CHARACTERIZATION OF STRAIN-RATE DEPENDENT SHEAR FAILURE OF PRESSURE SENSITIVE ADHESIVES

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Introduction

Characterizing pressure sensitive adhesives (PSAs) at high rates presents many challenges. The viscoelastic behavior of PSAs requires testing at rates relevant to the application of interest. However, standard mechanical test equipment is often not capable of reaching high rates. Those that do, such as the Izod impact test [1], are not instrumented to measure the force or duration of the impact. While Kolsky bar tests are becoming more common for investigating high rate tensile and compression deformations [2,3,4], test methods for high rate shear deformation are less prevalent. Recently an instrumented method for high rate shear deformations has been developed [5]. This technique requires a Kolsky bar apparatus and custom strain gage fixturing, making it difficult to run in an industrial setting.

The viscoelastic mechanical properties of PSAs are well-known and commonly characterized using rheology techniques such as shear-mode master curves [6, 7] to quantitate the rate dependency of these properties. Similar to the bulk mechanical properties, the ultimate failure of a PSA is also a rate dependent phenomenon [8]. For example, Kaelble demonstrated that the technique used to create master curves for bulk material properties can also be used to understand the relationship between peel rate and steady-state peel force [9]. Specifically, Kaelble demonstrated that the same WLF relationship used to define shear-mode master curves can be used to create peel rate master curves. This is somewhat surprising as the WLF relationship assumes that the material undergoes small strains and can be considered linear viscoelastic, whereas peel tests to failure typically involve large adhesive deformations where nonlinearities are observed. Despite this discrepancy, this technique has been successfully used numerous times to quantify rate-dependent failure properties of soft materials including PSAs [8,10].

In the present work, a custom high strain rate double overlap shear test method was developed using a drop tower. The drop tower was instrumented to provide force and displacement measurements over the duration of the impact, providing a more complete picture of the response of the pressure sensitive adhesive at high strain rates. The effect of temperature and the use of time-temperature superposition and the WLF relationship [6,7] were investigated to better understand PSA failure properties.

Test Methods

A small strain shear-mode master curve was obtained for the adhesion as follows: an 8-mm diameter disk was punched from a 1 mm thick sheet of the adhesive. The liners were removed from the adhesive disk, and the disk was then loaded between 8 mm diameter parallel plates on an ARES-G2 strain controlled rotational rheometer (TA Instruments, New Castle, DE, USA). Oscillatory shear frequency sweeps were conducted at temperatures from 150 °C to -80 °C in 5 °C increments. At each temperature step, the sample was subjected to oscillatory shear deformations with an initial strain amplitude of 5%, with frequencies ranging from 0.1 Hz to 50 Hz. Auto-strain was used to automatically adjust the strain amplitude downward to limit the torque on the rheometer to 20 g-cm. This insured that the measurements were conducted in the linear viscoelastic range of the material, regardless of the current testing temperature.

The resulting frequency sweeps were shifted onto a master curve using time-temperature superposition (TTS) with a reference temperature of 25 °C, optimizing for superposition of the shear storage modulus (G'). Prior to TTS, a baseline y-axis shift was applied to the dynamic modulus terms (shear storage modulus G' and shear loss modulus G''), according to the following equation:

$$b_T = T_{ref}(in\ K)/T(in\ K) \qquad \text{Eq. 1}$$

Where b_T is the vertical shift factor by which the dynamic moduli at temperature T were multiplied before shifting, and T_{ref} was the reference temperature of 25 °C. This vertical shifting was performed as to account for the entropic origin of polymer elasticity, and hence improves the overall superposition of the dynamic moduli.

Impact tests were performed using a CEAST 9340 instrumented drop tower (Instron, Norwood, MA, USA) with a 22 kN piezoelectric load cell. The specimens were constructed by bonding three 10 mm x 10 mm x 20 mm plasma treated stainless steel blocks with two layers of 10 mm x 10 mm PSA (Figure 1). The 10 mm x 10 mm faces on the blocks had a mirror finish polish. The outer blocks were constrained with the drop tower's built-in pneumatic clamp and the center block was only constrained by the adhesive bond. A striker with 13 kg mass was dropped from a 100 mm [11]. The striker contacted the center block with a 15 mm x 15 mm square face. Temperature was varied from -15 °C to 100 °C using the drop tower environmental chamber. 5 tests were completed at each level. During the test, force and acceleration were recorded as a function of time. Displacement was calculated from the acceleration measurements. The accuracy of the calculated displacement was verified using an optical particle tracking technique to independently measure the displacement of the center block during a test. Peak force and peak energy density, which is defined as the energy dissipated per unit volume from initial contact between the striker and specimen until peak force is reached, were calculated for each test run. Tests were conducted on an acrylic PSA composition commercially available for consumer electronic displays.

