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Effects of Nanoparticle Coverage on the Adhesion Properties of Emulsion Aggregation Toner Particles

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Abstract. *Toner is a critical material in printing and copying processes. Compared to the conventional pulverized toner, polymerized emulsion aggregation (EA) toner provides more precise control over the shape and surface roughness of particles. Due to its roundness and low surface roughness, the near-spherical EA toner appears to adhere more strongly to flat substrates, which is desirable for efficient toner transfer and development. However, in the cleaning and transport processes, due to enhanced adhesion, it is difficult to remove the residual EA toner from surfaces of the photoreceptor and transport belt. To reduce the adhesion of the EA toner in a controllable manner, the surfaces of the toner particles are coated with silica nanoparticles with a diameter range of 15–32 nm. In the current study, a technique based on the rolling resistance moment of the particle-substrate adhesion bond is utilized for quantifying the effect of nanoparticle surface coating on the adhesion of individual toner particles. With the aid of a custom-made nanomanipulation system, an increasing lateral pushing force was applied to an individual toner particle adhered to a silicon substrate while the pre-rolling and rolling motions of the particle in response to the lateral pushing force were recorded. The work of adhesion between the toner particle and silicon substrate was extracted from the corresponding lateral force-particle displacement curve. The technique was used to characterize the adhesion properties of both uncoated (bare) and nanoparticle-coated model toner particles with a specified surface area coverage of 50%. It is found that the work of adhesion values between the surface-coated experimental toner particles and the silicon substrate are almost an order of magnitude lower than those for the bare ones. © 2010 Society for Imaging Science and Technology.*
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INTRODUCTION

In printing/copying applications, the adhesion bond of toner particles to the surfaces of photoreceptor, transfer belt, and paper plays a key role in the overall performance of printers and copiers. The adhesion property of toner particles is thus a critical factor for controlling the quality of resulting images. In the printing/copying process, toner particles are transferred from a cartridge to the photoreceptor drum, then from there to the transfer belt, and finally to the surface of paper. For conventional pulverized toner, the transfer efficiency is reported to be in the range of 85%–90%.[1] The residual toner particles are left on the photoreceptor surface and have to be mechanically removed. In the past decade or so, new generation chemical toners such as the polymerized emulsion aggregation (EA) toner have become available.[2] Such toners are energy efficient to produce and provide tighter particle size and shape distributions. Consequently, they have the potential to form sharper images with better resolution on the photoreceptor than with the pulverized toner. However, the near-spherical EA toner is more difficult to clean because such toner particles appear to adhere more strongly to the photoreceptor surface than the irregular-shaped (jagged) pulverized toner particles with the same effective diameters. To reduce adhesion in a controllable manner and improve the toner flowability and transfer efficiency, the near-spherical EA toner particles are surface-coated with an external additive such as silica nanoparticles, which can significantly reduce the particle-substrate adhesion. For conventional pulverized toner, the influence of additive coating on its adhesion has been studied by a number of researchers with colloid probe, centrifuge, and electric field detachment techniques.[3–5] However, prior to the reported investigation, the adhesion property of the EA toner and the effect of nanoparticle surface coating on the adhesion of the EA toner have not been systematically characterized at an individual particle level.

The particle-substrate adhesion has been theoretically investigated in the past few decades, and several contact mechanics models such as the Johnson, Kendall and Robert (JKR) model,[6] Derjaguin, Muller and Toporov (DMT) model,[7] and Maugis and Dugdale (MD) model[8] have been proposed. Experimentally, several particle adhesion characterization techniques have been used to study the toner particle adhesion, including the colloid probe technique, electric field detachment method, and centrifugal detachment method.[1,9–12] Among these techniques, the electric field detachment and centrifugal detachment methods can provide only the (bulk) adhesion properties for a large group of toner particles. While with the colloid probe technique with an atomic force microscope (AFM) the adhesion property of an individual toner particle can be studied, it is quite laborious to attach a particle to the probe and it is also a destructive method.

