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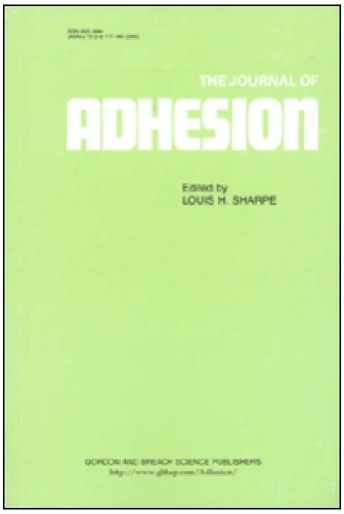

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A Method of Determining the Contact Area Between a Particle and Substrate Using Scanning Electron Microscopy*

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A new technique for determining the contact radius between a micrometer size particle and a contacting substrate using scanning electron microscopy has been developed. The Contact Area Measurement (CAM) technique, which is especially suited for small surface-force-induced contact radii, involves evaporating a thin, uniform coating of a conductive material, such as aluminum, over a sample comprised of particles on a substrate while the sample is rotated slowly. The sample is examined before and after particle removal to determine both the radii of the particle and its respective contact. Where the particle contacted the substrate, no metal deposition occurred. The resulting differences in the secondary electron emissions provide a contrast mechanism that the SEM can image. The CAM technique is shown to be useful in examining rigid particles on rigid substrates, where the inherent contacts are small, making measurements difficult, and for examining irregularly-shaped particles and contact areas.

KEY WORDS particle; contact area; deformation; SEM; adhesion; polystyrene; silicon

INTRODUCTION

Adhesion of particles to each other and to other surfaces is an area of science that affects many aspects of everyday life. For example, the cleaning of silicon wafers in the manufacturing of integrated circuits is important in maximizing device yields. This is critical as device miniaturization continues and even smaller particles can cause a device to fail.

Hertz,[1] assuming an ideal, elastic response, was the first to study how an indentor interacts with a surface. Derjaguin[2] assumed that particles interacted with substrates as Hertzian indentors, with the applied load coming from van der Waals interactions. Johnson, Kendall, and Roberts (JKR)[3] proposed that, in addition to the compressive forces exerted by a particle resting on a surface, tensile forces acting near the contact periphery can contribute significantly to adhesion. Derjaguin, Muller, and Toporov

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(DMT)[4] incorporated tensile forces into Derjaguin's earlier model.[2] Both the JKR and DMT models assume elastic responses.

Krupp[5] was the first to realize that the stresses associated with the surface forces could exceed the yield strength of one or both materials, resulting in a nonelastic (*i.e.*, plastic) interaction. Maugis and Pollock (MP)[6] generalized the JKR theory to include nonelastic type interactions.

One critical part to evaluating the theories is being able to measure the particle and contact radii. Once their relationship has been determined, along with certain mechanical properties of the involved materials, the appropriate theory can often be used to calculate parameters such as the work of adhesion. Direct observation of the particle-substrate interactions using microscopy allows accurate measurement of both the particle and contact radii simultaneously.

Optical microscopy has long been used to gather data for particle-substrate interactions. Hertz[1] studied contacting glass spheres using an optical microscope. Johnson *et al.*[3] measured the contact radii between gelatin and rubber spheres and planar surfaces and measured the interactions using an optical microscope. Chaudhri and Yoffe[7] studied millimeter spheres of steel or tungsten carbide on fused silica, soda lime glass, and sapphire using a modified optical microscope to measure the contacts.

The advent of scanning electron microscopy and its significantly greater spatial resolution extended the range of particle sizes and types of substrates that could be studied. Glass, polymeric, or other types of particles, ranging from submicrometer to a hundred micrometers in radius, could now be examined on a variety of substrates such as silicon wafers, cast polymer sheets, or polished metal surfaces. This is especially important because with particles in this size range, surface force deformations can be studied without complications arising from "external" loads such as gravity or electrical charges. Recent studies of a variety of particle-substrate systems, using the SEM to determine particle-substrate interactions, have been published.[8-11]

The technique of Rimai *et al.*[8] typically involves depositing spherical particles onto a substrate of choice, sputter coating a 10 nm Au/Pd film onto the sample, (which was necessary to avoid any space charge buildup that can distort the image) and examining the sample at high tilt to observe the particle-substrate interface.

