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Release Force Understanding – Recent Findings

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Abstract

The practical application of elastomeric polydimethylsiloxane coatings for the release of organic adhesives has been advanced at an accelerated rate by applying fracture mechanic principles to what was previously viewed as a complicated surface energy problem. Through the efforts of several research groups, including the work spearheaded by M. Chaudhury, new insights have been gained about the role release plays in applications ranging from bio-fouling to organic pressure-sensitive adhesives. The rapid increase in learning can be attributed to the reduction of diverse release problems to a single, unifying approach and by considering the frictional and viscoelastic forces operating across the siloxane organic interface.

Introduction

For many years, the primary considerations in the development of pressure-sensitive adhesives (PSAs) were the ultimate substrate (including paper, metal, the human body and organic plastics) and operating temperature. Typically, little or no consideration was given to the nature of the elastomeric polydimethylsiloxane (PDMS), or silicone, release coating that was in contact with the PSA for storage and pre-positioning purposes. However, two recent trends have brought about a change in focus: 1) the globalization of the paper, label and tape industries, and 2) increasing regulatory pressures to convert to environmentally friendly products.

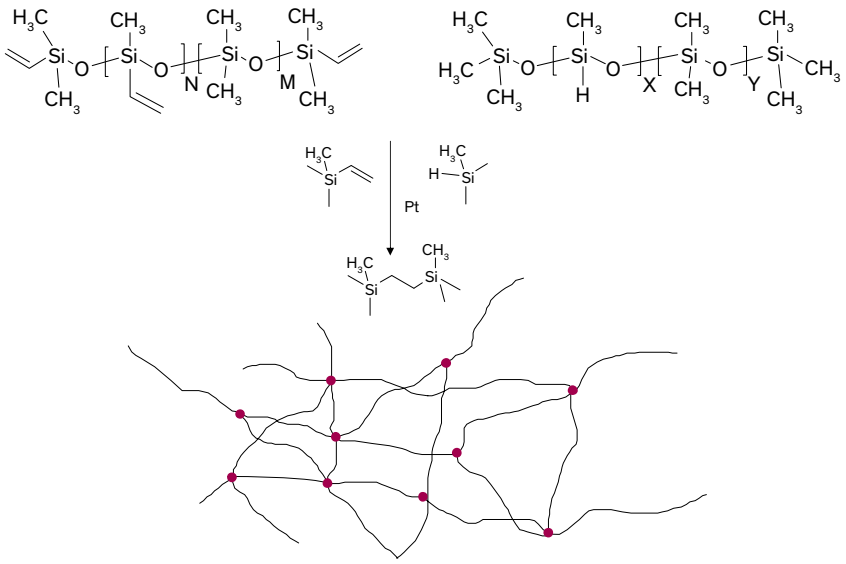
The globalization of the PSA industry has led to a greater emphasis on economy of scale. This has resulted in reduced or simplified product offerings and a greater emphasis on the production process. High-speed production methods employing low-temperature cure solventless coatings applied to the cheapest substrates available have placed practical constraints on silicone release coating designers. As a consequence, studies on wetting/adhesion, substrate poisoning, film splitting and network formation have received intense consideration. While the palette of available polymers has been shrinking, expectations for release force levels and degree of release force separation have not. In fact, the release coating is being asked to compensate for the economic-based modifications of the organic PSA; e.g., the incorporation of extending monomers (which add little to PSA viscoelastic properties) or the reduction in PSA thickness/coat weight. To meet the growing demands of the PSA industry, a greater understanding of the molecular basis of release is necessary so network and process demands can be better matched with optimum conditions for controlled release.

The greatest technological challenge is applying a 1-micron-thick silicone coating homogeneously over a macroporous substrate composed of acrylic resin, mineral particulates and cellulosic fibers. The degree of cure and homogeneity of coverage are the two most studied attributes. This is because deficiencies in either can have catastrophic consequences. Cheaper substrates generally have a less well-defined surface structure and a greater incidence of substances that poison the release coating's precious metal catalyst. These substrates can also negatively impact anchorage to the backing. High-speed coating processes (with their shorter residence times in costly gas-fired ovens, short wet-out times and atomization of the coating solution during roll splitting) impose viscosity—and therefore viscoelastic—limitations. Environmental constraints have dictated the use of solventless systems, which has created additive solubility issues and reduced the effective molecular weight of polymers and crosslinkers.

The PDMS elastomeric network is typically treated as if it were a single, unambiguous entity. However, no single polymer system offers the degree of freedom afforded by silicone chemistry. Figure 1 illustrates the silicone chemistries utilized in the majority of the industrial and academic studies. There is a great deal of controversy surrounding adhesion hysteresis and the relative role of bulk versus interfacial effects. This lies in the lack of a clear understanding of the subtleties of the roles Si–H and Si–Vi play in the architecture of the starting materials, which include silicone hydride crosslinkers and vinyl-functional polymers, high-release additives (HRAs) based on methyl silicate resins and a precious metal catalyst. In silicone release applications, there are unprecedented architectural options available, from the mundane degree-of-polymerization to the more complex degree-of-branching. These freedoms can be combined with reactivity changes between vinyl- or hexenyl-functional polymers, likewise between $-OSi(CH_3)_2H$ and $-O_{4/2}CH_3SiH$, and $-O_{3/2}SiH$. This, coupled with their low glass transition temperature (T_g), is why PDMS-based polymers are often used to study ideal networks. The same degree of freedom is also observed in HRAs based on methyl silicate resins. All major silicone release coating manufacturers have commercial versions of the methyl silicate resin. While these versions perform the same function, they may vary significantly in composition, molecular weight, degree of reactive functionality and, hence, in ease of dispersion, reactivity and efficacy in modifying release.