In current study, we present a method for adhesion characterization of an individual toner particle using a recently developed rolling resistance moment-based technique[13] involving a custom-made nanomanipulation setup under an inverted optical microscope. The rolling re-

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sistance moment acting on a particle to obtain detachment has been traditionally used as a criterion for predicting the initiation of rolling-based detachment of the particle. This criterion assumes no resisting moment to rolling at the adhesion bond prior to rolling. In 1995, Dominik and Tielens analytically showed that the adhesion bond between a microparticle and a flat surface indeed generates a resistance against the rolling initiation of the particle prior to free rolling.[14] Recently, Cetinkaya and co-workers experimentally confirmed the existence of the rolling resistance moment of an adhered polystyrene latex (PSL) microsphere using a noncontact acoustic excitation technique.[15,16] Previously, this technique was adopted to investigate the adhesion between the PSL microspheres and a silicon substrate in the vacuum chamber of a scanning electron microscope (SEM).[13] In the current study, the technique is employed to characterize the toner particle adhesion in the ambient under an inverted optical microscope and to investigate the effect of nanoparticle coating of the toner particles on the strength of particle adhesion. While the spatial resolution of the optical microscope is inferior to that of the SEM, performing measurements in the ambient can avoid the complications and possible inaccuracies associated with the charging of the nonconductive toner particles by the SEM electron beam. Moreover, working in the ambient provides the flexibility to explore the effects of certain realistic operational parameters such as the temperature and humidity on particle-substrate adhesion, which is also important for toner adhesion characterization but difficult or impossible to achieve in high vacuum.

The surface roughness of the particle and the substrate has a significant effect on the particle-substrate adhesion. In most contact mechanics-based adhesion models, both the particle and the substrate are assumed to have perfectly smooth surfaces. For a particle with a rough surface settled on a flat substrate, the contact area between the particle and the substrate is much smaller than that between a smooth particle of same diameter and the substrate, which results in a significantly lower particle-substrate bond strength. Because of its practical importance, the effect of surface roughness on particle-substrate adhesion has attracted considerable research attention in recent decades.[1,3,5,12,17–19] Significant reduction in adhesion force has been observed with the increase of the surface roughness of the particle and/or the substrate.

EXPERIMENTAL

Materials and Experimental Setup

The schematic of the rolling resistance moment-based lateral pushing experimental setup is depicted in Figure 1. When a lateral pushing force is applied to an adhered particle, the stress distribution in the particle-substrate contact area becomes nonuniform. Such nonuniform stress distribution creates a restoring moment (also referred to as resistance moment) to resist the free rolling motion, and this moment is proportional to the angle of rotation of the particle.[14] When the applied lateral force exceeds a certain threshold, the rolling resistance moment is unable to resist the free rolling motion and the particle begins rolling on the substrate surface. The accurate prediction at this rolling threshold is still an open problem.

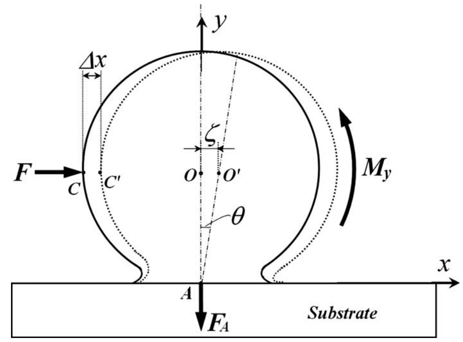


Figure 1. An adhered particle subjected to a lateral applied force (F) generating a resisting moment (M_y) to the free rolling at a lateral translation of (ξ) (contact area is not to scale).

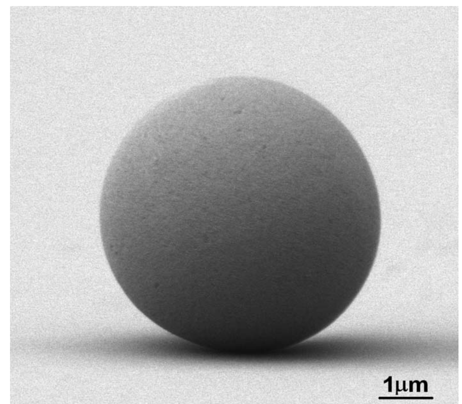
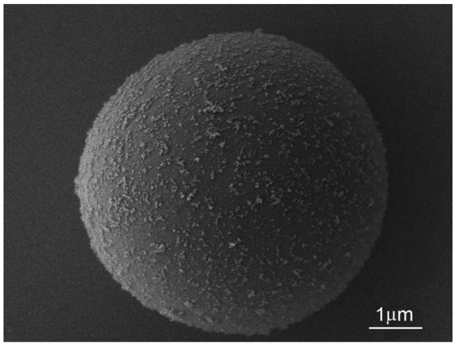


