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BLISTER CONTACT TEST FOR CHARACTERIZING PRESSURE SENSITIVE ADHESIVES COATED ON THIN SHEETS

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Introduction

There are many applications and products that involve pressure sensitive adhesives (PSAs) coated on thin flexible substrates. For example, postage stamps and mailing labels consist of a layer of PSA coated on thin sheets of paper. While PSAs are commonly used in a variety of commercial products, it is still difficult to fully characterize the adhesive behavior of PSAs on thin substrates. Characterizing the adhesive properties is challenging because the substrate properties often influence the measured properties of PSAs in many test configurations. For instance, the properties measured from a peel test [1] often depend on the stiffness of the backing layer. Alternatively, double cantilever beam tests can be employed, but specimen preparation with adhesive coated sheets is often difficult [2]. In the present work, we present a blister contact test to measure the adhesive properties of PSAs coated on thin flexible sheets. The basic principle of the proposed test is a clamped circular specimen subjected to uniform pressure loading that controls contact between the adhesive coating and a stiff reference surface. The test is different from the blister test [3, 4] which uses applied pressure to separate a film from a substrate. In the blister contact test presented here, an adhesive contact is formed and then separated by modulating the applied pressure. This allows measurements of work of adhesion during contact and the interface toughness during separation. This test has some similarities to other tests [5, 6, 7]. This paper describes the mechanics and implementation of the blister contact test and demonstrates its application to characterizing a PSA coating on a thin paper substrate.

Theory

During the course of a single test, we perform a bulge test (Fig. 1a) to establish the elastic properties of the specimen and a blister contact test (Fig. 1b) to measure the adhesive properties of the specimen. Bulge test data is collected before the membrane makes contact to the reference surface and the blister contact test data is obtained after contact is made. The specimen is clamped at the edge and a uniform pressure is applied on the bottom of the specimen. During the bulge test, the deformation of the pressurized membrane is measured and the elastic response is determined [8]. This elastic response data is needed for analyzing the blister contact test data. If the deflections of the specimen are small, the data can be analyzed by linear plate theory [9]; for this, the out-of-plane displacement profile of the bulge is

$$w(r) = \frac{p}{64D}\left(b^2 - r^2\right)^2 ; D \equiv \frac{Eh^3}{12\left(1-\nu^2\right)} \tag{1}$$

where b is the radius of the specimen, p is the pressure, r is the radial position, D is the flexural rigidity as a function of the elastic modulus E, Poisson's ratio ν and specimen thickness h. In the case of maximum deflections greater than approximately the specimen thickness, membrane stresses develop and alter the deflection response. In these cases, we use large deformation finite element simulations to relate the measured pressure and deflection data to the elastic properties.

In the blister test configuration (Fig. 1b), the adhesive makes contact with a stiff reference surface positioned a short distance from the specimen and limits the deflection to δ. Contact between the specimen and reference surface advances as pressure increases. Under the assumptions of linear plate theory, the strain energy release rate, G, is:

$$G = \frac{1}{512b^4 D\left[\left(\alpha^2-1\right)^2 - 4\alpha^2 \ln^2 \alpha\right]^2} \times \left\{ \begin{array}{l} qb^4\left[\begin{array}{l}\left(\alpha^2-3\right)\left(\alpha^2-1\right)^2 \\ -2\ln\alpha\left(1+2\alpha^2-3\alpha^4+4\alpha^4\ln\alpha\right)\end{array}\right] \\ +64D\delta\left(-\alpha^2-1+2\ln\alpha\right)\end{array}\right\}^2 ; \alpha \equiv \frac{a}{b} \tag{2}$$

When out-of-plane deflections are large, membrane stresses develop and Eq. 2 is no longer valid. In the event of very large deflections in the specimen, the strain energy

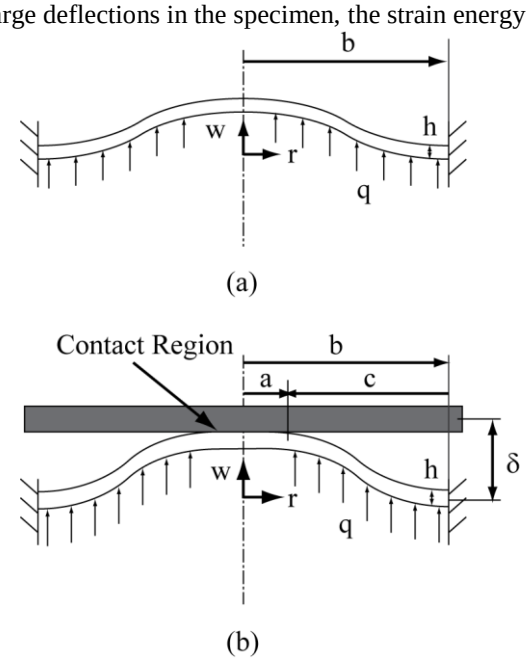

Figure 1. Schematic of the two tests performed: (a) bulge test without contact; (b) blister contact test with contact radii between 0≤r≤a.

