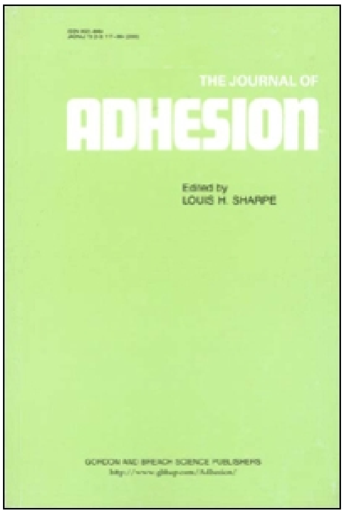

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The Effect of Relative Humidity on Particle Adhesion and Removal*

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The removal of small particles is vital for contamination-free manufacturing. In humid environments liquid can condense between the particle and substrate and give rise to a very large capillary force, which increases the total force of adhesion. The removal and adhesion forces of polystyrene latex (PSL) particles and pigmented coating chips were measured on silicon, polyethylene terephthalate, metallized and polyester coating substrates as a function of humidity. The results indicate that the capillary force is significant at a relative humidity above 50% and dominates at a relative humidity above 70%. At relative humidity below 45%, the electrostatic force becomes significant. The adhesion forces varied depending on the particles and substrates used, but the trend of high adhesion at high and low relative humidity was observed for all PSL particles/substrate systems. The pigmented coating chips/substrate system however, exhibited high adhesion at high relative humidity and low adhesion at low relative humidity.

Keywords: Particle adhesion; Humidity; van der Waals force; Capillary force; Electrostatic force; Adhesion force measurements; Particle removal

1. INTRODUCTION

Adhesion between small particles and solid surfaces is a concern to many technologies. Understanding of particle adhesion and removal

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is vital for contamination-free manufacturing in many industries such as semiconductor manufacturing, xerography, pharmaceuticals, aerospace, *etc*. Efficient removal of particles from surfaces requires knowledge of the magnitude of the forces involved. Experimental studies on the removal of particles and a detailed review of particle-surface interactions have been widely reported [1 – 12]. Kurz [13] used a rotating silicon wafer to generate the hydrodynamic force required to remove 1 μm and larger particles. Removal rates above 90% were reported for particles larger than 2 μm. Taylor, Busnaina and coworkers [13 – 15] measured the removal force for sub-micrometer particles on silicon substrates and correlated it with the theoretical van der Waals force. That same technique is used in this study to determine the total adhesion force including the van der Waals force.

The primary adhesion forces for a dry uncharged particle on a dry uncharged surface are the van der Waals and electrostatic forces. The van der Waals forces can increase due to particle and/or surface deformation that increases the particle contact area. Electrostatic forces, although they predominate for particles larger than 50 μm, play a significant role in bringing particles to surfaces. In humid environments, liquid can condense between the particle and substrate, giving rise to a very large capillary force, which increases the total force of adhesion. Zimon [7] concluded that when the relative humidity is above 70%, the capillary force dominates and should be the only adhesion force considered. The capillary force consists of two main components: the surface tension at the perimeter of the meniscus and the pressure difference between the liquid and vapor phases [18]. The effect of relative humidity on the removal force has also been studied by Bowden and Throssel [17]. They suggested that the force required to remove 98% of 50-micron glass particles from a glass substrate increases with increasing relative humidity. The study, however, did not provide the adhesion force as a function of humidity. Many studies have been conducted to investigate particle adhesion and removal. Very few studies, however, considered the effect of relative humidity. The overall objective of this study is to develop an understanding of the effects of the relative humidity on particle adhesion for different particle/substrate systems. The specific objective of this study is to determine the effect of relative humidity on the adhesion force between the following particle/substrate systems:

1. Polystyrene latex (PSL) particles/silicon substrate system.
2. Polystyrene latex (PSL) particles/polyethylene terephthalate (PET), metallized, and polyester coating layer (PCL) substrates.
3. Pigmented coating chips (selenium)/polyethylene terephthalate (PET), metallized, and polyester coating layer (PCL) substrates.
4. Skin flakes/polyethylene terephthalate (PET), metallized, and polyester coating layer (PCL) substrates.

They were obtained from CP Films, Fieldale, Virginia, USA. The metallized (Ti/Zr) and polyester coating layers are coatings on PET. All experiments were conducted in a class 10 clean room. Relative humidity between 10% – 90% was generated in a Plexiglas humidity chamber built for the purpose of conducting these experiments.