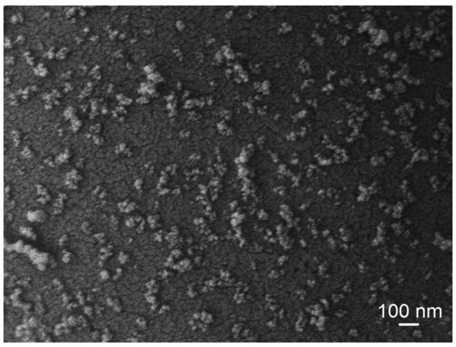
Figure 2. SEM image of a bare polymer particle on a silicon substrate with a 10 nm gold layer coating for charge dissipation during SEM imaging (at 10,000× magnification).

In the current work, three types of model toner particles are considered, including uncoated (bare) polymer particles with nominal diameters of 6.0 and 9.0 μm and surface-coated polymer particle with a nominal diameter of 6.0 μm and a 50% nominal surface area coverage (SAC) of silica nanoparticles. The outer layer of the bare polymer particle consists of a polyester resin, cyan pigment, and wax. The average diameter of the silica nanoparticles used in this study is approximately 24 nm, but the nanoparticles occasionally can form aggregates as large as 100 nm in effective diameter. All the model toner particles are prepared by the EA process and, subsequently, surface-coated with silica nanoparticles using a toner blender at the Xerox Research Center (Webster, New York).[2] SEM observations reveal that the bare polymer particles have fairly smooth surfaces at the nanometer scale and are nearly spherical (Figure 2), while, as expected, the surfaces of the nanoparticle-coated polymer particles are bumpy (Figure 3).

For the particle-substrate adhesion study, the toner particles are used as-received with no additional aging and/or chemical treatment. They are dry-deposited on a plasma

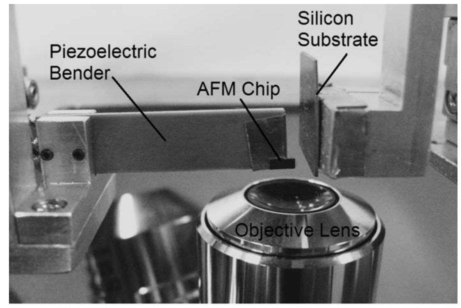


(a)

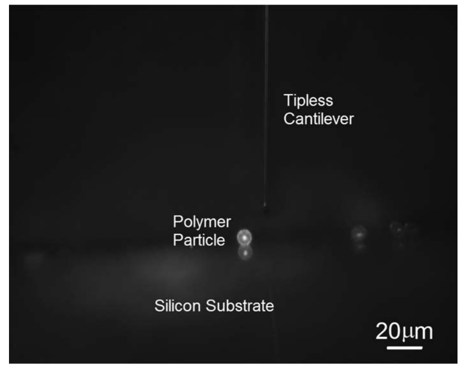


(b)

Figure 3. SEM images of a polymer particle coated with silica nanoparticle (50% SAC) and a 10 nm gold layer coating for charge dissipation during SEM scans: (a) the top view at 14,000× magnification; and (b) the close-up of the particle surface at 50,000× magnification.



(a)



(b)

Figure 4. (a) A photograph of the optical manipulation setup; (b) an optical image of lateral pushing test experimental setup; (c) an optical image of the pushing of an adhered polymer particle with a tipless cantilever probe.

cleaned [*p*-type doped (100) oriented wafer] single-crystal silicon substrate immediately before the experiments. The experimental setup is composed of two opposing *xyz* linear positioning stages (122–1135/1155, OptoSigma Inc., Santa Ana, CA) mounted on top of an inverted optical microscope (Epiphot 200, Nikon, Japan). These positioning stages are driven by six piezoelectric actuators (MRA 8351, New Focus, Inc., San Jose, CA) that provide linear motion with a displacement resolution of approximately 30 nm. A piezoelectric bender (CMBP 05, Noliac A/S, Denmark) that provides fine positioning at a sub-nanometer resolution is mounted on one of the axes of the *xyz*-linear stage. The positioning and particle pushing processes are monitored through a 100× objective lens using a high-resolution 10 megapixel digital camera (DXM 1200, Nikon, Japan) attached to the optical microscope. For pushing experiments, the base chip of a tipless AFM cantilever (CSC 12, length 350 μm, nominal force constant 0.03 N/m, MikroMasch, Inc., Wilsonville, OR) is attached to the free-end of the piezoelectric bender, and the silicon substrate with particles deposited is mounted on the opposing linear positioning stage [Figures 4(a) and 4(b)].

