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Hysitron Incorporated

Adhesion Measurement of a Pressure Sensitive Adhesive

Development of a Nanoindentation-based Testing Technique for PSA Materials

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Introduction

Repeatable testing of pressure sensitive adhesives (PSAs) is important to accurately predict the adhesive strength of a material for an intended application. However, adhesion testing often involves peel tests that depend on careful control of the peel angle, peel rate, thickness of the coating and modulus of the coating. Indentation testing is proposed as an alternative method for characterization of adhesion.

Procedure

The instrumentation used for this testing was a Hysitron TriboIndenter® equipped with Feedback Control. The TriboIndenter is a stand-alone nanomechanical characterization instrument that allows measurement of force and displacement of a probe relative to a fixed sample. This capability is most commonly used for investigation of physical material properties. A sphero-conical diamond probe with a radius of 650 nm, as calculated from Hertzian contact mechanics, was utilized based on the desired contact area for adhesion measurements. Poly-cis-isoprene was selected for testing based on its widespread use for PSA applications. Poly-cis-isoprene with a molecular weight of approximately 800,000 was acquired from a commercial distributor.

A one-centimeter square sample of the poly-cis-isoprene was mounted on a steel stub and placed into the TriboIndenter. The probe was brought into contact with the sample by utilizing the automated functionality of the instrument typically used for scanning probe imaging.

The Feedback Control capability allows one to define a withdrawal height of the tip from the initial position of the probe when it is in contact with the sample surface. A withdrawal height of 1400 nm was defined to ensure that the tip was free of any

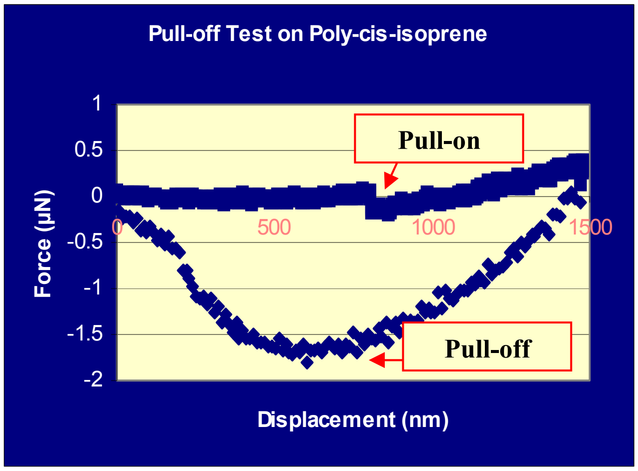

Figure 1. Typical pull-off adhesion test with points identified as pull-on and pull-off forces and displacements.

residual contact or surface forces. The transducer used for testing was then actuated under displacement control toward the surface with a total displacement of 1500 nm. The force is observed to be zero until the transducer brings the probe near enough to the surface for the probe-surface interactions to be sensed. When the attractive forces reach a critical value, the sample is pulled upward into contact with the probe, as observed at the point labeled "Pull-on" in Figure 1. In open loop or force-controlled devices, the probe would be pulled onto the surface in a snap-to-contact phenomenon, thus disallowing measurement of the attractive forces before contact. After the maximum displacement is reached, the transducer again withdraws the probe to the maximum withdrawal height. As also seen in Figure 1, this shows a maximum pull-off force due to the adhesion between the probe and the sample. This maximum adhesive force, or minimum force measured during withdrawal of the probe, is recorded as the pull-off force.

Each test was performed on the aforementioned specimen within a twenty-four hour period to ensure that degradation of the sample did not affect the results. Each test location was chosen to be at least two times the contact radius from the previous testing location to ensure that previous tests did not affect the local property measurements. The forward displacement was maintained at 10 nm/sec, while the withdrawal displacement was varied. Measurements were taken at 10, 100 and 500 nm/second withdrawal rates.

Results and Discussion

Initial analysis of the results showed a significant distribution in the maximum adhesive force for identical withdrawal rates. However, good repeatability was observed for each withdrawal rate when analyzed as a function of maximum sample penetration. Figure 2 shows the maximum adhesive force plotted as a function of sample penetration for each of the withdrawal rates tested. The sample surface was defined as the point at which the "pull-on" phenomenon was observed. The force clearly increases beyond this point, implying increasing stiffness due to increasing contact area with the sample. The measured adhesive forces correlate with the sample penetration, because increased penetration causes increased contact area of the tip/sample interface. The area of the interface will clearly affect the force or energy required to break the interface. Figure 2 also shows a clear dependence of the adhesive force on the withdrawal rate due to the dynamics of bond breaking and the viscoelastic nature of poly-cis-isoprene.

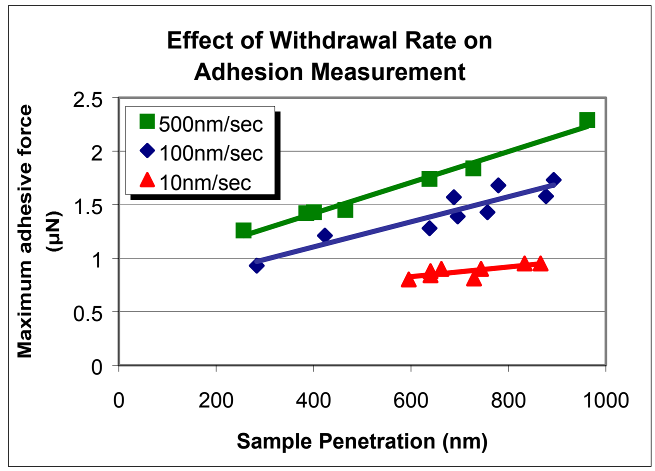

Figure 2. Adhesion measurements from pull-off tests showing effect of sample penetration and withdrawal rate.

Conclusions

Nanoindentation has been successfully implemented as a tool for measuring adhesive pull-off forces. This technique eliminates some of the effects that complicate traditional testing techniques. Future work will include modification of the test probe for adhesion measurements between other materials. Additional work will also include incorporation of a technique that allows continuous measurement of contact area. This will provide the capability of calculating surface energy from the pull-off test.

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