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Polyacrylamide hydrogels. III. Lap shear and peel

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ABSTRACT

Lap shear and peel are common tests for soft materials. Their results, however, are rarely compared. Here we compare lap shear and peel as tests for measuring toughness. We prepare specimens for both tests by using stiff layers to sandwich a layer of a polyacrylamide hydrogel. We introduce a cut in the hydrogel by scissors, pull one stiff layer at constant velocity, and record the force. In lap shear, the force peaks and then drops to zero, the cut grows unstably through the entire hydrogel, and the peak force is used to determine toughness. In peel, the force peaks and then drops to a plateau, the cut grows in the hydrogel in steady state, and the plateau force is used to determine toughness. Our experimental data show that the average values of toughness determined by lap shear and peel are comparable. The peak forces in both tests scatter significantly, but the plateau force in peel scatters narrowly. Consequently, toughness determined by lap shear scatters more than toughness determined by peel. We hypothesize that the peak forces scatter mainly due to the statistical variation of the cuts made by scissors, and test the hypothesis using two additional sets of experiments. First, after a cut is made by scissors, we pre-peel the specimen to extend the cut somewhat, and then measure toughness by lap shear and peel. The peak force in lap shear scatters less, and the peak force in peel is removed. Second, we prepare cuts using spacers of various thicknesses, and find that the peak forces in both lap shear and peel vary with the thickness of the spacer. These findings clarify the use of lap shear and peel to characterize soft materials.

1. Introduction

When we spread water by hand on a plastic film, the plastic film can adhere to another plastic film. Shearing them apart is hard, but peeling them apart is easy (Video 1). This everyday experience illustrates two tests, lap shear and peel, commonly used to characterize soft materials. In either test, a layer of a soft material is sandwiched between two layers of a stiff material. The thickness of the soft material is much smaller than its length. In lap shear, the shear force is distributed over the entire layer of the soft material. In peel, the peel force is localized at the peel front. This difference accounts for the everyday experience described above. Lap shear and peel also represent distinct loading conditions imposed on soft materials in many applications, such as adhesives between stiff materials, and matrices in composites.

Both lap shear and peel can be used to measure the toughness of a soft material. Their results, however, are rarely compared.

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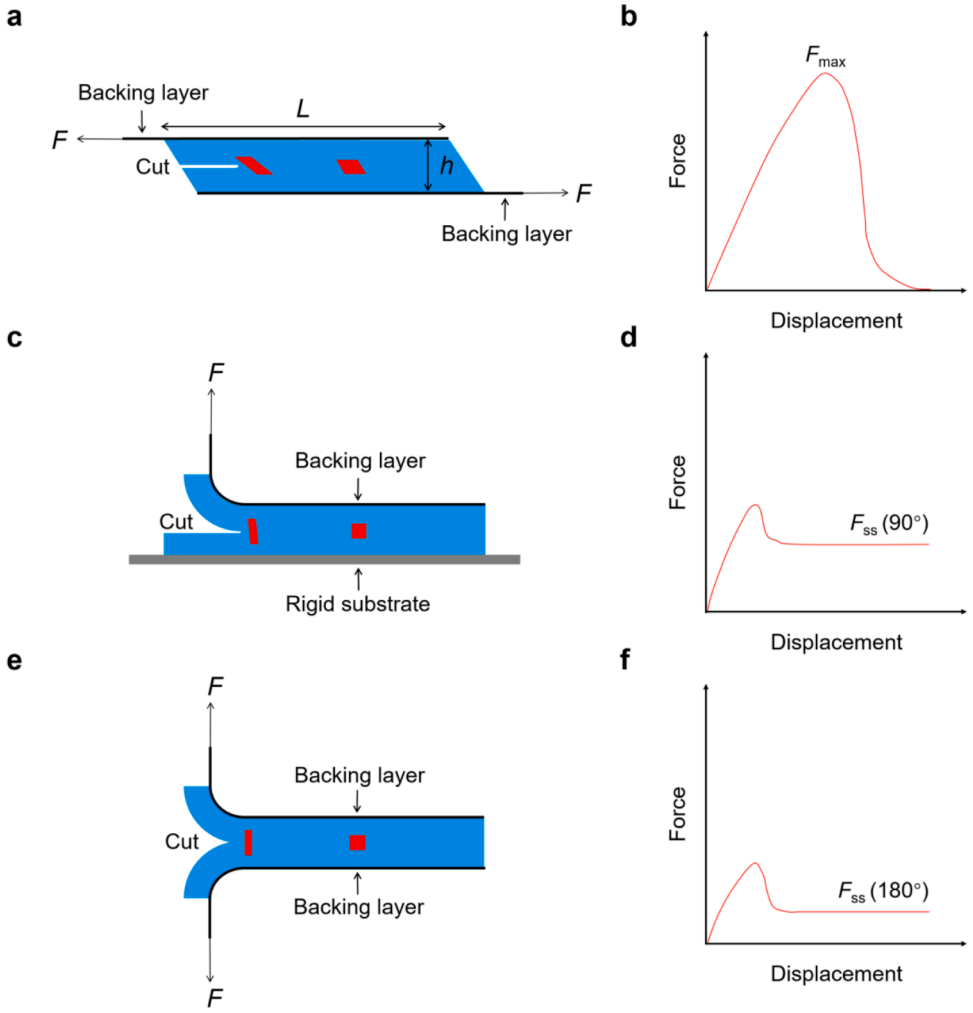