In the lateral pushing experiments reported here, a dc voltage increased in discrete steps was applied to the piezoelectric bender to displace its free end. The tipless AFM cantilever beam attached to the free end of the bender was actuated to push the particle adhered to the substrate. During the test, the pushing force was increased in discrete steps (5–15 nN/step) due to the deflection of the cantilever beam with a time interval of approximately 30 s (for image recording and voltage increase). By acquiring a series of digital images, the entire pushing process was recorded for each particle tested. The AFM cantilever served as the force sensing element, and the applied force (F) was calculated from the relative cantilever deflection with respect to the cantilever base using the linear bending stiffness of the cantilever beam. The relative deflection of the AFM cantilever beam at each pushing step was obtained with a piezoelectric bender response calibration procedure;[20,21] and the stiffness constant of the AFM cantilever beam was calibrated in the ambient prior to the test with a resonance method developed by Sader et al.[22] The displacement of the particle in the x direction (Δx) was obtained from the processing of recorded images by tracking the pixel positions of the left edge of the particle in each recorded digital image with an image analysis software, IMAGEJ.[23] The digital images taken by the 10 megapixel digital camera under the 100× objective lens were 3840×3072 pixels in size, and the corresponding pixel resolution is ~35 nm/pixel. Because of the small depth of focus of the optical microscope, it is difficult to precisely position the polymer particle along the central axis of the AFM cantilever probe. However, the error induced by possible "off-center" load on the cantilever deflection should be negligible because the AFM cantilever beam has much higher torsional stiffness relative to its bending stiffness.

THEORY

With the pushing force and particle displacement information at each pushing step, a lateral force-displacement (F-Δx) curve can be constructed for each particle tested. The work of adhesion between the particle and substrate can then be extracted from the lateral pushing force-displacement curves.[13] For the lateral pushing test experiment, assuming no sliding, the slope of the force-displacement curve (k) can be approximated in a displacement range corresponding to the pre-rolling phase of motion as

$$k = \frac{F}{\Delta x} = \frac{M/(D/2)}{\theta(D/2)} = \frac{4M}{\theta D^2}, \tag{1}$$

where M is the moment generated by the pushing force with respect to the particle-substrate contact area, θ is the angle of rotation of the particle with respect to the center of particle-substrate bond, and D is the diameter of the spherical particle. According to Dominik and Tielens,[14] the rolling resistance moment as a function of the angle of rotation θ can be approximated as

$$M \approx 6\pi W_A (D/2)^2 \theta, \tag{2}$$

where W_A is the work of adhesion between the particle and substrate. Therefore, from Eqs. (1) and (2), the work of adhesion is directly proportional to slope of the force-displacement curve, i.e.,

$$W_A = \frac{k}{6\pi}. \tag{3}$$

It is noteworthy that, with this approach, no knowledge of the particle diameter is required for extracting the work of adhesion between the particle and substrate.

RESULTS AND DISCUSSION

The experimental procedures detailed above were applied to the three types of polymer particles of different dimensions and surface roughness conditions. The force-displacement relationships of uncoated (bare) polymer particles with the nominal diameters of 9.0 and 6.0 μm are presented in Figures 5(a) and 6(a). The force-displacement curves of coated polymer particles with 50% nominal SAC are presented in Figure 7(a). While the displacements of the particles kept increasing with increasing pushing force, the slopes of the force-displacement curves decrease significantly after the first few pushing steps. This behavior is consistent with previously reported observations[13] in the SEM experiments. Based on these data, we believe that at the first few loading steps there exists a resistance against the rolling initiation of the particle, and the initial particle displacement is due to the pre-rolling motion of the particle. The decreases in slopes following the first few pushing steps indicate that the adhesion bond between the particle and the substrate is broken and the adhesion bond cannot resist free rolling any longer. The particle begins free rolling on the substrate.

