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Powder Adhesion Measurement Using a Metered Air Pulse

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Abstract. *A blow-off tool has allowed for the measurement of the cumulative distribution of charged toner particle adhesion to a polymer belt. The tool methodology and design are described. The tool is calibrated to obtain the adhesion force using two different techniques, and the measured values agree well with published values using other adhesion measurement techniques. Measurements taken in different environments have yielded significantly different particle adhesions for the same average particle charge.* *© 2013 Society for Imaging Science and Technology.*
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INTRODUCTION

Chemically processed toners (CPTs) used in electrophotographic printing are fairly uniform microparticles with repeatable and predictable electrostatic charge. Present toner particles made for office printing systems are generally 5–7 μm in diameter, and are composed of a resistive polymer base and an outer coating of small silica additives that provide asperities to reduce the cohesion and improve system performance. These particles are moved through an electrophotographic printer by electrostatic forces, and significant controversy has arisen as to the relative magnitude of the electrostatic forces as compared to the van der Waals or dispersive forces also acting on the particles. Electrostatic forces are moderately easy to simulate, but dispersive adhesion forces are more elusive. Measurement of adhesion in toner has taken multiple forms, and all of these have indicated forces on the order of 10× what would be predicted from a model of simple Coulombic attraction to a substrate.[1]

The lack of agreement between Coulombic predictions and measured adhesive forces has led to a long discussion as to the dominance of toner adhesion being electrostatic or dispersive forces. The principal mechanism of particle adhesion depends upon the particular experimental configuration. For example, the limiting case of zero net charge results in the dispersion forces prevailing. However, as the surface charge of a particle increases, so do the electrostatic forces. As for adhesion of toner in electrophotography, there is still some debate as to the dominant mechanism, even within the last five years.[2,3] Since the purpose of this correspondence is to present a new method for measuring microparticle adhesion, it is constructive to briefly discuss the nature of such forces and how they affect the development of a new adhesion measurement procedure.

When particles are within a few nanometers of a substrate, van der Waals intermolecular dipole forces contribute to the attachment of the particle to the substrate. Since these forces are short range in nature, the magnitude of the force can vary greatly with the contact surface area.[4] The simplest mathematical representation of electrostatic adhesion force, the point-image model, consists of treating the total particle charge as a point charge interacting with its image charge by Coulombic attraction. Several proposals have been put forward to explain the order of magnitude difference between measurements and results from the point-image model. The charge patch model introduced by Hays attributes the difference to nonuniform charging (e.g. charge patches).[5,6] Schein criticized the charge patch model because the size of the charge patches required to fit experimental data would produce local electric fields exceeding air breakdown.[7] Instead, Schein and colleagues introduced the proximity force resulting from the discrete nature of charge, which produces a 2× increase over the point-image model.[1,8] Using an analytical model, Kemp and Whitney recently showed that, while including nonuniform charge, multiple particle interactions, and dielectric polarization produces enhancements over the image theory model by 2× to 5× individually, combining all of the effects into one model produces a nearly order of magnitude increase over the point-image model.[9] This analytical model was also used to explain the nonlinear nature of the attachment and detachment force of particles in an applied electrostatic field E_0.[10] Nonlinear field detachment physics is often approximated by a formula which is quadratic in the applied electric field:

$$F \approx -\alpha \frac{Q^2}{16\pi\epsilon_0 R^2} + \beta Q E_0 - \gamma \pi \epsilon_0 R^2 E_0^2, \qquad (1)$$

where Q is the particle charge, R is the particle radius, and the coefficients α, β, and γ are commonly determined

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empirically from experiments. Such nonlinear behavior implies that adhesion force measurements by electrostatic detachment are difficult to interpret without the aid of a full model of the electrostatic fields.

To aid in the separation of these two force components, a new adhesion measurement method based on an air pulse blow-off of toner has been developed to quantify the distribution of adhesion forces acting on a sample of charged toner developed onto a flat surface by an electrophotographic process. This air pulse method produces average adhesion measurements comparable to those of other published techniques. Because the test sample is produced by electrophotographic printing directly on a desired substrate (e.g. an intermediate transfer belt), it is much more controllable than other methods, and comparable measurements can be taken with different charge distributions and at different temperatures and humidities. The system has been found to be repeatable for equivalent samples even when measured in different systems or at different times. In this article, we describe a novel tool and methodology for microparticle adhesion measurement using a metered air pulse.

