Morphology and Elasticity of Waterborne Acrylic Pressure-Sensitive
Adhesives Investigated with Atomic Force Microscopy

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Running head: AFM of waterborne pressure-sensitive adhesives

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Abstract

The morphology of pressure sensitive adhesives (PSAs), especially at the surface in contact with a release liner, is expected to have a dominant influence on the tack strength and energy in an application. We have used tapping-mode atomic force microscopy to determine the morphology at the surfaces of freshly-cast waterborne acrylic PSAs over lateral length scales of a few μms . We demonstrate that topographical features on silicone release liners can be used to pattern the PSA surface in contact with it. Control of the texture of a PSA surface can potentially be exploited to tailor its properties. Latex particle boundaries are much better defined at the air surface of the PSA in comparison to its back face. A series of experiments suggest that this difference results from the distribution of water-soluble species within the dry film. The pressures and processes involved in the transfer lamination process do not alter the PSA morphology. The first reported AFM images of the response of these materials to pressure and shear provide insight into the deformation mechanisms. Amplitude-distance curves on PSA surfaces show that there is a small decrease in tack and an increase in stiffness after ageing for 13 months.

Key words: acrylic, latex, pressure-sensitive adhesive, atomic force microscopy, indentation, roughness

1. Introduction

Pressure-sensitive adhesives (PSAs) adhere quickly and firmly to a surface under the application of light pressure. They exhibit high adhesion strengths without necessarily any chemical bond formation at the interface [1]. PSAs are increasingly being used in more demanding applications that require enhanced performance characteristics [2]. Much progress has been made in understanding the mechanisms of tack and debonding in PSAs through a combination of modelling [3, 4, 5, 6] and experiment [7, 8, 9, 10, 11, 12]. This present work demonstrates a method to control the surface topography of a waterborne PSA. The topography and elasticity are then examined using atomic force microscopy. The relevance of this work will become clear in the following discussion.

Recent theoretical models [13, 14] and experimentation [15, 16] have shown that the surface roughness of the *adherend* (or probe surface) profoundly affects adhesion strength and tack energy. Likewise, the surface roughness of an *adhesive* is likely to influence the PSA performance in one or more ways: (1) decreasing the contact area between the adhesive and adherend; (2) creating interfacial microbubbles that provide suction; and (3) nucleating cavities in the de-bonding process. Each of these effects will be considered separately, although they are inter-related.

Contact Area. The tack energy of PSAs has been found experimentally to be proportional to the square of the *true* microscopic contact area with the adherend (as measured optically) [7]. In turn, the contact area is a function of the surface roughness of the adhesive, according to a recent model [3]. Other models have taken

into account the effect of the surface roughness of the adherend in influencing the contact area with the adhesive [4].

Microbubbles and suction. A theory of tackiness developed by Gay and Leibler [5] assumes that the wavelengths of the roughness of the adhesive and adherend differ. When the two surfaces are placed in contact, two populations of microbubbles are created. It was proposed that these microbubbles produced suction that increased the tack energy and the force required to separate the two surfaces.

Cavitation. Probe tack tests have yielded great insight into the debonding mechanisms of PSAs. It has been clearly established that the formation of cavities at the interface, followed by fibrillation at higher nominal strains, is responsible for high tack energies [10]. Microbubbles at the interface, partially the result of adhesive roughness, are potential sites for cavity formation [5]. Internal interfaces in the film, resulting from poor particle coalescence, might serve as "weak points" that determine the number of cavities that are formed [6].

In the case of waterborne (*i.e.* latex) PSAs, another factor must be considered. The presence of surfactants (and other low molecular-weight species) is likely to weaken the interface between the PSA and adherend and also might create nucleation sites for cavitation. Depending on the latex and surfactant types and the drying conditions, excess surfactant can be found at the PSA/air interface [17, 18] but also at the PSA/substrate interface [19, 20]. The presence of surfactant has been found to have a pronounced influence on the adhesion properties [21, 22]. Holl and co-workers [23] have shown that the interfacial effects resulting from the weak boundary

layer of surfactant dominates the other effects influencing the adhesion properties. It is indeed noteworthy that surfactant is found at the locus of failure of the film [24].

