

EXPERIMENTAL CHARACTERIZATION OF ADHESION HYSTERESIS IN SOFT ELASTOMER CONTACTS

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1. Introduction

Mechanical characterization of adhesion is crucial in the development of nearly all pressure sensitive adhesives (PSAs) [1] and emerging bioinspired adhesives [2]. Adhesion is frequently characterized via probe tack and peel tests that provide a measure of the strength and adhesion energy, respectively. For PSAs coated onto flexible plastic or paper substrates to form tapes or labels, the peel test is frequently used for mechanical characterization. In the peel test, a thin strip comprised of a backing layer and an adhesive is peeled at a controlled speed from a substrate and the force during peeling is measured. Through analysis of the test data, the adhesion energy (i.e., interface toughness with units of J/m^2) can be quantified. Methods for performing and reporting data from peel tests are summarized in ASTM standards (ASTM D903, ASTM D3330) and there are countless papers discussing the complexities of analyzing data from peel tests (e.g., [3]). Peel tests, as well as most other standard measurements of the mechanical behavior of adhesives, quantify the adhesion during separation of the interface. When an interface is separated, the adhesion energy that is measured is a function of the both the interfacial bonding, which can be described in terms of the thermodynamic work of adhesion, as well the energy dissipated due to deformation of material at the interface. PSAs are generally based on materials that are highly viscoelastic, thus the adhesion energy is typically dominated by the energy dissipated in deformation of the adhesive rather than the energy needed to break the interfacial bonds.

In order to understand and improve the performance of adhesives, it is important to be able to quantify the various contributions to interfacial adhesion. One approach to developing a better understanding of the mechanics of interface adhesion is to characterize the behavior of the contact during both formation and separation of the interface. The adhesive deforms significantly less during formation of the interface than during separation, thus mechanical measurements of adhesion during contact formation can be used to understand the non-viscous contributions to adhesion. Previous work, in which JKR tests (sphere on flat contacts) were used to quantify both adhesion and separation behavior, has shown that many materials systems exhibit strong adhesion hysteresis [4]. With the exception of the JKR test, there are few experimental approaches that allow for detailed characterization of the adhesion during both the formation and separation of an interface. Here, we present a blister contact test that can be used to characterize the adhesion energy as the contact is formed and then subsequently separated. The blister contact test is well-suited for characterizing adhesion of PSAs coated on flexible backings as well as elastomers that are used in many bioinspired adhesives. This paper briefly summarizes the blister contact test (BCT) and provides example measurements performed using the technique.

2. The Blister Contact Test

Fig. 1 illustrates the basic configuration of the BCT. A thin membrane of thickness, h , is clamped along a circular boundary of radius R and pressurized by q , to deform the membrane. If the deformation of the membrane is sufficiently large, it will contact the stiff plate that is positioned a distance δ from the

undeformed membrane and form a contact area with radius b . The radius of the contact that forms is a function of the geometry, elastic properties, applied pressure and the adhesion between the membrane and the plate. We have used the BCT to characterize elastomer adhesion, in which the membrane is simply an elastomer sheet (e.g., a silicone rubber with modulus of 1-2 MPa), as well as the adhesion of PSAs coated on backing layers made of paper or polymer sheets. The stiff contact plate forms one side of the interface and we have previously used plates made of glass, coated glass, and various polymers (e.g., acrylic).

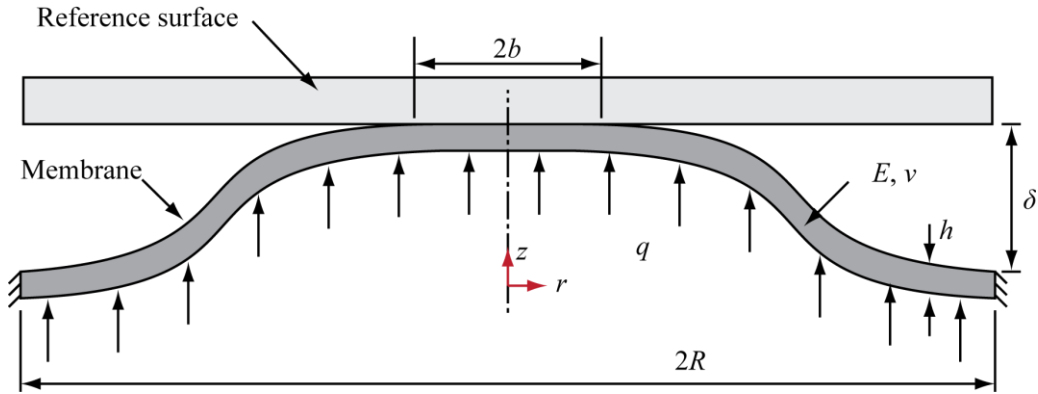


Figure 1. Schematic of blister contact test.