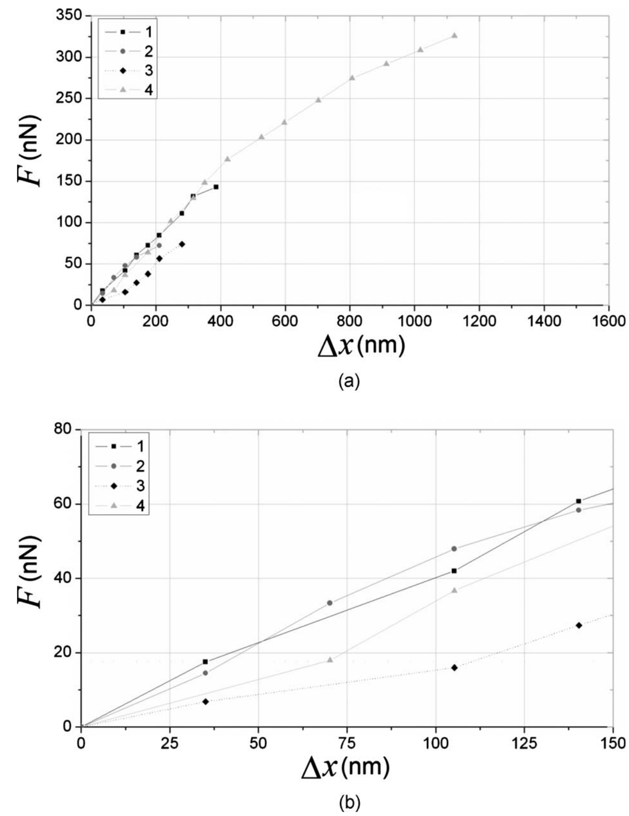


Figure 5. (a) The lateral force-displacement curves of four uncoated (bare) polymer particles with a nominal diameter of 9.0 μm under the lateral pushing force F, and (b) the close-up of the initial portion of the curves.

The characteristic results for the three types of experimental polymer toner particles are summarized in Table I. The work of adhesion between the polymer particle and silicon substrate is calculated from the pre-rolling slope of each force-displacement curve [Figs. 5(b), 6(b), and 7(b)]. As reported in Table I, the work of adhesion between uncoated polymer particles and silicon are in the range of 5.3–34 and 9.0–37 mJ/m^2 for the 9.0 and 6.0 μm particles, respectively. The average work of adhesion values for these uncoated polymer particles are around 20 and 23 mJ/m^2, respectively. A two-tailed Student's T-test with a confidence level of 95% shows that there is no significant difference between the work of adhesion for the 9.0 and 6.0 μm particles. Based on the JKR adhesion model, the corresponding average pull-off forces for the 9.0 and 6.0 μm particles are around 420 and 330 nN, respectively. While there is no other adhesion measurement data for these specific toner particles in literature, the measured work of adhesion values are close to those of similar toner particles,[24] and the estimated pull-off forces are comparable to the pull-off forces measured with AFM for other types of toner particles.[3,4]

For the coated polymer particles with 50% SAC, the work of adhesion between the particle and silicon substrate is in the range of 1.2–12 mJ/m^2, with an average value of approximately 4.0 mJ/m^2, nearly an order of magnitude lower than that for the uncoated ones. A one-tailed Student's