Further development of PSAs requires an analytical technique able to probe the topography of the soft, tacky surfaces of PSAs. Optical microscopy can characterise surface features over length scales larger than the diffraction limit of light. But a technique to analyse the surface roughness of PSA surfaces and to characterise the surfactant distribution, at the nanometer scale, is also required. Detailed studies of adhesive roughness and topography are lacking in the literature.

Atomic force microscopy (AFM) has been extensively applied to many materials within the last decade, including latex films. However, in the field of PSAs, its use has been restricted by the problem of the AFM tip sticking to the adhesive surface and by deformation of the very soft surface by the tip. There are only a few reports of AFM applied to PSA surfaces [25,26,27,28]. Often AFM has been restricted to aged PSA surfaces that are less tacky and thus present less experimental difficulty [25, 26].

We have recently shown that it is possible to image latex PSA surfaces by using some rather extreme AFM tapping parameters [17]. In the present paper we demonstrate how AFM can be used as a characterization tool for PSA surfaces. Surface topography, the distribution of surfactant phases, and elasticity are all simply evaluated by this single technique. We provide the first AFM images showing the response of waterborne acrylic PSAs to pressure and shear.

2. Materials and Methods

2.1. PSA characteristics

A model acrylic PSA latex (referred to in previous publications [17, 29] as PSA A) was investigated. The latex dispersion has a bimodal particle size distribution (average particle sizes of 180 nm and 350 nm) and a solids content of 60 wt.%. It was prepared by standard semi-batch emulsion polymerization. The polymer is a random copolymer based on an acrylic ester, 2-ethylhexyl acrylate, which yields a soft and tacky material with a low glass transition temperature (T_g). High T_g monomers (such as methyl methacrylate) are added in a smaller proportion to increase the T_g , in order to improve the cohesive strength as well as the peel and tack properties. A small amount of polar monomers (acrylic acid and methacrylic acid) are also copolymerised to improve adhesion on polar substrates and to provide sites for cross-linking reactions with other groups [13]. Dissolution of the latex polymer in organic solvent has revealed a low insoluble fraction, which indicates the presence of a significant amount of polymer chains with very high molecular weight and/or a partially cross-linked network. It is noteworthy that previous unpublished work in our laboratory has found that reducing the molecular weight did not enhance particle coalescence, as was observed by AFM. The T_g of the copolymer, as determined by DSC, is -45 °C. The loop-tack strength of the PSA film, determined according to Finat Test Method No. 9 (FTM9), is 512 N/m.

2.2. Preparation of samples

Films were cast onto silicone-coated release liners (30 cm x 20 cm) using a 40 μm hand-held bar coater. Both paper and polyester substrates were used. Hereafter, the release liners will be therefore be referred to as silicone/paper and silicone/polyester. The films were dried under laminar air flow for 3 min. on heated plates at 60 °C in a controlled humidity chamber maintaining a relative humidity of 40 %. (This drying temperature was found to be the average temperature experienced by the latex on an industrial coater.) The dried films were about 20 μm thick.

PSAs laminates are usually manufactured via a lamination transfer process, which is illustrated in Figure 1. First, the film is dried on a release liner (usually silicone/paper), then laminated on a facestock (paper or films of polymers such as poly(vinyl chloride), oriented polypropylene or poly(ethylene terephthalate)). In this work we have characterised both faces of the film: the PSA/silicone release liner face, which becomes the adherend contact face in an application, and the original top face that comes into contact with the facestock. Poly(vinyl chloride) sheets were used as the facestock in the transfer lamination process in the present work. Our sample preparation method replicated, as closely as possible, the transfer lamination process that is used in industrial production (Figure 1). In some AFM experiments, the PSA was subjected to a pressure of 3×10^6 Pa before analysis. The surface of interest was placed in contact with a silicone release liner and compressed in a vise.