2. BACKGROUND

2.1. Adhesion Force

The van der Waals force is the dominant adhesion force for small particles (less than 50 μm). It arises from the high frequency movement of the electrons in the atoms or molecules giving rise to momentary areas of charge concentrations called dipoles [9]. The van der Waals force for a spherical particle attached to a planar substrate is given as:

$$F_{\mathrm{vdw}} = \frac{AR}{6z^2} \tag{1}$$

where A is the Hamaker constant, R is the radius of the spherical particle, and z is the separation distance between the particle and the substrate. The average separation distance between the two surfaces is taken as 4 Å (for smooth surfaces). Electrostatic force constitutes the main force of attraction for particles larger than 50 μm in diameter. For dry particle-substrate systems the electrostatic force becomes important. The presence of electrostatic charge can drastically alter the total adhesion force. Zimon [7] reported that the force of adhesion could be increased by a factor of two when the net number of unit charges per particle on 40 – 60 µm particles increases from 700 to 2500. Most particles carry some electric charge, and some may be highly charged. Particles at low humidity were found to retain their charge and are held to surfaces by an attractive electrostatic force [9]. A charged particle experiences an electrostatic force in the vicinity of a conductive or insulating surface. The electrostatic image force (F_{el}) is given as [20]:

$$F_{\mathrm{el}} = \frac{Q^2}{6(D + z_0)^2} \tag{2}$$

where Q is the particle charge, D is the particle diameter and z_0 is the separation distance. This electrostatic force will deteriorate with time due to the dissipation of the charge.

When moisture is present in the air medium, condensation can take place between the particle and substrate. The capillary condensation gives rise to a capillary force, F_C, which is given by [20]:

$$F_C = 4\pi r \gamma_{\mathrm{LV}} \tag{3}$$

where γ_{LV} is the surface tension (for the liquid–air interface), and r is the radius of the spherical particle. Equation (3) is applicable to smooth surfaces and represents the maximum force that could be experienced as a result of capillary condensation. However, the force of adhesion approaches the values predicted by Eq. (3) only at relative humidity near 100% [19]. The capillary force consists of two components, as mentioned previously: the surface tension at the perimeter of the meniscus and the difference in pressure between the liquid and vapor phases. The existence of surface tension in a liquid–gas interface causes a difference in hydrodynamic pressure across the interface if the interface is curved. The quantitative expression for this pressure difference, as a function of surface tension and curvature, was derived by Laplace in 1806. He postulated an intermolecular force of attraction that diminishes rapidly with increasing distance [18]. Laplace's equation is given by:

$$P_C = P_1 - P_2 = \gamma\left(\frac{1}{r_2} + \frac{1}{r_2}\right) \tag{4}$$

where P_C is called the capillary pressure, P_1 is the pressure within the liquid phase, P_2 is the pressure within the gas phase, and r_1 and r_2 are the principal radii of curvature of the surface at the point of interest. The resulting capillary force depends on several parameters such as particle size, surface tension of the condensed fluid and the wettability (contact angle) of the substrate surface. Capillary forces are proportional to particle size, and according to Corn [19] the adhesion of large particles increases with relative humidity.

2.1.1. The Effect of Capillary Force on Adhesion

Equation (3) applies only to smooth spheres in a saturated atmosphere. Zimon [7] found that in air at a relative humidity near 100%, the majority of particles are held with forces less than that predicated by Eq. (3). Kordecki and Orr [22] observed that capillary condensation begins to appear at relative humidity above 50%. Luzhnov [24] reported that adhesion due to capillary force occurs when the relative humidity exceeds 70%. The same effect was also reported by Zimon [7], who concluded that at relative humidity of 50%, and particularly at humidity below 50%, capillary forces have no effect on the adhesion force. But all agreed that at relative humidity between 50% – 65%, the capillary force starts to have an effect on the total adhesion force. Zimon went on to conclude that between 70% – 100% relative humidity, the capillary force dominates the other adhesion forces and should be the only adhesion force considered.

2.2. Removal Forces

The removal forces required to dislodge particles off the substrate were generated by spinning the substrate about its axis and using air as the fluid medium, as shown in Figure 2.1.

