

PROBE TESTS INVESTIGATIONS OF BULK AND SURFACE CAVITATION PROCESSES ON MODEL PSA'S

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The adhesion mechanisms of a model pressure-sensitive-adhesive, based on a styrenic block copolymer and a tackifying resin, have been investigated with two mechano-optical methods. The simultaneous observation of the debonding mechanisms and the measurement of the real contact area and the force show, that the debonding starts with cavitation in the bulk of the adhesive layer. Subsequently a second population of cavities appears at the interface between the probe and the film and the film eventually separates adhesively from the surface of the probe.

After the pioneering work of Zosel¹, a series of recent experimental^{2,3} and theoretical⁴⁻⁶ investigations have shed some light on the detailed mechanisms by which a soft adhesive can dissipate large amounts of energy upon debonding from a hard surface even when interfacial interactions are weak Van der Waals bonds. The key to a large energy dissipation is the formation of bridging fibrils which can transfer stress from one surface to the other over large distances.

In this study we have used two complementary probe techniques to investigate the process of formation of these fibrils in the case the debonding of a styrene-isoprene-styrene (SIS) based pressure-sensitive-adhesive (PSA) from a glass surface. Probe techniques are ideally suited for the testing of very soft materials since everything can be assumed to be rigid and non-dissipative except the adhesive film. Furthermore, for very soft adhesives which can form fibrils upon debonding, a flat probe (alignment problems notwithstanding) has the advantage of applying a uniform displacement field to the film⁵.

Both testers used a flat ended probe to test the tackiness of the PSA. One of the devices was equipped with a video camera, which allowed visualization (through the film thickness) of the cavitation inside the film during the debonding stages as shown schematically on figure 1. In the other device, shown on figure 2, the contact probe was a total reflexion prism which allowed the quantitative measurement in real time of the fraction of the probe actually in molecular contact with the adhesive⁷. The combined use of both devices can give clear insights on the location where cavities appeared first (in the bulk of the adhesive or at the interface with the probe) and how they evolved with time.