2.3. Atomic force microscopy of PSA surfaces

Small pieces (1cm x 1cm) of the cast PSA were cut from the large-area films and were analyzed with an atomic force microscope (Nanoscope IIIa, Digital

Instruments, Santa Barbara, CA, USA) within three hours of casting. No significant evolution of the sample surface over a period of up to 12 hours after casting was observed. All measurements used a silicon cantilever (NT-MDT, Moscow, Russia) equipped with an ultrasharp, conical silicon tip having a radius of curvature of about 10 nm. The nominal resonant frequency f_o of the cantilever is 320 kHz and its spring constant k is 48 N/m.

As indicated in Figure 1, AFM analysis was performed on either the original interface with air (at Stage 2) or on the back face of the PSA after release of the silicone liner (at Stage 5). Images were recorded simultaneously in the topographic (height) mode and in the phase mode, with scan sizes ranging from 5 μm to 30 μm . The optimum method for obtaining images of latex PSA surfaces has been reported previously [17]. Parameters needed to describe the tapping conditions are the "free" amplitude A_o and the setpoint value d_{sp} , which are defined in Figure 2.

The high tack of the PSA surface makes it necessary to use high tapping amplitudes to impart enough energy to the tip to "pull off" of the adhesive surface. A high setpoint ratio is required to minimise indentation of the tip and thereby to reduce deformation of the soft surface. To obtain values of A_o and d_{sp} in metric units, a systematic calibration of the cantilever was obtained from amplitude-distance curves on a clean silicon wafer, assuming no deformation of the silicon surface and no bending of the cantilever during tapping [30].

All AFM images presented here have been obtained with nearly the same tapping conditions, as follows. The setpoint distance d_{sp} was between 90 and 100 nm

for all measurements. A_o was typically 20 nm above the d_{sp} for the air surfaces of the PSAs and 30 to 40 nm above the d_{sp} for the face delaminated from the release liner. These tapping conditions ensure that the indentation of the AFM tip into the PSA surface is small enough to avoid significant distortion of the morphology. It is, however, important to realise that the surface is necessarily indented by contact with the AFM tip [17].

Amplitude-distance curves obtained with the AFM have been used to monitor the adhesion and mechanical properties of the surfaces. In an amplitude-distance curve, the variation of amplitude of the cantilever oscillation is monitored as a function of the scanner displacement as the sample surface is brought closer to the cantilever tip. When the tip is far from the sample surface, the amplitude of the cantilever oscillation is, by definition, A_o . When the tip enters into the range of interaction forces with the surface, there is usually a slight decrease in the free amplitude value and a phase shift in the oscillation. The contact point is defined as the start of the amplitude decrease.

Amplitude-distance curves were obtained from PSA surfaces using different A_o values (and hence different tapping energies). A qualitative comparison of fresh and aged PSA surfaces was made. The curves were repeated in five different areas [31], and examples presented here are representative of typical results. Amplitude-distance curves obtained from the PSA surface can also be used to evaluate the indentation depth of the tip into the PSA surface, as described elsewhere [17], and thereby can probe the surface hardness.

3. Results and Discussion

This paper is divided into three inter-connected sections. The first section shows that surface features on the silicone substrate are permanently imprinted in the PSA cast on it. The second section compares the particle coalescence between the top and the back faces and considers the origin of the observed differences. The third section qualitatively explores whether the elasticity of the PSA surfaces evolves over time.

3.1 Imprinting of Substrate Patterns on the PSA Surface

We first consider the morphology of the surface of the silicone/polyester release liner. Figure 3 shows a large area AFM scan showing the topography of the silicone/polyester surface. Channels with a depth of about 80 nm and a width of 1-2 μm are observed in topographical images. The pattern and the density of channels vary across the surface of the release liner. The channels are attributed to imperfections resulting from the silicone coating process. There is no evidence that the surface chemistry varies laterally across the channels.

We have discovered that this pattern of channels in the silicone/polyester release liner can be imprinted into the back face of a PSA film that is cast upon it. Figure 4 illustrates the effect as characterised by AFM. The top face of the PSA (obtained during Stage 2 of the process) has no dominant features except for the texture of the latex particles. The back face (obtained during Stage 5), by contrast, shows ridges with an appearance and dimensions that are comparable to the

silicone/polyester surface. It is apparent that these ridges result from “moulding” in the 80 nm deep silicone channels, but they are only about one-half the height expected if accurate replication had occurred. Hence, there is evidence for some relaxation or flow of the surface after delamination. The apparent differences between the topography of the channels and the moulded ridges, however, can also be partly attributed to deformation of the PSA induced by indentation of the AFM tip. In any case, the PSA surface does not fully flatten under the action of capillarity [32] when in contact with air, as would be expected for a strictly viscous liquid.