Fig. 1. Lap shear and peel. A soft elastic layer is sandwiched between two stiff layers, pulled by a force F. A cut is introduced in the soft layer. Marked in the undeformed state are two small red squares. (a) In lap shear, the red squares become parallelograms. The parallelogram near the cut tip deforms more than that far from the cut tip. (b) As the displacement increases, the cut blunts but does not grow, and the force increases. When the force reaches the peak, F_{max}, the cut grows unstably through the entire soft layer, and the force drops to zero. (c) In 90-degree peel, the red square at the cut tip deforms into a parallelogram, but the one far away from the cut tip remains square. (d) As the displacement increases, the cut blunts but does not grow, and the force increases. Once the cut starts to grow, the force drops and plateaus at a steady state, $F_{ss}(90^\circ)$, and the cut grows stably in the soft layer. (e) In 180-degree peel, the red square at the cut tip deforms into a rectangle, but the one far away from the cut tip remains square. (f) The trend of the force-displacement curve of the 180-degree peel is similar to that of the 90-degree peel, but the level of force is typically lower.

Toughness is commonly regarded as a material property, but lap shear and peel give markedly different critical forces. Will the two tests measure the same value of toughness for a given soft material? Here we answer this question by using lap shear and peel to measure the toughness of a polyacrylamide hydrogel. Polyacrylamide hydrogels are used in many applications and routinely synthesized in many laboratories. We have used polyacrylamide hydrogels as model materials to study the mechanical behavior of elastic hydrogels (Liu et al., 2019; Yang et al., 2019). For both types of test, we use two stiff layers to sandwich a layer of a polyacrylamide hydrogel. To use such a specimen to measure toughness, the thickness of the hydrogel must exceed the fractocohesive length, which is $\sim$ 1 mm for the polyacrylamide hydrogel (Liu et al., 2019; Yang et al., 2019).

We review the mechanics of lap shear and peel (Section 2). We introduce a cut in the hydrogel by scissors, pull one stiff layer at constant velocity, and record the force (Section 3). In lap shear, the force peaks and then drops to zero, the cut grows unstably through the entire hydrogel, and the peak force gives toughness (Fig. 1a and 1b). We consider two types of peel: 90-degree peel (Fig. 1c and 1d), and 180-degree peel (Fig. 1e and 1f). In either type of peel, the force peaks and then drops to a plateau, the cut grows in steady state, and the plateau force gives toughness. Our experimental data show that the average values of toughness determined by the three tests are comparable. The peak forces in both lap shear and peel scatter significantly, but the plateau force in peel scatters narrowly. As a result, toughness determined by lap shear scatters more than toughness determined by peel. We hypothesize that the peak forces

scatter mainly due to the statistical variation of the cuts made by scissors. To test the hypothesis, we conduct two additional sets of experiments. First, we introduce a cut in a specimen by scissors, pre-peel the specimen to extend the cut somewhat, and then measure toughness by lap shear and peel (Section 4). The peak force in lap shear scatters less, and the peak force in peel is removed. Second, we prepare cuts of finite tip diameters using spacers of various thicknesses, and find that the peak forces in both lap shear and peel vary with the cut tip diameter (Section 5). We describe the experimental approaches (Section 6).

2. Mechanics of lap shear and peel

2.1. Lap shear

Lap shear has been widely used to measure strength (Jeevi et al., 2019; Vakalopoulos et al., 2015; Yuk et al., 2019) and toughness (Hutchinson and Suo, 1991; Kendall, 1975a,b). The shear force is limited by either strength or toughness, and the transition is analyzed recently (Golovin et al., 2019). In this paper, we will use lap shear to measure toughness.