However, this technique has some limitations. The relative size of the contact must be large compared with the particle. This precludes studying systems consisting of relatively stiff particles and substrates. Moreover, stiff particles larger than approximately 50 μm, interacting with relatively noncompliant substrates, can cause problems when examined at high tilts because of gravitational effects. The finite thickness of the thin, metal coating must be taken into account when examining small contacts. Finally, the contact of irregularly-shaped particles on substrates cannot be determined. In order to facilitate studying systems involving relatively stiff particles and substrates, a new method of measuring the contact radius was needed.

EXPERIMENTAL

For this study, nominal 7 μm radius polystyrene particles were gently deposited onto a silicon substrate. The sample was then placed in a vacuum evaporator and, while rotating slowly (1 rev/s), coated with approximately 50 nm of aluminum. The particles were then sized using the SEM. The sample was removed from the SEM and the particles removed by gently blowing them off from an oblique angle using canned air and the identical areas re-examined. Where the particles contacted the substrate, (*i.e.*, the contact area) there was no metal coating. This metal *versus* bare (substrate) surface resulted in a different secondary electron emission, which provides a contrast mechanism for the SEM. By initially monitoring the particles positions, each particles and its corresponding contact area could be measured.

An illustrative example comparing the high tilt examination method and this new technique is shown in Figures 1 and 2. Figure 1 shows a SEM micrograph, taken at high tilt, showing a typical polystyrene particle contacting a silicon substrate. The contact radius, corrected for the 10 nm thick conductive coating, was approximately 0.6 μm. Figure 2 shows an SEM micrograph of a typical contact area after particle removal using the new Contact Area Measurement (CAM) technique. A "bulls-eye" effect comprised of three distinct regions, resulting from the coating process, is observed in this micrograph. The first, outermost region, received the most aluminum coating material and appears bright. The middle annular region appears darker, because it was shadowed from the direct influx of aluminum by the particle. However, some aluminum appears to have diffused into this "shadow" region. This effect is discussed in detail later in this paper.

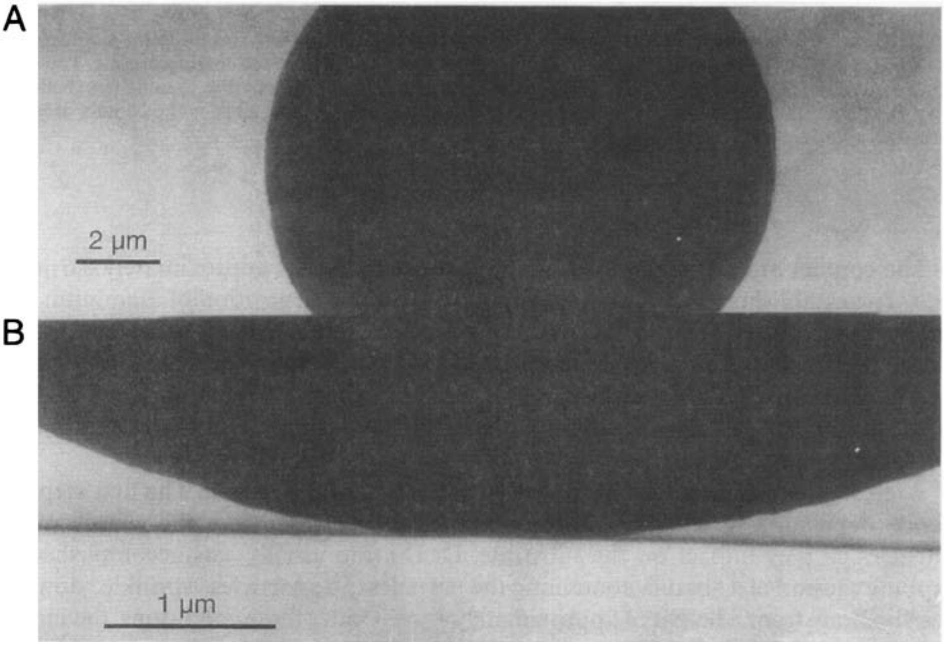

FIGURE 1 A SEM micrograph taken at high tilt showing a typical polystyrene particle contacting a silicon substrate. Most of the particle was first scanned at lower magnification (A) so that the particle size could be determined, and then the magnification was increased (B) near the particle- substrate interface to facilitate observation of the contact area. The contact radius, compensating for the conductive coating, is approximately 0.6 μm.