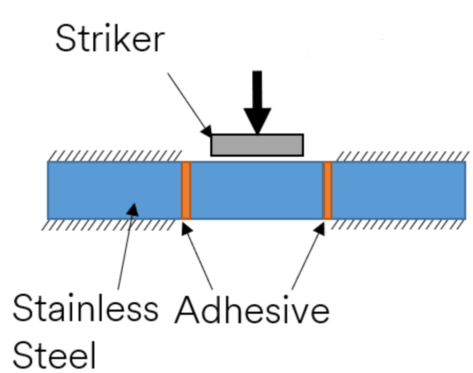

Figure 1: Side view of the shear impact specimen

For comparison, quasistatic double overlap shear tests were performed using an MTS Acumen III load frame (MTS Corporation, Eden Prairie, MN) using a +/- 3 kN strain gage load cell and an MTS Acumen DMA double lap shear fixture (Model Number 100332946, MTS Corporation, Eden Prairie, MN). All tests were completed at room temperature. Specimens compatible with this fixture were made of plasma treated stainless steel with 10 mm x 10 mm areas that were bonded with the PSA. Again, the 10 mm x 10 mm faces of the stainless steel posts had a mirror finish polish. Tests were run at constant displacement rates between 0.002 mm/s and 10 mm/s. Like the drop tower experiments, time, force, and displacement were measured and peak force and peak energy density were calculated for each test run.

Results

Figure 2a shows the shear-mode master curve and shift factors for the PSA. The WLF relationship,

$$\log(a_T) = \frac{-C_1 * (T - T_{ref})}{C_2 + (T - T_{ref})} \qquad \text{Eq. 2}$$

was fit to the data where a_T is the shift factor by which the frequencies (or rates) at temperature T are multiplied to create the master curve at the reference temperature, T_{ref}, of 25 °C. The fit parameters were C_1 = 7.63 and C_2 = 108.1 K. As shown in Figure 2b, the WLF relationship fits the data well for temperatures above -30 °C, which is the region of interest for shear impact tests.