A soft elastic layer, width w and thickness h, is sandwiched between two stiff backing layers, thickness H (Fig. 1a). A cut is introduced in the soft layer by scissors. A force F is applied to the backing layers to shear them apart. In the undeformed state, we mark two small pieces of the soft layer by red squares. When the backing layers are pulled, shear stress develops in the soft layer. The soft layer undergoes finite shear deformation, γ, distributed over its length, so that the red squares become parallelograms. The red square near the cut tip deforms more than that far away from the cut tip. As the displacement increases, the cut blunts but does not grow, and the force increases. The force then reaches the peak, $F_{\max}$, and drops to zero, while the cut grows unstably through the entire soft layer (Fig. 1b).

The energy release rate is commonly calculated using the modulus and thickness of the stiff layers, neglecting those of the soft layer (Kendall, 1975a, b). However, we have recently shown that such an analysis is inappropriate when the soft layer is much softer than the stiff layer (Wang et al., 2020; Wang et al., 2021). A characteristic length exists, called the shear lag length, $L_s = \sqrt{\frac{EhH}{\mu}}$, where E is Young's modulus of the stiff layers and μ is the shear modulus of the soft layer. For a lap shear in which two layers of a plastic sandwich a hydrogel, $E/\mu \sim 10^6$, so that the shear lag length is enormous and often much larger than the length of the soft layer, L. When $L \ll L_s$, the backing layers behave like rigid bodies and the elasticity of the soft layer gives the energy release rate (Wang et al., 2020): $G = hW(\gamma)$, where $W(\gamma)$ is the elastic energy of the deformed soft layer per unit volume. When the soft layer is modeled as a neo-Hookean material, the energy release rate is

$$G = \frac{h}{2\mu}\left(\frac{F}{wL}\right)^2. \tag{1}$$

To ensure the validity of the use of the energy release rate, the cut needs to be long compared to the thickness of the soft layer, but short compared to the length of the soft layer (Wang et al., 2020). As we noted before, the thickness of the soft layer must exceed its fractocohesive length, which is $\sim$ 1 mm for the polyacrylamide hydrogel.

2.2. Peel

Peel has long been used to measure toughness of soft materials and is commonly performed at peel angles of 90 degree and 180 degree (Gent, 1974; Gent and Hamed, 1977). In 90-degree peel, a soft elastic layer, width w and thickness h, is sandwiched between an inextensible backing layer and a rigid substrate (Fig. 1c). A cut is introduced in the soft layer and the backing layer is pulled by a force F. In the undeformed state, we also mark two small pieces of the soft layer by red squares. When the backing layer is pulled, the deformation of the soft layer localizes at the cut tip, but vanishes elsewhere. The red square at the cut tip deforms into a parallelogram, but the one far away from the cut tip remains to be a square. As the displacement increases, the cut blunts but does not grow, and the force increases. Once the cut starts to grow, the force reaches a peak, then drops and plateaus, $F_{ss}(90°)$, and the cut peels in steady state (Fig. 1d).

The energy release rate in peel has been analyzed (Rivlin and Thomas, 1953). When the backing layer is pulled by a distance $\mathrm{d}x$, the peel force does work $F\mathrm{d}x$, and the elastic energy in the specimen changes by $\mathrm{d}U$. The energy release rate G is defined by the equation $Gw\mathrm{d}c + \mathrm{d}U = F\mathrm{d}x$, where $\mathrm{d}c$ is the length of the cut growth. In the steady state, $\mathrm{d}c = \mathrm{d}x$ and $\mathrm{d}U = 0$, because the backing layer is inextensible and the deformation region travels with the cut tip. Therefore, the energy release rate of the 90-degree peel is

$$G = \frac{F_{ss}(90°)}{w}. \tag{2}$$

In 180-degree peel, a soft elastic layer with a cut is sandwiched between two inextensible backing layers, pulled by a force F (Fig. 1e). The trend of the force-displacement curve is similar to that of the 90-degree peel, but the level of the force is typically lower (Fig. 1f). The energy release rate of the 180-degree peel is