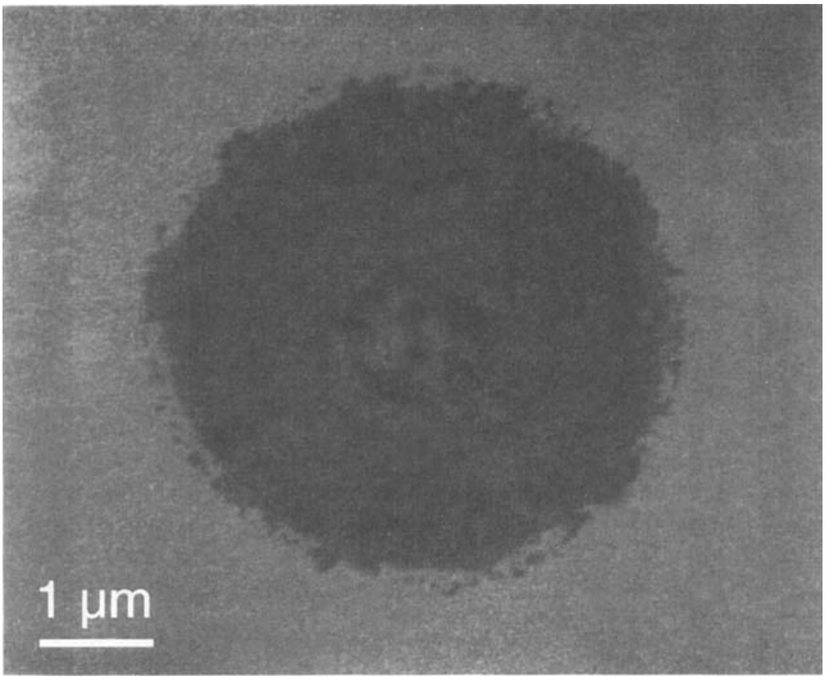

FIGURE 2 A SEM micrograph of a typical contact area after particle removal using the Contact Area Measurement (CAM) technique. Note the "bulls-eye" effect resulting from the coating process. The outer area received the most coating material and appears bright. The inner ring is darker, because it is shadowed from the direct influx of coating material by the particle. The inner, bright circle is the contact area and measures approximately 0.6 μm in radius.

The contact area is represented by the inner circle and is approximately 0.6 μm in size. The establishment of a contact zone precludes the deposition of aluminum into this region. Note the noncircular contact area of Figure 2. This was not uncommon and presumably results from the fact that the particles used were not all perfectly spherical, and may have an indented surface or asperities that cause a noncircular area to form. Also, during particle removal, the aluminum film could tear, resulting in formation of a noncircular area.

Each step in this new procedure will now be discussed in detail. The first step, the gentle deposition of the particles onto the substrate, minimizes the energy of the particles as they impact on the substrate. Deposition usually was accomplished by tapping the end of a spatula containing the particles. The particles "sprinkle" down to the substrate from a height of approximately 1 cm. Under these conditions, the impact energy was small and the effect on the contact area was negligible.[8] This was important because particles of sufficient kinetic energy impacting on a surface could deform non-elastically[12, 13] and thus affect the contact areas. A submonolayer of particles was desired so that single, isolated particles could be studied. After particle deposition the sample was allowed to equilibrate for 7–10 days.

The metal coatings were deposited using an Edwards E306A high vacuum evaporator. A vacuum of at least 10^{-6} torr was achieved prior to coating. The sample was slowly rotated (1 rev/second) to achieve a uniform coating. Slowly rotating the sample assured minimal centrifugal and precessional forces acting upon the particles that could cause the particles to "roll" around the surface of the substrate. A minimum of three inches (7.6 cm) separated the evaporating source and the sample.

A variety of metals were tried (gold, gold/palladium, platinum/palladium, chromium, aluminum) and were found to work, but the results presented here were done with aluminum coatings. Aluminum was chosen for several reasons. Aluminum evaporates at a lower temperature, and this reduces the radiant heat that the sample is exposed to (a consideration when dealing with polymeric particles or substrates). Aluminum also oxidizes rapidly to aluminum oxide, which increases the yield of secondary electrons, and thus the SEM sensitivity, between 2–9 times over elemental aluminum or other metals.[14] Finally, aluminum is substantially less expensive.