Equilibrium of an adhesive contact requires that the strain energy release rate (units of J/m^2) at the bond/crack front be equal to the interfacial adhesion energy (i.e. toughness). Thus, the adhesion energy during both the formation and separation of the contact can be characterized in terms of the strain energy release rate. In general the strain energy release rate of PSAs and other polymer adhesives are both time and rate dependent. The BCT is a pressure controlled test and the loading/unloading times can be varied to achieve different loading rates in the test and characterize rate dependent behavior.

The strain energy release rate is calculated from the applied pressure, q , and measured contact radius data, b , with knowledge of the membrane thickness, h , elastic properties, E and ν , membrane radius, R , and separation gap, δ . There are several mechanics models in the literature that can be used to calculate strain energy release for certain limiting cases [5, 6] of the BCT. The deformation of the membrane can be quite complex and involve multiple deformation modes, such as membrane stretching, bending, and transverse shear. As such, we have found that finite element modeling, which captures all of these deformation modes, is convenient for calculating strain energy release rates from test data [7]. For the purposes of this paper, we simply want to emphasize that

$$G = f(q, b, \delta, R, h, E, \nu) \quad (1)$$

and is calculated from a mechanics model based on values measured in the BCT. If either the out-of-plane or the in-plane displacements of the membrane are measured during the test, the elastic properties of the membrane can be determined from data measured in the test as well.

A typical BCT experiment is performed by increasing the pressure at a constant rate and monitoring the initial contact of the membrane with the plate and the subsequent increase in contact area that occurs as

the pressure is increased. The pressure is then held for a fixed period of time to monitor time-dependent behavior of the adhesive. Finally, the pressure is decreased at a constant rate and the reduction in contact radius with decreasing pressure is measured. The contact radius is determined via optical imaging and the pressure is controlled and measured throughout the test, thus a set of data for a single test consists of contact radius as a function of pressure. The loading rates, hold times, pressures, and specimen dimensions used in the experiment depend on the specific material being investigated.

3. Experimental Setup

The results reported in this paper were obtained in experiments performed on the custom BCT apparatus shown in Fig. 2. The specimen is clamped between two precision washers with the adhesive surface of the specimen facing the reference surface. The thickness of the upper precision washer defines the separation gap, δ , between the specimen and the reference surface. The specimen is positioned over a small chamber that is pressurized with dry nitrogen. The pressure is controlled via an electronic pressure regulator and is also measured independently via a pressure sensor. The contact is imaged with a CCD camera fitted with a telecentric lens. If the reference surface is transparent, the contact is illuminated through the reference surface, as shown, however it can also be illuminated from the camera side.

In the setup used here, the membrane has a radius of 9.5 mm, δ is between 0.5 and 1.5 mm (depending on the specimen), and pressures up to 15 kPa are applied. Images of the contact are acquired digitally and then analyzed using a custom image processing script to extract the contact radius.