$$G = \frac{2F_{ss}(180°)}{w}. \tag{3}$$

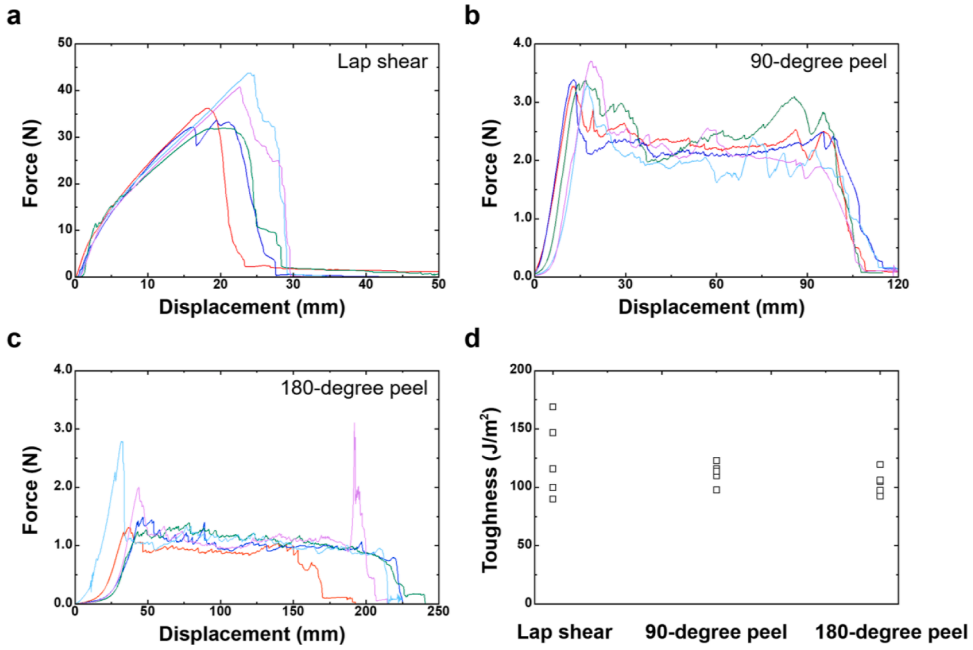

Fig. 2. Lap shear and peel using specimens with cuts made by scissors. Force-displacement curves of (a) lap shear, (b) 90-degree peel, and (c) 180-degree peel. (d) Lap shear determines toughness by the peak force, which scatters significantly. Two types of peel determine toughness by the plateau forces, which scatter narrowly. The crosslinker-to-monomer weight ratio is 0.058%.

3. Toughness measured using specimens with cuts made by scissors

We use two stiff layers to sandwich a polyacrylamide (PAAm) hydrogel, length $L = 10$ cm, width $w = 2$ cm, and thickness $h = 3$ mm. We introduce a cut, length c, in the hydrogel by scissors. The cut is long compared to the thickness of the hydrogel, but is short compared to the length of the hydrogel, $h < c \ll L$. For lap shear and 180-degree peel, the two stiff layers are identical polyester films, thickness $H = 50$ μm. For 90-degree peel, one stiff layer is the polyester film, and the other stiff layer is an acrylic sheet, thickness 3 mm. We pull one stiff layer at constant velocity, record the force-displacement curves, and compare the toughness measured by lap shear and two types of peel (Fig. 2).

In lap shear, as the displacement increases, the force rises, peaks, and drops to zero (Fig. 2a). The cut blunts but does not grow when the force rises, and then grows unstably through the entire hydrogel after the force peaks and drops (Video 2). The hydrogel is taken to obey the neo-Hookean elasticity. The shear modulus of the hydrogel is $\mu = 4256 \pm 238$ Pa and Young's modulus of the polyester is $E = 4.2 \pm 0.2$ GPa (Wang et al., 2020). The shear lag length is $L_s = \sqrt{\frac{EhH}{\mu}} \approx 38.5$ cm, which is much larger than the length of the hydrogel, $L = 10$ cm. Consequently, the backing layers can be regarded as inextensible and the peak force gives the toughness by Eq. (1).

In each type of peel, as the displacement increases, the force rises, peaks, and then drops to a plateau (Fig. 2b and 2c). The cut blunts but does not grow when the force rises, and then grows in steady state when the force plateaus (Video 3 and Video 4). The plateau force gives the toughness by Eq. (2) for 90-degree peel and by Eq. (3) for 180-degree peel. In Fig. 2c, one of the curves (purple) shows a spike in the force before rapidly dropping to zero. This spike is due to the heterogeneity near the end of the specimen. For Videos 2-4, the crosslinker-to-monomer weight ratio of the hydrogel is 0.174%.

The average values of toughness determined by the three tests are comparable (Fig. 2d). For stiff materials undergoing small deformation, toughness varies substantially with the peel angle, which is attributed to the dependence of plastic dissipation on the peel angle (Gent and Kaang, 1987; Thouless and Jensen, 1992). For an infinitesimal material of linear elasticity, when the far field is antisymmetric with respect to the plane of the cut, a mode-II field exists; the combination of antisymmetry and linearity dictates that the cut should not open or close. This antisymmetry-linearity argument, however, does not apply to finite deformation of nonlinear elasticity. For finite deformation of nonlinear elasticity, no pure mode-II field exists, and the cut opens even when the far field is antisymmetric with respect to the plane of the cut (Long and Hui, 2015; Stephenson, 1982). As a result, the cut in the hydrogel is locally under tension for lap shear and two types of peel. These considerations may rationalize our experimental finding that, for a highly elastic soft material such as polyacrylamide hydrogel, lap shear and two types of peel give nearly identical toughness.