Another parameter investigated was the coating angle, θ; that is, the angle the evaporating source makes relative to the sample plane. Figure 3 shows a SEM

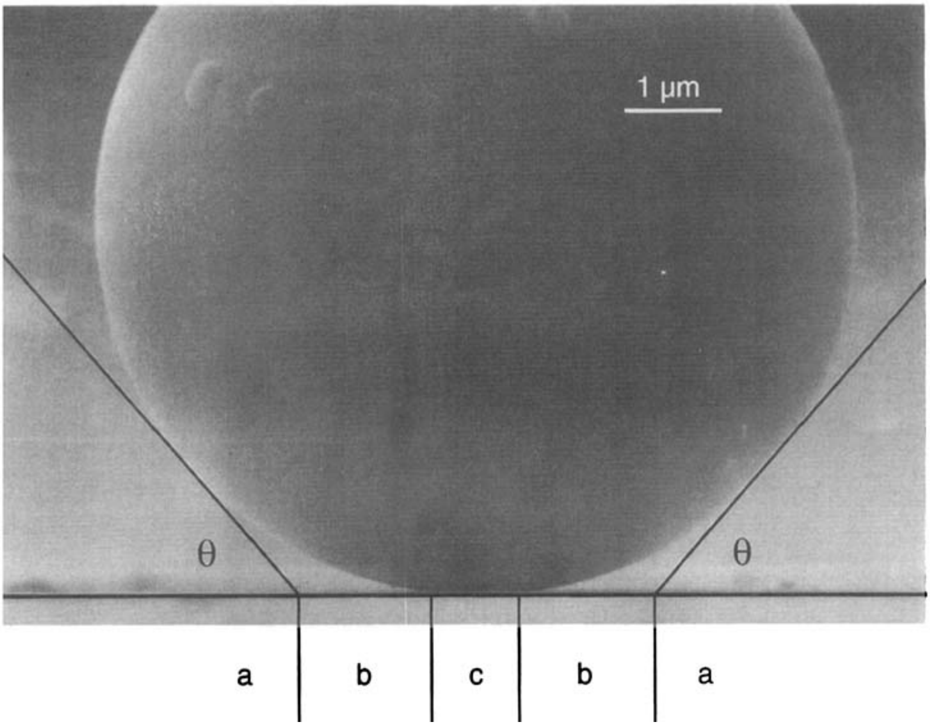

FIGURE 3 A high tilt SEM micrograph of a polystyrene sphere on a silicon substrate illustrating how the "bulls-eye" effect results from the coating process. At a coating angle θ, the "a" areas are directly exposed to coating material. Area "b" is the region shadowed by the particle. Area "c" is the particle-substrate contact.

micrograph of a polystyrene sphere on silicon viewed at high tilt and illustrates the effect of θ. Area "a" is the region directly exposed to the coating material. Area "b" is the region shadowed by the particle from the direct influx of coating material. Area "c" is the actual particle-substrate contact. As θ decreases, "b" becomes smaller until it equals "c". Initially, it was thought that the shallower the coating angle, the better the coating material would reach into the particle-substrate contact zone. Figure 4 shows the "bulls-eye" effect described earlier resulting from separate coatings at three different θ values. As expected, and shown in Figure 4, when θ decreases, the shadowed region "b" becomes smaller. It should be noted that the inner contact area, "c", is independent of θ. A θ value of 45° was chosen for two reasons: the large shadowed area "b" aids in locating specific contact areas after particle removal, and the shallow coating angles cause the aluminum coating to be more granular.

Nominal 7 μm radius spherical, cross-linked polystyrene particles were prepared using the limited coalescence[15] process with the particle size controlled by the quantity of SiO_2 particles added to the suspension. After polymerization any silica on the surface