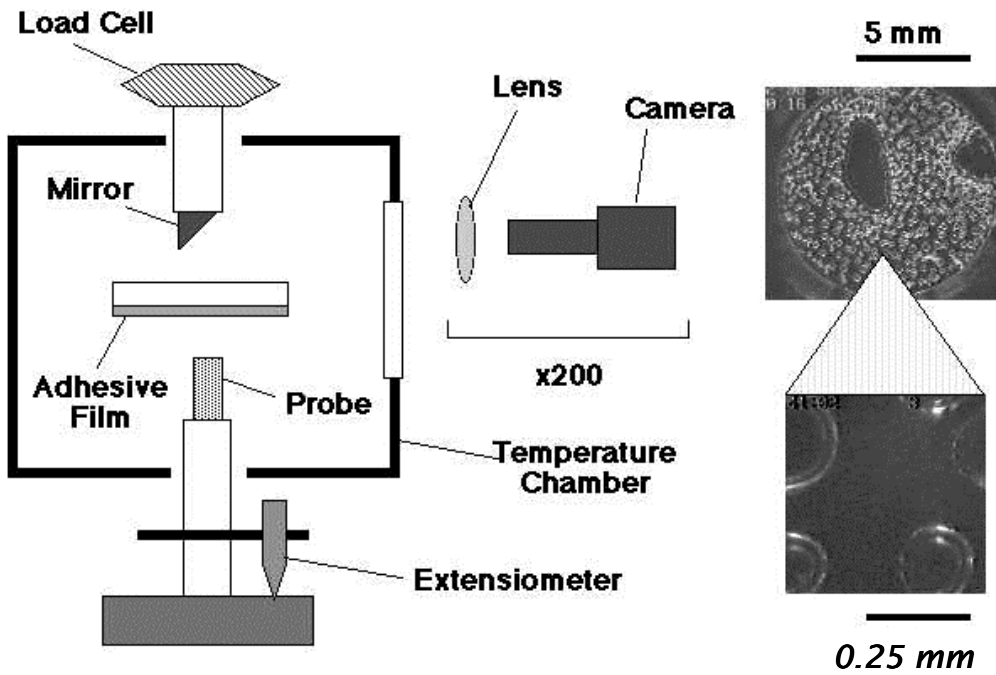


Figure 1: Schematics of the video probe tack tester

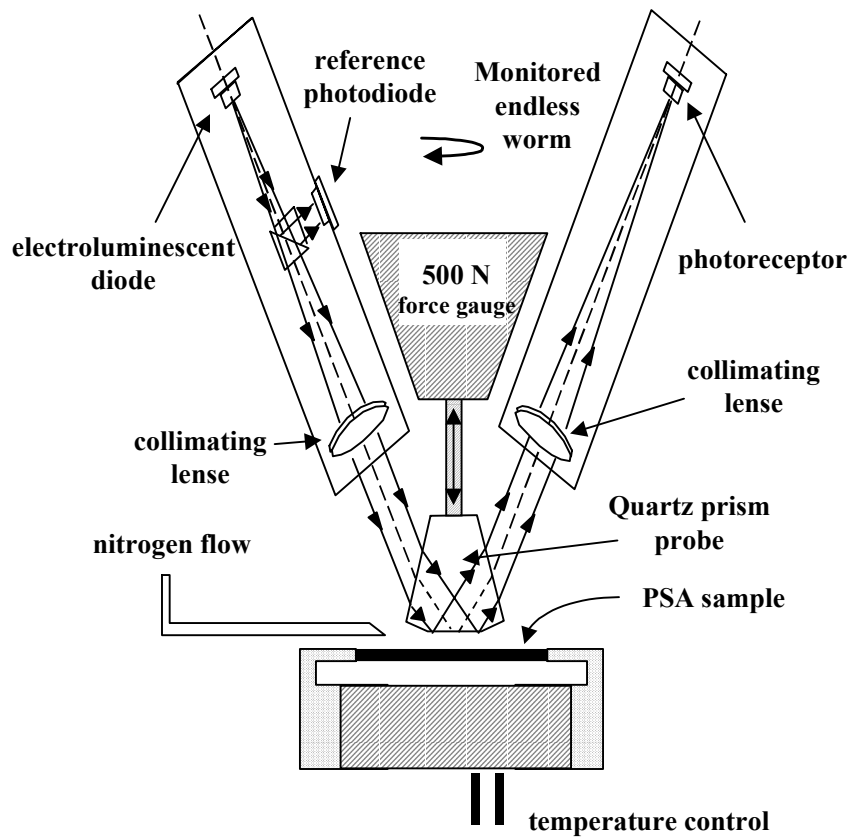


Figure 2: Schematics of the MOTT probe tack tester

Experimental

The styrene-isoprene-styrene block copolymer was purchased from Exxon Chemical (Vector 4211D). It is a pure triblock with 30wt% styrene. Its number average molecular weight (PS standards) was 114000 g/mole and its polydispersity index was 1.06. The tackifying resin was purchased from Hercules (Regalite R101) and was a low molecular weight C-5 hydrocarbon resin with a number average molecular weight $M_n = 1200$ g/mole and a polydispersity index measured at 1.2.

15wt% solutions were prepared of a 50/50 (by weight) blend of Vector and Regalite. Films were either prepared by solution casting the solution with a pipette onto precleaned standard microscope slides (75x25x1 mm) or were pressed from the melt in between two silicone release paper sheets. The resulting film thicknesses were around 150 μ m. Films were then stored in a vacuum dessicator and were not kept longer than two weeks.

Results

Probe tack experiments were performed on our custom-designed probe tester allowing the simultaneous observation of the debonding process through the transparent glass substrate and on the MOTT tester. A schematic of the test geometries are shown on figure 1 and 2 and further details on the experimental setup can be found elsewhere^{2,7}. For both instruments, the maximum area of contact was determined either from the video observation or from the laser, and the force-displacement curve was transformed into a nominal stress-strain curve by dividing the force by the maximum contact area during the compression stage and by dividing the displacement by the initial thickness of the adhesive film.

A first interesting result which we obtained is that stress-strain curves obtained on both instruments could be superposed very well if the actual area of contact was used to normalize the force. Since the measurement of contact area in the video tester is macroscopic while it is microscopic on the MOTT tester, one can infer that no significant air pockets were trapped at the probe/film interface during the compression stage.

