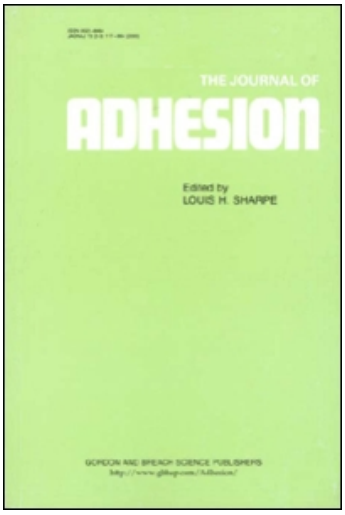

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Particle Adhesion to Elastomeric Substrates and Elastomeric Substrates with Semi-Rigid Coatings

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Particle Adhesion to Elastomeric Substrates and Elastomeric Substrates with Semi-Rigid Coatings*

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The force needed to remove micrometer-size polystyrene particles from elastomeric substrates having Young's moduli of 3.8 and 320 MPa was measured using atomic force techniques. It was found that the removal force was approximately an order of magnitude less for the more rigid substrate than for the more compliant substrate. In both cases the removal force was independent of applied load. However, when the more compliant material was overcoated with the stiffer material, the particle removal force was found to increase with increasing pressure, with the limit at low pressure commensurate with the removal force observed for the stiffer substrate and commensurate with the more compliant material at higher pressures. The results are interpreted in terms of the penetration depth of particle asperities into the substrates.

Keywords: Particle; adhesion; JKR; deformation; elastomer; Young's modulus

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1. INTRODUCTION

The adhesion of particles to substrates is of great importance both scientifically and technologically. Accordingly, it has been the subject of intense investigation for many decades [1–6]. It is intuitively obvious that the materials' properties such as the surface free energy, γ, and the thermodynamic work of adhesion, w_A, should affect the force needed to separate a particle from a substrate. Less obvious, perhaps, is the role the mechanical properties of the contacting materials, such as Young's modulus and yield strength, play in determining the separation force. Even more ambiguous is the effect of any load applied to the particle, in addition to the load generated by the surface forces, on the removal force. Specifically, since materials are not perfectly rigid, they will deform whenever a stress is applied. Such stresses can be generated by surface forces. How these deformations can contribute to the separation force for relatively rigid particles in contact with elastomeric substrates is the subject of this paper.

As discussed by Krupp [4], the adhesion-induced stresses can result in either elastic or plastic deformations. This subject was further pursued by Maugis and Pollock [7], who showed that, in the case of total plasticity, the contact radius, a, is related to the particle radius, R, and the yield strength of the yielding material, Y, by

$$P + 2\pi w_A R = 3\pi a^2 Y \tag{1}$$

where P is the applied load on the particle. It is readily seen that, if $P = 0$, the contact radius varies as the square root of the particle radius. Several examples of this type of behavior have been reported in the literature [8–10].

If, on the other hand, the adhesion-induced deformation is elastic in nature, it is generally describable by either the theory proposed by Johnson *et al.* [11, 12] (hereafter referred to as the JKR theory) or that proposed by Derjaguin and coworkers [13] (generally referred to as the DMT theory). The differences in the assumptions and predictions of these two theories has been adequately discussed in the literature [14–16] and further discussion is beyond the scope of this paper. It is sufficient to say that it has been demonstrated both theoretically [17] and experimentally [18, 19] that, for micrometer-size particles in contact

with elastomeric substrates (as is the case in the present study), the adhesional behavior of the system is described by the JKR model.

According to the JKR theory, the adhesion-induced contact radius, a, is related to the particle radius, R, the Young's modulus, E, and Poisson's ratio, ν, of the substrate (assuming that the particle is rigid compared with the substrate) and any externally applied load, P, by

$$a^3 = \frac{3(1-\nu^2)R}{4E}\{P + 3w_A\pi R + [6w_A\pi RP + (3w_A\pi R)^2]^{1/2}\} \quad (2)$$

where the work of adhesion, w_A, is related to the surface free energies of the particle and substrate, γ_p and γ_s, and their interfacial free energy, γ_{ps}, by

$$w_A = \gamma_p + \gamma_s - \gamma_{ps} \quad (3)$$

It is readily apparent from Eq. (2) that, upon application of a negative load, the contact radius decreases. However, the requirement that the contact radius be real precludes the radical in Eq. (2) from becoming negative. Rather, separation of the particle from the substrate occurs when the negative applied load reaches a critical value such that

$$P_s = -\frac{3}{2}w_A\pi R \quad (4)$$

In other words, the JKR theory predicts that the force needed to effect particle-substrate detachment is independent of the Young's modulus of the substrate. This result appears to contradict the observed effect of Young's modulus on adhesion (as is exemplified by the sticky surface of tape). Several mechanisms, including viscoelasticity [11] and adhesion hysteresis [20, 21] have been proposed to explain this apparent discrepancy.