The peak forces in all three tests scatter significantly, but the plateau forces in the two types of peel scatter narrowly. As a result, toughness measured by lap shear scatters more than toughness measured by peels (Fig. 2d). We hypothesize that the peak forces scatter mainly due to the statistical variation of the cuts made by scissors. To test the hypothesis, we report two additional sets of experiments in the following sections.

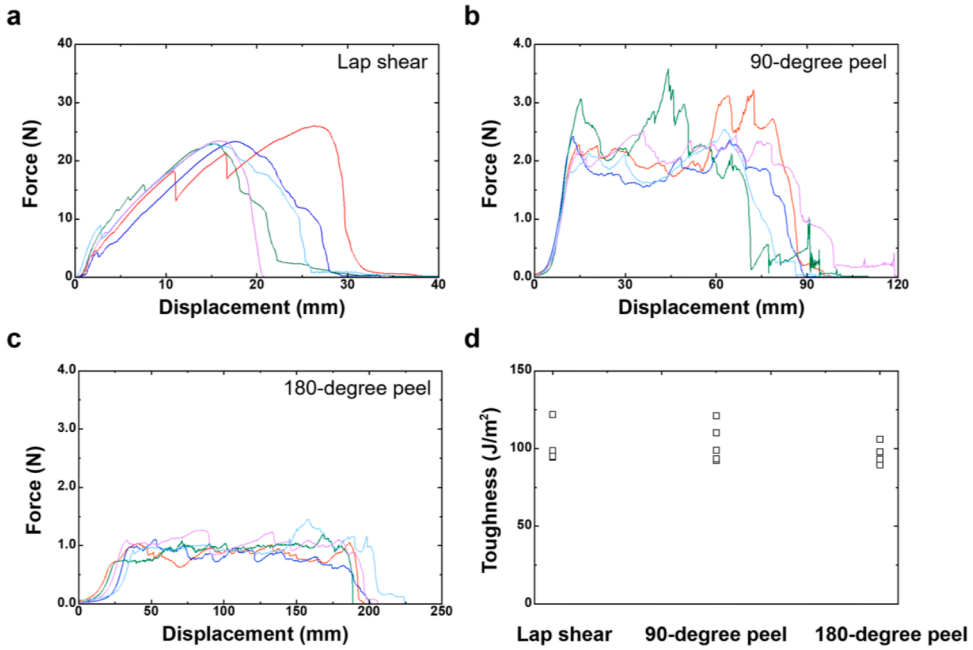

Fig. 3. Cuts are introduced by scissors to specimens, which are then peeled somewhat, before being used to measure toughness by lap shear and peel. Force-displacement curves of (a) lap shear, (b) 90-degree peel, and (c) 180-degree peel. (d) Toughness determined from the three types of specimens. The crosslinker-to-monomer weight ratio is 0.058%.

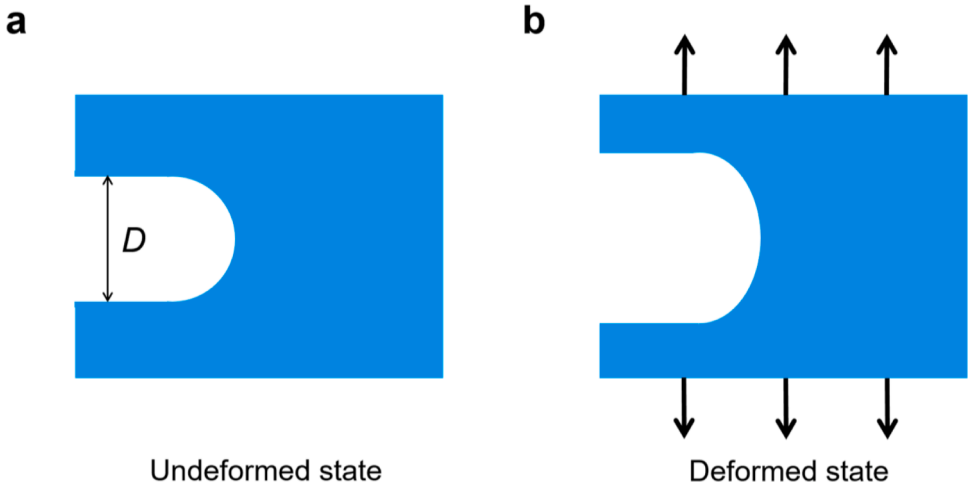

Fig. 4. A cut tip of a finite diameter. (a) In the undeformed state, a cut of the tip diameter, *D*, is introduced in the specimen. (b) In the deformed state, the cut deforms but does not grow.