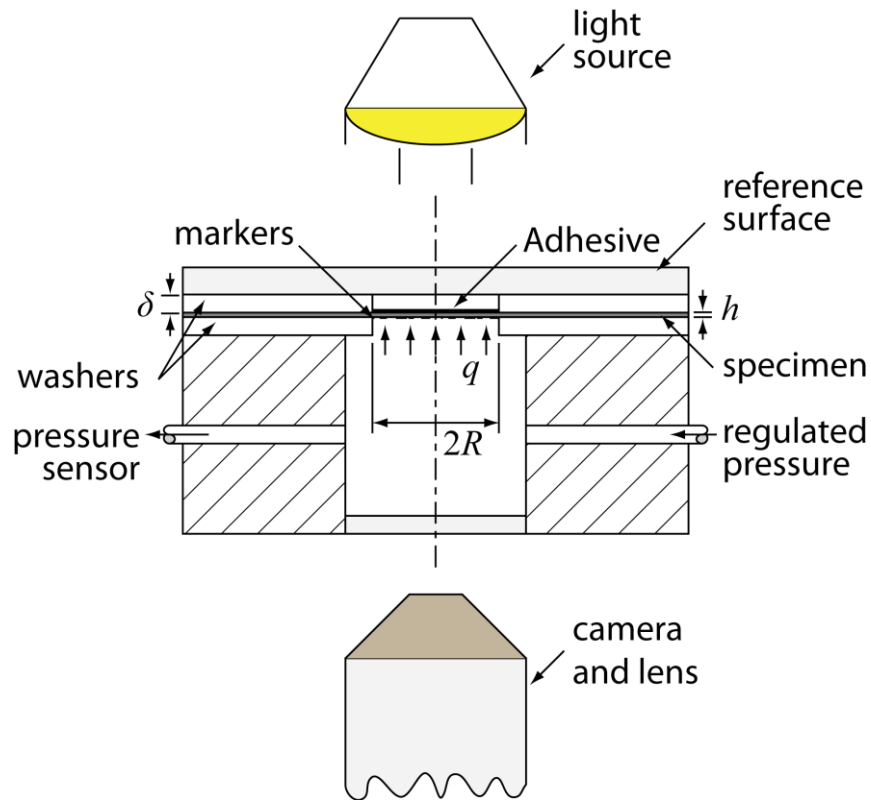


Figure 2. Experimental setup to for implementation of the blister contact test.

4. Results and Discussion

To demonstrate the technique, we first show data for adhesion between a 1 mm thick polydimethylsiloxane (PDMS) specimen and a glass reference surface. PDMS is frequently used in bioinspired adhesives [2] and semiconductor transfer printing processes [8], thus there is significant interest in its adhesion properties. Fig. 3 shows an example of an applied pressure sequence, measured contact radius and an image of the contact area in the test. Fig. 4 shows the measured contact radius as a function of applied pressure for a similar PDMS specimen. The results in Fig. 4 include data for three different depressurization rates.

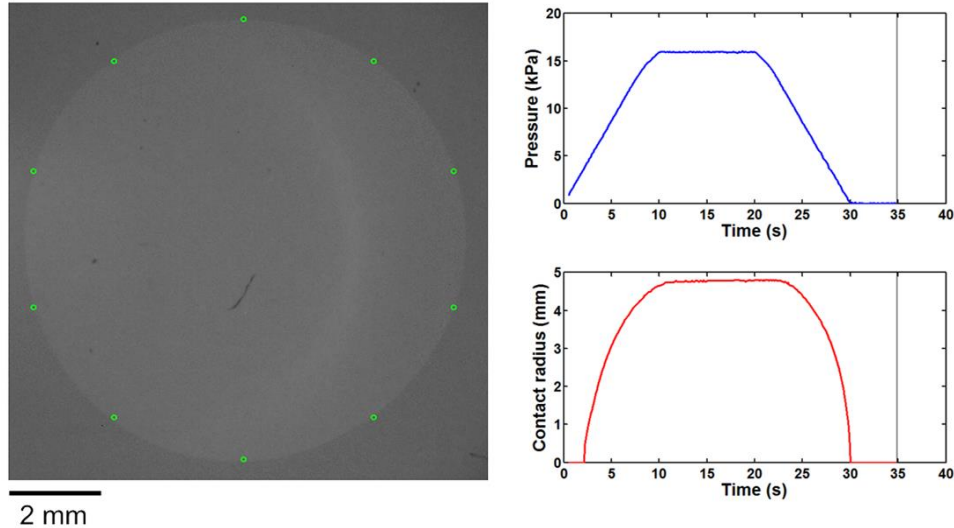


Figure 3. Example data from a BCT test. Right: Pressure and contact radius as a function of time. Left: An image of the contact area at time = 15 s (the small circular markers on the contact front are placed there by the image processing algorithm).

The results in Figure 4 demonstrate that there is hysteresis between the formation (i.e. advancing contact) and separation (i.e. receding contact) of the interface. At a given pressure, the contact radius is smaller during formation of the contact than it is upon separation. This suggests the strain energy release rate is lower during formation of the interface than it is during separation. This is consistent with the aforementioned idea that the separation is more strongly affected by viscous dissipation at the interface. For the three separation curves, the contact radius at a given pressure increases with decreasing separation time (i.e., faster separation rate). This indicates that the strain energy release rate increases with separation rate. Similar increases in strain energy release rate with faster separation rates are often observed in peel tests.

The color of the markers in Fig. 4 indicates the speed of the bond/crack front during the test. While the pressure rate, which is controlled in the experiments, influences the speed of the bond/crack front it does not fully determine the rate. As such, the speed of the bond/crack front is determined from the contact radius values that are measured as a function of time in the test. The strain energy release rate can be calculated from the data as described in Sec. 2. A strain energy release rate can be calculated for each data point in Fig. 4, which allows strain energy release rate to be determined as function of bond/crack front speed during both advancing and receding contact. Such calculations are beyond the scope of this conference paper and are summarized elsewhere [7].