A stationary particle in a moving fluid stream will experience a drag force, F_D, due to the difference in pressure across the surface of the particle. A gradient in the shear flow of the fluid will result in the Saffman or hydrodynamic lift force, F_L. Centrifugal force, F_{Cent}, is another contributing factor to the total removal force and is due to the angular velocity of the substrate about its axis. When the resultant removal force (drag, lift, and centrifugal force) exceeds the force

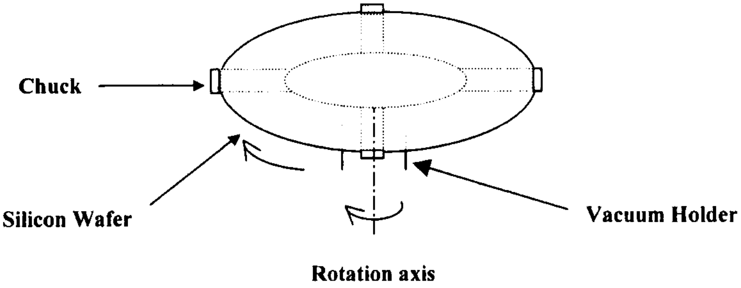

FIGURE 2.1 Schematic of the experimental setup for the particle removal experiment.

of adhesion, the particles are detached from the surface [15]. The Reynolds number based on the velocity at any radial distance, R, from the axis of rotation is given by [15, 16]:

$$\Re = \frac{R^2\omega}{\nu} \tag{5}$$

where R is the wafer radius, ω is the rotational speed of the substrate, and ν is the kinematic viscosity of the fluid. In the case of laminar flow, the following formulas are used for the drag and lift forces [15]. For the Stokes drag:

$$F_D = 3\pi\mu d V \tag{6}$$

For the Saffman lift:

$$F_L = 1.62\,\mu d^2\left(\frac{1}{\nu}\frac{du}{dx}\right)^{1/2} V \tag{7}$$

where μ is the viscosity of the fluid, d is the particle diameter, V is the relative velocity between the particle and the fluid, ν is the kinematic viscosity of the fluid and u is the fluid velocity. If the Reynolds number ($\Re^* = (dU^*/\nu)$ based on the particle diameter) exceeds 3×10^5, the flow is presumed turbulent. The two forces acting on the particle in turbulent flow are the drag force which is given as [15]:

$$F_D = 8\rho\nu^2\left(\frac{dU^*}{\nu}\right)^2 \tag{8}$$

and the Saffman lift force that is given as [15]:

$$F_L = 10.1\rho\nu^2\left(\frac{dU^*}{\nu}\right)^3 \tag{9}$$

where d is the particle diameter, ρ is the fluid density, and ν is the kinematic viscosity of the fluid. U^* is the friction velocity. The friction velocity has the dimensions of velocity and is defined as:

$$U^* = \left(\frac{\tau_0}{\rho}\right)^{1/2} \tag{10}$$

where τ_0 is the shear stress.

The centrifugal force in both flow regimes (laminar and turbulent) depends on the mass of the particle as well as the speed of rotation and is given as [20]:

$$F_{\text{Cent}} = mR\omega^2 \tag{11}$$

where m is the mass of the particle, ω is the rotational speed of the substrate, and R is the radial distance from the axis of rotation to the particle position on the substrate. The Reynolds number is calculated at any radial distances, R, from the center of the substrate. Equations (6) and (7) are used to calculate the drag and lift forces for laminar flow, and Eqs. (8) and (9) are used to calculate the drag and lift forces for turbulent flow. The centrifugal force is calculated using Eq. (11) and is vectorially added to the drag and lift forces to calculate the total removal force.

2.2.1. Adhesion Force Measurements

Busnaina, Taylor and Kashkoush [15] utilized the method outlined in Section 2.2 to determine the magnitude of the removal forces. They then correlated the hydrodynamic removal force with the removal efficiency and adhesion force, and introduced the following empirical correlation:

$$RE = 37 + 86.3\,FR - 39\,FR^2 + 5.7\,FR^3 \tag{12}$$

where FR is the force ratio ($F_{\text{removal}}/F_{\text{adhesion}}$). By using this correlation and calculating the hydrodynamic removal forces analytically and measuring the removal efficiency for any particle/substrate system experimentally, the particle adhesion force can be estimated [16]. The above equation is utilized to determine the total adhesion force of the particles considered in this study. Experimentally, the removal efficiency is defined as [20]:

$$\text{Removal efficiency} = \frac{n_{\text{before}} - n_{\text{after}}}{n_{\text{before}}} \qquad (13)$$

where n_{before} and n_{after} are the numbers of particles before and after cleaning in the area to be cleaned. Equation (12) is not valid at a removal force equal to zero.