Replication of the release layer topography was likewise demonstrated with a silicone/paper substrate having greater roughness than in the silicone/polyester. Figure 5 shows the results. Ridges, up to 300 nm high, are observed in the substrate (Figure 5A). This topography is replicated in the PSA surface that was delaminated from it (Figure 5B), although the ridges are less than 200 nm in amplitude.

As already described in the Introduction, microbubbles might result from a difference in the length scales of roughness at an adhesive/adherend interface. It has been suggested that these bubbles create suction that can increase the adhesion strength [5]. On the other hand, excessive surface roughness of either the adhesive or adherend will limit the contact area at the interface, which can lower the adhesion strength and tack energy [7, 3]. Our results indicate that patterns on a silicone release layer could be used to tailor the surface topography of the PSA surface in order to impart the desired properties. Recent models of PSA adhesion specifically consider the roughness of the adhesive by describing it as cylindrical columns [3]. Patterns in

the silicone release liner could be used to create any desired geometrical features at the optimum length scales.

3.2 Comparison of Top and Back PSA Surfaces

Film morphology on a shorter length scale is considered next. Films usually appear flat over lateral distances of a few μm . Figure 6A is a representative image of the top surface of the PSA (Stage 2). Previous analysis has concluded that the particles near the air surface are flattened and cylindrical in shape [17]. Particle identity is clearly retained. The reason for this non-coalescence of particles has been previously attributed to the presence of the latex serum, containing surfactants and other low molecular weight species (mainly residues from the emulsion polymerisation) [29]. This liquid-like phase is rather uniformly distributed near the top surface, as can be observed in the larger area AFM images in Figure 4A.

In PSA technology, the film face in contact with the release liner during the transfer process is of great interest because it will be subsequently placed into contact with the adherend (Stage 6 in Figure 1). Figure 6B shows such a surface after delamination from the release liner. We are not aware of any previous reports of an AFM image of the *back* face of a waterborne acrylic PSA. There is an obvious difference between this image of the back face in comparison to the top face (Figure 6A).

Individual particles can be seen on the back face, especially in the phase image. The back surface is less regular and apparently rougher than the top surface.

The dark areas in the height image and the bright regions in the phase image are interpreted as species formerly contained in the latex serum. This second phase is not uniformly distributed around the particles. Some aggregates have formed. In comparison to the top face, the particle boundaries are less well defined. At the back face, there appears to be less of the latex serum phase. This apparent difference in the latex serum concentration between top and back faces is currently under investigation in our laboratory using complementary analytical techniques. The silicone/PSA interface probably has a low energy, which would not favor segregation of surfactant. The distribution of water-soluble species is probably influenced by the water concentration profiles during drying.

We now consider the reasons for the observed differences in morphology when comparing the back and top surfaces. These differences could be the result of one or more stages in the processing that are experienced by the back surface but not by the top face. The back face experiences contact with the silicone release liner during drying (Stage 1), the pressure applied with the rubber roll during the lamination of the PSA on the PVC sheet (Stage 3), prolonged contact with the silicone release liner (mainly Stage 4), and the operative forces during the delamination of the silicone liner (Stage 5). These factors will be considered in the following discussion.

First, experiments were conducted to explore the effect of pressure and delamination on the morphology and arrangement of particles, as a way to mimic the effects of delamination in Stage 5. A silicone/polyester sheet was applied to the top face of a PSA under slight pressure for two seconds and then removed. This procedure was repeated three times, and then the surface was analysed by AFM.

Images obtained before and after this process appear very similar. The phase contrast is found to be slightly lower, but particle boundaries are still clearly defined, and there is nothing in common with what is observed at the back face. These simple experiments indicate that contact with a silicone surface, the application of light pressure, and de-lamination are not sufficient to disrupt the phase separating the particles at the top surface. It is not expected that these factors contribute strongly to the observed morphology at the delaminated back face (Figure 6B).

