Microstructured Shape Memory Polymer Surfaces with Reversible Dry Adhesion

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Supporting Information

ABSTRACT: We present a shape memory polymer (SMP) surface with repeatable, very strong (>18 atm), and extremely reversible (strong to weak adhesion ratio of >1 × 104) dry adhesion to a glass substrate. This was achieved by exploiting bulk material properties of SMP and surface microstructuring. Its exceptional dry adhesive performance is attributed to the SMP’s rigidity change in response to temperature and its capabilities of temporary shape locking and permanent shape recovery, which when combined with a micropip surface design enables time-independent control of contact area.

KEYWORDS: dry adhesives, shape memory polymer, reversible adhesion

INTRODUCTION

Reusable dry adhesives with strong adhesion and a high degree of adhesion reversibility are attractive for a wide range of applications including temporary bonding in domestic and industrial settings, the “feet” of climbing robots, and automated assembly at both macro- and microscale. Both adhesive strength and reversibility come from a combination of bulk and surface material properties, often aided by carefully designed surface micro/nanostructures.1–4 Because a dry adhesive relies primarily on noncovalent molecular interactions to create its adhesion force, it is important to maximize contact area at the molecular scale. To accomplish this, the adhesive material must be compliant enough to conform closely to the surface of the substrate. However, as the adhesive material becomes more compliant it also becomes more susceptible to failure from crack formation and propagation, leading to lower adhesion. A common strategy to overcome this contradiction is to create arrays of microscopic fibrillar structures on a relatively rigid backing layer;5 the microscopic fibrils are compliant enough to conform to the substrate, whereas the rigidity of the macroscopic structure helps to evenly distribute the load among the contact points, thereby delaying the onset of peeling. Although significant efforts have been made to study and manufacture compliant, hierarchical fibrils for dry adhesives,6 relatively few authors have demonstrated the importance of controlling backing layer rigidity.2,7 One such demonstration was performed using phase changing material as a backing layer, where the effective adhesive strength of the overlying elastomer was shown to increase substantially when rigidly supported.7

A change in elastic modulus can be effected in most polymeric materials by shifting the temperature across the polymer’s glass transition (T_g). A class of thermosensitive smart materials referred to as shape memory polymers (SMPs) are specifically designed to drastically change their mechanical compliance in this way at a convenient T_g.8 The change in an SMP’s elastic modulus is accompanied by another very important property from which its name is derived: it is ability to lock itself into an arbitrary “temporary” shape and to then recover its original, “permanent” shape. This ability can be utilized to reversibly change the surface morphology of SMP, leading to switchable surface properties such as dry adhesion.9,10 During an SMP’s transformation from a temporary shape to its permanent shape, the stresses generated during attachment are released, which can serve as a unique built-in adhesion detachment mechanism. Despite these attractive features offered by SMP, strong adhesion has only been previously demonstrated when SMP is combined with an intrinsically adhesive (or sticky) rubber layer or when the surface is treated with adhesion molecules.3,4,11,12 We hereby explore SMP as a single component to construct a strong dry adhesive, with no additional “adhesive” layer added.

RESULTS AND DISCUSSION

The SMP described above forms the basis for a not only strong but also highly reversible dry adhesive when its shape fixing and recovery capabilities are combined with a micropatterned surface design.13 Evenly spaced microscale pyramids – termed microtips – are patterned onto an SMP surface using a reusable silicon mold (see the Supporting Information). In the
fabrication of our SMP surface, we chose a particular thermoset epoxy-based SMP that experiences a change in elastic modulus from approximately 2.5 GPa (below 35 °C) to 10 MPa (above 65 °C) corresponding to the SMP’s $T_g$.