ADHESION MEASUREMENTS

Multiple attempts have been made to accurately measure small particle adhesion to a flat substrate. The most common methods used consist of ultracentrifuge,[2,11–13] electrostatic removal,[13–15] and microcantilever testing.[13,16–19] Each of these methods yield information about the nature of adhesion of small particles on smooth flat substrates. Toner particles have frequently been used as test particles due to their uniform size and composition. Adhesion measurements have been made for toner particles ranging from 6 to 20 μm in diameter, with and without small silica on the particle surface, and with and without electrostatic charge.

The ultracentrifuge method is the most studied method of measuring particle removal, and multiple tests have been reported using this technique. A key advantage of this approach is that it yields information about adhesion distributions in a sample, rather than individual measurements for individual particles. Ultracentrifuge measurements have found a 100× difference in adhesion forces between the first 20% removed and the amount of force needed to remove 80% for electrophotographic toner without surface additives on a flat substrate.[11] This work revealed that, while the size of the particle had a significant impact on the removal force,[2,11,18] no change in adhesion was found to be induced from neighboring particles,[13] although an amplification of about 5–7× has been predicted for an electrostatic adhesion model based on uniform charged dielectric spheres.[9,15,20] Due to the nature of the equipment used in this test, all testing published to date has been done at lab ambient conditions.

Other methods also produce adhesion values for toner and toner-like particles, but have various limitations. A microcantilever on an atomic force microscope (AFM) can be used to make adhesion measurements on individual toner particles.[13,16,17,19] The drawback of this method is that it results in measurements for individual particles and not distributions. Parallel plates, which generate an electrostatic field, can be used to obtain force distributions, but this method can only be used on particles with sufficiently high charge.[13–15] Air pulse removal is not limited to charged particles and results in removal force distributions. In this and previous work, air pulse removal was used to measure the electrophotographic adhesion of toner, with surface additives, to a variety of substrates in a variety of environments.[21] Testing yielded adhesion measurements very comparable to those found by the ultracentrifuge method. Air blow-off techniques have also been used successfully to measure paint powder adhesion for automotive applications.[22]

For all measurement techniques, the average adhesion value for particles with surface additives, suggesting minimal contact area, was on the order of a few hundred nanonewtons.[2,11,12,18,21] This value exceeds the predicted value of adhesion from the Coulombic attraction model (assumes a spherical particle with a uniform surface charge) for even the most highly charged particle. Image forces predicted by Coulombic approximations estimate adhesion at tens not hundreds of nanonewtons, and this order of magnitude difference ignited a debate as to the nature of the physics occurring with small particle adhesion.[1,12,15,20,21,23,24]

TOOL DESIGN AND FUNCTION

Blowing off toner with a pulse of air has several advantages over other adhesion measurement methods. The equipment we developed to do this measurement is easily constructed, portable, and configurable. To generate a controlled sample, toner is patterned in a uniform dot (isopel) halftone pattern onto an intermediate transfer belt inside an electrophotographic printer. The sample consists of an average 20% toner coverage such that the layer is approximately one particle thick. The sample is then placed in a fixture directly under a 1 mm diameter stainless steel tube connected to a device to meter a pulse of air. The air pulse is aimed directly downward, as that allows for a closed-form solution for the velocity profile. The toner removed at specific distances from the nozzle can be measured optically, and the images converted to toner removal (i.e. percentage removed) by the use of internally written image analysis software. Higher toner coverage reduces the accuracy of the toner removal measurement by the optical measurement technique. The photograph in Figure 1 shows the blow-off portion of the tool. The air pulse is held at 1 s, and the pressure can be adjusted from 10 psi to 20 psi. The choice of air pressure range was determined by varying the pressure and noting the range of maximum sensitivity without completely blowing off the stagnation point. The belt sample is placed in a frame that holds the material both during the blow-off phase of measurement and in a locating fixture on the *x*–*y* table of a microscope.