A further experiment aimed to determine the effect of prolonged contact with a silicone surface on the particle morphology and arrangement of the top surface of the PSA. One might expect that a long contact time with silicone substrate could allow reorganisation of the surfactant phase and simultaneous particle coalescence. However, after the top face was placed in contact for four hours with a silicone/polyester release liner, there was no significant change in the appearance after delamination, as revealed in Figure 7. Because the back faces in our experiments are usually analysed within two or three hours after delamination, we do not interpret the contact with the silicone liner as being related to the very different structure observed on the delaminated back face.

In another experiment, a silicone/polyester sheet was applied onto the top face of a PSA for 10 seconds under a high thumb-pressure that also induced shear forces. The silicone/polyester was then removed, and the PSA surface was immediately analysed with AFM. The start of the scan was delayed for four minutes after the removal of the silicone/polyester sheet while the AFM scanning parameters were being optimised. Two successive scans of the same area were performed and are

shown in Figure 8. The first scan was recorded, while moving from the top to the bottom (Figure 8A), and ended 11 minutes after the silicone liner removal. The second scan was started immediately after, moving from the bottom to the top (Figure 8B). The second scan finished 18 minutes after the silicone liner removal.

It can be observed in the first scan that particles are distorted from their circular shape by the shear stress that had been applied. As the scan progresses to the bottom of the image, the particles start to relax, and they appear less deformed. When the first scan is finished, the particles have almost completely relaxed, and so there is not much difference in the particle shape at the bottoms of the two images (8A and B).

A white line has been drawn on the images as a guide to the eye to illustrate the consistency of the position of the particles in relation to each other. Elastic contraction of the surface, primarily in the direction running from the top to the bottom of the image, is vividly demonstrated. Comparison of Figure 8B to images of the top surface after casting (Stage 2), such as in Figure 6A, reveals that the surface is not permanently altered by the application of pressure and shear forces great enough to deform the particles and to strain the film. The particles keep the same neighbors; they are displaced across the surface without changing their position in relation to each other. Apparently, stress is transmitted from one particle to another, but this stress does not disrupt the phase that surrounds the particles. Particle identity is retained.

These results provide convincing evidence that neither the pressure of the lamination process (Stage 3) nor the delamination of the silicone release liner (Stage 5) should alter the morphology of the back face. Instead, we propose that the differences observed between Figures 6A and 6B are due to differences in the distribution of the surfactant and other water-soluble species in the film that develop during film formation. In other work, we have shown that the surfactant phase at the top surface can stabilise particles against coalescence [29]. There appears to be less surfactant at the back face, and so there is a greater degree of coalescence. The reason for this difference in the surfactant distribution is currently under investigation in our laboratory.

In a similar experiment, the PSA surface was once again subjected to a shear force by a silicone sheet, but then the sheet was left in contact for 12 min. before delamination. AFM analysis of the delaminated surface found no evidence for particle deformation or strain of the surface, as was the case in Figure 8A when the surface was analysed shortly after delamination. We conclude that the PSA surface was initially deformed elastically during the application of the pressure and shear. During the storage, however, the PSA surface was able to relax to its original morphology by moving laterally along the silicone surface.

The elasticity and plasticity of the PSA surface was also examined on a larger length scale. The rough surface of a silicone/paper release liner (*cf.* Figure 5A) was pressed onto the top of a PSA film using a vise. The high resulting pressure (*ca.* 3 MPa) was found to have a limited effect on the individual particles, with only some partial particle aggregation being observed. On larger length scales, there was no

impression or indentation into the PSA surface from the topography of the silicone/paper substrate. The surface of the dry PSA behaved elastically and did not exhibit any plastic deformation.

3.3 Elasticity and Tack of Fresh and Aged PSAs

From a practical point of view, any stiffening of the PSA surfaces would have an impact on adhesion performance, particularly because it would inhibit the wetting of an adherend. In PSA films formed from rubber/tackifier blends cast from solution [33], stiffening was observed and was attributed to phase segregation during ageing. The phase separation also induced a loss of tack, which enabled the AFM analysis of the aged PSA films. In the acrylic latex PSA investigated here, there is no tackifying resin. Experiments were conducted to determine if the film properties at the surface were subject to change with ageing.