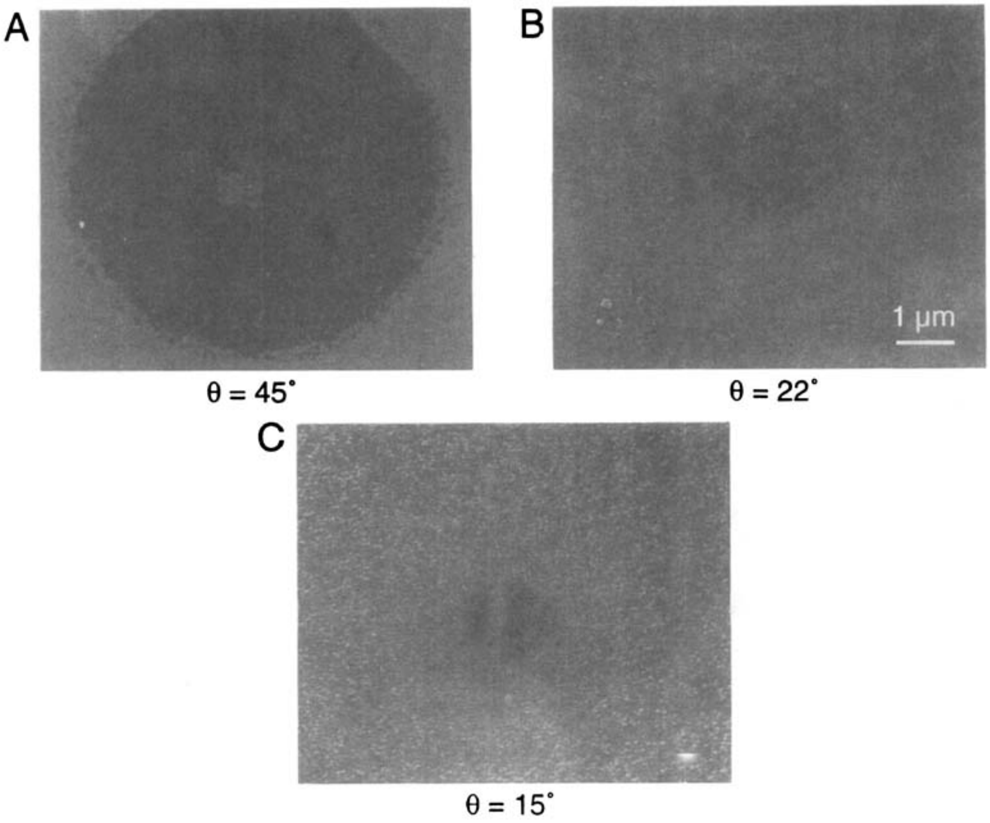

FIGURE 4 SEM micrographs of the silicon substrate after particle removal illustrating the effect of varying the coating angle θ on the "bulls-eye" effect. As θ decreases, the shadowed region becomes smaller, but the contact area remains the same.

of the particles was removed by base-washing the polystyrene particles in a one-normal solution of sodium hydroxide, followed by washing in a dilute base. Finally, the polystyrene particles were washed in water until a neutral pH was achieved and were subsequently dried. All washings were done at approximately 20°C. Careful SEM and TEM examinations of these particles and their cross sections, respectively, showed that the cross-linked polystyrene particles were spherical and that little, if any, residual silica particles remained on their surfaces.

RESULTS

The nominal 7.0 μm polystyrene particles were prepared onto silicon wafer pieces as previously described.

The samples were examined in a Philips 515 SEM at 30 KV accelerating voltage, 10 nm spot size, and 7 mm working distance. Initially, a number of particles were photographed at a high sample tilt (88°). More recently,[16] the CAM technique has compared very well with the high tilt technique for a different particle-substrate system. For that study, glass beads ranging from 3–25 μm in radius were deposited onto silicon substrates and the surface-force-induced contacts measured. Once again, within the 95% confidence interval, there was no statistical difference between the CAM and high tilt techniques, This strongly suggests that the CAM technique is a viable technique for examining particle-substrate interactions, and that the probability that the results are fortuitous is minimal.

Table I summarizes the results. Within the 95% confidence interval, there is no statistical difference between the two techniques. This also supports using the CAM technique for examining particle-substrate interactions.

As mentioned in the introduction, one application of the CAM technique is the study of irregularly-shaped particles. Whereas a spherical particle will have a circular contact, the contact area of an irregularly-shaped particle depends on the shape of the particle and how it rests on the substrate (*i.e.*, on an edge or an asperity). The high tilt technique is of limited use in these situations because it is not possible to image the entire contact region. For example, Figure 5 shows irregularly-shaped, ground polystyrene particles on a polyester substrate. Figure 5A shows a field of particles before removal and Figure 5B after removal. The particles labeled 1, 2, 3, and 5 (Fig. 5a) are nominally the same size, however, as shown in Figure 5B, there is a large variation in their contact areas. These variations can be due to a number of parameters including surface morphology, Young's moduli, and method of particle deposition. This example

TABLE I
Nominal 8.0 μm radius polystyrene particles on silicon substrate examined using SEM. The data here indicate that, within 95% confidence intervals, both analyses are statistically the same

	Number of particles	Avg. particle radius (μm)	Avg. contact radius (μm)
high tilt	6	7.0 (± 0.4)	0.52 (± 0.19)
CAM	15	7.4 (± 0.5)	0.53 (± 0.10)