Another apparent discrepancy between the predictions of the JKR model and observed behavior involves the scaling of the contact radius with the Young's modulus of the substrate. Specifically, in a recent paper [19] it was reported that the contact radius of glass particles on elastomeric substrates having Young's moduli of 3.83 and

41.7 MPa varied with $E^{-1/3}$, as is predicted by the JKR model. However, when those substrates were overcoated with a 5 μm thick layer of a more rigid thermoplastic [22] (Young's modulus = 320 MPa), the resulting contact radius was much smaller than predicted.

The resolution of these aforementioned discrepancies may be due, at least in part, to the role played by the microscopic roughness of the particle. The effect of asperities on the adhesion of particles to atomically-smooth, relatively-rigid, substrates (mica and highly oriented pyrolytic graphite, or HOPG) has been discussed by Schaefer *et al.* [23]. In that study it was shown that the particle removal force scaled with the surface free energies of the contacting materials as well as the radius of curvature, β, of the particle asperities, where β is related to the height of the asperity, h, and the radius of the asperity in the plane of the particle surface, a_p, by

$$\beta = a_p^2/2h. \tag{5}$$

It should be noted that, in those substrates, the Young's moduli of both the particles and the substrates were sufficiently large so as to preclude large deformations.

This study builds on the earlier studies of particle adhesion. Specifically, the removal force as a function of applied load was measured from two substrates differing principally (with respect to their adhesional properties) in Young's modulus. In addition, the effect of a thin coating of the more rigid material over the more compliant material on the particle removal force was also determined as a function of applied load.

2. EXPERIMENT

In this study, the force needed to remove a particle from various polymeric substrates was measured as a function of the Young's modulus of the substrate and the loading force applied to the particle.

The materials used in this study were all used in previous studies wherein the adhesion-induced contact radius was measured [19, 22, 24]. The particle separation forces were measured using atomic force techniques similar to those described previously [23].

Spherical, cross-linked, polystyrene particles, having radii of approximately 6 μm, were attached to atomic force microscope (AFM) cantilevers using a microscopic drop of Norland Optical Cement No. 68. The particle was pressed into close contact with the cantilever using a micromanipulator. The cement was subsequently cured by exposure to UV light for approximately 20 minutes.

The polystyrene particles were prepared using the limited-coalescence process [25], with the particle size determined by the amount of silica particles added to the suspension. The silica was then removed by washing the particles in a one-normal solution of NaOH, followed by washing in a dilute base. Finally, the particles were washed in distilled water until a neutral pH was obtained. All washings were done at approximately 20°C. The Young's modulus of the polystyrene comprising the particles, measured using a Sintech/20 tensile tester, was found to be 2.55 GPa [9]. This is in good agreement with the literature values [26].

The substrates were produced from commercially available polymers. The first polymer was a polyurethane (TU-500, produced by Conap, Inc.) This material had a Young's modulus and glass transition temperature of 3.83 MPa and −45°C, respectively [19,22]. The second substrate consisted of a thermoplastic (Permuthane, produced by Stahl Finish) with a melting temperature in excess of 150°C and a Young's modulus of 320 MPa [19,22]. In addition, a third substrate was prepared consisting of a 5 mm thick slab of the TU-500 over which was coated a 5 μm thick layer of the Permuthane. All substrates were made by casting and had an air interface. The surface free energies of the substrates, as determined using contact angle with distilled water and diiodomethane as the liquids, was 43 ± 3 ergs/cm^2.

The AFM used in this study was a custom-built instrument, capable of operating in constant force, constant displacement, and non-contacting modes. Clean substrates were attached to a segmented piezoelectric tube. The cantilever was held by a second piezoelectric tube. This allowed the nominal position of the particle with respect to the substrate to be varied. Deflection of the cantilever was measured using the laser deflection method [27–29]. A focused beam was reflected off the back of the cantilever into a United Detector Model SPOT2D split-photodiode position-sensing detector. System control and data acquisition were all done using a computer.