A tack curve obtained from the video tester as well as a series of images taken from underneath the glass probe are shown on figure 3. It is clear from this sequence that the a first population of cavities appear around the maximum in force and subsequently grow first laterally along the plane of the adhesive film and then normal to it, forming a 3-dimensional foam with extended walls. Since the video only gives a projected image, it is difficult to determine unambiguously the location of the nucleation of these cavities. It is possible however to measure the non load-bearing area from the projected area of the cavities.

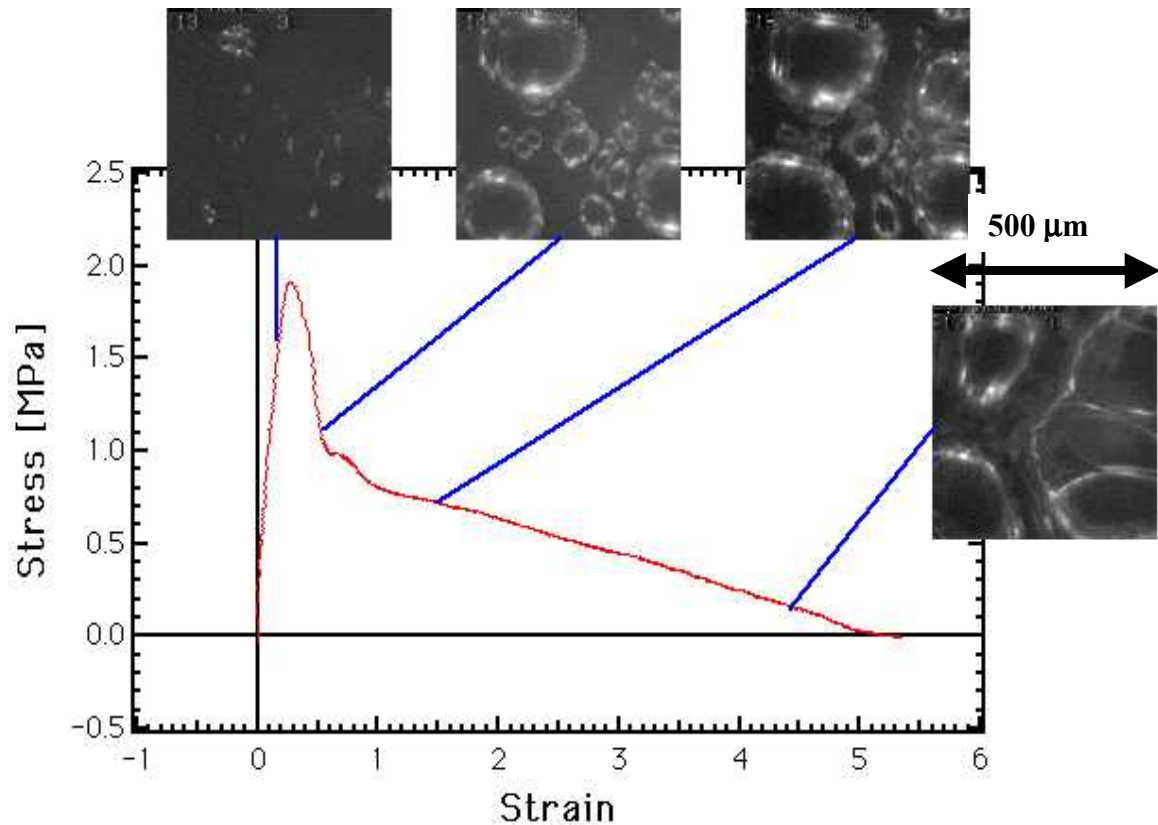


Figure 3: Stress-strain curve of an SIS+resin adhesive on glass.

If the same experiment is done on the MOTT, the force and the real contact area with the quartz surface are directly measured. Clearly, as shown on figure 4, there is a time lag between the appearance of the first population of cavities on the video images and the decrease in contact area. At the force maximum, where most of the cavities are already present, the contact area is still close to 100% and this contact area starts to decrease only at a later stage, when all the main cavities are fully expanded in the plane of the adhesive film. This result proves that, for this system, the cavities are initially nucleated inside the adhesive layer rather than at the interface between the adhesive and the probe.