As with most polymers, curing it in a mold captures surface details down to the nm (see the Supporting Information, Figure 3). A small section of this SMP in its rigid permanent shape is represented in Figure 1a. When heated above its $T_g$, it will become compliant (Figure 1b) and can be easily deformed to a temporary shape. This is depicted in Figure 1c, where the SMP is pressed against a mating substrate, thereby compressing the microtips and causing the flat region between them to collapse into contact with the substrate. Note that an essential step toward forming a strong adhesive bond has been accomplished by this collapse; namely, the generation of large contact area between SMP and substrate. However, the bond is not yet very strong or stable. If pressure is released, the heated SMP, like any other elastically deformed compliant material, is susceptible to peeling failure and will try to spring back to its original shape. It is at this stage that the SMP sets itself apart from other common materials by locking in its temporary shape through cooling below its $T_g$ (Figure 1d). The SMP will stay in this shape until it is again heated to resume its original shape, as shown in Figure 1e where the contact area, and therefore the adhesion, is nearly completely eliminated. Scanning electron micrographs of the fabricated microtipped SMP in both its permanent and temporary shapes are shown in images a and b in Figure 2, showing the microtips partially flattened and level with the collapsed intermicrotip region, all of which now make intimate contact with the substrate. The collapsed, temporary shape is reproduced using finite element software (see the Supporting Information) and is shown in Figure 2c, d along with the stress profile showing stresses concentrated near the microtips where deformation is greatest.

There is a minimum microtip height that is required to reliably cause the intermicrotip region to fully delaminate when the SMP is reheated. This height is a function of the SMP storage modulus, work of adhesion to the substrate material, detachment temperature and microtip spacing. In our case, the substrate material is glass and a target detachment temperature of 90 °C is selected for consistency with previous work. The stresses and strains generated during bonding near the microtips increase with microtip size, shown in Figure 3 with 100 μm spacing. Cooling below $T_g$ traps these stresses internally within the polymer’s molecular structure, eliminating the restoring force between SMP and substrate. When reheated, the stresses will be relieved and the restoring force reestablished. For delamination between SMP and substrate to occur, the released strain energy must exceed the work of adhesion of the contacting area. Experimentally, the size, measured by base-width, required for reliable delamination from glass was determined to be between 18 and 21 μm. FEM analysis was performed with the storage modulus of 10 MPa and the work of adhesion of 46 mJ m−2 measured using atomic force microscopy (see the Supporting Information). FEM results shown in Figure 3 indicate the critical size to be between 15 and 18 μm; a consistent result given the idealizations inherent in computational analysis (see the Supporting Information).

The adhesive strength and reversibility of the resulting microtip SMP surface to a glass substrate is demonstrated in Figure 4. First, the unpatterned face (back side) of a 6.35 mm diameter section of SMP is glued to an aluminum cylinder to provide a means of loading and unloading the SMP surface (see the Supporting Information, Figure 1). The microtip SMP surface is then bonded to a glass-topped 5 kg mass using the process described in Figure 1a–d. The SMP-to-glass interface can support the full weight of the 5 kg mass as it is lifted and held, representing an adhesive strength of more than 156 N cm−2. To reverse the adhesion, the load is removed and the SMP heated to 90 °C to initiate shape recovery. The adhesion is now essentially zero, as in Figure 1e, and the SMP is easily lifted away from the glass surface.

To quantify the adhesion, we performed tests using similarly constructed SMP samples with an aluminum holder. The bonding of the rigid aluminum to the side opposite to the adhesive interface of the SMP was found to have unintended consequences for the observed collapse behavior. Heating and applying pressure to the SMP during bonding causes radial expansion in our cylindrical SMP adhesive; however, this expansion cannot occur where bonded to the aluminum, and so a slight convex curvature develops on the adhesive and the contact pressure for adhesion cannot be perfectly even from center to edge (see the Supporting Information, Figure 2). This fact contributes to the observed relationship between the preload applied during bonding and the strength of the...
has been demonstrated previously. To ensure that its adhesive recover its original shape repeatedly and without deterioration through 20 bond/debond cycles and then tested to failure 10 consecutive times with results in Figure 4e. The tests indicate that the qualities are similarly robust, a single SMP adhesive was put on in the Supporting Information.

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resulting bond in Figure 4d. Adhesive strength increases steadily with increasing bonding preload because of the progressive radially outward collapse of the intermicrotip regions of the SMP to the substrate. As preload approaches approximately 30 N cm$^{-2}$, all intermicrotip regions are in contact with the substrate and further increases in bonding pressure yield no measurable increase in adhesion because gains in contact area become minimal. The magnitude of the preload required to reach this plateau in adhesive strength is expected to depend on the aspect ratio, i.e., the ratio of width to thickness, of the SMP adhesive layer. This point is elaborated on in the Supporting Information.