The X-Y calibration was performed using a diffraction grating with a periodicity of 278 nm for large displacements and by measuring the known lattice constant of highly oriented pyrolitic graphite for small displacements. The hysteresis of the piezotube and the calibration of the cantilever were done using the techniques described elsewhere [23]. The AFM was then placed into a chamber which could be evacuated to a pressure of approximately 500 mTorr. Alternatively, the chamber could be filled with a gas such as dry nitrogen. This allowed the detachment force to be measured in either vacuum or a controlled atmosphere.

The cantilevers were obtained from Park Scientific Instruments and had a nominal spring constant of 2.2 N/m. The precise value of the spring constant was obtained by measuring the resonance frequency of the bare cantilever, in the manner described elsewhere [23].

Figure 1 shows a typical loading – removal cycle, as performed in this study. The particle, attached to the AFM cantilever, was held at some distance above the substrate in question. The substrate was then moved towards the particle, as designated by Figure 1a. At some separation distance the particle snapped into contact with the substrate. Upon further movement of the substrate, the cantilever flexed in the manner illustrated in Figure 1b. This, in effect, resulted in a positive load being applied to the particle. After a predetermined load was applied (about 500 nN in this study), the substrate was moved back, resulting in the cantilever flexing in a manner (Fig. 1c) so as to exert a negative force upon the particle. At some separation distance this force is sufficiently great so as to result in the particle abruptly separating from the substrate (Fig. 1d). The force curve associated with the loading and unloading of the particle are shown in Figure 1e. Here, the portion of the curve representing the approach is shown by *a*. The snap-together of the particle and substrate occurred at point *b*. Loading and unloading are represented by the portion of the curve designated by the arrows. Upon exertion of a negative load, the force curve extends along portion *c* beyond point *b*, until region *d* is reached, whereupon the particle and substrate snap apart from each other. It should be noted that the loading and unloading portions beyond point *b* of the curve approximately superimpose, in general. This behavior was also observed in this study.

In this study the particles only contacted the air interface of the polymeric substrate. Because the Young's modulus of the particle was

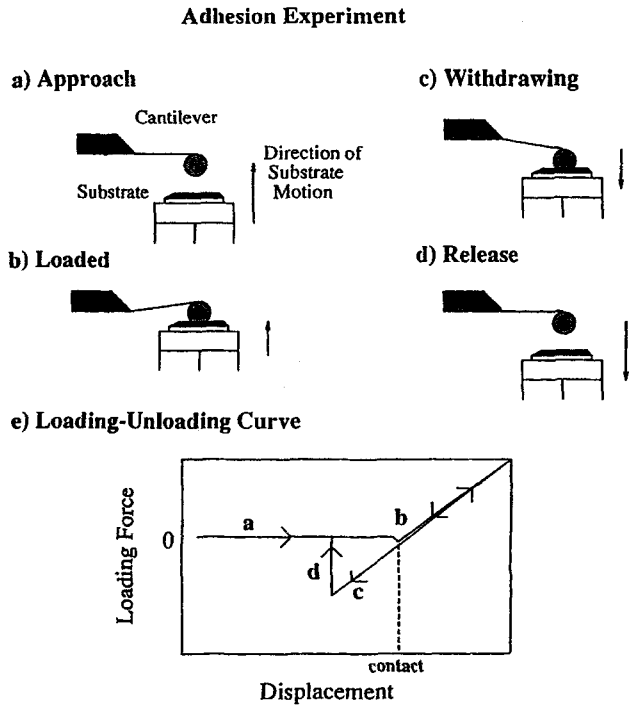

FIGURE 1 A schematic depicting the particle and cantilever of an AFM as the particle a) approaches the substrate, b) is in contact with the substrate, c) is withdrawn from the substrate, and d) finally separates from the substrate. Figure 1e shows a graph of the force on the particle as a function of particle/substrate separation.

an order of magnitude higher than the most rigid substrate, the only significant deformations resulting from either the applied load or the surface forces occurred in the substrates.

