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# Adhesion of Silica-Coated Toner Particles to Bisphenol-A Polycarbonate Films

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**Abstract.** *Particles of electrophotographic toner (black, silica-coated, number-weighted diameter of 7 $\mu$m) were electrostatically developed on the surface of a film of bisphenol-A polycarbonate coated on nickelized polyethylene terephthalate (PET). The thickness of the coatings varied from 0.8 to 22.0 $\mu$m. The force needed to remove the particles was determined using ultracentrifugation. We found that removal of half of the particles from the film surface required a force of approximately 250 nN irrespective of the thickness of the polymer film. This result indicates that removal of the toner from the film involves surface van der Waals interactions rather than forces associated with long-range electrostatic interactions of the charged toner with the grounded nickel electrode.*

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## INTRODUCTION

In a typical electrophotographic process small polymer-based charged particles (toner) are electrostatically transferred imagewise to the polymer-based surface of an organic photoreceptor (OPC).[1–3] The toner is subsequently transferred to a receiver, such as paper, using a combination of pressure and electrostatics. The mechanisms governing the adhesion of toner particles to the photoreceptor have been a topic of interest for many years, and interest has intensified as the demand for improved image quality has led to the reduction of the diameter of toner particles from approximately 20 $\mu$m several decades ago to about 8 $\mu$m today. The increased interest is due, at least in part, to the fact that smaller toner particles are both more difficult to transfer from the photoreceptor to a receiver and more difficult to remove from the photoreceptor during subsequent cleaning operations.[4]

Many theories have been proposed to explain the interactions giving rise to toner adhesion. The two mechanisms generally accepted to explain the adhesion of toner particles to a photoreceptor are 1) electrostatic interactions between the charged particles and the photoreceptor and 2) adhesion forces arising from van der Waals interactions. As discussed by Gady et al.,[5] both types of forces contribute to the attractive forces giving rise to toner adhesion. The appropriate questions are which of these interactions are dominant, under what conditions, and how can each force be measured independently? Indeed, the debate over the dominant mechanism has given rise to much research.[6–25] A summary of this debate has been presented elsewhere.[26]

Several recent papers have illustrated the interplay between electrostatics and van der Waals forces in toner adhesion. For example, Rimai et al.[26] showed that the force needed to remove 7-$\mu$m-diameter spherical toner, without a silica surface treatment, from a standard commercial OPC that had not been overcoated with any release agent such as zinc stearate was in excess of 750 nN, which was the maximum force they could exert. It was estimated, using Johnson-Kendall-Roberts (JKR) theory,[27] that van der Waals surface adhesion would require a detachment force in the range of 1100 to 1200 nN. Furthermore, by applying zinc stearate to the surface of the photoreceptor, the residual van der Waals forces were reduced to approximately 100 nN and the remaining detachment force varied as the square of the charge on the toner. These results highlighted the apparent contributions of both van der Waals and electrostatic forces to toner adhesion.

Rimai et al.[28] studied the effects of size, silica concentration, and charge on the force needed to detach irregularly shaped surface-treated toner particles from an organic photoreceptor. The toner particles had been prepared by grinding and classifying. They reported that van der Waals interactions dominated electrostatic forces in this study.

Recently Rimai and Quesnel[29] and Wright et al.[30] reported that surface forces between electrically charged toner particles and a photoreceptor are sufficiently strong as to prevent electrostatic transfer across an air gap to a receiver at voltages approaching electrostatic breakdown. In such circumstances, transfer from a photoreceptor to a receiver is accomplished by having the toner particle contact both the photoreceptor and the receiver, thereby at least partially balancing the surface forces.

These results demonstrate the interplay between surface van der Waals interactions and long-range electrostatic forces in toner adhesion to the OPC surface. Investigations utilizing materials modified to reduce the van der Waals forces, such as zinc stearate treatment of the OPC surface and the addition of small silica particles to the surface of the

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toner, have demonstrated that long-range electrostatic forces are also present but often dominated by the surface forces.

Van der Waals interactions have a range in the order of only a few nanometers while electrostatic interactions are long range. One can test for the relative importance of van der Waals and electrostatic interactions by measuring the force needed to remove toner particles from a series of substrates comprising an electrically conducting grounded layer overcoated with a dielectric varying in thickness. To carry out such a test we have electrostatically developed toner particles (7.1 $\mu$m median number-weighted average) onto a series of four substrates where the thickness of the dielectric went from 0.8 to 22 $\mu$m. As the distance between the toner and the ground plane is increased, adhesion forces due to electrostatic interactions should decrease while van der Waals interactions should remain constant. In the following experiments bisphenol-A polycarbonate was used as the dielectric because polycarbonates are commonly utilized as the surface layer (charge transport layer) binder in many commercially available OPCs.

