STICKING IN SPACE: MANUFACTURING DRY ADHESIVES AND TESTING THEIR PERFORMANCE IN SPACE ENVIRONMENTS

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ABSTRACT

Methods of fabricating adhesive with mushroom-shaped caps, offset-caps and hierarchical structures are described. The synthetic polydimethylsiloxane (PDMS) dry adhesives were tested in facilities at the European Space Agency using a 1.5 mm diameter quartz sphere and instrumented indentation equipment capable of operating at pressures of 1×10^{-5} mbar and temperatures between -50 and 75 °C. At a significance level of $\alpha = 0.05$, we found the adhesive forces to be unchanged for the temperature and pressure range tested, despite changes in PDMS properties. The lack of a significant effect of pressure on adhesion confirms that van der Waals forces, and not suction forces, are primarily responsible for dry adhesion. Endurance tests were performed on large-scale adhesive pads to simulate long term use by a robotic system. 12% degradation over 2000 adhesion cycles was observed. Best practices for indentation tests and endurance tests were discovered.

Key words: Dry adhesives; vacuum; temperature; space; outgassing; instrumented indentation.

1. INTRODUCTION

On the foot of a climbing gecko, small hairs called setae, each containing multiple spatulae, allow the gecko to adhere to surfaces [1]. Inspired by geckos, scientists have developed synthetic, dry adhesives [2, 3, 4, 5]. The gecko-like synthetic adhesives are passive mechanisms, do not outgas like pressure sensitive adhesives, and can potentially adhere to various materials and surfaces of many roughness scales. One application for dry adhesives is to integrate the adhesives with a climbing robot, as a method of adhering the robot to a climbing surface. In space applications, dry adhesives could be a method of adhering a robot to the exterior of an orbiting spacecraft [6]. To use synthetic dry adhesives in robotic applications, the long term performance of a dry adhesive patch must be studied. In addition, the performance of a dry adhesive in low pressure and at various temperatures must be understood.

A typical adhesion test involves indenting a spherical or flat probe into a sample of the adhesive, normal to the surface of the adhesive. The force and often depth are both recorded during the test. Various properties of dry adhesives have been characterized, for example repeated measurements in the same location show that the adhesive force of a dry adhesive decreases with use [7]. The adhesive performance of a gecko is affected by temperature [8] and humidity [8, 9, 10], however synthetic dry adhesives have a constant adhesion performance over a wide range of ambient humidity [11]. While the shear modulus of polydimethylsiloxane (PDMS), the substrate used for the dry adhesive in this study, changes with temperature [12], to the best of our knowledge dry adhesives have not been tested at temperatures other than room temperature. At low ambient pressures, the sticking ability of a synthetic dry adhesive has been shown by some authors to decrease [13, 14], and by others to remain unchanged [2, 15]. The lowest pressure tests of a dry adhesive, to the best of our knowledge, are at 10 mbar [2]: to better understand the usefulness of a synthetic dry adhesive in space, tests at lower pressures and varied temperatures are necessary.

In this paper, we first present a method for fabricating a synthetic dry adhesive (Section 2); modifications to the procedure for making offset caps or hierarchical structures are also described. Next, in Section 3, tests conducted in a simulated space environment are described. In Section 4, endurance tests on a large-scale patch of dry adhesive are presented. The paper concludes in Section 5 by drawing conclusions from the tests, and proposing future work.

2. FABRICATING A DRY ADHESIVE

2.1. Mould fabrication

To manufacture a mould for a mushroom-capped dry adhesive array (see Fig. 1a), the procedure described by Sameoto and Menon was employed [5]. First a 100 mm-diameter silicon wafer was selected. It was coated with a 50 nm/50 nm bi-layer of chrome/gold. This layer prevented the dry adhesive from sticking to the silicon wafer.

A layer of Polymethylglutarimide (PMGI) (1.5 µm) was spun and baked onto the bi-layer. Next a layer of AZ 9260 (18.5 µm) was spun onto the wafer and baked (step (a)I). The AZ 9260 was exposed (step (a)II) and developed (step (a)III); the PMGI was also sensitive to the developer, creating an undercut layer (step (a)III), which formed the mushroom caps in the dry adhesive.