3. RESULTS

The removal force is shown as a function of the applied load for the particle in contact with the TU-500, the Permuthane, and the Permuthane-overcoated TU-500 in Figures 2–5, respectively. Measurements were performed both in air and in vacuum. Multiple measurements were performed under each atmosphere and applied load. The reproducibility is shown by the superposition of the data in these

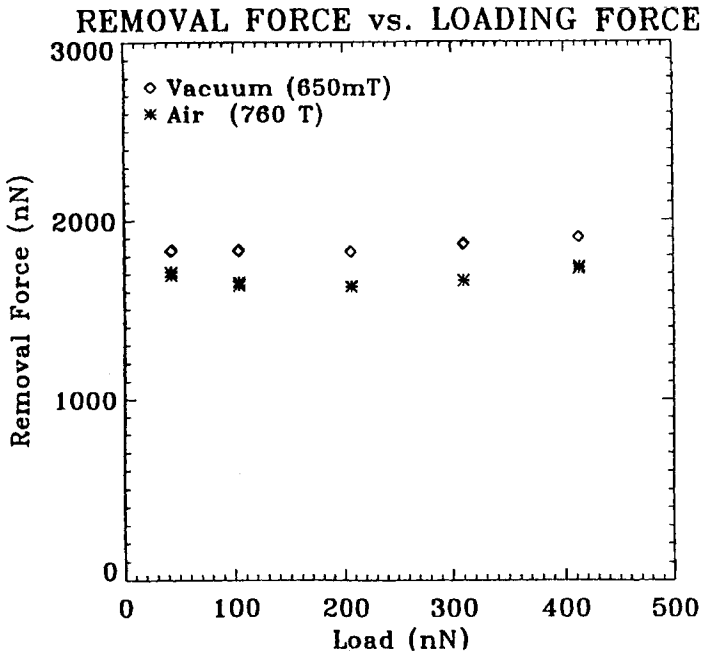

FIGURE 2 Particle removal force as a function of applied load for a polystyrene particle in contact with a polyurethane (TU-500) substrate.

graphs. As can be seen, there is little difference in the observed removal force between the samples in vacuum and those in air. It appears that the removal force is somewhat larger in vacuum than in air for the TU-500 substrate, but opposite for the Permuthane substrate. However, the nature of this experiment precludes adequate statistics to state categorically that this is the case and is, more likely, experimental error introduced when the atmosphere was changed. Moreover, the force of removal does not seem to be dependent on the applied load for either the TU-500 or the Permuthane substrates. This latter point is apparently consistent with the removal force predicted by the JKR model, as indicated by Eq. (4). However, this point will be revisited later in this paper.

It is also apparent from Figures 2 and 3 that the removal force of the particle from the more compliant (TU-500) substrate (approximately 1700 nN) is more than an order of magnitude greater than the particle removal force from the more rigid substrate (approximately

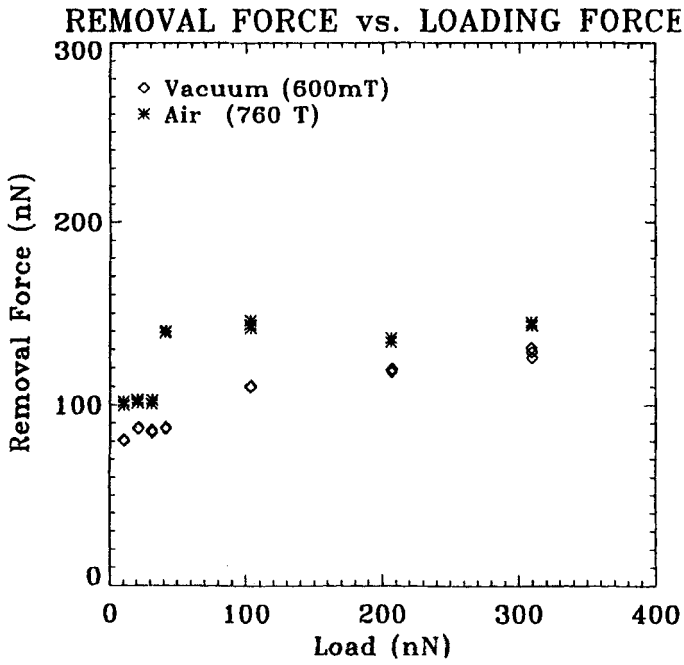

FIGURE 3 Particle removal force as a function of applied load for a polystyrene particle in contact with a thermoplastic (Permuthane) substrate.

100 nN). Since the surface free energies of these materials are virtually identical and the same particle was used in these studies, the difference in removal force can only be attributed to the difference in the Young's modulus of the substrates. This is in apparent contradiction to the predictions of the JKR theory. Moreover as seen in Figure 4, the removal force on the TU-500 substrate overcoated with the thin layer of Permuthane increases with applied load and appears to approach the removal force of the particle from the Permuthane after a small load had been applied and that of the particle from the TU-500 after the particle was subjected to a high applied load.