The conventional explanation for the efficacy of PDMS as a release coating is its low surface energy; PDMS release coatings have solid surface tensions in the region of 18 to 22 $mN{\cdot}m^{-1}$. However, low surface energy, by itself, does not guarantee excellent release performance; other factors, such as interfacial dynamics and viscoelastic considerations, also play important roles. Early studies by Chaudhury and Whitesides [1] of the adhesion between monolayer fluorocarbon and hydrocarbon films supported by elastomeric PDMS established that the adhesion hysteresis for the *fluorocarbon* monolayer was significantly higher than for the *hydrocarbon* monolayer. Chaudhury [2] further observed that when adhesives were peeled from PDMS surfaces, a sliding or lateral displacement, as tracked by fluorescent markers [3], could be observed several hundred microns from the crack tip. It was also revealed that, depending on the release surface, peel velocity and peel geometry, the magnitude of this induced sliding could account for 50 percent of the overall peel fracture energy.

Figure 1. General hydrosilylation chemistry used to form silicone elastomeric networks.

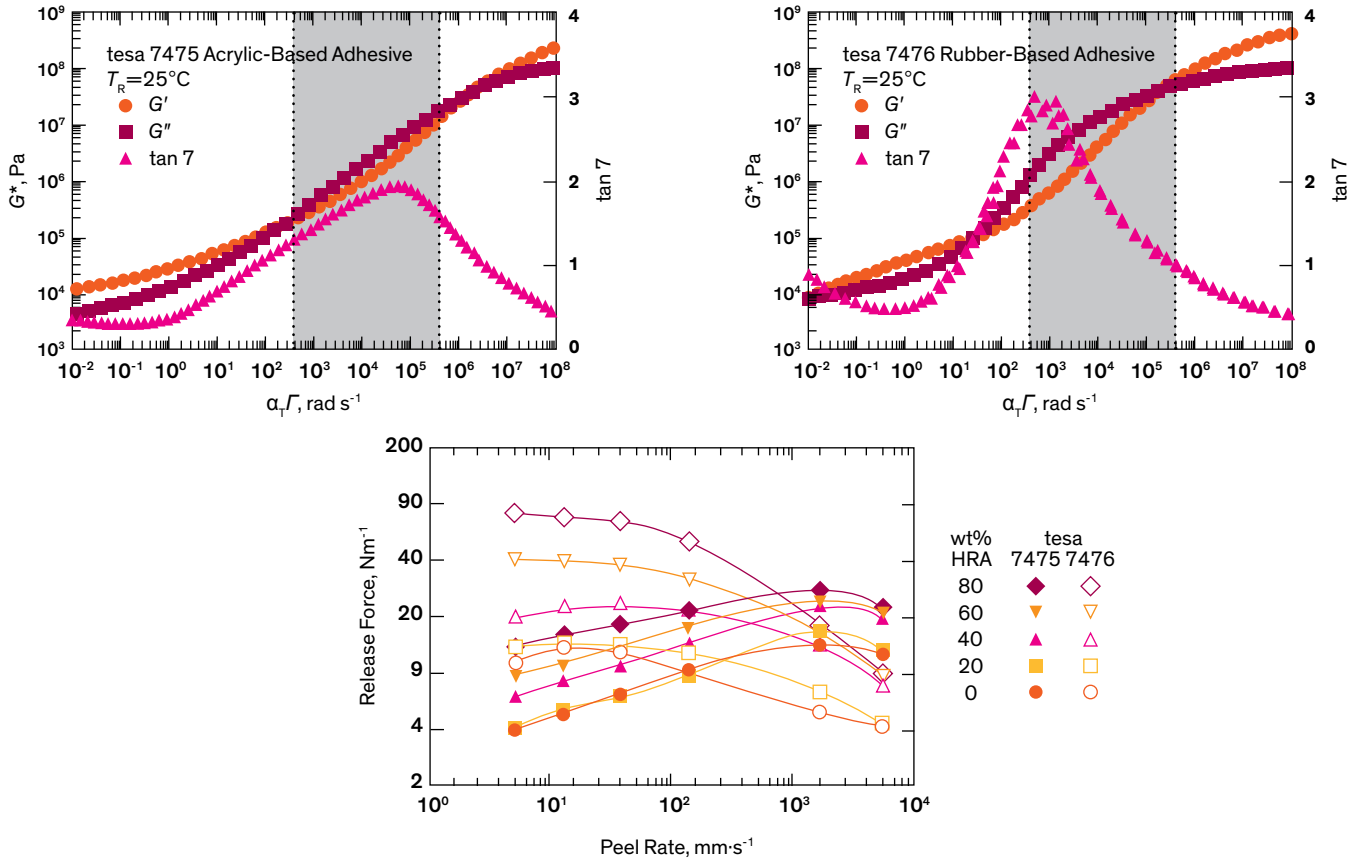


In Chaudhury's sliding experiment, a semispherical PDMS slider layer was slid over mica-supported fluorocarbon and hydrocarbon self-assembled silane monomers yielding friction and adhesion hysteresis data on both systems. The adhesion energies for compressive and decompressive loads with the fluoroalkyl monolayer were 32.2 and 62.7 $ergs{\cdot}cm^{-2}$, respectively, whereas the alkyl surface yielded equivalent values of 38.4 and 45.6 $ergs{\cdot}cm^{-2}$, respectively. In the former case, the hysteresis (D = 30.5 $ergs{\cdot}cm^{-2}$) was significantly higher. The normalized friction force (interfacial shear strength) for the PDMS/fluorocarbon monolayer was 21.4 $dyn{\cdot}cm^{-2}$, which was significantly higher than the value of 4.6 $dyn{\cdot}cm^{-2}$ obtained for the PDMS/hydrocarbon monolayer. The friction force values did not follow the trend of surface-free energies: the fluorocarbon surface had a value of 9.5 $ergs{\cdot}cm^{-2}$ compared to the hydrocarbon surface value of 19.3 $ergs{\cdot}cm^{-2}$. This was clearly in contradiction to Dupre's thermodynamic consideration of adhesion.

One polymer property that follows this trend, as well as the similar trend illustrated in Figure 2, is T_g. Wynne and co-workers [4] have proposed that such molecularly flexible substrates prevent adhesive mechanisms, such as mechanical interlocking on rough surfaces, from operating.

Professor Chaudhury's work on the influence of surface states on adhesion hysteresis and frictional properties has spurred work by others in these fields [5-8]. It was observed that in elastomeric PDMS systems, low-release surfaces also possessed the lowest friction [5,9]. Both adhesion and friction are determined by polymer chains close to the surface; the segmental mobility of these polymers is altered by their proximity to an interface. When a PDMS elastomer is placed in contact with either a similar or dissimilar surface, reconstruction of surface polymer chains will occur. The nature and extent of this reconstruction will determine the level of adhesion and its ultimate stability. The adhesion hysteresis of extracted and non-extracted PDMS substrates residing against the same and dissimilar substrates has been studied by many researchers [10-12]. In some studies, adhesion