To make a mould for adhesives with offset caps, a geometry that more closely resembles a natural gecko adhesive, a modified technique by Sameoto and Menon was employed [16]. This procedure is detailed in Fig. 1b). The wafer was coated in the chrome/gold bi-layer. A layer of PMGI (1.5 µm) was spun and baked onto the wafer. A layer of S1830 photoresist was spun onto the PMGI (step (b)I), and exposed and developed to mask the cap features in the PMGI. The wafer was exposed (step (b)II), making the unmasked PMGI sensitive to a future development step, and the S1830 was stripped. A layer of AZ 9260 (10.5 µm) was then spun onto the wafer and baked (step (b)III). A mask was aligned to be offset to the cap features, and the AZ 9260 was exposed and developed (step (b)IV). During the development step (step (b)V), the PMGI which had been previously exposed was rapidly undercut, forming the offset cap.

2.2. Adhesive fabrication

To fabricate a patch of PDMS adhesive using one of the moulds (see Fig. 1(a)IV-V or (b)VI-VII,) the procedure by Sameoto and Menon was followed [5]. PDMS was mixed 10:1 prepolymer to catalyst and degassed under light vacuum. The PDMS was then poured onto a mould. For a thin backing layer (0.5 mm), the mould was spun slowly to remove excess PDMS from the mould. The PDMS was again degassed in a light vacuum for 1 hour, and cured in an oven at 80 °C. Once cured, the adhesive was demoulded by hand, and the mould reused for future adhesive manufacturing.

In some instances, a hierarchical structure was desired. This was used, for example, during robotic integration, to increase the compliance of the dry adhesive. In this case, the method described by Li et al. was used [17]. A macro-post mould was made in PDMS using a laser cutter. Each macro-post had a width of 1 mm, a height of 3 mm, and was arranged in a square array with inter-post spacing of 3 mm. PDMS was mixed 10:1 and poured into the mould. Degassing under light vacuum ensured that any air bubbles were removed from the mould. The mould was baked for 3 hours at 80 °C and the PDMS macro-post array was demoulded by hand.

To manufacture the hierarchical structure, the PDMS adhesive was bonded to the macro-post array. The PDMS adhesive sheet was placed, posts downwards, on a flat PMMA sheet. A thin layer of Dow Corning 732 (silicone compound) was applied to top of the PDMS adhesive sheet, and the array of macro-posts were placed, posts down, onto the PDMS adhesive sheet. After 24 hours at room temperature, the structure was fully cured, resulting in a hierarchical structure.

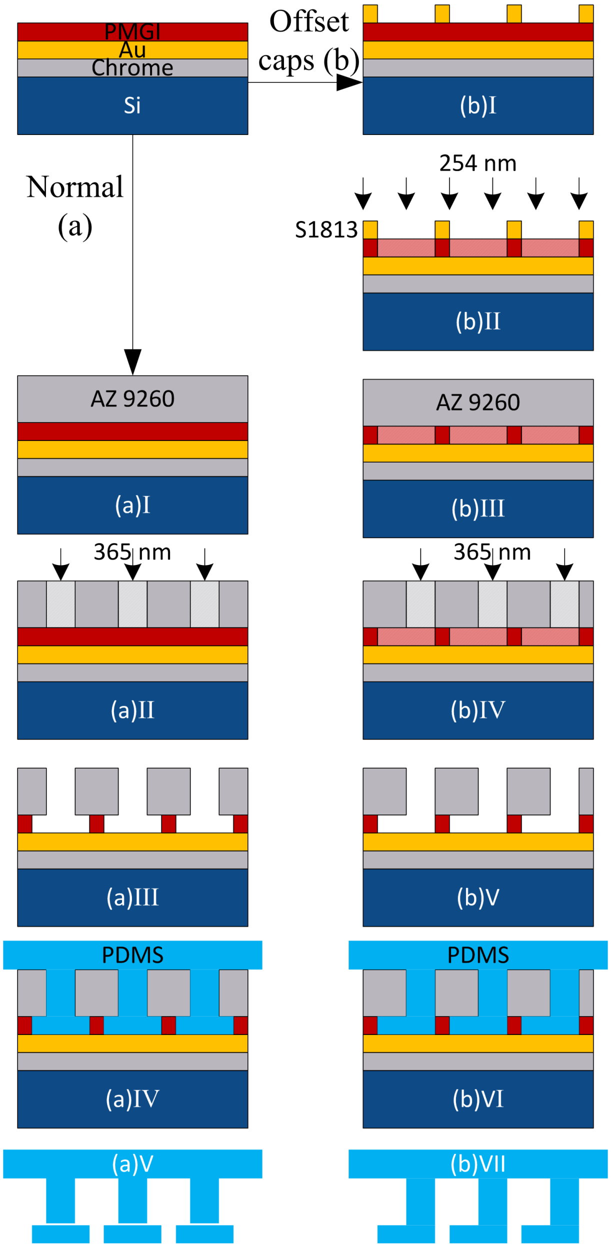

Figure 1. Fabrication procedure for dry adhesives. For the normal process, follow stream (a); for offset caps follow stream (b).