The observed effect of Young's modulus on removal force can, apparently, be explained in terms of the particle roughness and the indentation of the particle into the substrate. Specifically, particles are not atomically smooth. Rather, they have asperities which, in the case of relatively compliant substrates (as is presently the case), embed under the influence of surface forces, into the substrate.

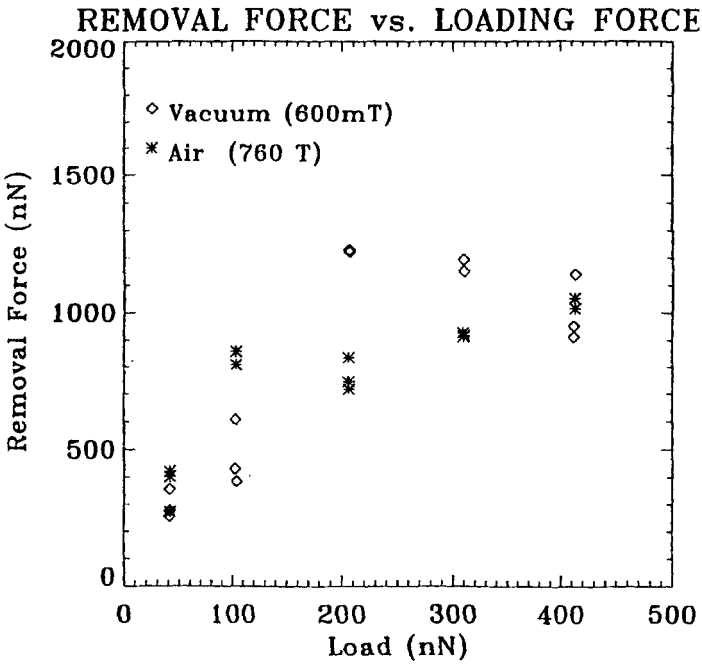

FIGURE 4 Particle removal force as a function of applied load for a polystyrene particle in contact with a substrate consisting of the TU-500 overcoated with a 5 μm thick layer of Permuthane.

The role of surface roughness on particle adhesion was originally studied by Greenwood and Williamson [30], who assumed that the asperities were spherical with a Gaussian size distribution and that their contact with the substrate was Hertzian. This work was later expanded to JKR contacts by Fuller and Tabor [31] and to DMT contacts by Maugis [32]. The present technique of mounting a particle onto a cantilever of an AFM allows one to study the adhesion of particles rather than asperities on broad surfaces, as done by Fuller and Tabor. Moreover, the embedment force applied to the particle can be precisely controlled, thereby controlling the depth of penetration of the particle into the substrate. This can allow the removal force to be quantitatively determined for the particle as a function of penetration depth. A limitation of this technique occurs because, at some separation distance, the particle will snap into contact with the substrate irrespective of the cantilever stiffness. The spring constant of the cantilevers used in this

study were chosen to maximize the precision of the measurement of the removal force while being able to remove the particle from the substrate.

If the substrate is highly compliant, the particle can embed to a depth greater than the height of the asperity. In that case the removal force is given by Eq. (4). However, if the substrate is somewhat less compliant, the degree of embedment is less than the asperity height. Because of the relatively short range of surface forces, the bulk of the particle would not, under these circumstances, contribute to the particle-substrate adhesion. Rather, the removal force should be related to the radius of curvature of the contacting asperity or asperities. In the present situation where the particle is constrained by the AFM cantilever so that there is only one contact point,

$$P_s^a = -\frac{3}{2}\pi w_A \beta \tag{6}$$

where P_s^a is the removal force when only the particle asperity embeds into the substrate.

This argument can be demonstrated more quantitatively. Figure 5 shows an SEM micrograph of a similar polystyrene particle. The micrograph was made using a Hitachi field emission microscope operating near the unity point of the polystyrene. This eliminated the need for any conductive coating and allowed a higher resolution micrograph to be made than can normally be produced. It is clear that the particle is not perfectly smooth. Rather, there appear to be numerous undulations on the surface of the particle. Figure 6 shows the surface profilometry of a polystyrene particle nominally identical to the one used in the present removal force determinations. As is evident, the surface is not smooth. Rather, there are asperities having a height of approximately 20 nm. The mean radius of curvature, calculated using Eq. (5), was determined to be approximately 0.30 μm. Accordingly, if the depth of penetration of the particle into the substrate is greater than approximately 20 nm, the entire particle can be considered to be in contact with the substrate and the separation force would be given by Eq. (4). Alternatively, if the penetration depth is significantly less than 20 nm, the van der Waals attraction between the main part of the particle and the substrate would be insignificant and the removal force would be determined by the radius of curvature of the asperity, as given by Eq. (6).