hysteresis due to induced hydrogen bonding was encountered; while in others, Pt catalyst complexation, or Pt catalyst-induced hydrosilylation across an interface, was reported as a root cause. In industrial release applications, Pt-catalyzed release coatings containing high residual levels of silicon hydride groups react with the carbonyl groups on some acrylic-based adhesives, leading to increasing peel forces and, ultimately, to acrylic "lock up." The release force observed in a freshly constructed laminate is typically lower than the release force observed in the same laminate after it has aged for several days. It is presumed that the non-equilibrium conditions encountered during the construction process lead to surface restructuring and a resulting increase in molecular contact. Calculations by Israelachvili [5] lead one to conclude

Figure 2. Profiles for a silicone-coated liner peeled at a 180-degree angle at ambient conditions from two different PSA tapes, in which release force increases with HRA concentration at a constant peel rate. Contrasting release profiles were observed between the tesa 7475 acrylic- and 7476 rubber-based adhesives.



that the interdigitation is not part of the surface restructuring between the two immiscible layers because we do not see a continuous increase in release forces after time. The reconstruction at the interface, which leads to the initial increase in release, is due to conformational changes in the organic polymers with higher T_gs. Pressure sensitive adhesives—including those that incorporate a soft rubber segment—are still less molecularly mobile than the low-T_g PDMS coating.

Chaudhury's work sought to quantify the energy directly related to the interfacial process and separate it from the bulk deformation process. We have sought to extend that knowledge to the fabrication and performance of industrial PSA laminates. With the help and guidance of Chaudhury and his research group, we have begun to deconvolute the interfacial and bulk contributions to the control of release force at higher peel velocities [13].

Experimental

Solventless silicone coating systems containing modulating levels of HRA were prepared via addition-cure chemistry. All vinyl-functional polymers were stripped on a wipe film evaporator to 99.5 percent or greater non-volatile content, as measured by 3-hour weight-loss measurements at 150°C. The mixing ratios for all systems involved 100 parts vinyl-functional PDMS (with or without HRA), 0.7 parts of a maleate inhibitor and 2 parts of SYL-OFF™ 4000 Catalyst. The crosslinker used throughout these studies was SYL-OFF™ 7678 Crosslinker, a dimethyl-, methylhydrogen-siloxane copolymer with an SiH content of 1.1 wt%.

The 1.5-µm-thick test specimens were cured onto 54.5-lb Nicolet paper at 205°C for 6 seconds. For viscoelastic measurements, 1.3- to 1.6-mm-thick samples were crosslinked in situ at 130°C for more than 3 hours, enabling real-time monitoring of the extent of cure and, more importantly, ensuring complete contact between the 8-mm diameter parallel plate fixtures of the rheometer. Samples for lateral-force microscopy, interfacial slippage, and indentation were prepared in accordance with published methodologies. Industry standard PSA tapes were selected to represent two major adhesive technologies: tesa 7475 acrylic and 7476 rubber-based adhesives with PVC and cloth substrates, respectively. The thicknesses of the tesa tape components was measured using scanning electron microscopy.