3. EXPERIMENTAL PROCEDURE

All experiments were conducted in the Microcontamination Research Laboratory's class 10 cleanroom at Clarkson University. Polished silicon wafers, 5 inches (125 mm) in diameter, were used as the deposition substrates in the experiments. The PET, metallized, and PCL substrates were glued on the silicon wafer. Polystyrene latex spheres (22 µm), pigmented coating chips and skin flakes were used to simulate particulate contaminants. The PSL spheres were manufactured by Duke Scientific. A standard pharmaceutical nebulizer consisting of a pump, reservoir and nozzle was used. To eliminate contamination from the pump, a 1 µm Millipore air filter was used between the pump and reservoir. A Headway Research photoresist spinner was used to spin the wafer up to a maximum speed of 10,000 rpm. A laser surface scanner, Particle Measuring System (PMS), provides a detailed color-coded map of particle sizes, positions, and exact count of particles. It then sorts them into different sizes ranging from 0.1 µm to 10 µm. Particles larger than 10 µm in diameter are shown as 10 µm. An Olympus BH-2-UMA optical microscope is used to count the particles on substrates other than silicon wafers. The microscope is connected to a CCD camera, a video monitor, and video microscaler. A calcium sulfate desiccant was used to reduce humidity below 40%. To study the effect of levels between 10% and 90% relative humidity, a humidity chamber was designed and built (using Plexiglas) to provide the required humidity.

The chamber is equipped with two independent humidity sensors, and an opening through which particles can be deposited, a platform to place the wafer during deposition, and an inlet for the moisture being supplied through a PVC valve from a humidifier. When the chamber sensor is set, the moisture input to the chamber is controlled to maintain the desired relative humidity. The following experimental procedures were used for each of the particle/substrate systems:

3.1. The PSL Particles/Silicon Substrate

1. To generate relative humidity between 10% and 50%, a half-pound (228 grams) of calcium sulfate desiccant is placed in the humidity chamber. For relative humidity between 50% and 90%, a humidifier is turned on, and in both cases the system is monitored until the desired humidity level is reached.
2. The silicon substrate is placed in the humidity chamber for two minutes.
3. Using a nebulizer, the 22 μm PSL particles are deposited and left for two minutes; 12–16 seconds are required to deposit more than 300 particles.
4. The laser surface scanner is used to obtain the exact number (n_{before}) of the 22 μm PSL particles deposited on the substrate.
5. Using air as the medium, the spin coater generates the removal forces that consist of drag, lift and centrifugal forces. The silicon substrate is rotated at 8500 rpm for 120 seconds.
6. The laser surface scanner is used to determine the exact number (n_{after}) of the 22 μm PSL particles remaining on the silicon substrate.
7. The above procedure is repeated at various humidity levels, 10% to 90% relative humidity.

3.2. The PSL Particles/(PET, Metallized and PCL) Substrates

1. The substrates (PET, metallized, and PCL) are cut into five-inch (12.7 cm) disks and glued onto the surface of a silicon wafer. A small (1.5 cm × 1.5 cm) square is marked on the substrate surface, at 4 cm (in the radial direction) from the center of the circular substrate.

2. Depending on the desired relative humidity, the substrate is left for two minutes in that humidity and then the 22 μm PSL particles are deposited using a nebulizer and left for two minutes.
3. The substrate is then taken out of the humidity chamber and a microscope is used to count the number (n_{before}) of PSL particles in the marked square.
4. Using air as the medium, the spin coater generates the removal force that consists of lift, drag and centrifugal forces. The substrate is rotated at 8500 rpm for 120 seconds.
5. The microscope is again used to count the number of PSL particles (n_{after}) still remaining in the marked square.
6. The above procedure is repeated for the PET, metallized surface and PCL substrates and at different humidity levels.

3.3. The Pigmented Coating Chips/ (PET, Metallized and PCL) Substrates

The procedure was the same as in Section 3.2 with the following exceptions:

1. The pigmented coating chips are placed on a glass plate and cut into smaller pieces. A tweezer was used to deposit the pigmented coating chips.

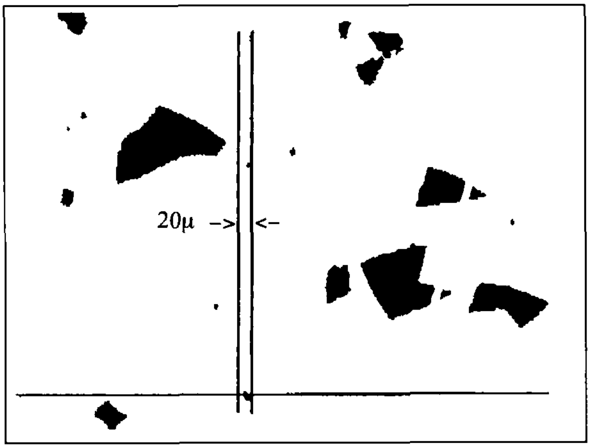

FIGURE 1 Pigmented coating chips.