release rate can be obtained in analytical form by considering the membrane stresses only [7]. Finite element modeling is primarily used in the current work to calculate G of the blister contact test as the experimental cases fall in a regime where a combination of bending and membrane stresses are important. Alternatively, a numerical model making use of the minimization of total energy can be used to solve the nonlinear problem [6]. Specifically, the FE model uses the virtual crack closure technique [11] and the commercial software Abaqus® [12]. The strain energy release rate is assumed to be equal to the work of adhesion when the contact is being formed and equal to the interface toughness (i.e., work of separation) when the contact is being separated. In general for PSAs, the values of work of adhesion and interface toughness are different.

Experiment

The experiment requires the ability to control and measure pressure as well as characterize the deformed profile and contact area of the sheet. Pressure is supplied from a compressed nitrogen tank, a regulator modulates the pressure, and a commercial sensor is used to measure the pressure. The full-field deformed profile is measured during the test using shadow moiré [13]. A camera with a zoom lens is used to image the moiré fringes and measure the contact area during blister test. The reference surface is a silanized glass coverslip and is transparent to allow imaging of the contact. The specimen is a sheet of paper coated with a thin layer (~0.01 mm) of pressure sensitive adhesive. Table 1 lists the dimensions of the specimens and key parameters in the experiment. The sheet is sandwiched between two identical circular arbor shims to hold the specimen in a clamped configuration during the test. The thickness of the top shim determines the spacing between the sheet and glass coverslip, and thus defines maximum deflection, δ, of the specimen in the contact test.

After the applied pressure is sufficient to bring the specimen into contact with the glass, the position of crack/bond front is monitored optically. Over a typical test the applied pressure is increased to cause the contact area between the specimen and the glass surface to increase. The pressure is increased until the contact radius reaches approximately 15% of the radius of the specimen. Then the applied pressure is decreased to separate the contact. Once the pressure is decreased sufficiently, the sheet will typically pull out of contact completely. When the interface toughness is larger than the work of adhesion, as is typical, the contact area does not immediately begin to decrease when the pressure is decreased from its maximum value. Rather, the contact stays fixed until the decrease pressure causes a large enough G to cause the contact to recede. The key data that is collected from the blister contact test and analyzed is the contact radius as a function of pressure.

Results and Discussion

Fig. 2 shows images of the contact of a pressure cycle for the test data shown in Fig. 3. The specimen is circular in shape, but as the light illuminates from one side of the specimen, the shadow of the edge covers part of the blister. The contact area is the darker region near the center of each image. The contact area in this case is not axisymmetric, but rather somewhat elliptical. As such, an effective radius is used when analyzing the data using the model, which is currently limited to the axisymmetric case.

Fig. 3(a) shows the measured contact radius as a function of pressure for a typical specimen. At point **A**, the specimen is first brought into contact with the glass surface. As the pressure increases, the contact size increases and eventually reaches the maximum size during the test at point **B**. The strain energy release rate is equal to the work of adhesion between point **A** and **B** because the contact front is advancing during this time. Between points **B** and **C**, the contact size remains constant as the applied pressure decreases, because the interface toughness is larger than the work of adhesion for this specimen. From point **C**, the contact size begins to decrease until the specimen detaches completely from the surface at point **D**. The strain energy release rate during stage **C**-**D** when the contact is receding is taken to be equal to the interface toughness.

Fig. 3(b) shows the corresponding strain energy release rate for the data in Figure 3(a). The strain energy release rate was calculated via finite element analysis

Table 1. Dimensions for sample experiment

Parameter name	Dimension
Blister Radius b	9.53 mm
Shim Thickness δ	0.79 mm
Specimen Thickness h	0.10 mm
Approx. Applied Pressure Range q	0 – 32 kPa
Approx. Contact Radius Range a	0 – 1.6 mm
Approx. Flexural Rigidity D	1.1e-4 N-m