4. Toughness measured using specimens after pre-peel

The cuts made by scissors have substantial statistical variation. Because the hydrogel is soft, thin, and wide, often each cut has to be made by scissoring multiple times, and thus, can have different sizes, locations, or orientations. To remove the effect of this statistical variation on the peak forces, after making a cut by scissors, we pre-peel the specimen to extend the cut by 3 cm, and then measure toughness by lap shear and peel. After the pre-peel, the remaining uncut hydrogel has a length ~ 7 cm.

After we pre-peel the specimen, the peak force in lap shear scatters less (Fig. 3a), and the peak forces in the two types of peel disappear (Fig. 3b and 3c). The drops of force in a few force-displacement curves of lap shear before fracture (e.g., the dark blue curve in Fig. 2a and the red curve in Fig. 3a) are caused by the slip between the specimen and the gripper of the loading machine. Most specimens are well gripped, so that there are no drops of force. These drops of force are not due to stick slip instability. One force-displacement curve in 90-degree peel is not as flat as the others, possibly due to the heterogeneity introduced during specimen

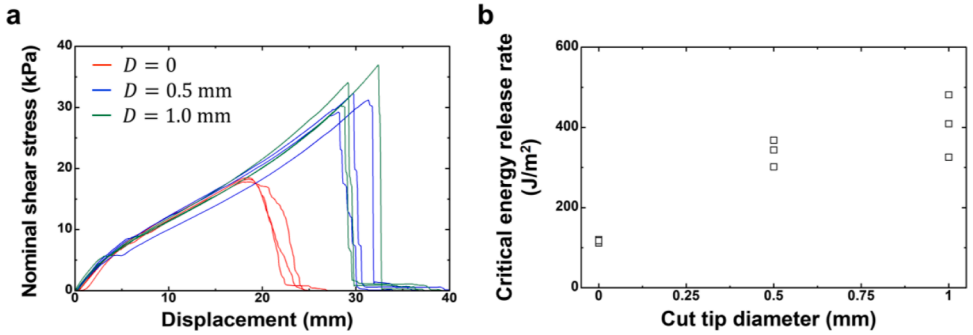

Fig. 5. Lap shear using specimens with cuts of various tip diameters. (a) Nominal shear stress as a function of displacement. (b) Critical energy release rate as a function of the cut tip diameter. The crosslinker-to-monomer weight ratio is 0.058% and the initial cut length is 1 cm.

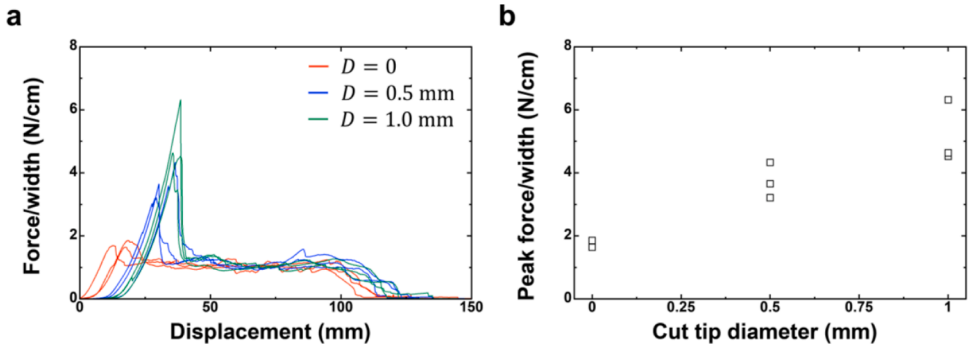

Fig. 6. 90-degree peel using specimens with cuts of various tip diameters. (a) Force-displacement curves. (b) Critical energy release rate as a function of the cut tip diameter. The crosslinker-to-monomer weight ratio is 0.058% and the initial cut length is 1 cm.