The air pulse is a laminar blast aimed directly down at the halftone pattern. Calculations and image analysis show that adhesion between the toner particles and the substrate is broken by a rolling motion of the toner due to the torque

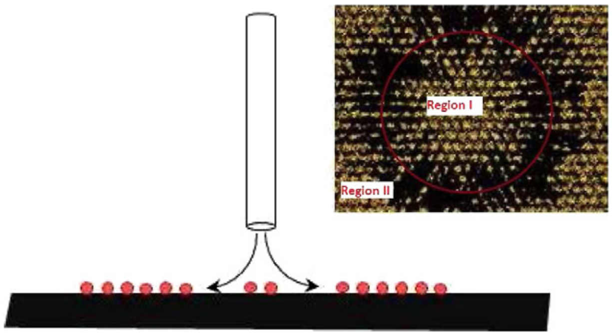

Figure 1. Diagram of blow-off tool with inset being a microphotograph of the stagnation point directly under the nozzle center, and a ring of removed toner. The inset microphotograph shows yellow toner, which provides a good contrast with the polyimide belt for determining percentage toner removal by the image analysis software. The stagnation area is shown in Region I, and the toner removal measurements are taken from Region II. The boundary line separating the two regions is defined as the local coverage minimum surrounding the point directly under the nozzle center, which is schematically depicted by the red circle.

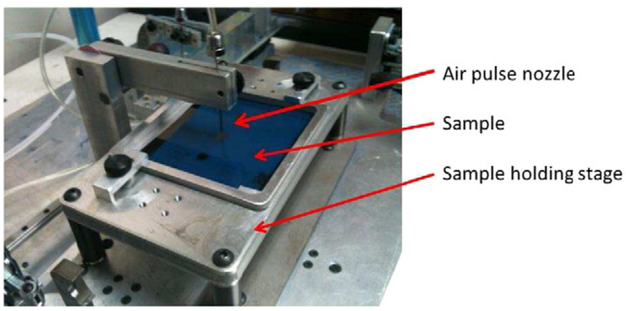

Figure 2. Blow-off tool. The air pulse nozzle is a 1 mm diameter stainless steel tube which delivers a 1 s metered pulse of air at a pressure ranging from 10 to 20 psi. The tip of the nozzle is located 4 mm above the sample. The sample holding stage includes a custom-built *x*–*y* positioning frame for precise positioning with the nozzle and the imaging microscope.

created by the drag profile. The small contact area with the substrate relative to the diameter of the toner gives a mechanical advantage to the drag-related torque. The lower the adhesion force of toner to belt, the smaller the stagnation spot and the larger the ring of removed toner that surrounds it. Figure 2 shows an image of the device developed for these measurements. The fixture allows for exact positioning of the nozzle and the sample. The lower end of the nozzle is located 4 mm above the sample surface, and toner removal is measured out to 7.2 mm from the center of the stagnation point in two or more directions. The fixture allows for three blow-off adhesion measurements per sample, and several measurements and samples are frequently averaged together to improve measurement accuracy.

CALIBRATION

There are two areas of toner removal that can be used in adhesion measurements, Region I and Region II, and correlation between the two is important for calibration. The first region is the size of the stagnation point directly under the nozzle, as shown in the inset image in Fig. 1. In this region, the velocity is increasing as a function of radial distance, and the adhesion removal forces on the toner are also increasing radially. The second region is outside the diameter defined by the nozzle size. In this area, the air velocity is decreasing as a function of radial distance, and the removal force on the particles is also decreasing. Additionally, in this area, removed toner rolls and impacts non-removed toner, effectively amplifying the removal force. Calibration in this area requires both an analysis of the air velocity, and compensation for collisions between toner particles. While this calibration is complex, the amplification factor allows for higher resolution. Calibration of the two regions was performed by a combination of determining the free-stream velocity by solving the Navier–Stokes equation, and by modeling the system using a finite-element solver.

The stagnation point solution yields an adhesion force at removal for a given radius of unremoved toner directly under the nozzle. Using an internally developed algorithm,[25] which digitally compares the number of toned pixels to non-toned pixels in the microphotograph, the stagnation spot is analyzed to determine the radius at which the population of toner left on the sample is decreasing at the fastest gradient. Since the removal is Gaussian, this spot is the 50% removal point for a sample of toner.

The removal of toner is done by the drag force F_D from the air pulse. Therefore, we need to know the velocity u, the area A of the toner being hit by the air, and the coefficient of drag C_D of the air as it hits the toner which is a few microns above the surface of the belt. The drag force is

$$F_D = \frac{1}{2} r u^2 C_D A, \tag{2}$$

where C_D is a function of the Reynold's number and is about 0.5 for our system. The solution for the higher-resolution outer removal area has been previously published,[21] and results in the velocity profile as a function of radial distance r and height z:

$$U_r(z) = -\frac{U_0^2 r_0^2}{r^3}\frac{z^2}{2\nu} + \left[U_r - \frac{\left(-\frac{U_0^2 r_0^2}{r^3}\right)\left(\frac{\nu r}{U_r}\right)}{2\nu}\right]\left[\frac{1}{\sqrt{\frac{\nu r}{U_r}}}\right] z. \tag{3}$$