## EXPERIMENTAL

In this study, the force needed to remove toner particles from a series of substrates comprising nickelized polyethylene terephthalate (PET) overcoated with varying thicknesses of bisphenol-A polycarbonate was determined using ultracentrifugation.

The developer (Kodak NexPress Black Developer, Cat. No. KH2102000) is commercially available with toner prepared by grinding and classification blended with a magnetic carrier. The toner particles comprise a polyester binder (mass density$=1.2$ g/cm$^2$) and have a median number-weighted diameter of approximately 7.1 $\mu$m, as determined using a Coulter multisizer. Assuming sphericity, the particle mass was calculated to be approximately $2.2\times10^{-10}$ g. The toner particles also comprised 1.2% by weight of Degussa Aerosil® R 972 silica applied on the surface. The charge-to-mass of the toner was approximately $-24.3$ $\mu$C/g, as determined using the method described by Maher.[31] This corresponds to a median particle charge of $-5.35\times10^{-15}$ C. Fresh developer was used for each experiment. The toner charge-to-mass was determined before each experiment and did not change during the course of an experiment.

The substrates were comprised of nickelized PET that had been coated from solution with four different thicknesses of bisphenol-A polycarbonate. The polycarbonate was chosen because of its high electrical resistivity (greater than $10^{16}$ $\Omega$ cm) and low dielectric constant (approximately 3).[32] These values ensure that the dielectric relaxation time of the substrates is very long compared to the duration of the experiment. The coating thicknesses, determined from the cross sections using optical microscopy, were approximately 0.8, 4.5, 9.0, and 22.0 $\mu$m.

The film sample (approximately $3\times6$ in.) was held by vacuum to a grounded aluminum platen and had an edge coated with electrically conducting paint to ground the nickel layer. The platen was positioned over an electrostatic voltmeter and the surface potential of the polycarbonate coating was measured. The coating was then repeatedly washed with distilled water and blown dry with canned air until the surface potential was zero.

A very light coating of toner particles was deposited on the substrate using a sumpless magnetic brush[2] biased at approximately $-10$ V. This potential was found to deposit a sufficient number of negatively charged toner particles to permit good statistical counting, but still left the particles generally isolated from each other. To ensure that the toner deposition and resulting adhesion were not influenced by a triboelectric interaction between the toner particles and the polycarbonate, the substrate was developed in a normal manner with the exception that the brush was also grounded. In addition, the development system was set up so the flow of the developer was matched to the speed of the sample. This was accomplished by adjusting the magnetic brush shell speed to counter the core speed until there was zero net flow of developer, and then adjusting the shell speed so that the speed of the developer matched that of the substrate. A small deposit of toner was developed, resulting in a postdevelopment potential of approximately $-5$ V. When this toner was removed using canned air, the substrate potential was again 0 V, indicating that no observable tribocharging had occurred and that the observed potential arose simply from the charge of the adhering toner particles. The detachment force was determined using a Beckman L8070M ultracentrifuge capable of speeds up to 70,000 rpm. At 70,000 rpm the acceleration of the centrifuge with the 6.45 cm radius rotor is 354,000 g.

The number of particles on a sample of the substrate was determined by counting the particles in five areas under a microscope using IMAGE-PRO particle-counting software. The sample was then placed in the rotor and spun at the desired speed. The sample was then removed and the number of residual particles was then determined in a similar manner. In order to eliminate the possibility of increases in adhesion caused by the occurrence of plastic deformations[33] or from the rotation of the particles on the substrate following deposition,[34] all measurements were made on the same day as the deposition. Moreover, the centrifuge speeds were randomized to ensure that any systematic deviation would be observed. The applied force needed to remove 50% of the particles initially on the substrate was considered to be the detachment force.

## RESULTS

Figure 1 shows the percentage of the toner particles removed as a function of the applied force for each of the four substrates.