To profile release force as a function of peel rates from 5.08 to 5080 mm·s^{-1} at ambient conditions, 180-degree angle peel testing was conducted using 3M90 Slip-Peel and ZPE-1000 High-Rate Testers from Instrumentors, Inc., on 25.4-mm-wide laminates aged for one day at room temperature. Isothermal, frequency-sweep viscoelastic experiments were performed within the linear viscoelastic region using Rheometric Scientific dynamic mechanical spectrometers from ambient to below the glass transition temperature.

Results and Discussion

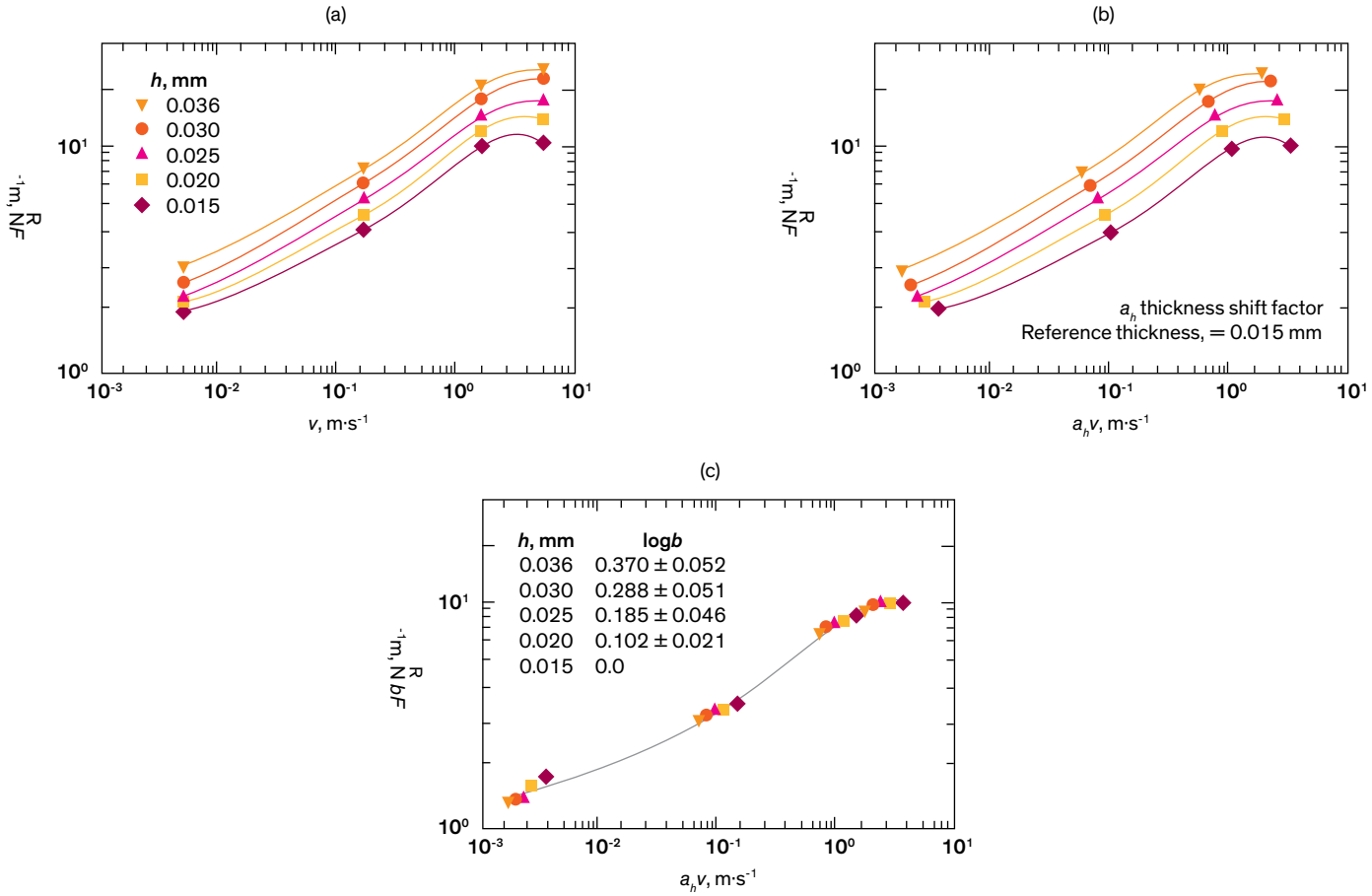
In an effort to maintain relevancy for the practical application of release coatings, the dynamic frequency (ω) from the viscoelastic measurements was related to the rate of delamination (υ) by accounting for the thickness (h) of the material subject to deformation in the peel test (Equation 1).

Equation 1. $$\omega = \frac{2\pi}{h}\upsilon$$

This made it possible to match viscoelastic events with the application time constant over the four decades observed in practical peel testing [14].