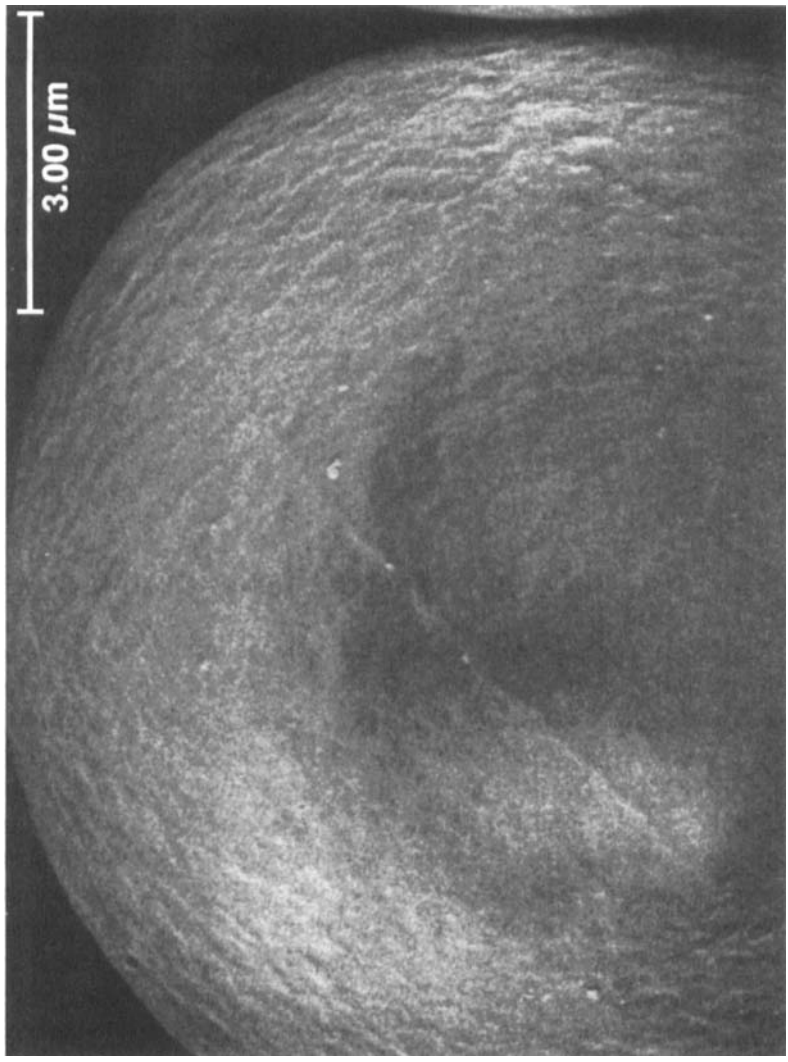

FIGURE 5 An SEM micrograph of a similar polystyrene particle to the one used in the removal force measurements.

The depth of penetration of the particle into the substrate, δ, can be calculated using the JKR theory. Accordingly,