In Eq. (3), $U_r(z)$ is the velocity at a given radius, U_o is the input velocity at the nozzle, r_o is the radial distance at the edge of the nozzle, U_r is the velocity at the edge of the boundary layer, and ν is the kinematic viscosity. The input to the system is considered to be the edge of the nozzle. Conservation of matter and the assumption of a Blasius boundary layer shape are used to determine the boundary conditions. Recent work by Sweeney and Finlay[26] revealed a relationship for the Reynolds number for very small spheres in a Blasius boundary layer. From their work, it is also possible to determine appropriate coefficients of lift and drag from the air velocity on the toner. The removal force due to air velocity will be the sum of the lift and drag forces. The force from the air velocity is augmented in calibration by the force from toner collisions, greatly enhancing the removal force as a function of distance. When added to the toner collision forces, the resulting net force represents the force needed to remove the particle.

Zoeteweij, van der Donck, and Verslusis[28] published the results of a series of experiments for removing small particles with an air flow. They found that rolling was the removal mode for particles in the size range of toner, and this is borne out by the moment calculation. The toner used is chemically processed toner, which is fairly spherical. Determining the actual adhesion force requires understanding the resultant moment arm leverage over the length of the contact area. Since the contact area is much smaller than that of the center of the drag force, the air has a mechanical advantage in removing toner. The contact area found in the scanning electron microscope (SEM) image and that found from Johnson–Kendall–Roberts (JKR) theory[27] were fairly close. This contact area must lift off as the particle rolls, defining a moment arm for rolling initiation at about a tenth of a micron for the average toner particle.

Calibration involves calculating the air drag and lift forces on the toner as a function of the percentage of toner removed. This can be done by looking at the distribution of removal as a function of radial distance from the center of the stagnation point and correlating that with the same removal percentage from thin toner lines outside the nozzle diameter. The assumption made is that the toner adhesion characteristics of the lines and the isopel toner are equivalent. This procedure allows the user to calibrate out the effect of collisions. At the stagnation point, the air velocity is zero, and it increases radially until the radial distance equals the diameter of the nozzle. For this reason, the removal force from the air velocity increases radially from the center of the stagnation point to the edge of the nozzle. From the edge of the nozzle outward, the velocity decreases as the velocity drops as the cube of the distance. Thin lines of toner placed 2 mm apart on the sample instead of a halftone pattern allow for calibration of the force outside the nozzle radius. These lines are the thinnest that the system can produce and are only about 8–10 toner particles wide. These two removal measurements can be correlated by the percentage of toner removed. The system is then calibrated for the actual distance that toner is removed due to the force enhancement of toner collisions. Toner removed rolls outward, striking other toner, and this increases the toner removal force. Particle collision is a statistical event, and is therefore calibrated as such.

Figure 3 shows the relationship between the halftone toner pattern removal and the removal of toner from a line sample. Doing this multiple times for samples with similar removal percentages gives a calibration curve for force at a distance in the halftone area. Since the number of collisions impacts the force multiplication, several calibration curves have been created for low, medium, and high removal at a given air pressure. Calibration is also sensitive to the toner mass in each halftone dot. Opacity measurements were used to insure that this level was kept constant throughout the test.

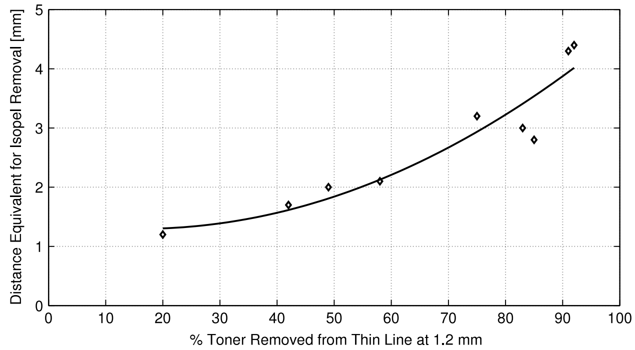

Figure 3. Example of a distance adjustment to correlate the isopel toner removal with thin line toner removal. Multiple samples create a calibration curve for halftone adhesion measurements converting distance values to force at that distance. This is one representative calibration curve used to account for collisions in the isopel samples.