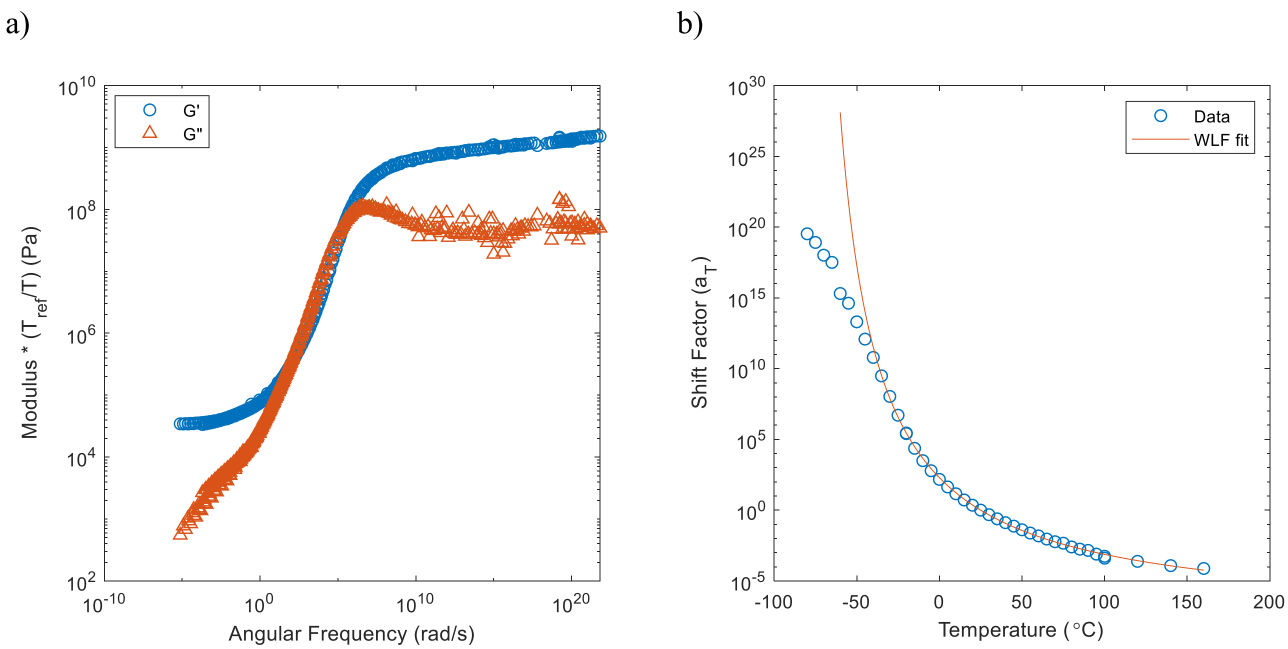

Figure 2: a) Shear-mode master curve and b) shift factors for the adhesive

Adapting Kaelble's approach to the shear tests, the vertical correction was applied to peak force and peak energy density and the shift factors from the WLF relationship were applied to the impact velocity, v, to create a shear impact master curve. Figure 3 shows peak force and peak energy density measured from the shear impact and quasistatic double overlap shear tests. Impact tests run between 60 °C and 100 °C had equivalent velocities to the 1 mm/s and 10 mm/s quasistatic shear tests run at room temperature. As expected from previous cases, the peak force and peak energy densities are in good agreement in the overlapping region indicating that the WLF relationship can be used to create shear impact master curves for PSAs.

a) b)

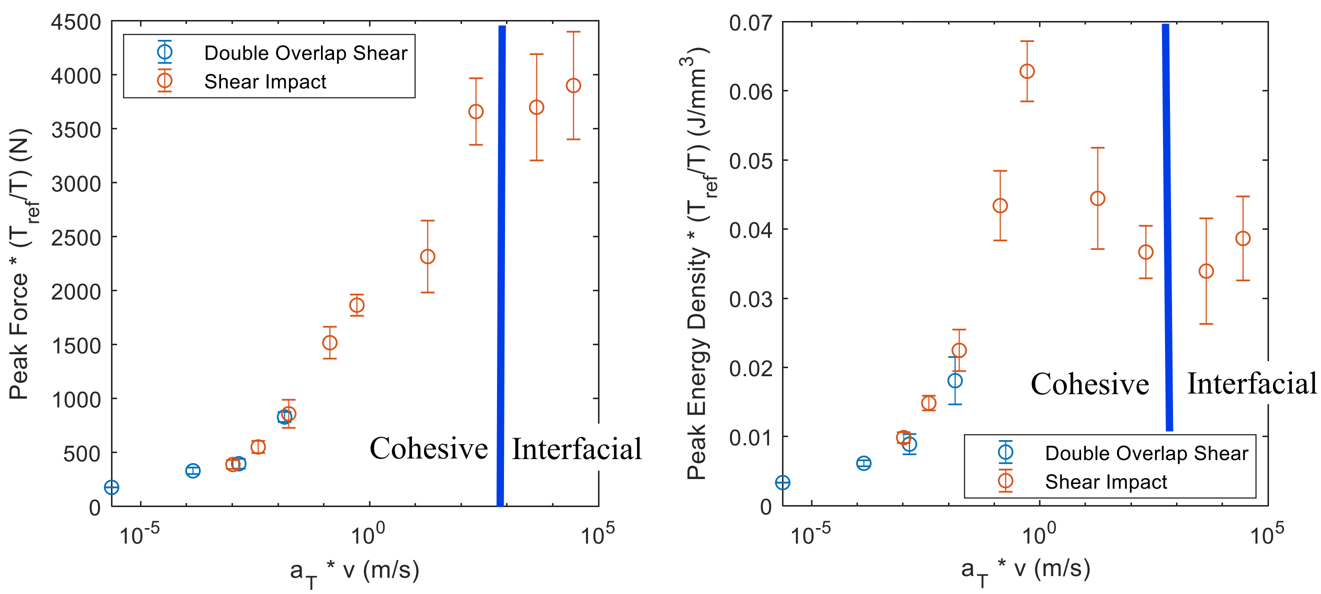

Figure 3: a) Peak force and b) peak energy density as a function of effective velocity for quasistatic double overlap shear and shear impact

At low rates, all failures were cohesive. When the effective rate increased beyond 1,000 m/s, the specimens begin to fail at the adhesive-steel interfaces. In the cohesive failure region, peak force increased as effective velocity increased. Peak energy density increased as effective velocity increased between 10^{-6} m/s and 1 m/s, but decreased at higher rates until reaching a local minimum at the cohesive-interfacial failure transition. The cause of the decrease in peak energy is unknown. For the cases examined, it appears that the peak force and peak energy density are approximately constant in the interfacial failure regime. Previous studies that have used this approach to investigate rate dependent failure in non-shear deformation modes have observed that the peak force and peak energy density increase in the interfacial failure regime [8]. It is possible that if additional tests were run at greater effective velocities that this trend would appear in the present data set, but the current data set is inconclusive.