The SMP’s ability to undergo solid state deformation and recover its original shape repeatedly and without deterioration has been demonstrated previously. To ensure that its adhesive qualities are similarly robust, a single SMP adhesive was put through 20 bond/debond cycles and then tested to failure 10 consecutive times with results in Figure 4e. The tests indicate an average adhesive strength of 184 N cm$^{-2}$, an exceptionally high adhesive force compared with other macroscale dry adhesives which range from 0.1 to 100 N cm$^{-2}$, where the upper portion of this range has only been achieved using carbon nanotubes and polymer-based adhesives are generally below 10 N cm$^{-2}$. Additionally, the sample does not show signs of degradation with repeated uses. In contrast to the high “temporary” shape adhesion strength (Figure 1d), the “permanent” shape adhesion strength (Figure 1e) was below the resolution of our equipment (1 mN). This corresponds to a residual adhesion less than $\sim 3 \times 10^{-5}$ N cm$^{-2}$, demonstrating more than 4 orders of magnitude difference between the adhesion of the temporary and permanent shape states. Shear data has not been explicitly included, but is expected to be of similar magnitude as the provided normal-direction adhesion data.

Substantial opportunities exist to expand beyond the work presented in this paper, including the characterization of the adhesive bond between the SMP material and other materials with varying chemical composition and surface roughness. It is important to keep in mind that the SMP formulation used here is but one of many formulations which have already been developed and are available in literature. Other formulations may exhibit superior adhesion by virtue of surface chemistry or bulk material properties. Likewise, the glass transition temperature, which dictates the detachment temperature, can be tailored for specific applications. The design of the adhesion interface geometry may be similarly be subject to improvement. For example, a more refined analytical/computational model may be developed to guide the optimization of the microtip size and pattern, and differently shaped microstructures may provide enhancements in adhesion through crack-trapping or other mechanisms while preserving reversibility.

CONCLUSION

In conclusion, shape memory polymers can offer excellent dry adhesive performance by virtue of their shape-fixing-recovery properties and dramatic shift in elastic modulus in response to temperature change. The magnitude of the reversibility can be enhanced with simple, robust, and easily molded microstructures. Our particular SMP adhesive demonstrates tensile adhesive strength to glass twenty times greater than the typically cited shear adhesion of gecko foot pads ($\approx 10$ N cm$^{-2}$) and far exceeding most other reusable macroscale dry adhesives, while the application of heat reduces adhesion to negligible levels when detachment is desired. There is no particular upper limit to the manufacturable size of our SMP adhesive, except that issues related to thermal expansion and

Figure 3. Von Mises stress near four sizes of microtip surface (diameter: 6.35 mm). (a) SMP is bonded to a glass surface applying preload initially at 90 °C, (b) 5 kg of mass is lifted by SMP bonded to a glass surface with the contact area of $\sim 3 \times 10^{-5}$ m$^2$. (c) Heating to 90 °C causes detachment with negligible residual adhesion. (d) Effect of preload on adhesion. (e) 10 consecutive cycling tests of a single SMP microtip surface.

Figure 4. Demonstration of adhesive performance of an SMP microtip surface (diameter: 6.35 mm). (a) SMP is bonded to a glass surface applying preload initially at 90 °C, (b) 5 kg of mass is lifted by SMP bonded to a glass surface with the contact area of $\sim 3 \times 10^{-5}$ m$^2$. (c) Heating to 90 °C causes detachment with negligible residual adhesion. (d) Effect of preload on adhesion. (e) 10 consecutive cycling tests of a single SMP microtip surface.
Poisson’s effect may necessitate mitigating design features at large scale.

**ASSOCIATED CONTENT**

S Supporting Information
Details of material property tests, finite element modeling, and the SMP adhesive preparation, geometry, and testing procedures are provided. This material is available free of charge via the Internet at http://pubs.acs.org/.

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The authors declare no competing financial interest.