2. The number of pigmented coating chips deposited (n_{before}) ranged from 65 – 120 in the marked square.
3. The size of the deposited pigmented coating chips ranged from 20 μm – 120 μm. There was a significant variation in the shapes of the pigmented coating chips. Figure 1 shows the different shapes and sizes observed under the microscope.

3.4. Skin Flakes/(PET, Metallized and PCL) Substrates

The same experimental procedure was followed as in Section 3.2, with the following exceptions:

1. Skin flakes were generated by rubbing dry skin over the substrate.
2. The number of skin flakes deposited (n_{before}) was from 10 – 12 in the marked square.
3. Different sizes and shapes were observed under the microscope. The size ranged from 30 μm – 100 μm.

4. RESULTS AND DISCUSSION

4.1. The Effect of Relative Humidity

A wafer surface scanner is used to measure the removal of PSL particles on silicon wafers which gives an accurate count of the number of particles on the five-inch (12.7 cm) wafer. This is the only part of the study which uses a scanner to count the particles. The rest of the experiments involved counting the particles visually using an optical microscope in pre-marked areas only. This produced a larger data scatter compared with the measurements using the surface scanner. For example, a typical standard deviation for the PSL/silicon substrate experiments is about 4 – 5%. For the rest of the data the standard deviation is 14 – 18%. Therefore, these experiments are at best qualitative since only the particles on that pre-marked area of the substrate are counted. However, the data show clear trends for the effect of relative humidity on particle adhesion and removal.

Figures 2 and 3 show the effect of relative humidity on the removal efficiency and adhesion force for 22 μm PSL particles on a silicon substrate. The relative humidity was between 15% and 85% in the humidity chamber. The highest removal efficiency is achieved at 45%

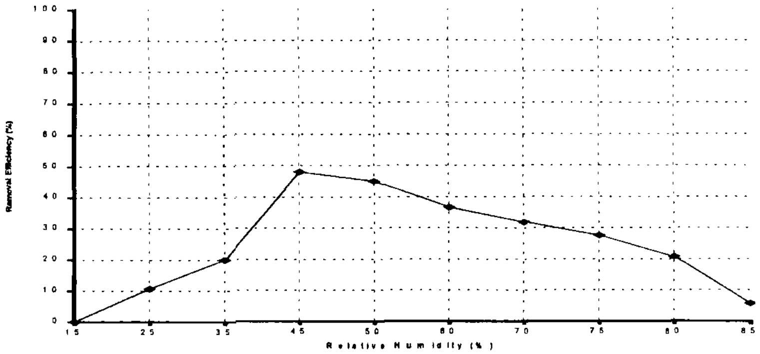

FIGURE 2 The effect of relative humidity on the removal efficiency (PSL particles/silicon wafer).

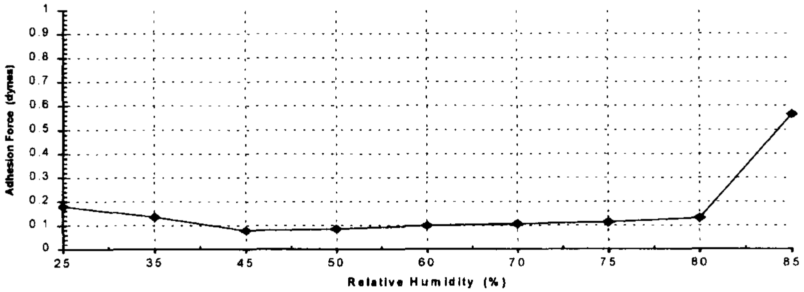

FIGURE 3 The effect of relative humidity on the adhesion force (PSL particles/silicon wafer).

relative humidity. As the relative humidity is reduced (below 45%), the adhesion force increases, and the removal efficiency decreases to zero at 15% relative humidity. The increase in the adhesion force is due to the charge build-up in low relative humidity since both the PSL particles and the silicon wafer (with native oxide film) are insulating.