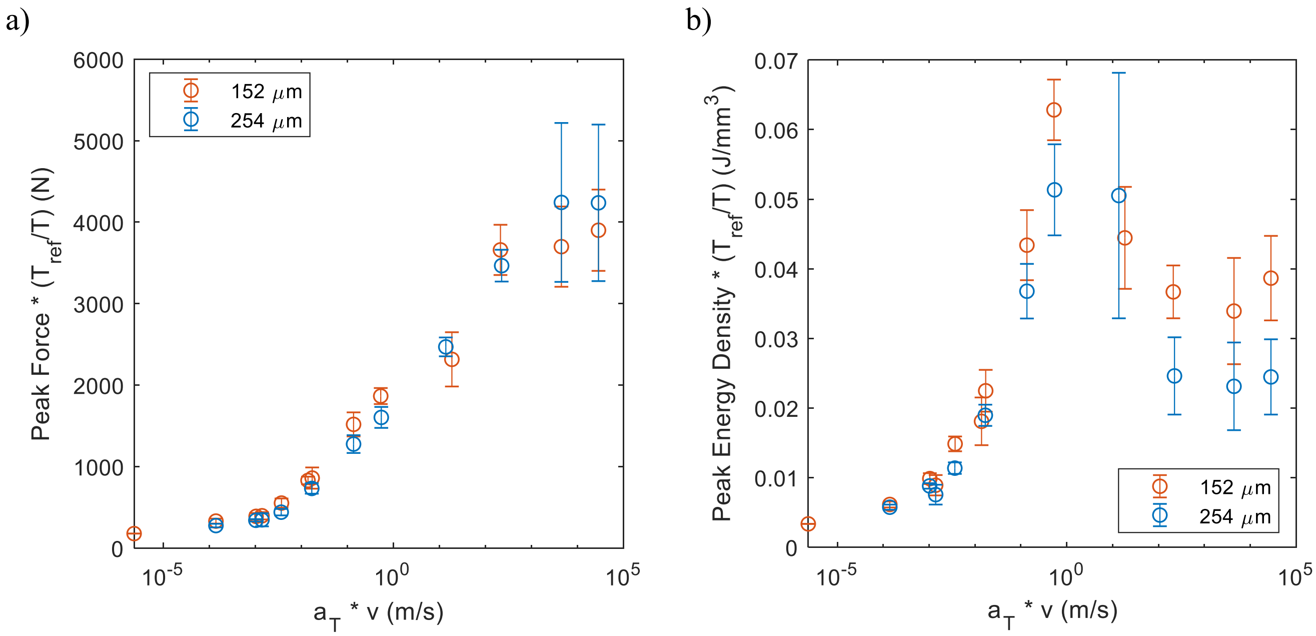

Figure 4: a) Peak force and b) peak energy density as a function of effective velocity for 152 μm and 254 μm thick PSA

This technique was also used to investigate the effect of adhesive thickness on peak force and peak energy density. Figure 4 compares shear impact master curves from 152 μm and 254 μm thick versions of the PSA. For the cases examined, it appears that changing the thickness did not have a significant effect on the measured peak force (Figure 4a). In contrast, peak energy density appears to decrease as thickness increases. For both metrics, the overall shapes of the master curves are consistent with each other suggesting that the thickness of the adhesive does not have a significant effect on the failure mechanisms.

Conclusions

In the present work, a custom high strain rate double overlap shear test method was developed using a drop tower. The drop tower was instrumented to provide force and displacement measurements over the duration of the impact, providing a complete picture of the response of the pressure sensitive adhesive at high strain rates. The effect of temperature on the impact performance was investigated. Using the WLF relationship calculated from shear-mode master curves for the PSA, the data was shifted to equivalent rates. Traditional quasistatic double overlap shear tests were completed on the PSA and showed good agreement with the impact tests run at elevated temperature, indicating that the WLF relationship can be applied to shear impact tests to better understand shear mode failure of PSAs.

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