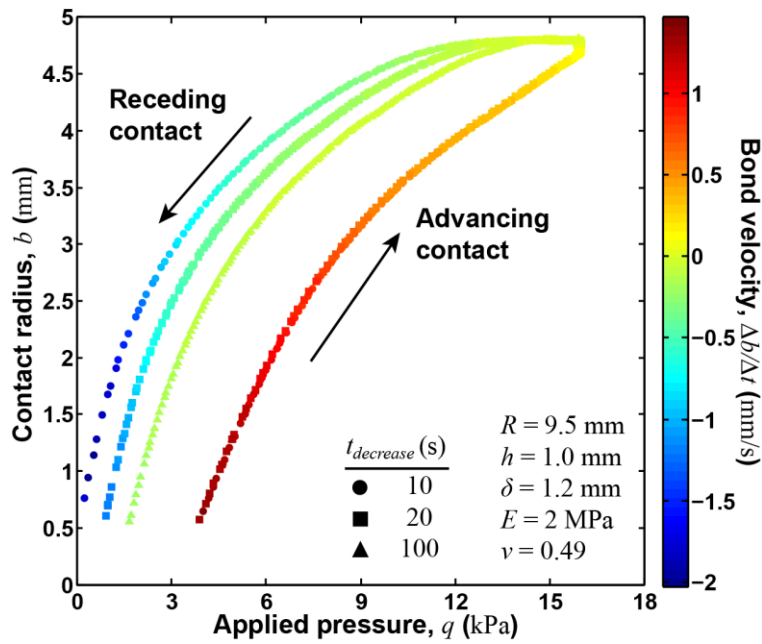


Figure 4. Example data from a BCT test of a PDMS membrane in contact with a glass plate.

We have also used the BCT to characterize the adhesion behavior of PSAs coated on flexible backings, including polymer and paper substrates. Figure 5 shows images of a PSA coated on a paper substrate in contact with two different reference surfaces. In the image on the right, the specimen is in contact with a stiff glass surface, while the image on the left shows a similar specimen in contact with a thin PDMS layer coated on a glass surface. These specimens are comprised of a paper substrate (~ 80 μm thick) coated with a ~ 15 μm thick PSA layer. When the specimen is contacted with the stiff glass plate there is only partial contact due to the roughness of the paper. The compliance of the PDMS layer in the test shown on the left accommodates the roughness and allows more complete contact. These images show the value of imaging the contact during the BCT as it provides insight into the true contact area. Full tests characterizing PSA adhesion during both contact formation and separation will be discussed in the conference presentation.

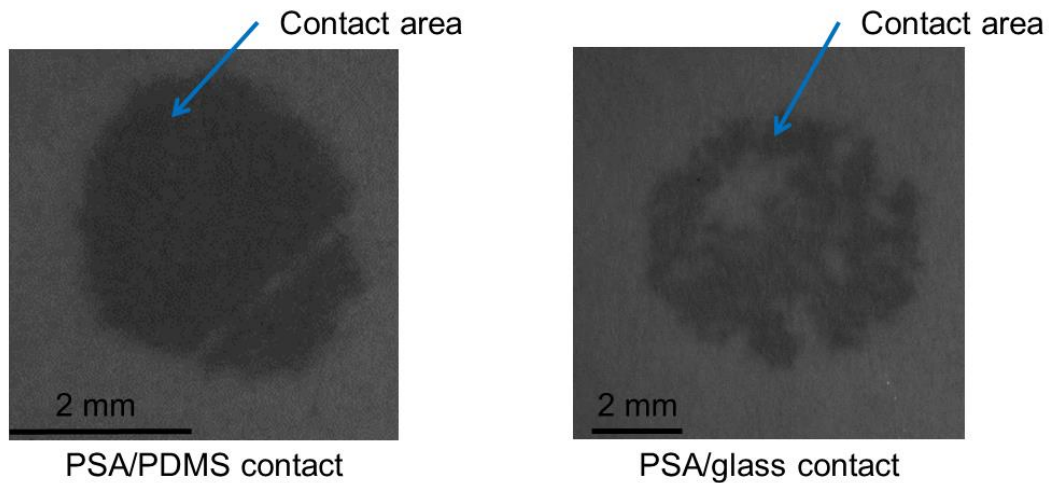


Figure 5. Images acquired during BCTs of a PSA (coated on paper) in contact with two different reference surfaces.

5. Summary

We have described a blister contact test for characterizing the strain energy release rate during both formation and separation of interfaces. The hysteresis between the advancing and receding contacts in the tests results from the fact that the separation behavior is more strongly influenced by rate-dependent viscoelastic deformation at the interface. The combination of adhesion energy measurements of contact formation and separation can allow for a more detailed understanding of the mechanics of adhesion of various material systems.

Acknowledgements

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