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**REFERENCES**

Silicon Microtip Pattern Molds:
A mold was prepared to generate the pyramid microtip pattern in the SMP. The mold was created by first depositing a silicon nitride layer on a clean Si (100) wafer. A layer of photoresist was spin-coated and patterned to form square openings each 20 µm across in a square pattern with 100µm center-to-center spacing. The silicon was exposed by etching the nitride briefly in a 10:1 BOE (buffered oxide etch) bath. The photoresist was then removed. Etching in a KOH solution (70g KOH, 190ml H₂O, 40ml IPA) at 80°C formed the pattern of pyramid recesses in the wafer using the remaining nitride layer as a mask. Finally, the nitride layer was removed, and the completed mold was coated with trichlorosilane for silanization in a vacuum chamber for 1 hour.

Mixing and Curing of SMP Precursor:
Prior to mixing, the EPON 826 (The diglycidyl ether of bisphenol A epoxy monomer; Momentive) was heated at 75°C for 30 minutes to remove any crystallization. The EPON 826, Jeffamine D230 (poly(propylene glycol)bis(2-aminopropyl) ether; Huntsman), and NGDE (Neopentyl glycol diglycidyl ether; TCI America) were then mixed at a 1:1:1 molar ratio. The mixture was shaken vigorously for no less than one minute, and then allowed to settle for 10 to 20 minutes. Once poured onto the mold to a depth of approximately 4mm, it was placed in a pre-heated oven at 100°C for 1.5 hours to cure, and then the oven temperature was increased to 130°C for an additional hour to ensure the curing was complete.

SMP Sample Geometry:
Cylindrical samples of 0.25 inch (6.35mm) diameter are cut from the patterned sheet of SMP. Cylindrical aluminum segments of 0.375 inch length were made and each segment then had a 0.125 inch diameter cross hole drilled through it. Each aluminum segment is termed a “sample holder,” and the unpatterned face of each cylindrical SMP sample is glued to the end of one sample holder using a general purpose epoxy (SI Figure 1). The epoxy is also used to affix a
0.25 inch diameter steel ball bearing to the center of the opposing end of the sample holder to minimize rotational moments applied during the bonding process.

SI Figure 1: a) An example of an SMP adhesive bonded to an aluminum cylinder with epoxy for handling purposes. b) The adhesive face of the SMP with microtip pattern (not visible). c) SMP bonded to a glass surface during an adhesion test with weight applied via string fed through the hole in the aluminum.

**Bonding Procedure:**
A clean glass slide is placed on a custom temperature controlled aluminum heater, and is heated to 90°C. The SMP sample is placed on the center of the glass slide so that the microtip patterned surface contacts the slide. The sample is allowed to sit on the slide for five minutes to come to thermal equilibrium, and force is then applied acting perpendicular to the SMP-to-glass interface by pressing on the top of the affixed ball bearing by applying a fixed weight. The weight is applied gradually, increasing over the course of several seconds. The heater remains on for two additional minutes while allowing the viscoelastic SMP to relax towards mechanical and thermal equilibrium in its collapsed state. The heater is switched off, and a gentle air flow is applied over the system to hasten the cooling process which lasts for seven minutes. The SMP and glass slide are now bonded.

**Macroscale Adhesion Test:**
The glass slide with bonded SMP sample are placed in a custom apparatus so that the glass slide is held in place with the SMP-to-glass interface parallel to the ground and with the SMP sample pointing downwards. A container hangs from the cross hole in the SMP sample holder, placing a small initial load (<20 N cm⁻²) on the adhesive interface. A small water pump is used to gradually fill the container with water at a rate of 12 mL s⁻¹, increasing the load on the SMP-to-glass adhesive interface at a rate of 0.37 N cm⁻² s⁻¹, until failure of the SMP-to-glass bond. The flow of water is stopped, and the adhesive strength is indicated by the combined weight hanging from the SMP sample at the time of failure.
The backside of our SMP surfaces are bonded to aluminum holders of the same diameter (0.25 inch) in order to apply tensile load during the adhesion testing. As temperature is increased, the SMP expands much more than the aluminum due to the thermal expansion coefficient mismatch, and so the free face of the SMP becomes subtly convex (SI Figure 2). During bonding, a preload is applied acting through the center of the SMP-substrate interface. This preload will initiate collapse of the microtips as described previously in a process we term “local collapse”.