In Figure 2, time-temperature superposition was used to characterize the viscoelastic response of two adhesive

Figure 3. Plots of release force using adhesive thickness and velocity as variables, with the addition of normalization and vertical shift factors to show superposition of data at high peel speeds.



families (solvent-based acrylic and styrenebutadiene rubber) within the time scale of the peel test. The shaded data symbols in Figure 2 represent the practical peeling velocities used in generating the 180-degree peel data. It was evident from the general shape of the resulting release profiles that the inclining or declining profile observed in release force versus peel speed had its origins in the bulk viscoelastic properties of the PSA. Later in this paper, we will show that control of the release profile, while dominated by the organic adhesive, can be tuned through the choice of the silicone release surface and its viscoelastic characteristics.

Other practical considerations, such as the effect of adhesive thickness on release force, could also be accounted for through simple geometric considerations. Figure 3a shows the release profile of a hot melt adhesive from a vinyl-endblocked PDMS as a function of adhesive thickness (h). In Figure 3b, the profiles have been normalized for thickness, using the data from the thinnest adhesive (h = 0.015 mm) as a reference. Because the data did not collapse into a single curve, it was apparent that other factors influence the observed release force (F_R). Figure 3c shows that by applying a vertical shift factor (b), there is a reasonable superposition of data in the intermediate and high peel speeds.

If adhesive failure energy (G_a) is assumed to be independent of adhesive thickness at a particular peel velocity (υ), this would suggest that the actual peel angle (θ) decreases as h increases. Hence, from the relationship illustrated in Equation 2, an increase in release force values should be expected.

Equation 2. $$G_{a,} = (1 - \cos\theta)\, F_R$$

Recent works by Shull [15, 16] have used nano-indentation with hemispherical and flat punch probes to advance the understanding of the molecular basis for the viscoelastic process observed in the debonding process. The work, which combined forced-displacement measurements with visual observations, was able to delineate the energetics of the various processes involved in separation/release of glass and metal probes from organic adhesives. In Figure 4a, an initial dewetting process was followed by the formation of singularities at the interface, which in turn became the site of fibrils formation. The bulk of the dissipation process being observed involved the stretching of the fibrils. Working in Shull's laboratory, we modified the hemispherical glass probes with a PDMS release coating. Two coatings were chosen: a nominally 130 *dp* (degree of polymerization) vinyl-endblocked PDMS, and the same polymer matrix with a given percent of a high-release modifier (HRA). In Figure 4b, the low-release coating exhibited a characteristic circular contact area that simply decreased in size upon displacement until the surfaces separated at a critical interfacial shear force. In the HRA-containing experiment, Figure 4c, a similar circular contact area was observed; however, upon displacement, the dewetting process advanced to a greater degree. Displacement advanced to the point of singularity formation, but actual fibrils were not observed in the systems studied. These PDMS release coatings were further probed using lateral-force microscopy and interfacial-slippage experiments detailed in the following section.

The majority of commercial release coatings are based on platinum-assisted silicone vinyl/silicone hydride addition-cured chemistries [17]. The Pt and inhibitor molecules are used to tune the coating's bath life and cure temperature to optimize the operation of the coating line. The inhibitor's only impact on release performance is associated with its impact on extent of cure and, in some cases, manifests itself in better coverage. The main variable in the polymer design is the degree of polymerization (*dp*) of the vinyl-endblocked polymer. Figure 5 reveals several trends occurring in the release profile (release force versus peel speed) by simply increasing the *dp* between crosslinks. As the *dp* between crosslinks increased, the release force in the high-speed domain increased. Conversely, release force decreased in the low-speed domain. The net result of these two opposing trends is a general increase in the release profile with increasing *dp*.

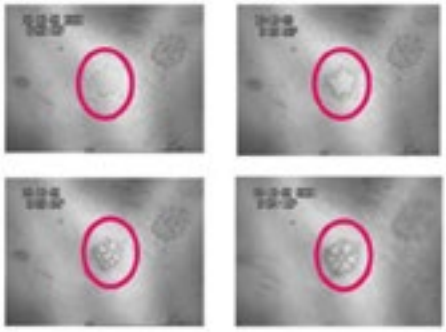
Figure 6 plots the bulk viscoelastic properties of these networks, and others with even higher *dp*, between crosslinks.

Bistac [7] used pin-and-wheel tribometry measurement together with peel testing to study adhesion and friction of PDMS against glass. In polymer-only networks, a more pronounced increase in peel force was found in networks with greater distances between crosslinks, a trend that increased with increasing separation rates. Higher *dp* leads to a greater quantity of free and pendent chains linked by one end only. Bistac argued that this increase in surface mobility creates better surface wetting and increased contact point, hence higher adhesion strength on separation. The separation energy was greatest at higher speeds because, at high speeds, the viscous resistance to chain slippage becomes more important. It was also observed that the higher-*dp* polymers had lower friction, yet higher adhesion. This finding was attributed to the formation of a highly mobile surface layer, a consequence of free ends with poor cohesion and shear resistance. Bistac's measurements were made at low peel speeds just bordering on those typically used in practical PSA applications shown in Figure 2.