We have noticed that the scanning conditions necessary to image *fresh* PSA films are not the same that are required for *aged* films. When the PSA is aged it requires lower tapping amplitudes, which suggests that it becomes stiffer and less tacky over time.

Amplitude-distance curves obtained from 13-month old PSA samples were compared to data obtained from freshly cast surfaces to explore whether there was any change in tack or stiffness with ageing. The analysis was performed on the back surfaces after delamination from the silicone release layer. The aged PSA was delaminated immediately before the AFM analysis to ensure that there was no surface

contamination during the period of ageing, which would interfere with the measurements. The amplitude-distance curves for four different A_o are shown in Figures 9A-D.

When $A_o = 22$ nm, the tapping amplitude falls to zero shortly after contact between the tip and the surface. The amplitude for the fresh surface falls after a displacement that is about 1 nm less than found for the aged surface. This result indicates that the aged surface is slightly *less* tacky (as detected by the AFM tip). With such a relatively low amplitude, the energy of the cantilever is quite low. Therefore, the attractive forces between the tip and the PSA surface are sufficient to trap the tip, causing the amplitude to fall to zero after a small displacement.

With $A_o = 46$ nm, there is similarly evidence that the aged surface is slightly less tacky, as indicated by the drop in amplitude on the fresh surface after a smaller displacement. Only small differences between the indentation into the fresh and aged surfaces are observed.

For the larger amplitudes (A_o of 77 nm and 131 nm), the energy of the cantilever is high enough to overcome the adhesion forces, and thus the amplitude does not drop abruptly after small displacements with the AFM tip being trapped on the surface. It then becomes possible to use the tip to indent into the surface to probe qualitatively the surface hardness. If a hard surface is moved toward the tip, then the amplitude would fall by the same distance as the displacement. The slope of the amplitude-distance curve consequently is unity for a hard surface. With a soft surface, the amplitude falls by an amount that is less than the distance of the scanner

displacement. In Figures 9C and D, the amplitude decreases less with the scanner displacement for the fresh PSA in comparison to the aged PSA, which means that the indentation depth is greater in the fresh surface. This result suggests that there has been some stiffening in the aged PSA.

The information yielded by the amplitude-distance curves is then two-fold. There is a slight decrease in tackiness of the PSA during ageing, and it is accompanied by a slight stiffening of the surface. It is important, however, to note that the changes measured in these waterborne acrylic PSAs are very small. This result is in good agreement with the observation that the bulk properties of acrylic PSAs are stable over time.

Amplitude-distance curves provide a useful indication of the optimum tapping conditions for PSA surfaces. They allow the microscopist to define the zone of instability of the scanning parameters where the adhesion forces can provoke steep changes in amplitude. For example, with $A_o = 22$ nm, the amplitude falls to zero after a variation of only 1 nm! When A_o is small, there is a high sensitivity in the tip amplitude to a small scanner displacement, mainly because the energy of the oscillating tip is not high enough to overcome the adhesion forces. This is the reason why the use of a large A_o is necessary to image adhesive surfaces.

4. Summary

AFM is a powerful technique to characterise PSA surface topography and to reveal the extent of particle coalescence during film formation. We have discovered

that corrugations on silicone substrates leave an imprint on the back face of PSA films, which persists over time and even after being compressed. Many applications of PSAs (labels, tapes, films) use a silicone-coated substrate as a release liner. Any defects or roughness transferred to the back face of a PSA film will be placed in contact with the adherend over the lifetime of the PSA. In turn, any interfacial voids associated with the surface roughness are likely to influence the adhesion strength and the tack energy.

Particles are not coalesced at the top face of a waterborne, acrylic PSA. The particles are less distinct at the back face after delamination from the release liner. This different appearance of the back face can be attributed neither to the pressure during the lamination process nor to the removal of the silicone layer. It is very likely that the difference results from less surfactant (and other low molecular-weight species in the latex serum) being present at the back face.