As can be seen from Fig. 1, the detachment force for 50% removal on all samples was approximately 300–350 nN. Moreover, the actual detachment force of the particles varied randomly with substrate thickness, suggesting that the variations were due to experimental error rather than arising from the thickness of the polycarbonate coating. Finally, the percent removed at a given applied force appears

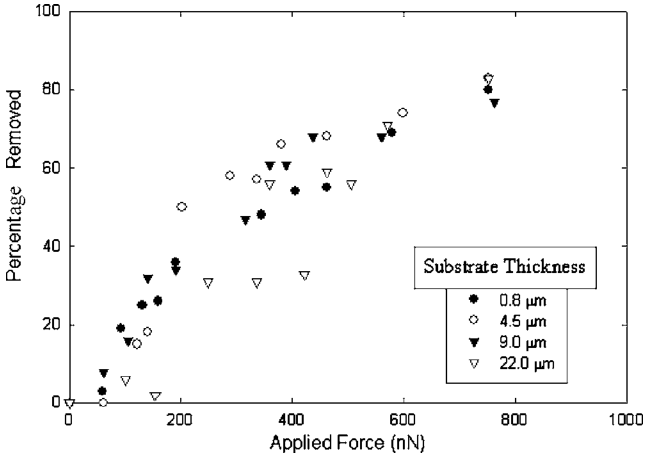

Figure 1. The percentage of particles removed as a function of the applied force for each of the four bisphenol-A polycarbonate thicknesses.

to be (within experimental error) independent of the coating thickness. These results clearly show that the force of adhesion between the toner particles and the polycarbonate-coated nickelized PET is independent of the thickness of the polycarbonate coating, and therefore independent of the distance from the particle to the grounded nickel layer.

As is well known, the electric field outside an ideal parallel plate capacitor is zero. While such a capacitor is a reasonable model for the substrate used in this study, one must be cautious about the effects of nonuniform charge distributions on the surface or trapped charges within the bulk of the polycarbonate. To determine whether or not such charges can play a role on the adhesion of toner particles, the substrate with the 4.5-$\mu$m-thick polycarbonate coating was charged with a grid-controlled DC corona charger[1] to a potential of −112 V. The development station bias was then set to approximately −125 V, resulting in a similar difference of potential between the substrate and the development station as in the previous experiment. A similar sample was prepared with the substrate initially charged to approximately −500 V and the potential on the development station adjusted accordingly. Toner particles were then developed onto the substrate at about the same density as in the previous experiments. This puts charged ions in the immediate proximity of the toner particles. The percentages removed as a function of the applied force for the uncharged and charged substrates are shown in Figure 2.

It is apparent from Fig. 2 that (within experimental error) there is little difference in either the detachment force or the percentage of particles removed at a given applied force for the charged and uncharged substrates. As the charges giving rise to the potential on the substrate reside on its surface, the negative ions present should repel the negatively charged toner particles, thereby decreasing the detachment force if electrostatic contributions to the toner adhesion were significant in this instance.

## DISCUSSION

Theories of toner adhesion to surfaces invoke two main concepts: short-range van der Waals and long-range electrostatic forces. The former is influenced by the composition and geometry of the surfaces in contact and the latter by electrostatic interactions of the charged particle with the ground plane of the film. The thrust of the experiments described here is to address the question of the importance of the separation distance of the charged toner from the ground plane in the force required for toner removal. In the following discussion our results will be interpreted within existing theories of toner adhesion.

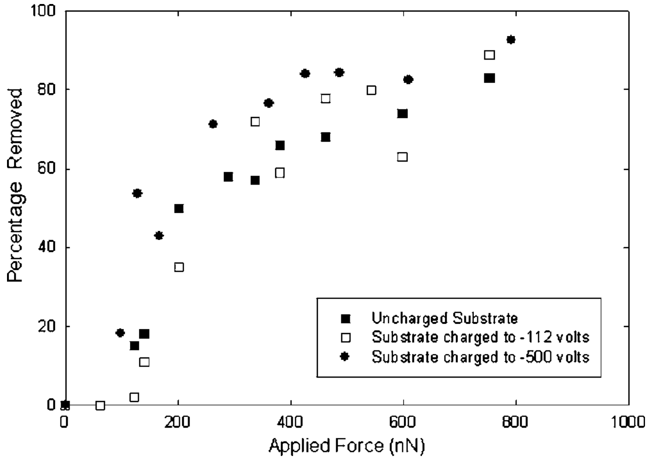

Figure 2. The percentage of toner particles removed as a function of the force applied to the 4.5-$\mu$m-thick bisphenol-A polycarbonate-coated substrate that was at 0, −112, and −500 V prior to the toner deposition.

At this point it is instructive to examine the various mechanisms that have been proposed to explain toner adhesion by calculating the force needed to detach the particles from the substrate, assuming the dominance of a) van der Waals interactions, b) electrostatic charged patches, and c) uniformly charged particles.