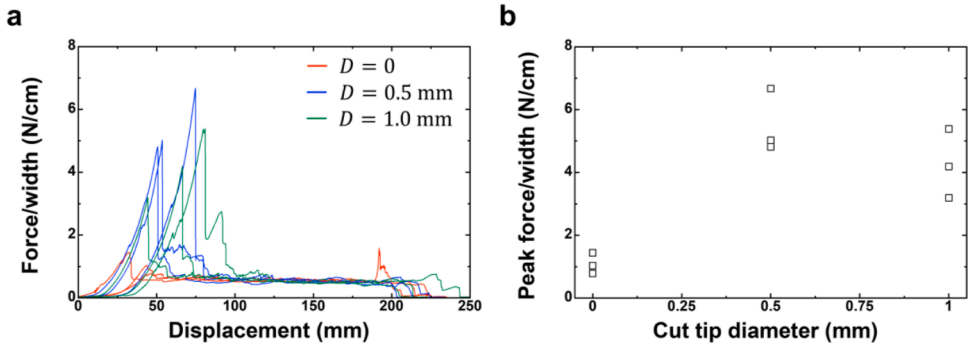

Fig. 7. 180-degree peel using specimens with cuts of various tip diameters. (a) Force-displacement curves. (b) Critical energy release rate as a function of the cut tip diameter. The crosslinker-to-monomer weight ratio is 0.058% and the initial cut length is 1 cm.

preparation. Lap shear and peel give nearly identical toughness (Fig. 3d). These results indicate that pre-peel is effective to narrow the scatter in toughness obtained in lap shear and to remove the peak force in peel.

5. The effect of cut of a finite tip diameter

The cuts made by scissors may have tips of large diameters. To study the effect of the tip diameter on the critical energy release rate, we prepare cuts of finite tip diameters by casting the hydrogel over spacers of various thicknesses. In the undeformed state, the cut tip diameter is D (Fig. 4a). In the deformed state, the cut deforms but does not grow (Fig. 4b). The material near the cut tip stretches more than the material far ahead of the cut tip. When the cut tip diameter D is small, stretch concentrates at its tip. When the cut tip diameter D is large, stretch concentrates less. The tip diameter of a cut prepared by scissors without using a spacer is denoted as $D = 0$ for comparison.

In lap shear, the maximum nominal shear stress, F_{max}/wL, increases with the cut tip diameter, D (Fig. 5a). We derive the critical energy release rate, G_c, by Eq. (1) using the peak force (Fig. 5b). As expected, G_c is no longer a material constant, but increases with the cut tip diameter, D.

In 90-degree peel, the peak force increases as the cut tip diameter increases, but the plateau force is insensitive to the cut tip diameter (Fig. 6a). The peak force is affected by the initial cut made by the scissors or spacers. By contrast, the plateau force is due to an advancing peel front, which is independent of how the initial cut is introduced. A peak force corresponds to a critical energy release rate, G_c, which increases with the cut tip diameter, D (Fig. 6b).

In 180-degree peel, as the cut tip diameter D increases, the plateau force is unaffected, but the peak force first increases then decreases (Fig. 7). At $D = 1.0$ mm, fingering is observed when the cut is about to grow, causing the peak force to decrease. An in-depth study of this fingering instability is beyond the scope of this paper.

For a rubber, Thomas related the critical energy release rate for rupture to initiate from a semicircular incision tip, G_c, to the incision diameter in the undeformed state, D (Thomas, 1955):

$$G_c = DW_f, \tag{4}$$

where W_f is the work of fracture of the rubber. When the incision grows by a small amount, the corresponding part of the deformed specimen is removed and a new surface is formed and stressed. The above equation is given by the change in the elastic energy of the deformed specimen per unit volume caused by the growth of the incision. Thomas experimentally measured the critical energy release rate, G_c, at various incision diameters, D, and confirmed that G_c/D was comparable to the work of fracture, W_f. For lap shear at $D =$ 1 mm in this paper, $G_c/D \sim 4 \times 10^5$ J/m^3. The work of fracture of the hydrogel is $W_f \sim 1.2 \times 10^5$ J/m^3.

6. Experimental section

6.1. Preparation of hydrogel

All the chemicals, including acrylamide (AAm; Sigma-Aldrich, A8887), N,N'-methylenebisacrylamide (MBAA; Sigma-Aldrich, M7279), and alpha-ketoglutaric acid (Sigma-Aldrich, 75890), were purchased.

The precursor of the polyacrylamide (PAAm) hydrogel was an aqueous solution of AAm monomer (1.916 mol/L), MBAA (0.058%, 0.116%, or 0.174% the weight of AAm) as the crosslinker, and alpha-ketoglutaric acid (0.2% the weight of AAm) as the photoinitiator. The PAAm hydrogel was made by pouring the precursor solution into a 3 mm thick plastic mold glued on a plastic substrate and gelled under UV. A glass sheet was used to the seal the mold.

6.2. Preparation of cut

6.2.1. Scissor-made cut

A cut was introduced in the specimen by scissors. The cut was long compared to the thickness of the hydrogel, but was short compared to the length of the hydrogel.

6.2.2. Pre-peeled cut

A cut was first introduced in the specimen by scissors, and then was extended by 3 cm by 90-degree peel.