3. SPACE ENVIRONMENT TESTING

The most adhesive contact shape of a synthetic dry adhesive is a mushroom cap [18], however a visual examination of mushroom-capped dry adhesive shows that the caps resemble suction cups [2]. If a dry adhesive sticks to smooth surfaces with both suction and van der Waals force contributions, in a vacuum where the suction force cannot be supported, a decrease in overall adhesion is expected. Varying the temperature of a dry adhesive could

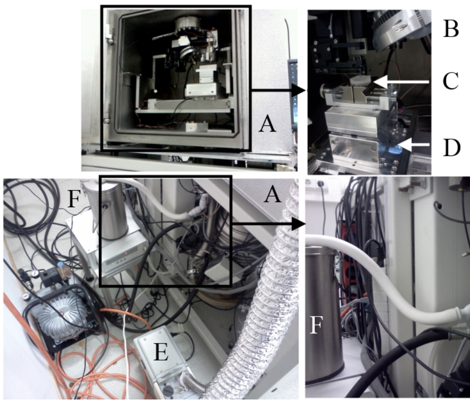

Figure 2. The NST equipment showing the vacuum chamber (A), microscope (B), sample (C), and 3-axis stage (D). At the rear of the NST is a roughing pump (E), dewar for liquid nitrogen (F) and diffusion pump (G).

also change its adhesion strength. The material properties of PDMS have been shown to change with temperature [12], and certain material properties have been shown to affect adhesion [11]. In this section, adhesion tests performed at various temperatures and low pressures to examine these potential effects are presented.

3.1. Experimental setup and considerations

Instrumented indentation apparatus called a NanoScratch Tester (NST, see Fig. 2), described by Henrey et al. [19], was used to perform adhesion tests of a synthetic dry adhesive. A 100 mm^2 piece of PDMS adhesive was bonded to a copper block using Bison High Temperature Silicone. A 1.5 mm diameter quartz sphere was bonded to a steel pin using cynaoacrylate for use as an indentation probe. During tests, the probe's speed and final depth were controlled, and both the probe's depth, and force on the probe were recorded at 100 Hz.

During a representative test (Fig. 3), the probe was indented into the sample and retracted, at least 20 times, to depths between 1 and 20 µm. Then the sample was moved and the indentations were repeated on a new section of the adhesive. Various sections of the adhesive were tested because it was observed that, due to manufacturing imperfections, the adhesive had slight variances over its surface.

During pilot testing, best practices were determined. While preload and detachment forces were in the mN-range, if the sample was not mounted to the copper block, it was observed to move slightly with the probe and affect measurements. This can be seen by examining the detaching profile of an adhesion test, as shown in Fig. 4, where the detachment profile of an unmounted sample is different than the profile of a mounted sample. As a second best practice, before testing, the adhesive patch was examined under microscope to find an area without any missing or damaged regions. As expected, in areas where posts or caps were missing, the adhesion force was substantially reduced.

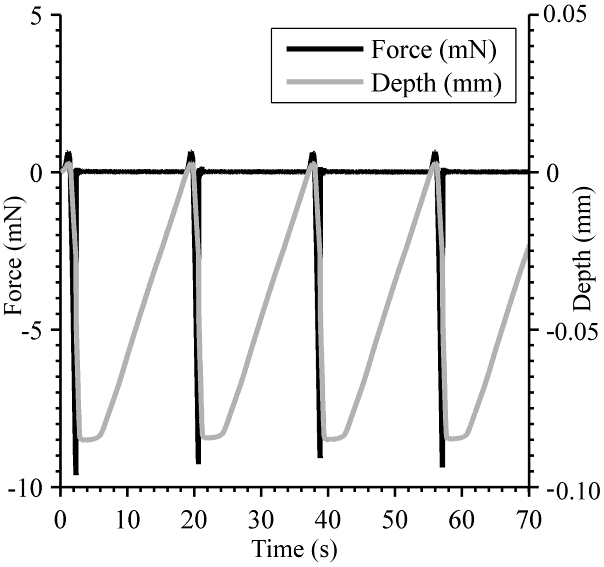

Figure 3. Data from four representative indentations during an adhesive test; positive forces are preloading forces, negative forces are adhesion forces.