The detachment force for van der Waals interactions is calculated with the assumptions that the substrate is planar and that the particle is a sphere of radius 3.5 $\mu$m without silica on the surface. According to JKR theory, the detachment force $F_d$ is related to the radius of the particle $R$ by

$$F_d = \frac{3}{2} w_A \pi R, \tag{1}$$

where $w_A$ is the work of adhesion and is related to the surface energies of the particle $\gamma_P$ and substrate $\gamma_S$ and their interfacial energy $\gamma_{PS}$ by

$$w_A = \gamma_P + \gamma_S - \gamma_{PS}. \tag{2}$$

If the contacting materials are very similar, no interface is formed when they come into contact and $\gamma_{PS} \approx 0$ and the two surface energies are approximately equal, so Eq. (2) reduces to

$$w_A \approx 2\gamma. \tag{3}$$

For materials such as the polymers used in this study, $2\gamma \approx 0.08\ \mathrm{J/m^2}$. With these approximations, the detachment force of a spherical toner particle from the polycarbonate substrate is estimated to be approximately 1300 nN. The real value for the case of a perfect sphere is expected to be somewhat lower because $\gamma_{PS}$ will have a finite value. Even so, the calculated detachment force is well in excess of what is observed and indicates that van der Waals forces alone are of sufficient magnitude to potentially account for the adhesion of the toner to the polycarbonate. Moreover, because of the short-range nature of van der Waals forces, the detachment force would be expected to be independent of the thickness of the polycarbonate layers used in this study.

In reality, the toner particles used in this study are not spheres. Moreover, they also comprise silica nanoclusters on the surface. Both of these factors will also tend to decrease the detachment force.[26] While a detailed explanation of the effect of the particle shape and silica concentration on adhesion is beyond the scope of this article and has been presented elsewhere,[35] it suffices to say that detachment forces arising from van der Waals interactions are typically several hundred nanonewtons for particles and substrates similar to those used in this study.

The electrostatic contributions to the force of adhesion can also be calculated. As discussed by Johnson et al.,[27] the force needed to remove a particle from a substrate is not the same as the force of attraction. That is to say, one cannot simply balance the attractive and the applied detachment forces to determine the force at which separation of the particle from the substrate occurs. This is because the force balance approach does not take into account the energy stored elastically in the contacting materials. Rather, one must equate the energy within the system with the work needed to be done to effect separation. While the JKR theory does take into account applied forces, it does not consider long-range interactions such as those caused by electrostatic forces. Amending the JKR theory to include electrostatic forces is beyond the scope of this article. However, Rimai et al.[26] have shown that simply treating the long-range electrostatic forces as an applied load in the JKR equation is a reasonable approximation. Accordingly, the electrostatic contribution to the adhesion of the toner particles will be treated as an additional force balance effect, although this is not strictly correct.

First, let us assume that the charge on the toner particles, $q$, is uniformly distributed over the surface of each particle and that each particle is spherical. Furthermore, assume that the particle of radius $R$ is in contact with a polycarbonate coating of thickness $d$. From the method of images, the attractive force $F_A$ between the surface charge on the particle and the grounded nickel layer alone is given by

$$F_A = \frac{1}{4\pi\varepsilon_0}\left(\frac{q}{2(R+d)}\right)^2, \tag{4}$$

where $\varepsilon_0$ is the permittivity of free space. Shielding of the conductive layer by the dielectric coating decreases this force. This can be understood as simply the result of the polarizability of the polymeric coating on the grounded conductive nickel layer. For $q=-5.35\times10^{-15}C$, $F_A$ is calculated to be less than 0.3 nN if the polycarbonate coating is 0.8 $\mu$m thick. This force is obviously inconsequential. Moreover, it will decrease by over an order of magnitude with the 22 $\mu$m coating. This calculation is qualitatively correct, but it is, of course, a simplification of the exact solution.[36] Feng and Hays[37] have calculated the effect of an intervening dielectric material on the electrostatic force between a charged particle and a conductive ground plane. They report that large changes will occur when the thickness of the dielectric is less than the particle diameter. We do not observe such an effect.

As discussed by Hays,[17] the surface charge density of toner particles need not be constant. Rather, Hays proposed that the charge can be localized, thereby creating "charged patches." Assuming that the charged patch is in close proximity to the photoreceptor so that parallel plate capacitor geometry could be assumed, Hays then showed that

$$F_A = \frac{\sigma^2 A_C}{2\varepsilon_0}, \tag{5}$$

where $\sigma$ is the surface charge density within the area of contact $A_C$ between the toner particle and the photoreceptor. This could give rise to a substantially higher force than that given by Eq. (4).