The elasticity of the PSA surface has been demonstrated in a very visual manner. When subjected to a shear stress, the particles are distorted in shape, but they relax to their original shape within 12 minutes after release of the shear stress. The particles retain the same neighbors as they move laterally across the surface, during relaxation of the stress.

After ageing, PSA films do not lose their elastic properties. Only minor changes are detected in indentation curves after ageing PSAs for 13 months. Aged samples appear to be slightly less tacky and slightly stiffer than the fresh ones.

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This work has illustrated that AFM provides numerous insights into the film formation and properties of PSA films. Further uses of AFM include the investigation of the influence of tackifiers on morphology and the origins of water whitening.

Figure Captions

Figure 1. Schematic diagram showing the production of a PSA from the casting step and the transfer lamination process (Stages 1 to 4) to the end use (Stages 5 to 7). AFM analysis was performed at Stages 2 and 5.

Figure 2. The free amplitude A_o is the oscillation amplitude of the cantilever when there is no interaction with the surface of the sample. The setpoint ratio r_{sp} for a soft surface has been defined as d_{sp}/A_o , where d_{sp} , the setpoint value, is the distance between the tip and the sample [17]. A_{sp} is then the setpoint amplitude of the cantilever when it has been reduced by contact with the sample. (b) When the tip is lowered and it first comes into contact with the surface, $A_{sp} = d_{sp}$ and $r_{sp} = 1$. (c) As the tip is lowered further, it indents into the surface over a distance z_{ind} . A_{sp} is then equivalent to d_{sp} plus z_{ind} and $r_{sp} < 1$.

Figure 3. Top view (left) and 3D-view (right) of the irregular silicone/polyester substrate. Channels, with an average depth of 80 nm, are observed at the surface. Scan size: 30 μm x 30 μm ; height = 300 nm (left) and 200 nm per division (right).

Figure 4. AFM images of the PSA showing the (A) top surface (Stage 2) (vertical scale: height = 50 nm; phase = 90 degrees) and the (B) back surface de-laminated from the silicone/polyester release liner (Stage 5) (height = 100nm; phase = 90 degrees). In both A and B, the height image is on the left, and the phase image is on the right. Scan size for both images is 30 μm x 30 μm . In 4B, features of the substrate

are clearly apparent in the topographical image. The ridges are an average of 40 nm high. The distinct white spots are interpreted as dust contamination.

Figure 5. (A) AFM image of the silicone/paper release liner. (Vertical scale: height = 300 nm; phase = 50 degrees). (B) AFM image of the delaminated face of a PSA cast on the silicone/paper, 1 hour after delamination (Stage 5). (Height = 200 nm, phase = 50 degrees). In both A and B, the height image is on the left, and the phase image is on the right. Scan size for all images: 30 μm x 30 μm .

Figure 6. AFM images of (A) the top face of the PSA (Stage 2) and (B) the back face after delamination from a silicone/polyester release liner (Stage 5). (Vertical scale: height = 50 nm, phase = 90 degrees. Scan size: 5 μm x 5 μm .) In both A and B, the height image is on the left, and the phase image is on the right.

Figure 7. AFM images of the PSA top face that had been pressed lightly with a silicone/polyester sheet and left in contact for 4 hours before delamination. (Scan size: 5 μm x 5 μm ; height = 50 nm, phase = 90 degrees). The height image is on the left, and the phase image is on the right.

Figure 8. AFM images of the PSA top face after being pressed with silicone/polyester (under combined shear and pressure) (A) 4 min to 11 min after removal and (B) 11 min to 18 min. after removal. After 12 min, the particles have returned to their original state. (Scan size: 5 μm x 5 μm ; height = 50 nm, phase = 90 degrees) In both A and B, the height image is on the left, and the phase image is on the right.

Figure 9. Comparison of indentation curves on the PSA's back surfaces of samples freshly cast (●) or aged 13 months (■), with four different free amplitudes: (A) 22nm, (B) 46nm, (C) 77nm and (D) 131nm. (The motion of the scanner moving upward is represented as moving from the right to the left in all figures). A more complete description of the amplitude-distance curves can be found in reference [17].

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

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