813. All the others chemicals were used as received.

Lithography with SU-8 resists was performed using a mask aligner MA6 (SUSS MicroTec), while lithography with v-S1813 was performed using a laser writer DWL 66FS (Heidelberg Instruments Mikrotechnik GmbH).

The patterned surfaces were characterized by Scanning Electron Microscopy (SEM) using a Helios NanoLab 650 Focused Ion Beam SEM (FEI Company). A dynamic mechanical analyzer Q800 (TA instruments) was used to perform adhesion measurements of surfaces textured with dimples arrays. An Ultra Nanoindentation Tester (CSM Instruments SA, Switzerland) was used to measure the pull-off force onto precisely positioned dimples. The height of the fabricated features was measured by a XP-2 Profiler (Ambios technology). Photos of the replicas were taken with an optical microscope DM2500 (Leica Microsystems).

2.2. Fabrication of spherical micro-dome arrays

Arrays of micro-domes were fabricated in v-S1813 by thermal reflow technique. In a first step, a silicon wafer was chemically cleaned with acetone and afterwards dried with nitrogen. A liquid solution of v-S1813 was dispensed on the wafer, and a uniform thin layer of resist was formed by spinning the wafer at 2000 rpm. The thickness of the film obtained was 9 μm . Circular features having a diameter $2a = 48 \mu\text{m}$ were imaged on the resist by laser writing ($\lambda = 405 \text{ nm}$) and cylindrical pillars were obtained after development, followed by washing the sample in distilled water. Micro-domes were produced by thermal softening and rounding of the pillars at 150 $^{\circ}\text{C}$ for 30 min on a hotplate. Due to the reflow, the final height of the domes was 13 μm . SEM images of selected arrays are shown in Fig. S1 in Supporting Information.

2.3. Fabrication of square-shaped micro-pillar arrays

Arrays of square-shaped micro-pillars having four different heights were fabricated as follow. (1) SU-8 type 100 was dispensed on a silicon wafer. The wafer was spin-coated at 2500 rpm. Next, the sample was soft-baked at 65 $^{\circ}\text{C}$ for 10 min and at 95 $^{\circ}\text{C}$ for 30 min on a hotplate. The thickness of the film obtained was 110 μm . A sodalime masks (Deltamask, The Netherlands) of square-shaped patterns (42 μm side) with various spacing were used for the exposure of the resist. Patterning was performed by exposing the samples to UV radiation ($\lambda = 365 \text{ nm}$) using an exposure dose of 300 mJ cm^{-2} . Exposure was followed by a bake on a hotplate at 65 $^{\circ}\text{C}$ for 1 min and at 95 $^{\circ}\text{C}$ for 10 min. The samples were allowed to cool and, finally, pillars of $d = 110 \mu\text{m}$ and $l = 42 \mu\text{m}$ were

Table 1

Topographical features of the different textured surfaces investigated in this work: inter-dimples spacing (s), spherical dimples radius (a), spherical dimple depth (h), square-shaped dimple side (l), square-shaped dimple depth (d).

Spherical micro-cups			Square-shaped dimples		
s (μm)	a (μm)	h (μm)	s (μm)	l (μm)	d (μm)
28	24	13	77	42	4
50	24	13	77	42	6.5
63	24	13	77	42	10
77	24	13	77	42	110
90	24	13	50	42	4
120	24	13	50	42	6.5
			50	42	10
			50	42	110
			28	42	4
			28	42	6.5
			28	42	10
			28	42	110

obtained by washing the sample for 10 min in SU-8 developer, followed by rinsing in 2-propanol. (2) Pillars in SU-8 type 25 with $d = 10 \mu\text{m}$ and $l = 42 \mu\text{m}$ were obtained following the same procedure described in (1) using the following process parameters: spin coating at 3000 rpm, soft bake at 65 $^{\circ}\text{C}$ for 2 min and at 95 $^{\circ}\text{C}$ for 5 min on a hotplate, exposure dose 200 mJ cm^{-2} , post exposure bake at 65 $^{\circ}\text{C}$ for 1 min and at 95 $^{\circ}\text{C}$ for 2 min, developing for 3 min in SU-8 developer followed by rinsing in 2-propanol. (3) Pillars having $d = 6.5 \mu\text{m}$ and $l = 42 \mu\text{m}$ were fabricated in v-S1813 by laser writing. First, v-S1813 was dispensed on a silicon wafer. The wafer was spin-coated at 2000 rpm. Squares with the desired side were imaged by shining a laser beam on the resist film. The pillar array was obtained by washing the sample in MF-319 for 315 s, followed by rinsing in distilled water. (4) Squares-shaped pillars having $d = 4 \mu\text{m}$ and $l = 42 \mu\text{m}$ were obtained in v-S1813 by laser writing as described in (3), using a spin-rate of 6000 rpm and developing in MF-319 for 150 s.

2.4. Replica molding for the fabrication of dimple arrays

A PDMS solution was prepared by mixing prepolymer and crosslinker with a weight ratio of 10:1. After degassing to remove air bubbles, the solution was poured on the master and negative replicas were obtained by carefully peeling the elastomeric film after thermal curing. Curing was performed on a hotplate at 70 $^{\circ}\text{C}$ for 1 h. Fig. 1a and b shows representative optical micrographs of arrays of spherical and square-shaped dimples, respectively. The key geometrical features of the fabricated surfaces are depicted in Fig. 1c, while the dimensions of the dimples used in this study are specified in Table 1.

2.5. Adhesion measurements on dimple arrays

Pull-off forces were obtained by measuring quasi-static force versus displacement curves. In Fig. 2 schematics of the key steps involved in a typical adhesion test are shown. A square-shaped flat glass having a surface area of 13.5 mm^2 glued to a metallic shaft by means of $\sim 1 \text{ mm}$ thick wax layer was used as probe. The shaft can be moved up and down in displacement or force control. During a typical adhesion test, the probe was put in contact with the surface of the sample applying a force of 0.1 N. The nature of the probe makes the measured pull-off forces very sensitive even to slight misalignments. To align the glass punch to the surface of the samples, the wax was first softened with an air flow at 50 $^{\circ}\text{C}$, and then it was left to solidify for a few minutes being in contact with the surface of the sample under the preload of 0.1 N. At this point, after an additional compressive displacement of 20 μm , the probe was retracted at a constant rate of 50 $\mu\text{m min}^{-1}$. The magnitude of

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