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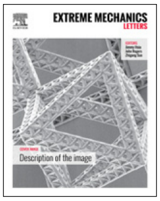

Micro-wedge array surface of a shape memory polymer as a reversible dry adhesive

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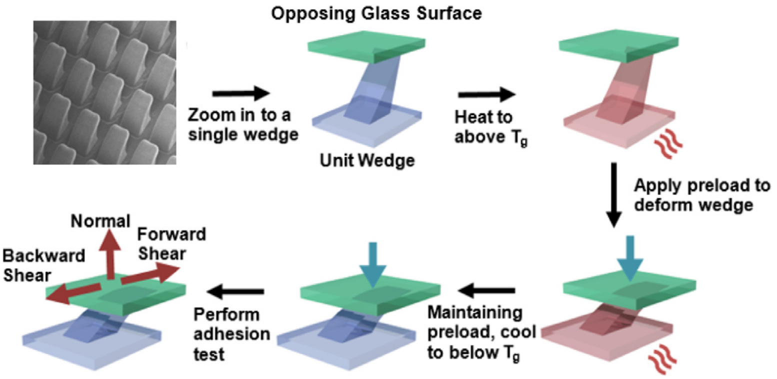
HIGHLIGHTS

- Asymmetric wedge geometry gives rise to varying adhesion strength, depending on loading direction.
- Fabricated shape memory polymer (SMP) micro-wedge arrays are used to perform adhesion tests to characterize adhesion strength based on applied loading direction.
- Experimental data is compared with finite element method results, confirming directionality.

GRAPHICAL ABSTRACT



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ABSTRACT

A shape memory polymer (SMP) surface with geometrically asymmetric micro-wedge array is fabricated as a reversible directional dry adhesive through a double exposure angled lithography technique. The unique shape fixing and recovery properties of SMPs and surface microstructuring enable highly reversible adhesion strength upon thermo-mechanical loading. The tilted wedge geometry gives rise not only to its capability for varying adhesion strength based on loading direction, but also the reduction of strain energy input necessary to achieve contact area saturation with the opposing surface. To characterize the directional adhesion strength of the fabricated micro-wedge surface, adhesion tests are performed in the forward shear, backward shear, and normal directions based on the tilting direction of the micro-wedges. The adhesion strength is measured as a function of the applied preload for the three directions investigated, and is compared to a computational analysis by modeling the adhesive failure as the initiation of crack growth in linear elastic fracture mechanics. Additionally, reversibility is demonstrated by heating the micro-wedge surface above its T_g, allowing the structure to recover its original shape after being deformed, resulting in almost zero adhesion strength. The adhesion tests demonstrate that the forward shear direction is capable of adhesion strengths that are greater than that of the backward shear direction by a factor of over 3, confirming its capability for directional adhesion.

1. Introduction

Dry adhesives offer various advantages over wet adhesives including reusability, longevity, greater adhesion, robustness, and potential for reversibility [1]. A reversible dry adhesive design that

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has shown great success in past studies is a biomimetic array of micro- or submicro-scale fibrillar structures, inspired by nature's gecko foot hairs [2–12], which themselves exhibit directional adhesion during shear loading. These designs make use of individual fibers to enhance the robustness of the adhesive interface, particularly when mated to microscopically rough or uneven surfaces, compared with chemically similar flat-surfaced adhesives. The bulk surface adhesion strength is then primarily dependent upon the collective contact area of the individual deforming fibers.

In past studies on fibrillar-structured dry adhesives, the most favorable results were found through the fabrication of a compliant microfiber array on a relatively rigid backing layer, whereby the former structure contributes to its conformity to the opposing surface, while the latter structure inhibits peeling by providing more uniform load distribution, thus increasing its overall adhesive strength [11]. However, as evidenced in published past experiments, applications of such designs still typically yielded relatively low adhesion, within the range of sub-1 atm adhesive strengths. Although some studies demonstrating higher maximum adhesive stresses exist [12–14], there remains substantial room for improvement, as past studies have yet to develop versatile systems suitable for general use; strength is but one aspect of an adhesive, among other qualities such as directionality [15,16], reusability, and reversibility.

Advancements to dry adhesives can be found through the implementation of responsive materials, which enables material property "switchability" of the structure [17]. One such investigation was done, utilizing a shape memory polymer (SMP) as the primary adhesive material [18]. SMPs refer to a broad range of such responsive materials that, as their name implies, exhibit a behavior that allows for the storing of a configurational memory of a certain "permanent shape" that is recovered from a deformed shape, i.e. "temporary shape", through an external stimulus that is specific to the material. One common stimulus to activate the shape memory behavior is temperature: heating the polymer beyond a certain glass transition temperature (T_g) initiates its material compliance transition from "rigid", on the order of 1 GPa, to "compliant", on the order of 10 MPa. These unique properties have been utilized in combination with surface microstructuring, e.g. microtip patterning, to produce an SMP-based dry adhesive with both high normal adhesive strength and reversibility [18].