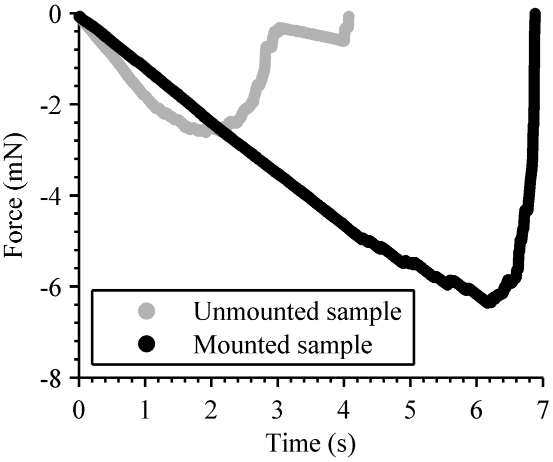

Figure 4. Plot showing the effect of adhering the sample to a copper block during testing. Without adhering the sample, there is a secondary detachment as the sample slightly lifts off the substrate during probe retratction. The overall adhesion is also lower when the sample is not adhered. In this plot, only the forces during probe retraction are presented. Adhesion forces are negative forces.

3.2. Effect of vacuum

In one test, the effect of low pressure was studied [19]. A sample was tested at 20 locations in each of low pressure (1×10^{-5} mbar) and at atmospheric pressure (980 mbar) environments. The total number of adhesion data points collected was 488 at 1×10^{-5} mbar, and 408 at 980 mbar. To compare the two data sets, the model developed by Schargott et al. [20], and written as Eq. 1, was fit to the data:

$$f_a = 2\sqrt{f_{a(sat)} f_p} - f_p, \tag{1}$$

where f_a is the adhesion force, f_p is the preload force and $f_{a(sat)}$ is the saturation adhesion. The saturation adhesion is a measure of the maximum expected adhesion force, independent of the preload force. For each data set, a fit was computed using linear regression, as shown in Fig. 5a, allowing $f_{a(sat)}$ and its 95% Confidence Interval (CI) to be estimated for low and atmospheric pressure tests. In low pressure, the estimate was 20.02 mN and in atmosphere the estimate was 19.81 mN. The confidence intervals of the estimates (shown as error bars in Fig. 6a) overlapped, indicating, within the pressure range and accuracy of this test, that adhesion was not affected by the ambient pressure. This means that, for the adhesive geometry used, there was no discernible contribution of suction to adhesion.

Using the same data set, the effect of vacuum on a material property (the effective Young's modulus, E^*) of the PDMS was studied. This was possible because the indentation apparatus measured both force and displacement, allowing the method proposed by Greiner et al. [11] to be used to estimate E^* for the PDMS adhesive in atmosphere and low pressure. Using Eq. 2:

$$f_p = \frac{4}{3} E^* \sqrt{rs^3}, \tag{2}$$

where r is the indenter radius (0.75 mm) and s is the indentation depth, E^* was estimated to be 2.131 MPa ($n = 488$, $\sigma = 0.1324$) in low pressure and 2.258 MPa ($n = 408$, $\sigma = 0.2122$) in atmosphere. Graphically, in Fig. 5b, the fit of Eq. 2 to the data is shown. In Fig. 6b, the estimates of E^* and its 95% CIs are shown. A t-test showed these two estimates to be different, with 95% confidence. The changes were expected to be due to outgassing of the PDMS, or the reduction of humidity in the low pressure environment, or a combination of the two.

3.3. Effect of thermal vacuum

To conduct measurements at temperatures other than room temperature, a thermal microscope stage was used with the indentation apparatus, as described by Henrey et al. [19]. To avoid condensation and frosting on the sample at low temperatures, low pressures (1×10^{-5} mbar) were used in conjunction with all the temperatures tested. Testing at various temperatures was difficult because sample heating and cooling also affected the measurement ability of the indentation apparatus. Indenting a quartz sphere onto fused silica (two hard materials, so the indentation depth was predicted to be approximately zero) helped determine the uncertainty in measurement introduced by the heated or cooled sample. This uncertainty was estimated to be 0.6 nm mN^{-1} °C^{-1}.