At relative humidities between 45% and 80% the removal efficiency decreases, and the adhesion force gradually increases. Above 80% relative humidity, the adhesion force increases linearly due to capillary condensation which gives rise to capillary forces. This is due to the fact that both surfaces are hydrophillic, which gives rise to a large capillary force. Also, above 70% relative humidity the capillary force becomes the dominant force of adhesion. The theoretical capillary force value obtained using Eq. (3) is 1.0 dynes while the capillary force obtained from Figure 3 is 1.2 dynes. The difference is about 16%, which could be attributed to the van der Waals force. Using Eq. (1), the van der Waals force was calculated to be 0.4 dynes. Figures 4 and 5 show the effect of relative humidity on the removal efficiency and adhesion force for 22 μm PSL particles and three substrates (PET, metallized and PCL). The bell-shaped curve is again observed with the highest removal efficiency, with lowest adhesion force for all three substrates occurring at 45% relative humidity. As the relative humidity increases above 45%, the capillary force increases the total adhesion force. The PSL particles and the substrates (PET, metallized and PCL) are hydrophilic which gives rise to a large capillary force resulting in a low

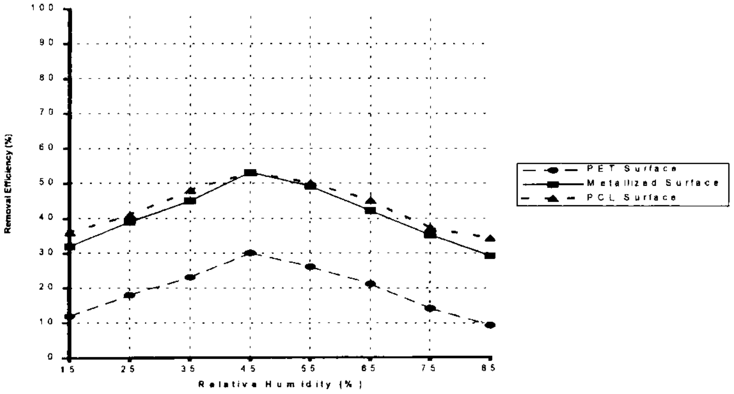

FIGURE 4 The effect of relative humidity on the removal efficiency (PSL particles/(PET, metallized, and PCL) surfaces).

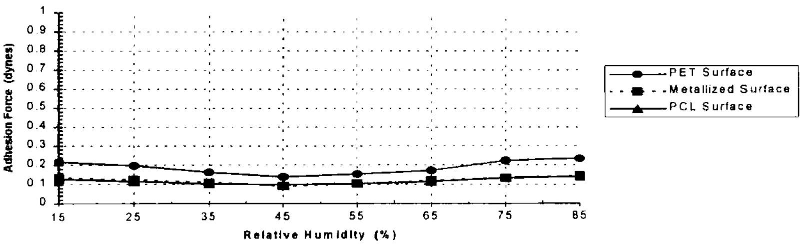

FIGURE 5 The effect of relative humidity on the adhesion force (PSL particles/(PET, metallized, and PCL) surfaces).

removal efficiency at high relative humidity. As the relative humidity decreases (below 45%) the electrostatic force increases the total adhesion force and decreases the removal efficiency. Figure 4 shows that the PSL particles on a PET substrate were the hardest to remove, compared with the metallized and PCL substrates that had similar removal efficiencies. This is due to the fact that the PET substrate and the PSL particle are both insulating (nonconductive) materials. At high relative humidity, the PET surface is more hydrophilic (a contact angle of 35°) as compared with the metallized (contact angle of 50°) and PCL substrates (a contact angle of 60°).

Figures 6 and 7 show the effect of relative humidity on the removal efficiency and adhesion force for pigmented coating chips on the three considered substrates (PET, metallized and PCL). The shape of the curve is different from that observed in the previous two figures where the removal efficiency is very high at low relative humidity below 25% (a removal efficiency above 90% can be achieved at 15% relative humidity). As the relative humidity is increased (above 25%), the adhesion force steadily increases. The high removal efficiency at low humidity could be attributed to the composition of the pigmented coating chips, which are made from selenium (a p-type semiconductor). The selenium will not build an electrostatic charge (when exposed to light) as compared with the PSL particles and skin flakes, which are not conductive (insulating). The conductivity of selenium is sensitive to light. The conductivity increases when the