6.2.3. Cut of a finite tip diameter

A cut of a finite tip diameter was prepared using a polyester spacer (McMaster Carr) of a finite thickness. The spacer was glued to the short edge of the mold using the cyanoacrylate (krazy glue) before pouring the precursor solution of the hydrogel. After gelation, the spacer was in the hydrogel. Cohesive fracture of the hydrogel always happened.

6.3. Lap shear test

We used the cyanoacrylate to glue the PAAm hydrogel to the polyester films (50 μm thickness; McMaster Carr) as the backing layers. Since the PAAm hydrogel was acidic, the surfaces were neutralized by applying a few drops of 0.1 mol/L $NaHCO_3$ solution (Sigma-Aldrich, S5761) and then dried by blowing air before the use of cyanoacrylate. A cut was introduced in the PAAm hydrogel. Such PAAm hydrogel with the backing layers was loaded by a tensile machine (Instron 5966; 100 N load cell) using lap shear test. The loading rate was 10 cm/min. The toughness was calculated by Eq. (1) using the peak force in the force-displacement curve.

6.4. 90-degree peel test

We used the cyanoacrylate to glue the PAAm hydrogel to a rigid acrylic substrate and a polyester film (50 μm thickness; McMaster Carr) as the inextensible backing layer. The acidic PAAm hydrogel surfaces were neutralized by applying a few drops of 0.1 mol/L $NaHCO_3$ solution (Sigma-Aldrich, S5761) and then dried by blowing air before the use of cyanoacrylate. A cut was introduced in the PAAm hydrogel. Such PAAm hydrogel with the rigid substrate and the backing layer was loaded by a tensile machine (Instron 5966; 100 N load cell) using 90-degree peel test. The loading rate was 10 cm/min. The toughness was calculated by Eq. (2) using the plateau

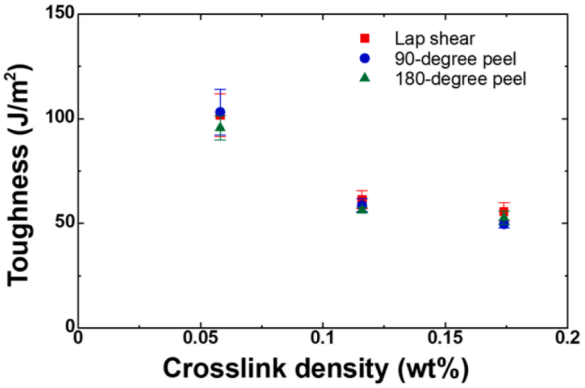

Fig. 8. Toughness measured by lap shear and peel of hydrogels of various crosslink densities. Specimens with cuts made by scissors are peeled somewhat, before being used to measure toughness by lap shear and peel. All data stand for the mean and standard deviation of 5 specimens.

force in the force-displacement curve.

6.5. 180-degree peel test

We used the cyanoacrylate to glue the PAAm hydrogel to the polyester films (50 μm thickness; McMaster Carr) as the inextensible backing layers. The acidic PAAm hydrogel surfaces were neutralized by applying a few drops of 0.1 mol/L $NaHCO_3$ solution (Sigma-Aldrich, S5761) and then dried by blowing air before the use of cyanoacrylate. A cut was introduced in the PAAm hydrogel. Such PAAm hydrogel with the backing layers was loaded by a tensile machine (Instron 5966; 100 N load cell) using 180-degree peel test. The loading rate was 10 cm/min. The toughness was calculated by Eq. (3) using the plateau force in the force-displacement curve.

7. Concluding remarks

For the polyacrylamide hydrogel with cuts made by scissors, we have shown that the peak forces in both lap shear and peel scatter significantly, but the plateau force in peel scatters narrowly. As a consequence, the toughness determined by lap shear scatters more than the toughness determined by peel. We have hypothesized that the large scatters in the peak forces are mainly due to the statistical variation of the cuts made by scissors and conducted two additional sets of experiments to test the hypothesis. Using specimens after the cuts made by scissors are pre-peeled, we show that the toughness determined by lap shear scatters less, and lap shear and peel give nearly identical toughness. We have also used specimens after the cuts made by scissors are pre-peeled to measure the toughness of hydrogels of several crosslink densities (Fig. 8). Lap shear and two types of peel give nearly identical toughness for all the crosslink densities. Using specimens with cuts of finite tip diameters, we find that the peak forces in both lap shear and peel vary with the cut tip diameter. These findings will provide guide to the use of lap shear and peel to characterize soft materials.

Declaration of competing interest

The authors declare no competing interest.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jmps.2021.104348.

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