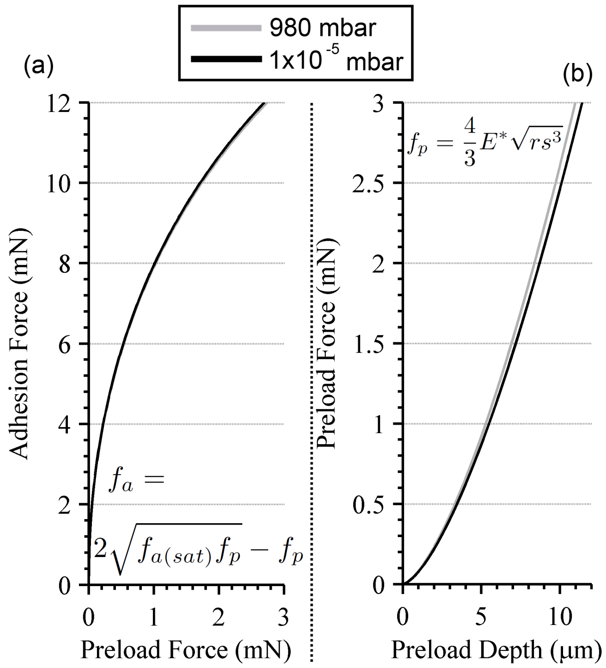

Figure 5. A fit is applied to adhesion data (a), and preloading data (b). In (a), Eq. 1 is used used to model the saturation adhesion in both low and atmospheric pressures. In (b), Eq. 2 is used to compute an effective Young's modulus for PDMS in both low and atmospheric pressures.

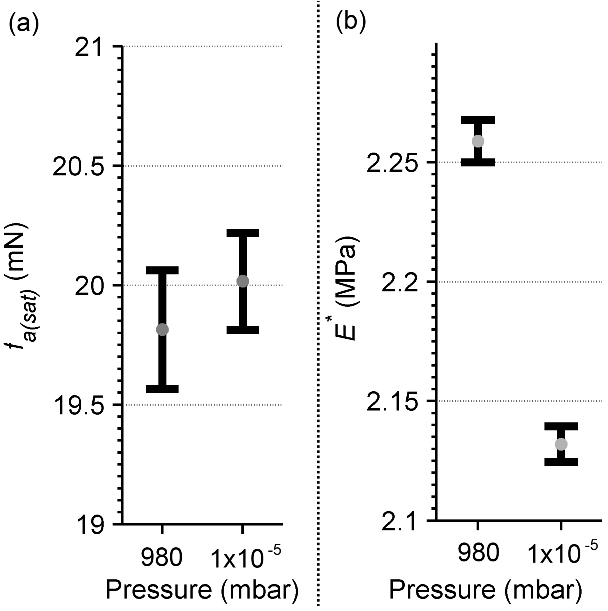

Figure 6. Estimates and 95% confidence intervals for the saturation adhesion (a) and effective Young's modulus (b) in low and atmospheric pressure indentation experiments.

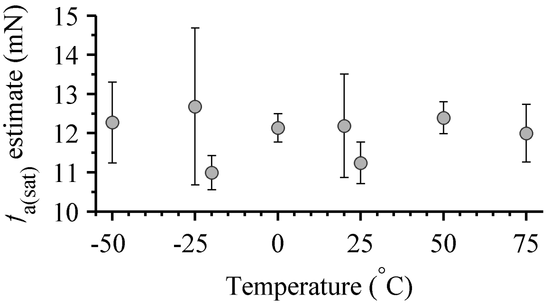

Figure 7. The saturation adhesion for each test (temperatures ranging -50 to 75 °C) is estimated, and its 95% CI is indicated by black error bars.

Eight different temperatures from −50 °C to 75 °C were tested, through a total of 219 measurements. As with the ambient pressure tests, the saturation adhesion and its 95% CI were estimated for each temperature tested. Because there were fewer tests and more categories, the statistical power was reduced (in comparison to the ambient pressure tests), and CIs are wider. However the results (see Fig. 7) showed no relationship between temperature and adhesion. It was inferred, to the power of this test, that temperature had no effect on the adhesion of a dry adhesive over the range −50 °C to 75 °C.

4. ENDURANCE ADHESIVE TESTS

While a test with a 1.5 mm diameter indenter is useful for investigating environmental factors affecting synthetic dry adhesives, practical adhesives are typically larger, and require larger contact surfaces for testing. For a practical dry adhesive (for example an adhesive used with a wall-climbing robot), it is necessary to understand how its adhesive performance changes over time. We used a linear stage to bring a flat microscope slide into contact with a dry adhesive patch (which was 300 mm, the size of adhesive required for the foot of a climbing robot), and recorded forces with a strain-gage load cell. To conduct a long-term adhesive test with a PDMS adhesive, two best practices were developed: keeping the preloading force constant, and keeping the preloading time constant. These are shown visually in Fig. 8a, where longer preloading times result in slightly larger detaching forces, and in Fig. 8b, where it is not possible to study the long term behavior of an adhesive as the preload force is drifting.