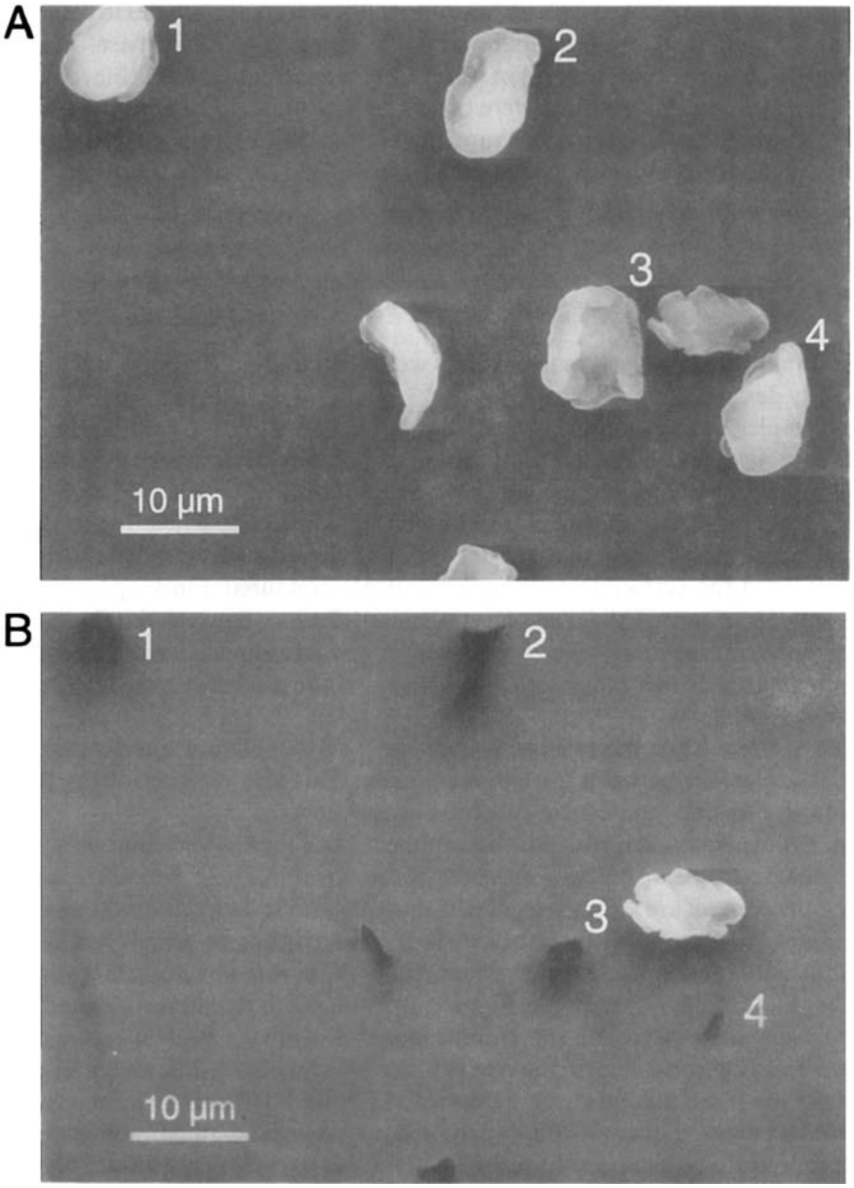

FIGURE 5 A SEM micrograph showing irregularly-shaped ground polystyrene particles on a polyester substrate (Fig. 5A). Figure 5B is the same field of view after particle removal showing the large variation in contact areas, even though Figure 5A shows the particles labeled 1, 2, 3, and 4 to be nominally the same size.

illustrates the power of this new CAM technique for mapping statistically the distribution of contact areas. Such a distribution could be correlated to process efficiency in technology applications, such as the removal of particulate contamination from silicon wafers or the transfer of toner particles from a photoconductor to a receiver.

CONCLUSIONS

A new technique for determining particle-substrate contact radii using the SEM has been developed. This new technique involves evaporating a thin, uniform coating of aluminum onto a sample comprised of particles gently deposited onto a substrate. The sample is then examined in the SEM and a number of particles are sized. The particles are then removed and the sample is re-examined in the SEM. Those areas where the particles contacted the substrate (*i.e.*, contact area) have no aluminum coating. This results in a contrast mechanism seen by the SEM and allows the contact area to be determined. The validity of this new technique was confirmed by comparing the contact radii obtained using the CAM technique with those found using the high tilt technique. Within the 95% confidence interval both analyses produced the same results, indicating the new CAM technique is a viable means to examine particle-substrate interactions.

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