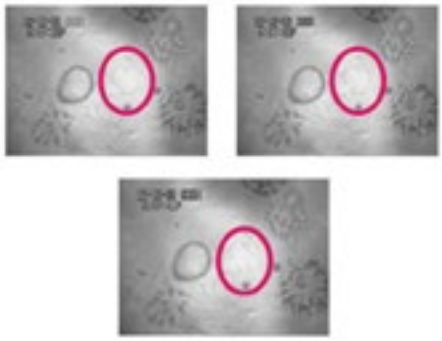
Chaudhury [18], working across a much broader velocity range (10^{-7} to 10^{-1} m·s^{-1}), also found that friction *decreased* with *increasing* molecular weight, while increasing as a function of velocity. A peak in shear stress as a function of velocity, and independent of molecular weight, was observed. The peak at 10^{-1} m·s^{-1} coincided with the inversion zone of the peel results

Figure 4. Visual observations combined with forced displacement showing the energetics of processes involved in the separation and release of glass probes from organic adhesives.

(a) Glass on adhesive by probe tack.



(b) Base polymer by probe tack.



(c) Resin-based network by probe tack.

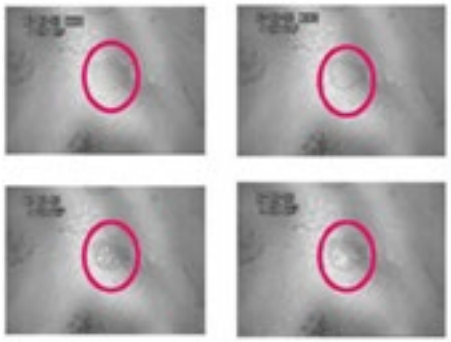
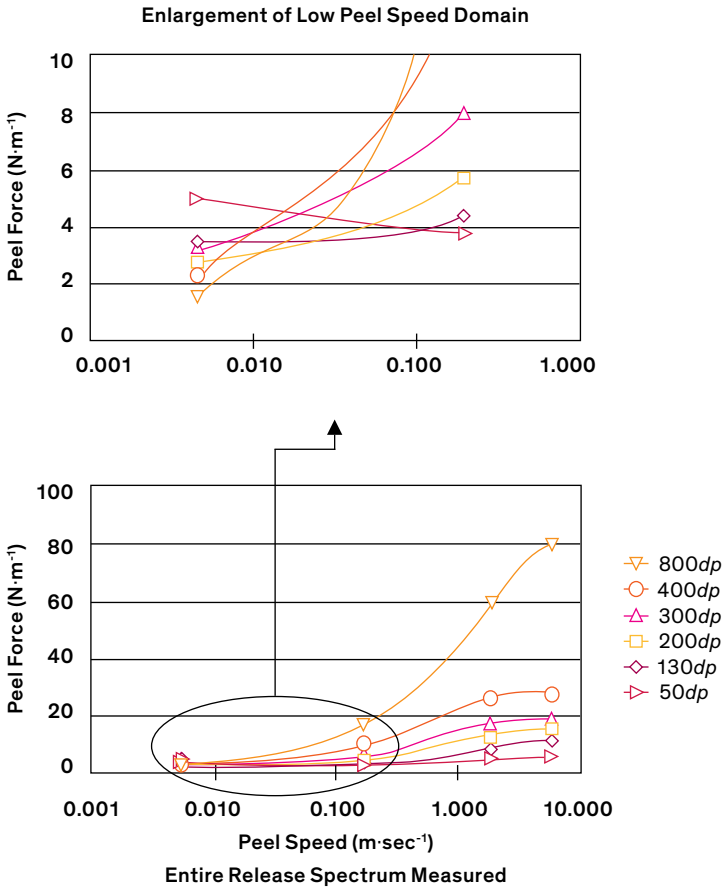


Figure 5. Release force versus peel speed as a function of polymer *dp*.



shown in Figure 5. While it is dangerous to make comparisons between two very different separation methodologies, it would appear that there is a broad transition from slippage-controlled release in the low speed domain to dissipative properties associated with higher-molecular-weight polymers. It is unclear whether this is describing the transition from interfacial to bulk rheological control. Considering the sample configuration and peel angle, it is possible (as previously suggested) that the high-speed results in this system are a result of a coupling of the bulk viscoelastic properties of the release coating and the adhesive.

In practical applications, the viscosity limitation engendered by multi-roll, high-speed coaters and the decrease in cure speed with increasing *dp* results in mainly PDMS polymers with *dp*s around the entanglement limit being used in solventless release coatings. Without the ability to broadly tailor molecular weight, the level of adhesion best suited for a given PSA application is achieved through the addition of an HRA to the silicone release coating. Figure 7 shows the combined effect of changing the *dp* of the vinyl-endblocked polymer and the HRA loading levels measured using an SBR tape aged for 28 days. Peel force versus peel speed reveals a trend toward an increase in low-speed peel force and a reduction in high-speed peel force as HRA levels are increased. The net effect is a reduction in the maximum peel force at intermediate peel speeds with almost flat profiles at the highest HRA loading level. Practical coat weight limitations prevent the study of higher loadings of HRA and polymer *dp*s under this set of conditions.