SI Figure 2: Diagram showing the progression of collapse for our particular testing procedure, contrasting global and local collapse.

The process of local collapse to generate adhesion, followed by reconstitution of the original shape to reverse the adhesion, is fundamental to the operation of our reversible dry adhesive. However, local collapse does not occur simultaneously for all regions of the sample surface due to the global curvature of the sample. In general, the central region of a sample will experience local collapse first as a preload is applied. As the preload is increased, the locally collapsed region expands outward toward the sample edges in a process we term "global collapse." Poisson's effect also works to inhibit full collapse by causing outward radial motion of the SMP as preload is increased. The result is that the necessary force to fully bond our SMP adhesives to glass is primarily dictated by global collapse, rather than local collapse. Likewise, the presence of the aluminum holder has an effect on the initial detachment process, which progresses as the reverse of the collapse process. However, we stress that the aluminum cylinder inclusion is not a prerequisite for detachment. In its absence, bonded microtip SMP can consistently and completely detaches from a glass substrate upon heating above approximately 70 °C.

The SMP adhesive layer, referred to as a 'backing layer,' for our gathered data was approximately 4 mm thick. There are a variety of factors to consider when choosing an appropriate thickness, some of them specific to our production and testing methods. A very thin backing layer may increase the force necessary to compress the microtips when compared with a thicker layer. Our FEM model and rough analytical estimates indicate that a backing layer on
the order of several hundred microns is sufficient to avoid this issue, and so it was not a concern during our tests. The curvature and distortion due to the bonding process is reduced by a thinner backing layer, but with the trade-off that the backing layer becomes less compliant and therefore any imperfections in the surface are more difficult to "flatten out" during bonding. Very thin, high-aspect ratio samples were more prone to warping during our production process (prior to bonding to the aluminum holder), and coupled with the reduced compliance appeared to negatively impact the consistency of adhesion between samples. In addition, it proved difficult to precisely control the thickness of the backing layer, and so choosing a greater thickness reduced the importance of tightly controlling this variable.

On the other hand, thinner backing layers are appealing since we would expect an increase in adhesive strength for a well-made and well-bonded adhesive sample with a very thin backing layer based on the principles of crack propagation by elastic energy release. In addition, by reducing or eliminating the convexity formed during bonding, a very thin backing layer would further highlight the utility of our pyramid microstructures since release by peeling would become exceedingly difficult without them. Many of the thinner samples (≈1 mm) showed excellent adhesive performance, though not noticeably better than the thicker samples. High-quality samples with very thin backing layers may exhibit improved performance.

**Material Property Tests:**

An Asylum Research MFP-3D AFM was used to produce the surface roughness and microscale adhesion results. An SMP surface cured against a silicon wafer was used for both AFM roughness and microscale adhesion testing. During adhesion testing, the SMP surface was additionally left exposed to air at 100°C for two hours to reduce the possibility of air-to-SMP chemical interactions affecting surface chemistry during testing. A typical AFM image of 4.7 Å root mean square (RMS) roughness of SMP surfaces is shown in SI Figure 3.

![AFM image of SMP surface roughness](si_figure_3.png)

**SI Figure 3:** AFM measurement of SMP surface roughness, cured against polished silicon. (RMS roughness = 4.7 Å)

The work of adhesion ($\gamma$) between a 1 µm diameter silica sphere and SMP near its glass transition range is calculated from atomic force microscope (AFM) adhesive force measurements in conjunction with the JKR theory of elastic contact using Equation 1:\(^1\)

$$F_c = -\frac{3}{2} \gamma \pi R$$  

(1)
where $R$ is the radius of the silica sphere, and the relationship is independent of elastic modulus. Measurements were taken in a grid, with 256 individual measurement locations using a 1µm diameter silica particle tip. The indentation load is ~45nN for each test with a speed of 2µm/s (SI Figure 4). The measurements were taken at 30°C, while the polymer is in its rigid state. From the collected data, the work of adhesion is estimated to be 46 mJ/m².