Once calibrated, adhesion measurements are made on uniform halftone dot pattern equivalent to about 20% area coverage. The samples used for measurement are taken from color electrophotographic printer systems stopped in the process of transferring an image from an intermediate transfer belt to paper or from a photoconductor to an intermediate transfer belt. Both transfer belt samples and photoconductor samples have been successfully used in toner adhesion measurement. In order to determine a practical adhesion for microparticles, it is important to measure the adhesion of particles as they exist in real systems, which is in groups or clusters, and at different charge levels.

The toner has been charged and placed in a halftone pattern by a full-color printer imaging mechanism. A particular color toner printed in this manner is primarily a monolayer, but it can stack slightly. It is believed that while tribo charging occurs in the developer, post-nip air breakdown is responsible for the recharging process at the subsequent nonprinting transfer stations normally associated with the transfer of other color toners. The imaging analysis of toner remaining has been found to be 97% accurate for halftones of 30% coverage equivalent or less. Additionally, by controlling the voltages at nonprinting stations, the toner charge can be manipulated, giving a range of charge for equivalently mechanically adhered toner.

The stagnation area diameter can also be used as a simpler metric of toner removal force for a given halftone sample. The diameter is found by mathematically rotating a small control area around the image center, calculating the pixel count as a function of radius, and averaging each step for a full sweep. A numerical algorithm is used to find where the second derivative of the curve is zero, and this correlates to the radius where 50% of the toner has been removed. The drag force at this radius can then be correlated with the force calibration for the 50% removal level for the collision-based measurement. While this method is simpler to use, it results in a measurement that is only for the 50% removal force, and it does not give the distribution of adhesion forces for the

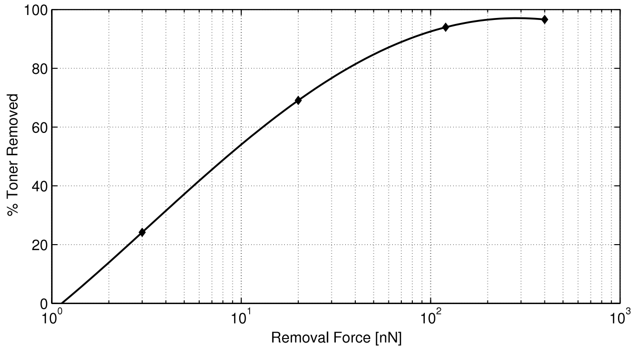

Figure 4. Toner removal as a function of force. The toner is at ambient temperature and humidity (approximately 72°F/50% relative humidity) after being printed onto a polyimide belt sample in a production printer.

entire sample. It has the advantage of being less sensitive to changes in the amount of toner in the sample.

MEASURED ADHESION FORCES

A wide range of adhesion force values has been reported for toner, with average toner requiring anywhere from 40 nN to 8000 nN depending on the external particle silica coverage, particle size, and charge. Silica-covered particle adhesion forces range from 40 nN to 600 nN, and uncoated toner from 100 nN upwards. Measurements taken with the blow-off system also demonstrated a wide range of removal forces, both within a distribution and as parameters such as silica coverage. The toner removal curve in Figure 4 demonstrates the type of output typical from the toner adhesion tool. The toner being tested is from a production printer, and the sample was taken at ambient temperature and humidity. The measured values of adhesion correspond well to the predicted values.

The toner removal curve shown in Fig. 4 is an example of a toner that is working very well in this printer system, and should have a typical to good measured adhesion. The transfer efficiency is roughly 97%, and the data reflects this. Approximately 95% of the sample is removed by a force at or below 350 nN. This particular sample had a mean charge of −27 μC/g, which would give a image force of about 12.5 nN for a small stack of toner, and this graph shows a 50% removal force close to that amount, about 10 nN. The data above suggests that half of the toner had only a minimal mechanical adhesion. SEM images of toners from these samples, shown in Figures 5 and 6, reveal the cause of much of the difference between the easily removed toner and that which is tightly held. As had been anticipated, the average toner in the sample is strongly impacted by silica additive coverage and how well that coverage, or the shape of the toner, minimizes the contact area with the transfer belt. Loosely held toner can be seen sitting upon a few silica nodules or resting on only a tiny portion of the toner radius. These toners exist in high percentages in toner that is removed with little force. The toner that is tightly held can frequently, but not always, be seen to have high mechanical adhesion. This can be caused by lesser quantities of silica