It has been shown elsewhere that the methyl silicate used as an HRA effectively tunes the viscoelastic properties of PDMS polymers and networks by impacting the segmental mobility of the flexible PDMS backbone. The damping of segmental motion is a bulk phenomena; and the methyl silicate, with its trimethylated silyloxy-covered surface, exhibits a slightly lower surface tension than the dimethylsiloxy polymer. Various surface analysis techniques have so far been unable to find evidence of unique methyl silicate surface states. The methyl silicate resin improves polymer wet-out by reducing polymer-polymer interactions. This increase in wetting should lead to increased release as greater numbers of polymer segments interact with the substrate. In release applications, the reduction in segmental mobility manifests itself as a systematic increase in friction as HRA loading is increased.

Figure 6. Effect of crosslink density of PDMS networks from vinyl-terminated precursors on the loss modulus (G'') at a frequency of 10^3 rad·s^{-1} and a reference temperature of 25°C.

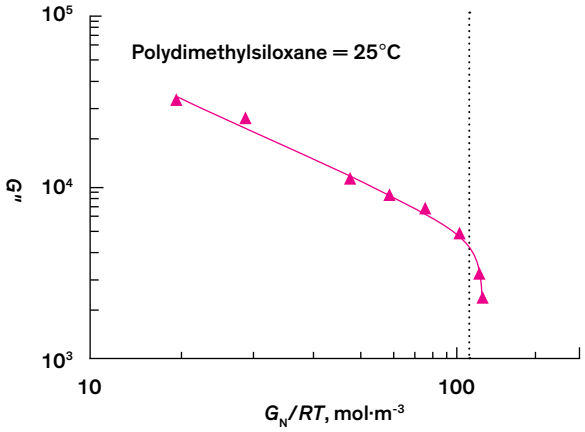
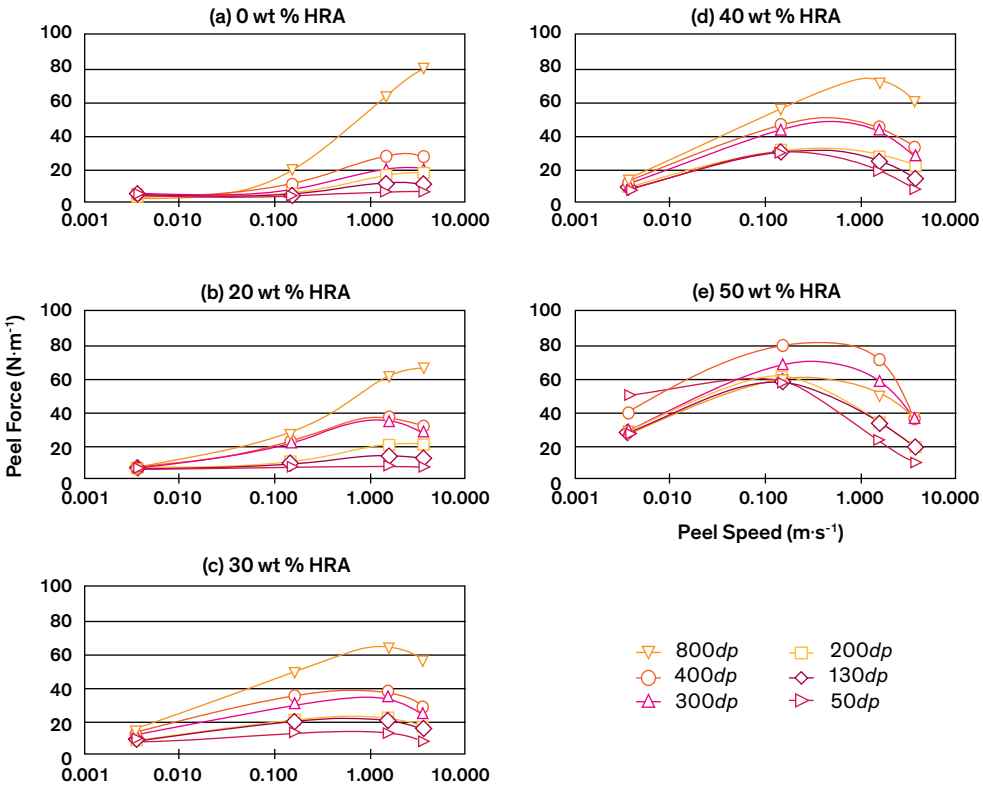


Figure 7. Release force versus peel speed as a function of polymer *dp* and HRA loading level.



Lateral-force microscopy clearly illustrates this increase in friction (Figure 8), where the force to move the LFM tip increases with added methyl silicate resin. This added methyl silicate prevents interfacial slippage as is shown in Figure 9. The particle displacement goes to zero with only a 17 wt % addition of methyl silicate resin and does not change with added methyl silicate. The shear stresses are also increased with added methyl silicate, particularly at higher velocity and higher resin concentration (Figure 10). It should be noted that the effectiveness of the methyl silicate is both rate and adhesive dependent. The interfacial slippage and shear stress data were generated at low peel velocities where the methyl silicate is most effective. The greatest impact of the methyl silicate on release force typically occurs at low to intermediate peel velocity (0.005 to 0.17 m·s^{-1}).