Dry adhesives generally rely on van der Waals forces, rather than chemical means to generate adhesive forces. It follows that a superior adhesive surface design is achieved by maximizing the area of contact between the adhesive surface and the opposing surface with as little input energy as possible. As seen in the results of past studies, the utilization of SMP as the primary adhesive material combined with surface microtip patterning allows for direct manipulation of the material compliance and the contact area, which contributed to their impressive results in adhesive strength and reversibility [18]. This paper strives to propose a method to expand upon that past investigation by introducing an asymmetrically tilted wedge-shape structure, in order to exhibit directional shear adhesion capabilities. The structures fabricated in the past demonstrated high normal adhesion strength and reversibility. However, because of the symmetric shape design of the microtip, the surface not only requires high preload to achieve contact area saturation but also is intrinsically incapable of directional shear adhesion, which would otherwise enable multiple adhesion strength states as a function of loading conditions [18]. Here, we present a micro-wedged SMP surface capable of high reversible adhesion based on the shape memory effect, and directional shear adhesion upon loading conditions based on asymmetric shape design in the micro-wedge. Such a surface allows not only multiple high shear adhesion states upon loading conditions, but also zero-adhesion states via thermo-mechanical loading. Furthermore, the tilted wedge geometry gives rise to the reduction of strain energy input necessary to achieve contact area saturation with the opposing surface. In other words, by using deflection of thin wedges, rather than bulk compression, as the primary mode of deformation, a large contact area can be obtained with a relatively small preload applied.

A two-step angled exposure technique is utilized to fabricate a mold comprised of an array of tilted asymmetric wedge structures. Further steps produce a positive SMP replica, the surface of which is tested for its forward shear, backward shear, and normal adhesion capabilities as a function of the preload, as indicated in Fig. 1. The introduction of asymmetric wedge structures implies that there will be inherent directionality of the adhesive, i.e., the shear adhesive strength should be higher for the forward shear direction than the backwards direction. This behavior is verified through both experiment and computational analyses and will be presented in the upcoming sections.

2. Fabrication procedure

The micro-wedge structure is shown in Fig. 2. It consists of a periodic array of SMP wedges fabricated as part of a continuous thin layer of SMP on a glass backing layer. The fabrication of the master mold relies on a multi-step angled exposure technique through the use of SU-8 50 negative tone photoresist (MicroChem) [12,19,20]. The SU-8 mold then undergoes a double-molding process to obtain a flexible polydimethylsiloxane (PDMS) negative mold structure that would facilitate the peeling of the negative mold from the more rigid final SMP structure.

The SU-8 negative mold was first fabricated by spincoating a negative photoresist, i.e., SU-8 at a spin speed of 1000 rpm for 30 s. After softbaking at 65 °C for three hours, the SU-8 was then left to rest for five minutes before undergoing a two-step angled exposure as shown in Fig. 3 through the use of a custom fabricated stage, with the UV illumination source being provided by a Model 60 Flood Exposure (ABM). The iron-oxide photomask used was of a periodic array of opaque squares with side and spacing dimensions respectively corresponding to the base and lateral pitch lengths of the wedge in Fig. 2(A). Following the exposure, the mold underwent a post-exposure baking step of three minutes at 65 °C and 10 min at 95 °C. After allowing the mold to rest at room temperature for five minutes, it was then developed in a SU-8 developer, and gently rinsed with isopropanol, obtaining the final negative mold structure.

The SU-8 mold was used to produce a positive PDMS wedge array that corresponds to the shape of the desired final structure. A double molding procedure then follows in order to cast the SMP into the desired shape, with a visual representation of the process shown in Fig. 4. The positive PDMS mold was cured with an 8:1 base prepolymer to curing agent mixing ratio in order to obtain a more rigid polymer compared to the standard 10:1 ratio, as to prevent the protruding PDMS wedges from sticking to adjacent wedges. Similarly, the negative PDMS mold was cured with a 13:1 mixing ratio to facilitate the peeling process by allowing the mold a higher strain before tearing occurs. The final SMP structure is cured on a glass backing layer in order to ensure the adhesive surface is level and parallel to the surface of the surrounding testing setup.

The particular formulation of thermosensitive SMP utilized in this paper is a thermally-activated type whose precursor is created by mixing a 1:1:1 ratio by molar mass of EPON 826, Jeffamine D230, and neopentyl glycol diglycidyl ether (NGDE), respectively [22]. This particular mixing ratio, referred to as "NGDE2", is one among several mixing recipes, chosen for its relatively convenient glass transition temperature (T_g) of about 40 °C, which is low enough to enable rapid heating and easy handling but high enough to prevent undesired reconstitution at room temperature, at which the adhesion tests are performed [22].

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