SI Figure 4: AFM adhesive force histogram of 256 individual tests in a grid pattern at 30°C with a Gaussian curve-fit and mean of 108.7 nN.

Thermal expansion was measured using a TA Instruments Q800 DMA by tracking the linear motion of a bar-shaped SMP sample with rectangular cross section of 0.7 mm² under a static load of 1 mN while it is incrementally heated and cooled. Temperature increments of 5°C between -20°C and 110°C were used, except near $T_g$ (35°C to 60°C) where temperature increments were reduced to 1°C. The temperature was held isothermally for 5 minutes at each increment. The temperature range was spanned from -20°C to 110°C, then back to 20°C, with the values in SI Figure 2a reflecting the average of the two sets of data. (SI Figure 5)

SI Figure 5: Elongation due to thermal expansion is shown where zero strain is at 20 °C.

**Finite Element Modeling:**
A quarter-symmetry finite element model of the large area SMP surface was developed using ABAQUS, with symmetry planes shown in SI Figure 6 relative to the microtip locations. ABAQUS was used as it is particularly well suited for simulating transient dynamic events with
an ability to handle severely nonlinear behavior such as contact and large deformation. The model was modified for 12µm, 15µm, 18µm, and 21µm microtip sizes as measured at the base. The backing layer thickness of the adhesive was modeled to be 400µm, which is sufficiently far from the microtips for the top boundary to have a negligible impact on the microtip deformation. Adhesive force between substrate and SMP is modeled with linear springs to approximate the measured work of adhesion that was described earlier.

The SMP is simulated in its hot state with elastic modulus of 10 MPa and a Poisson's ratio of 0.40. A force is applied to the top of the SMP, opposite the microtip surface, pressing the SMP together with a substrate. The force is increased from 0 to 30 N cm\(^{-2}\) to simulate collapse, and then decreased to 0 N cm\(^{-2}\) to simulate re-heating following bonding where the elastic energy stored during the compression of the microtips acts to overcome the adhesive force to separate SMP and substrate.

The mesh is composed of both tetrahedral and structured quadrilateral elements. The area adjacent to the microtip was meshed using linear tetrahedral elements and distortion control was enabled for these elements to ensure that these elements could withstand high deformation. The elements away from the microtip were meshed with structured linear quad elements without any distortion control to ensure optimal computational performance.

As the SMP adhesive has preload applied, the inter-tip areas collapse to contact the substrate and seal off a volume of air surrounding the base of each microtip. As collapse proceeds, the air becomes pressurized, causing a repulsive force between adhesive and substrate. An estimate of the air pressure versus preload for several microtip sizes is shown in SI Figure 7. Larger microtips require a larger preload before the intertip region collapses to seal the volume of air. The values are calculated from nodal positions using an FEM model that does not explicitly include the effect of the air pressure, and therefore are expected to be conservatively large.
From Figure 2 and SI Figure 2, it may be seen that for a cross section at the SMP-substrate interface, the air pockets are < 10% of the total area. Assuming trapped air at a pressure of 3 bars acting over 10% of the interface, a conservatively high repulsive force of 3 N cm\(^{-2}\) (0.3 bar) is calculated. The total effective strength of our SMP adhesive is on the order of 200 N cm\(^{-2}\) (20 bar), and therefore it is concluded that the trapped air does not have a significant direct effect on the strength of adhesion.

It may be noticed in Figure 2 that the FEM appears to predict shallower air pockets than the SEM images indicate. This is most easily explained by noting that the FEM mesh is large relative to the feature size in question, thus it is unable to capture such fine detail. Two other factors not present in the FEM are expected to contribute to the shape seen in experimental SEM images. The FEM does not include the force of the compressed air, which should act to create more circular, slightly deeper pockets. However, it is also evident from SI Figure 2 that the line of contact between SMP and substrate along the global collapse front is similarly well defined even though no trapped air is present. The discrepancy in shapes may be better explained by the fact that in the case of the SEM images, the SMP is cooled to complete the bond. During the cooling process, the polymer contracts slightly and pulls back away from the substrate, enhancing the “sharpness” of the interface edges.

References