This implies that the presence of defects or trapped air at the surface is not necessary to nucleate cavities and initiate the highly dissipative fibrillar structure. On the contrary one expects that if the cavities can be initiated in the bulk, the adhesion energy should be maximized.

On figure 3, a second population of cavities is observed to nucleate in between the first population. From the MOTT data, this second population of cavities nucleates later at the interface between the probe and the film (stage B on figure 4) and is then responsible for the interfacial failure which is eventually observed (stage C on figure 4). In this last stage, the contact area drops to zero when the small interfacial cavities coalesce and the fibrils debond so that no adhesive remains on the probe surface.

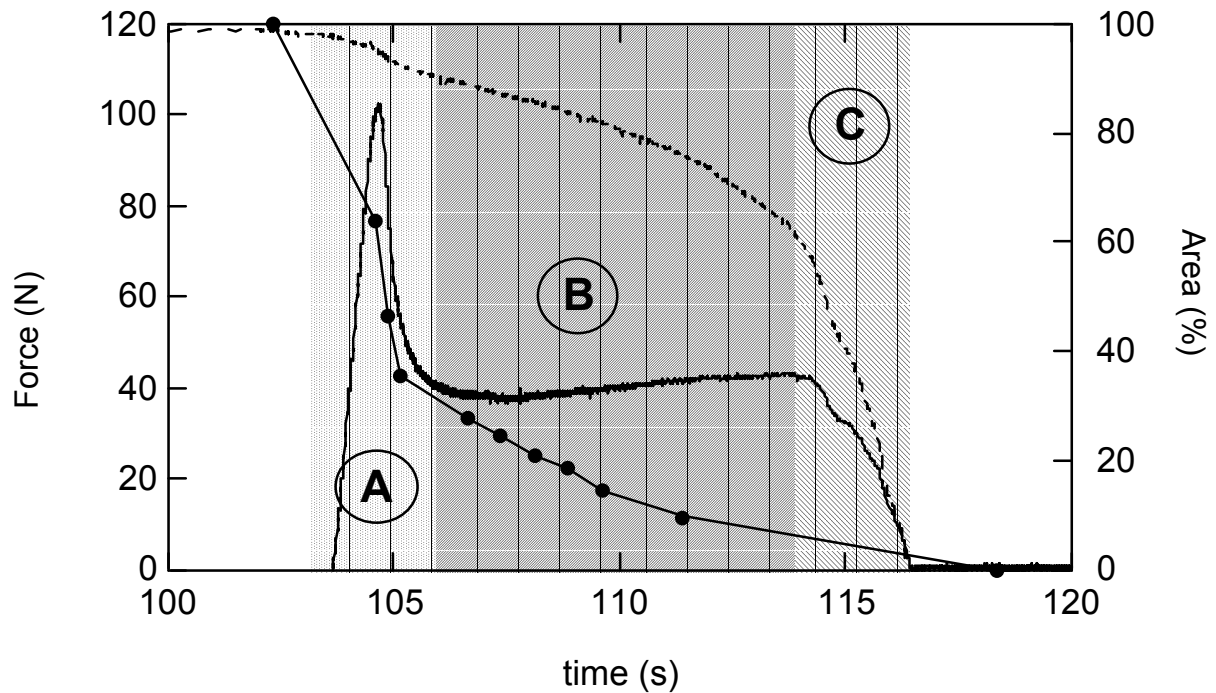


Figure 4: Force (full line), load-bearing area from the video (filled circles) and real contact area (dashed line) as a function of time during a probe test. The force and real contact area data are obtained from the MOTT tester. The debonding occurs in three stages: stage A (nucleation of cavities in the bulk of the adhesive layer); stage B (progressive nucleation of a second population of cavities at the interface, from figure 3), stage C (coalescence of these interfacial cavities and debonding of the fibrils).

While one must keep in mind that a change in topography of the surface (to a rough sanded surface for example) can lead to the nucleation of cavities at the interface and for much lower levels of stress, we anticipate that a combination of direct visual observation of the mechanisms which provides a spatial information, and of a surface analysis techniques which can analyze a locus of failure, will prove to be very useful tools in such investigations.

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