An endurance test of a dry adhesive patch (Fig. 9) shows interesting results for integration with a practical system undergoing multiple cycles of attachment and detachment (for example a robot). Degradation of the adhesive strength over time (in this case 12% over 35 hours, or 2000 attaching and detaching cycles) may be due to dust accumulation, which will have to be dealt with by autonomous robotic systems. This test was not performed at low pressure or various temperatures, because the force measuring equipment for larger sample sizes was not capable of operating in vacuum.

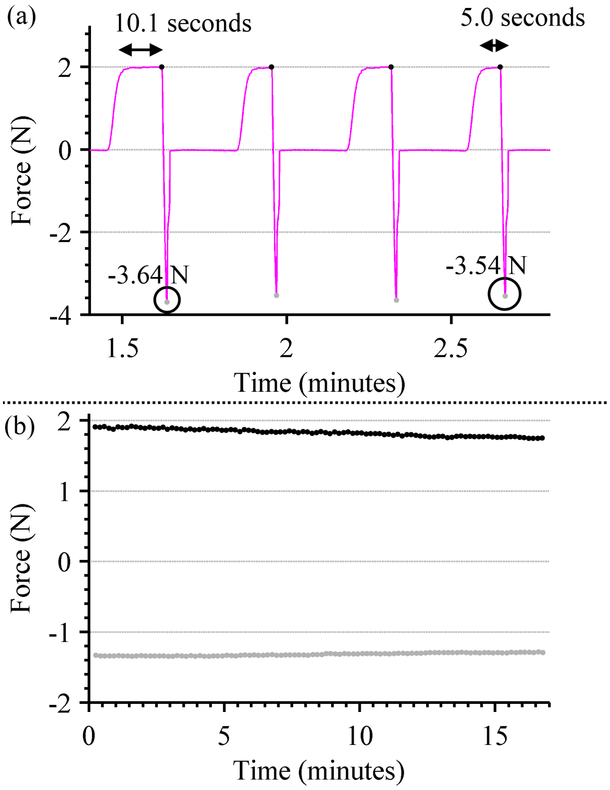

Figure 8. Two best practices for endurance testing of PDMS adhesives are shown. In (a), it can be seen that time of preload affected the peak adhesion forces, and therefore must be carefully controlled during measurement. In (b), the summary of 100 tests is shown, where black dots are the preload force, and grey dots are adhesion forces. The black dots drift over time, making it difficult to understand the effect of time on adhesion (grey dots).

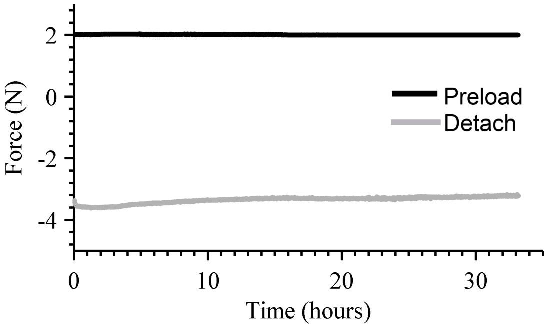

Figure 9. While the preload forces (black) are held constant at 2.0 N, the adhesive force (grey) decays over 2000 cycles by about 12%.

5. CONCLUSIONS AND FUTURE WORK

In this paper, fabrication procedures for manufacturing mushroom-capped, offset, and hierarchical synthetic, dry adhesives were outlined. The manufacturing methods described were low-cost and created reusable moulds for repeatable fabrication. Samples of the dry adhesives were tested in low pressure (1×10^{-5} mbar) and temperatures from −50 °C to 75 °C without showing any loss of adhesion compared to samples tested at room temperature and atmospheric pressure. This suggests that suction does not play a role in the adhesion of these dry adhesives, and that the dry adhesives have potential to work in space environments. Small changes in sample properties (effective Young's modulus) were observed as a result of placing the samples in vacuum. An endurance test on a larger patch of dry adhesive was also performed. This test used larger apparatus than the indentation apparatus. A long term degredation of 12% was observed over 35 hours of testing. Future work includes longer duration adhesive tests in low pressure, as well as endurance testing of adhesives in low pressure or at extreme temperatures.

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