$$= \frac{1}{3K^{2/3}R^{2/3}} \frac{P_1 + 2P}{P_1^{1/3}} \tag{7}$$

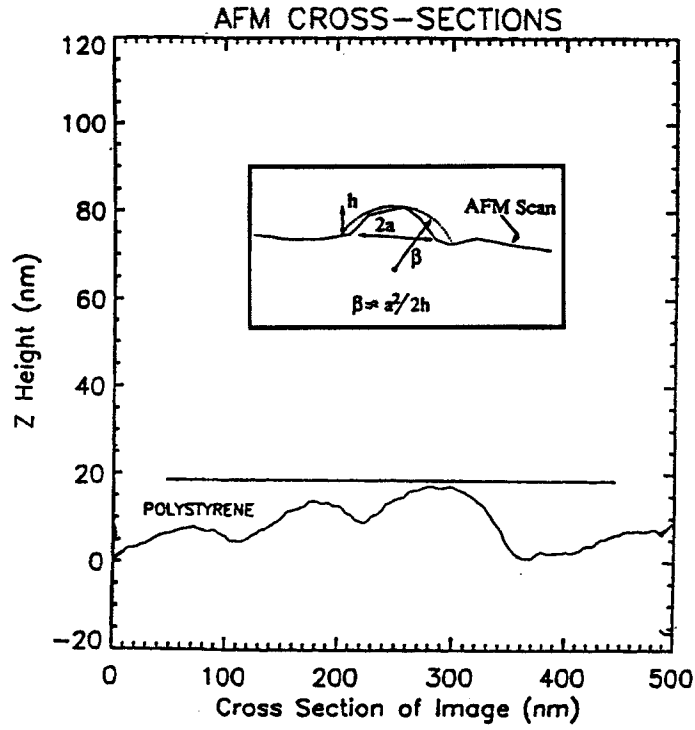

FIGURE 6 Profilometry of the polystyrene particle, as measured with an AFM.

where P is the applied load, E is the Young's modulus of the substrate,

$$P_1 = \frac{a^3 K}{R} \tag{8}$$

and

$$K = \frac{4E}{3(1-\nu^2)R} \tag{9}$$

where ν is the Poisson ratio of the substrate.

Experimentally-determined values for the contact radius, a, of these specific particles on these substrates are not presently obtainable. However, these radii can still be estimated from the measured contact

radii of glass particles having similar size asperities [23] on these identical substrates [22] and scaling them to the appropriate particle size using Eq. (2) with $P = 0$. Accordingly, it was estimated that $a = 2.21$ μm on the polyurethane substrate and 0.36 μm on the Permuthane substrate. It should be noted that errors introduced in the estimated contact radii due to different surface free energies of the glass and polystyrene particles should be minor due to the rather weak dependence of the contact radius on the surface free energy of any one of the materials.

The calculated penetration depth is shown as a function of applied load for the polystyrene particle on the Permuthane and TU-500 substrates in Figures 7 and 8, respectively. It is seen that, in both cases, most of the indentation arises from the surface forces, with the incremental penetration due to a 500 nN applied load being less than 30% of the no-load penetration depth. Moreover, the depth of penetration of the particle into the Permuthane is only approximately 8 nm with no applied load and 10 nm under the 500 nN force. This is small compared with the height of the asperity and leaves a sufficient separation between the bulk of the particle and the substrate (> 10 nm) so that the van der Waals attraction between the main part of the particle and the substrate would be insignificant. Accordingly, the separation force should be governed by the radius of curvature of the asperity and given, therefore, by Eq. (6). Assuming that $w_A = 0.050$ J/m^2, the removal force is estimated to be 71 nN. Moreover, since the particle never penetrates the substrate to the point where van der Waals attraction from the bulk becomes significant, the removal force should be independent of applied load. This is in good agreement with the measured removal force (approximately 100 nN). Any discrepancies between the predicted and observed removal forces can readily arise from differences between the statistically-determined radius of curvature and its specific value for the contacting asperity and from differences between the estimated and actual work of adhesion.

In contrast to the case where the particle is contacting the relatively rigid Permuthane substrate, the penetration depth of the particle into the more compliant TU-500 was estimated to be approximately 150 nm with no externally applied load. This is well in excess of the asperity height and suggests that the bulk of the particle is in intimate contact with the substrate. Accordingly, the removal force should be