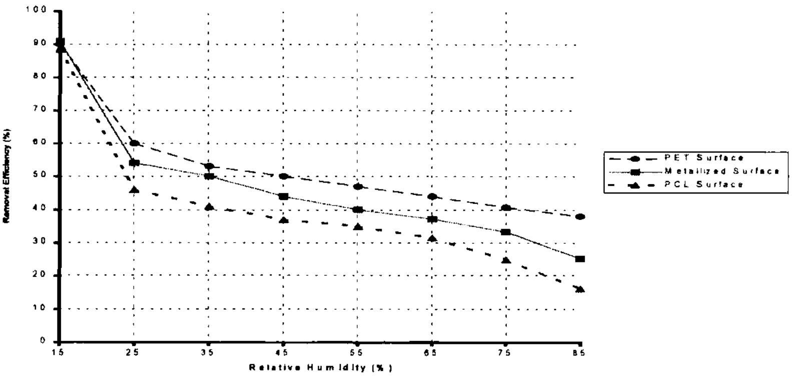

FIGURE 6 The effect of relative humidity on the removal efficiency (pigmented coating chips/(PET, metallized, and PCL) substrates).

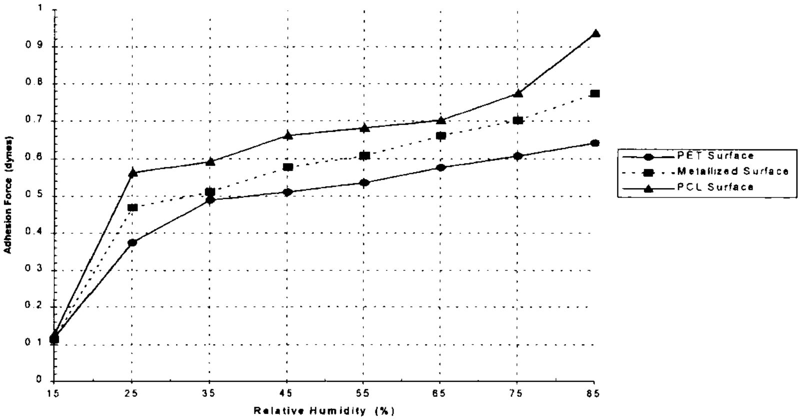

FIGURE 7 The effect of relative humidity on the adhesion force (pigmented coating chips/(PET, metallized, and PCL) substrates).

selenium is illuminated [25] which occurred throughout the experiments. The removal experiment leads to electrostatic charge build up by the high-speed rotation of the substrates and the particles.

Figures 8 and 9 show the effect of relative humidity on the removal efficiency and adhesion force for skin flakes on the three considered substrates (PET, metallized and PCL). As the relative humidity decreases (below 45%) the adhesion force also increases. Between 45% and 75% relative humidity the removal efficiency increases and then

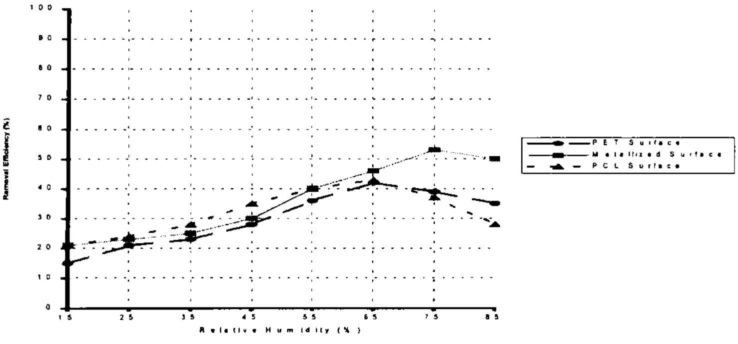

FIGURE 8 The effect of the relative humidity on the removal efficiency (skin flakes/ (PET, metallized, PCL) substrates).

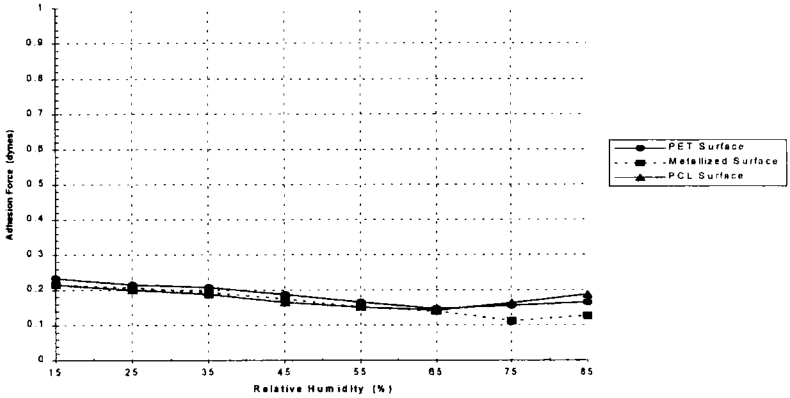

FIGURE 9 The effect of the relative humidity on the adhesion force (skin flakes/(PET, metallized, PCL) substrates).

starts to decrease. For the PET and PCL surfaces the adhesion force starts to increase at 65%, and for the metallized surface at 75%. The reason the removal efficiency is higher at higher humidity (65% – 75%) than for the other particles may be due to the fact that skin flakes absorb water and, consequently, may slightly increase in size and mass.