Let us now calculate the force of attraction between the charged toner particle and the substrate, assuming the interactions occurred according to the charged patch model. The actual surface charge density distribution for toner particles has never been determined. Moreover, because of the polycarbonate coating, one cannot simply assume parallel plate capacitor geometry, so Eq. (5) cannot be used to estimate the interaction force in the present study. Instead, let us assume the extreme case where all the charge on a toner particle is located at a point that is directly in contact with the surface of the polycarbonate coating. With this assumption, the force of attraction is given by

$$F_A = \frac{1}{4\pi\varepsilon}\left(\frac{q}{d}\right)^2, \tag{6}$$

where $\varepsilon$ is the dielectric constant of the polycarbonate ($\approx 3\varepsilon_0$), and $\varepsilon_0$ is the permittivity of free space. For the 0.8 $\mu$m thick polycarbonate coating, $F_A$ is calculated to be 134 nN, or approximately 40%–45% of the measured detachment force. This should decrease with the square of the coating thickness to 4.2 nN for the 4.5 $\mu$m thick coating, 1.1 nN for the 9 $\mu$m thick coating, and 0.18 nN for the 22 $\mu$m thick coating. Certainly, this decrease in the detachment force with increasing polycarbonate thickness would have been observed had it occurred. Instead, the detachment force is seen to be independent of coating thickness. Moreover, the present set of calculations assumes conditions that would give rise to the maximum force obtainable for these toner particles under the charged patch model. With a more uniform distribution of charge on the toner surface, the contribution to the toner adhesion caused by localized charged patches should be less.

Recently Schein has proposed that there is a long-range electrostatic force associated with each point at which a toner particle contacts a conductive surface that is equal to $4/\pi$ times the force of attraction of Eq. (6) in vacuum for small values of $d$.[38] The detachment force predicted by this model also depends on the square of the distance between the toner contact points and the ground plane.

As is well known,[39] a charged particle can polarize a neighboring dielectric substrate, thereby resulting in a force of attraction between that particle and the substrate. However, for a particle in intimate contact (i.e., there is no intervening dielectric medium), the polarization force depends on the difference between the dielectric constants. In this study, both the polycarbonate substrate and the polyester particles have a dielectric constant of approximately 3, so the polarization force should vanish.

If, on the other hand, one assumes that the silica physically separates the entire particle from the substrate, thereby creating an air gap, it is possible to create a polarization force. Bowen et al.[40] has shown that the contact area between particles similar to those used in this study and planar substrates is approximately 5% of the cross-sectional area of the particle. According to Hays,[37] in the presence of an applied field, E, the total electrostatic force of attraction between a uniformly charged particle, with net charge Q and radius R, and a conductive surface is given by

$$F_E = \frac{\alpha Q^2}{16\pi\varepsilon_0 R} + \beta QE - \gamma\pi\varepsilon_0 R^2 E^2, \tag{7}$$

where the dimensionless coefficients $\alpha$, $\beta$, and $\gamma$ are functions of particle characteristics. The first two terms of this expression correspond to the attraction due to the particle charge in the absence and presence of an applied filed. The third term describes the polarization of the particle in the applied field. In the absence of an applied field the contribution to the force of attraction due to the second and third terms of Eq. (7) vanish.

## CONCLUSIONS

We have found that the force required for removal of the silica-coated toner particles (median volume-weighted diameter of approximately 7 $\mu$m) from a film of bisphenol-A polycarbonate on nickelized PET is approximately 300–350 nN and is independent of the thickness of the coating or negative corona charge on the polycarbonate surface. Thus, in this system toner adhesion does not involve long-range electrostatic interactions but is dominated by surface van der Waals interactions. While the magnitude of these forces can vary somewhat depending on the substrate, the specific toner used, and the type and concentration of the silica coating on that toner, the values we observe are comparable to those reported elsewhere in the literature.

The nature of the forces governing toner adhesion to a polymer surface has generated considerable debate as to the dominance of either van der Waals or electrostatic forces. We have previously demonstrated that both interactions contribute and are important. Either may dominate depending on the exact circumstances. In the present system, van der Waals forces seem to be the dominant mode of interaction between the toner particles and the substrate. In future experiments we will investigate the effects of toner size, charge-to-mass, silica coverage, zinc stearate treatment of the dielectric surface, etc. on the force required for toner removal with varying thickness of dielectric.

## ACKNOWLEDGMENTS

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