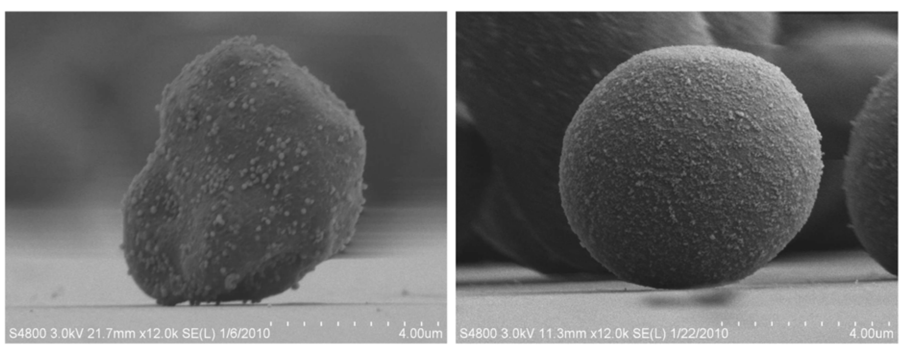

Figure 5. Scanning electron microscope image of weakly held toner particles (i.e. low dispersive adhesion forces).

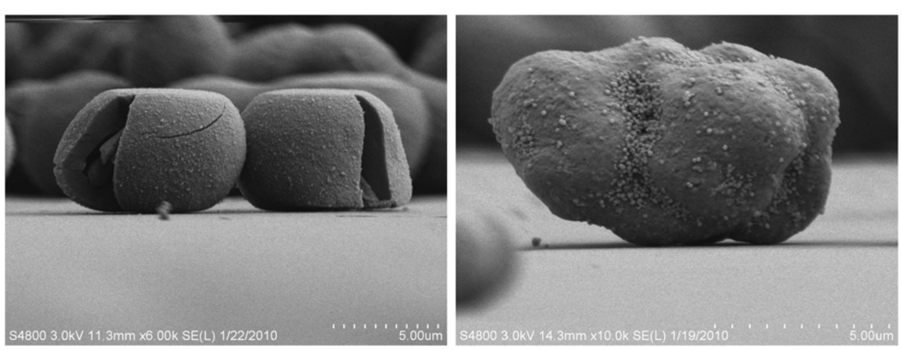

Figure 6. Scanning electron microscope image of strongly held toner particles (i.e. high dispersive adhesion forces).

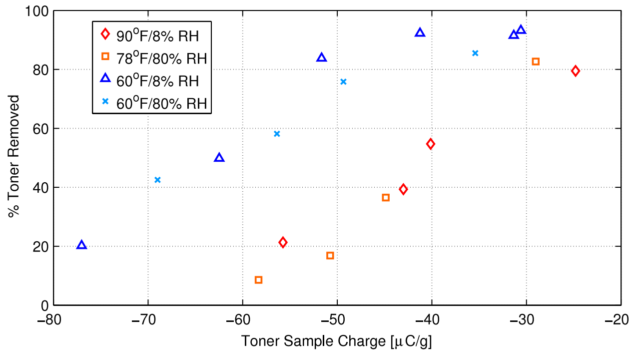

Figure 7. Data from toner adhesion testing at 90°F/8% relative humidity; 78°F/80% relative humidity; 60°F/8% relative humidity and 60°F/80% relative humidity. Data is shown as a function of toner charge for each sample, which was manipulated with transfer voltages.

additives on the particle surface or from damage to the toner that happens during the electrophotographic process.

Adhesion differences as a function of ambient temperature were also noted in sample measurements. Samples created in warmer environments had increased adhesion over samples made with the same materials and charge levels at colder conditions. The graph in Figure 7 shows the difference in adhesion as a function of charge for samples made at four different combinations of temperature and humidity levels. The charge is varied by adjusting the down-stream transfer voltages resulting in ion charging. This method of charging is believed to result in nonuniform particle surface charge.[9] The data naturally groups into high and low temperature components, and there is almost no variation for humidity. It is hypothesized that the increased contact area which arises from small plastic deformation in the warm environments contributes significantly to this adhesion increase.

CONCLUSIONS

We have developed an adhesion measurement technique for toner as processed in a full-color electrophotographic printer. Toner adhesion measurements were taken using an air pulse blow-off tool that delivered a metered pulse of air to a patterned sample of toner on a transfer belt. Calibration of the force was done by calculating the velocity profile, validating it with stagnation area diameter measurements, and correlating it with values from published literature. This tool predicts a correlation of the adhesion to the contact area between particles and the substrate as expected from previous studies. It also indicates a difference in adhesion for particles depending on the ambient temperature. As theoretically predicted, toner adhesion is a strong function of both electrostatic and non-electrostatic forces.

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