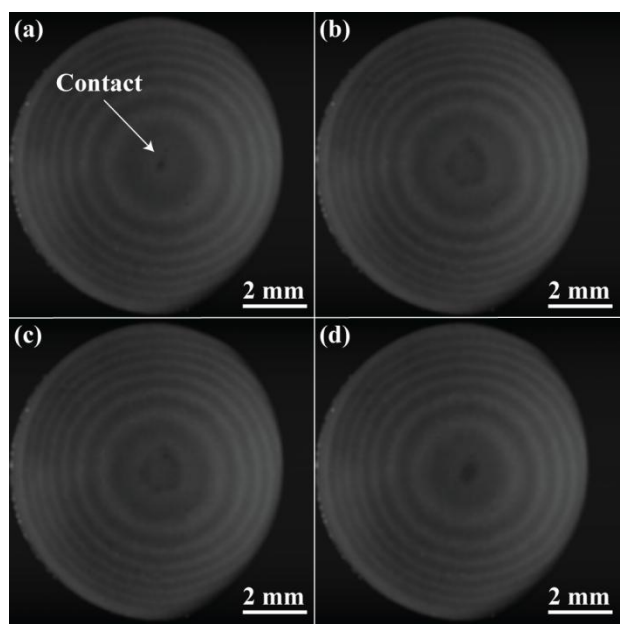

Figure 2. Typical loading sequence from a blister contact test: (a) contact initiated at the center at 26.7 kPa; (b) maximum contact area at 30.7 kPa; (c) contact area same as (b) at 27.6 kPa; (d) contact area just before pull-off at 21.0 kPa.

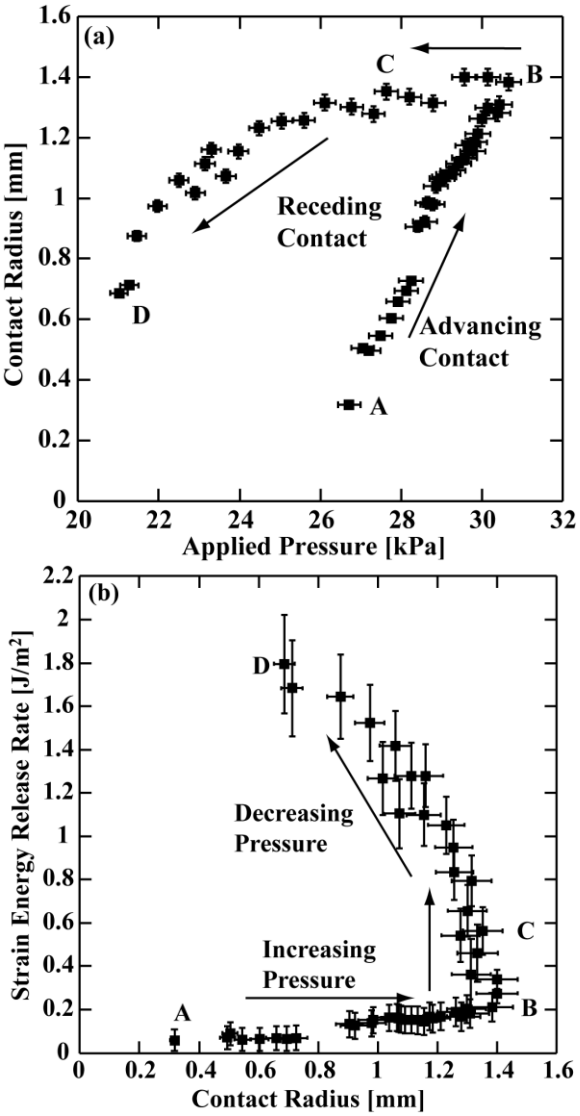

Figure 3. Data from a typical experiment: (a) measured contact radius versus applied pressure; (b) strain energy release rate versus contact radius.

based on properties obtained in the bulge test and the data shown in Fig. 3(a). The strain energy release rate during advancing contact (between **A** and **B**) increases slightly as pressure increases. Between **B** and **C**, the strain energy release rate increases while the contact radius remains constant. After point **C**, the contact radius begins to decrease and the interface toughness increases significantly. The significant increase in *G* during the separation phase is surprising, but may be due to several factors, including bridging at the interface and non-uniform adhesion across the contact area. Non-uniform adhesion, with higher adhesion at the center, may be due to the fact that the central region is in contact for a longer period time during the test. It is well-known that PSA adhesion depends on contact time [14].

Repeated tests on the same specimen show that the shape of the contact area is repeatable. Three consecutive tests were performed over a period of two days on the same specimen. The environmental conditions varied by three percent in relative humidity and less than one degree Celsius. In these tests, the minimum pressure to achieve contact is lower in the subsequent tests but the contact shape evolution and pull off pressure remain the same. The repeatability of the elliptical shape of the contact area suggests that the mechanics of specimen may be causing the shape. For example, the anisotropic properties of the specimen may influence contact shape; similar noncircular contact shapes due to anisotropic properties have been shown previously [15]. Our future work will include a three dimensional finite element model that can capture the anisotropic elastic properties and the asymmetric contacts. Additional experimental and modeling results will be provided in the presentation.

Conclusions

A blister contact test for characterizing the adhesion between a PSA coated on thin flexible substrate and a reference surface is described and results are reported. An applied pressure is used to control contact between the PSA and a reference surface. A fracture mechanics analysis is used to extract the work of adhesion and interface toughness from the test data. Data from a typical test was shown to illustrate the technique. In this test, the measured interface toughness is significantly larger than the work of adhesion and the shape of the contact area is noncircular.

Acknowledgements

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