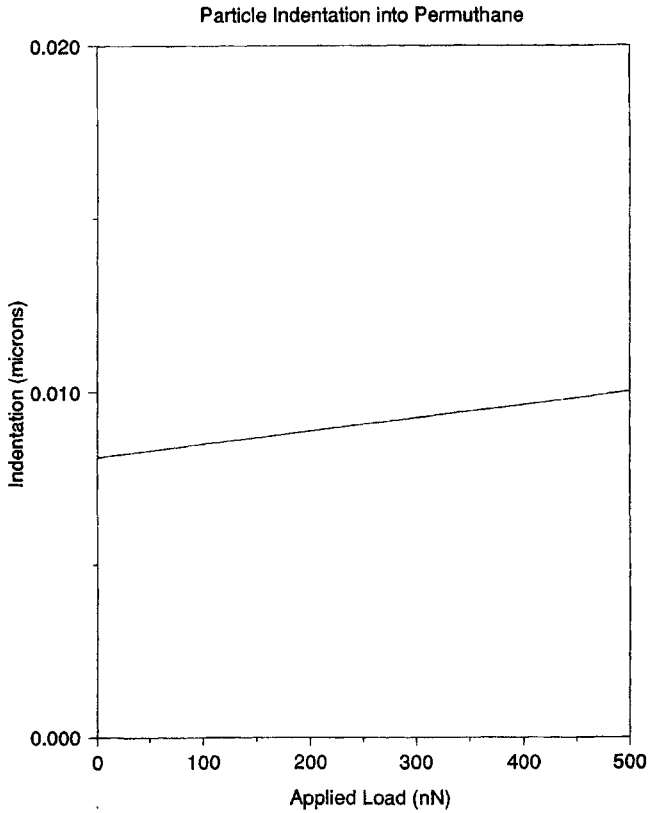

FIGURE 7 Indentation of the polystyrene particle into the Permuthane substrate, as calculated from the JKR theory.

given by Eq. (4). As before, because the deformations are elastic, the removal force should be independent of applied load. The predicted removal force (1400 nN) was found to be in good agreement with the experimentally-determined value (approximately 1700 nN), with the discrepancy readily attributable to errors in the work of adhesion and radius of the particle.

The removal force of the particle contacting the Permuthane-over-coated polyurethane may also be explainable, albeit only qualitatively, in terms of increased penetration depth of the particle under the applied load. As indicated previously, the removal force increases, in this

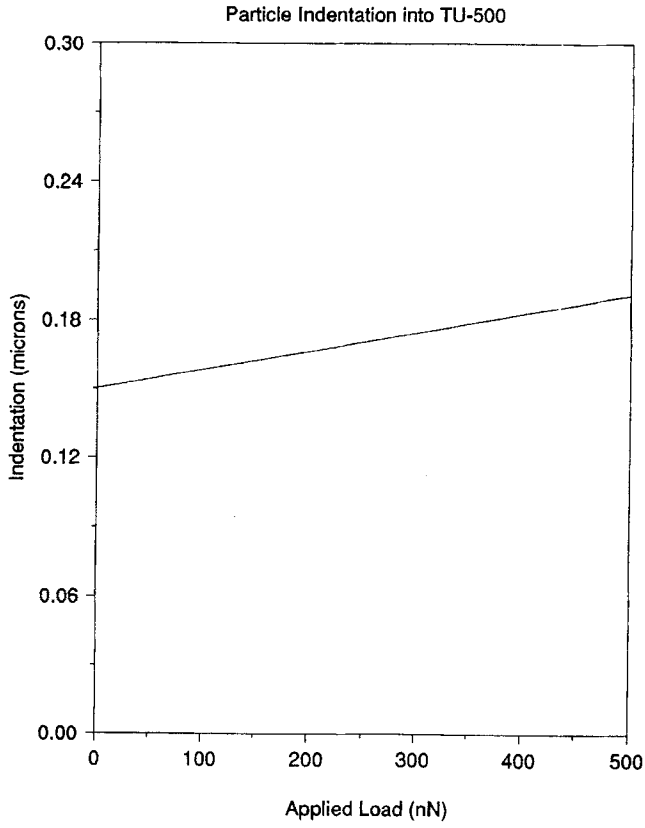

FIGURE 8 Indentation of the polystyrene particle into the TU-500 substrate, as calculated from the JKR theory.

case, with increasing applied load and appears to approach that of the particle on the Permuthane under no-load conditions and that of the particle on the TU-500 under the high-load conditions. To calculate the force needed to separate the particle from the substrate for a composite material, such is presently the case for the substrate, would involve nonlinear contact mechanics. Such a theory for particle adhesion has yet to be developed. However, it seems intuitive that some bending of the substrate around the particle might occur, which can account for this observation.

4. CONCLUSIONS

The force needed to separate a polystyrene particle from two polymeric substrates having Young's moduli differing by two orders of magnitude was measured using atomic force techniques in both air and low vacuum conditions. It was observed that, while no significant differences were found in the removal forces under the different atmospheres, the removal force differed by about an order of magnitude between the two substrates. When the more compliant substrate was overcoated with a thin layer of the more rigid material, the removal force was found to increase with increasing applied load. These apparent discrepancies with the predictions of the JKR theory appear resolvable by taking into account the roughness of the particle and the amount of embedment of the particle into the substrate.

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