4.2. The Effect of Particle Size

The size of a particle has a profound effect on the adhesion force. To evaluate this effect, two different experiments were conducted using the three considered surfaces (PET, metallized and PCL) and the pigmented coating chips as contaminants. Figure 10 shows the effect of the particle size of the pigmented coating chips on the removal efficiency. At first, the pigmented coating chips (sizes between 100 and 500 μm) were deposited directly at different values of relative humidity. This is represented by the dotted line in Figure 10. The same experiments were conducted under the same conditions, but using smaller pieces of pigmented coating chips (that are chopped into sizes between 20 and 60 μm). The removal efficiency is lower for the small particle size (dotted lines). This is because the hydrodynamic removal force is a function of the particle size (cross sectional area of the particle). This results in higher removal force for larger particles [9].

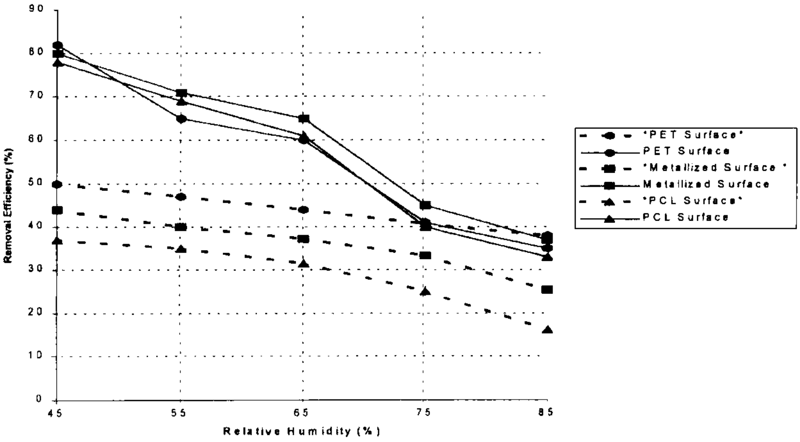

FIGURE 10 The effect of particle size on the removal efficiency (pigmented coating chips/(PET, metallized, PCL) surfaces). The smaller particles are indicated by the dotted line.

5. CONCLUSIONS

The results indicate that the capillary force is significant at a relative humidity above 50% and dominates at a relative humidity above 70%. At relative humidity below 45%, the electrostatic force became significant. Removal and adhesion results varied depending on the particles and substrates used but the trend of high adhesion at high and low humidity was observed for all particle/substrate systems. The results also indicate that the larger particles removal efficiency is higher overall.

1. The effect of relative humidity on the adhesion and removal for the 22 μm PSL particles on silicon substrates was very significant. The removal of PSL particles was very low at high and low relative humidity. The lowest adhesion force (highest removal efficiency, 49%) occurs at 45% relative humidity.
2. The effect of relative humidity on the adhesion and removal for the 22 μm PSL particles on the PET, metallized, and PCL substrates is very similar to PSL on a silicon substrate, showing the same bell-shaped curve. The removal of PSL particles was very low at high and low relative humidity. The highest removal efficiency (lowest

adhesion force) for the all three substrates occurred at 45% relative humidity. The PSL particles on a PET substrate surface were the hardest to remove, compared with the metallized and PCL substrates that had similar removal efficiencies.

3. The effect of relative humidity on the removal efficiency for pigmented coating chips on the three considered substrates (PET, metallized and PCL) was different from the PSL particles on the same substrates. The removal efficiency is very high at 90% (low adhesion force) at low relative humidity (below 25%). The removal efficiency is low (between 16 and 40%) at high relative humidity.
4. The effect of relative humidity on the removal efficiency of skin flakes from the three considered substrates (PET, metallized and PCL), was somewhat similar to the PSL behavior. At low relative humidity (below 45%) the removal efficiency was low. The maximum removal efficiency (lowest adhesion force) occurs between 45% and 75% relative humidity. For the PET and PCL surfaces the removal efficiency starts to decrease at 65%, and for the metallized surface at 75%.
5. The effect of particle size on removal efficiency is significant. The data indicate that when the size of particles decreases, the removal efficiency decreases.

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