Amouroux [19] carried out many of the same experiments on similar PDMS based release coatings with and without HRAs. Amouroux's system differed significantly

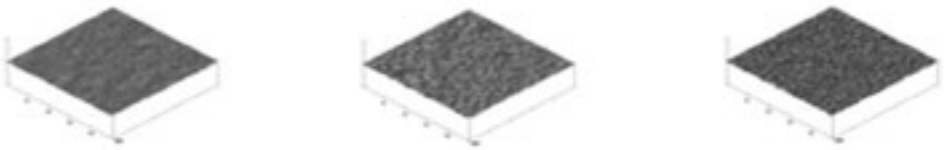
from that presented here and in previous publications by the same authors in that the SiH:SiVi ratio used for cure was 1.6:1 compared to 1.2:1. More significantly, however, was the nature of the methyl silicate HRA. The methyl silicate structure used in this study contained no soft-section dimethylsiloxy units, and reactive vinyl functionality was present only as vinyldimethyl silyl groups. Such gross changes in architecture make direct comparisons difficult. An example of this would be the reduction in slippage that Amouroux reported when going from 20 to 40 wt % HRA; whereas Figure 9 shows no slippage at any of the HRA levels tested. In this paper, we advocate surface pinning of the adhesive due to a universal reduction in surface mobility rather than direct, HRA-only, pinning of the adhesive as postulated by Amouroux. In previous work [20], no evidence was found for systematic changes in surface tension, nano-roughness or chemical composition in our systems that would explain the systematic change many groups have observed. The data presented is consistent with Brown's [21] observations that increasing the rigidity, i.e., reducing the flexibility, of the surface increases friction. This freezing of the surface results in an increase in the threshold stress supported at the interface and practical release force level.

Conclusion

Low peel speed results showed a decrease in peel (release) force as a function of increasing polymer molecular weight. At intermediate peel speeds of 10^{-1} m·s^{-1}, this dependence changed from decreasing to increasing force with increasing molecular weight. It is proposed that the molecular conformation of the silicone network at the separating interface controls the transition in the fracture process from one in which surface slippage circumvents the dissipation of force (resulting in easy release) to a more rigid surface capable of sustaining higher stress levels.

Chaudhury had proposed a release mechanism for cured PDMS-based release coatings in which interfacial slippage minimizes the bulk shear deformation experienced by the organic adhesive. The data presented here are consistent with this mechanism. Commercially used silicone release coatings exhibit interfacial slippage. Traditional HRAs used in release coatings "freeze out" interfacial slippage, resulting in increased adhesive deformation. The HRA reduces the segmental mobility of the PDMS chains within the cured network leading to increased shear stress and an increase in practical peel force. It is proposed that the mobility of the surface states, as controlled by the *dp* between crosslinks and the HRA content, simply allows for more efficient use of the viscoelastic dissipation states of the adhesive.

Figure 8. Lateral-force microscopy data for PDMS with 0 to 34 wt % methyl silicate high-release additive.



(a) 0 wt% HRA (b) 17 wt% HRA (c) 43 wt% HRA

Figure 9. Interfacial slippage data for PDMS with 0 to 34 wt % methyl silicate high-release additive.

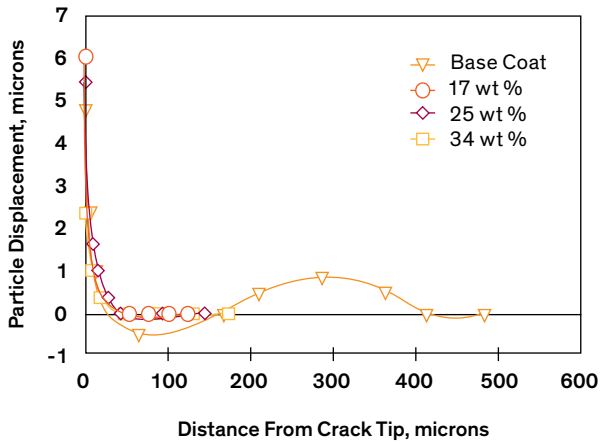
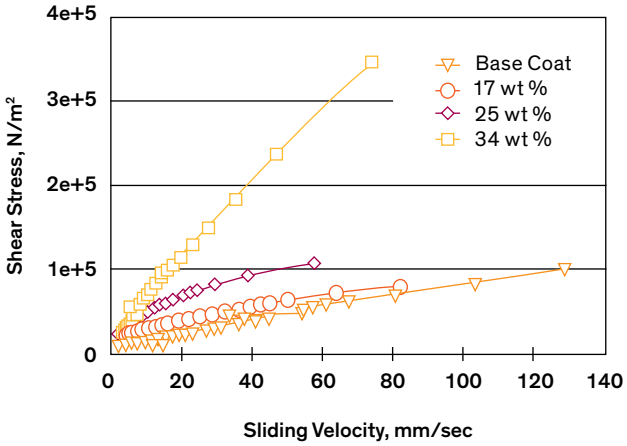


Figure 10. Shear stress as a function of sliding velocity for a PDMS coating with 0 to 34 wt % methyl silicate high-release additive.



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