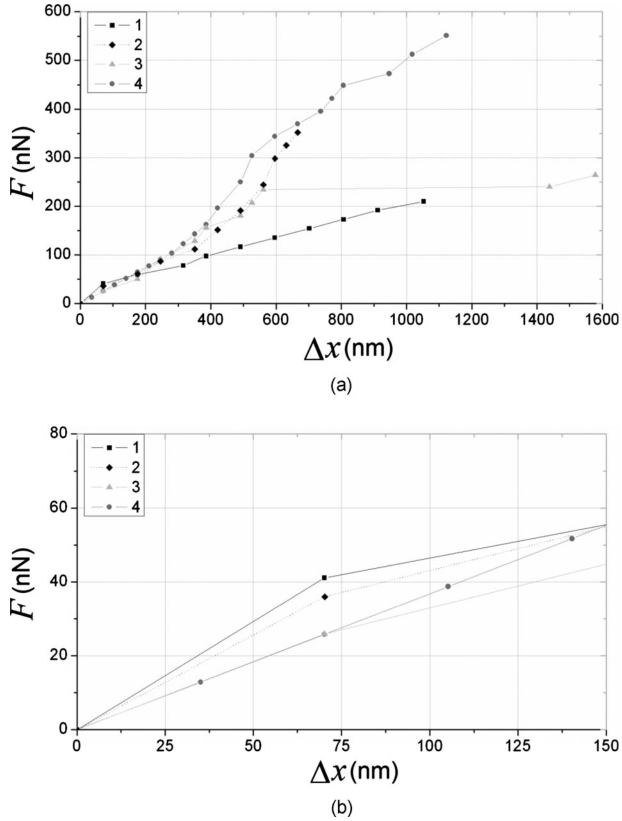


Figure 6. (a) The lateral force-displacement curves of four uncoated (bare) polymer particles with a nominal diameter of 6.0 μm under the lateral pushing force F, and (b) the close-up of the initial portion of the curves.

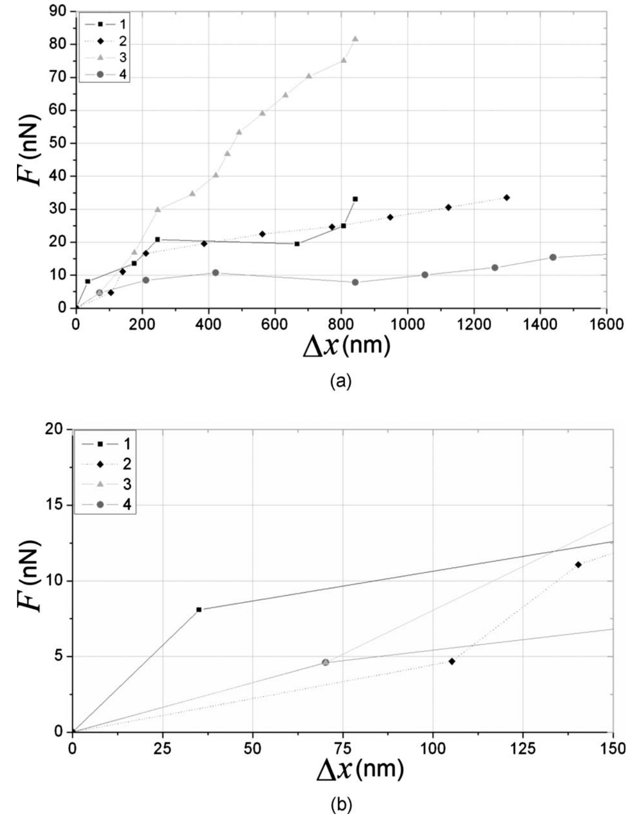


Figure 7. (a) The force-displacement curves of four surface-coated polymer particles (50% SAC) with a nominal diameter of 6.0 μm under the lateral pushing force F, and (b) the close-up of the initial portions of the curves.

T-test with a confidence level of 95% shows that the work of adhesion of silica-coated polymer particle to silicon is significantly greater than zero. However, note that the reported work of adhesion between the surface-coated polymer particle and silicon substrate is calculated with the smooth particle adhesion model and thus is an "effective" value for the work of adhesion. Because of the silica nanoparticle coating, the actual particle-substrate adhesion is dominated by the silica nanoparticle-substrate interaction rather than the direct polymer particle-substrate contact. These "effective" work of adhesion data provide a means to directly compare the adhesion performance of a toner particle with and without surface coating, and such measurement is valuable for the toner design in order to optimize its adhesion and printing performance. Accurate modeling of the actual work of adhesion between the nanoparticle and substrate is also important, but is outside the scope of the current study.

CONCLUSIONS

The effect of the surface roughness due to silica nanoparticle coating on the adhesion properties of near-spherical model EA toner is investigated with a recently developed rolling resistance, moment-based adhesion characterization technique in the ambient environment. A lateral pushing force was applied to an adhered polymer particle with a custom-made nanomanipulation system under an inverted optical microscope. The response of the particle displacement to the lateral pushing force was obtained, and the work of adhesion between the particle and silicon substrate was deduced from the slope of the pre-rolling part of the force-displacement curve. Three types of toner particles with different sizes and surface roughness conditions were studied, and the corresponding particle-substrate adhesion properties were determined. The experiments on silica-coated EA particles with 50% SAC revealed that silica nanoparticle coating on the polymer particle surface can significantly reduce the adhesion between the polymer particle and silicon substrate, which provides a means for optimizing the toner performance by controlling its surface roughness. The adhesion reduction provides a technique for optimizing the near-spherical EA toner design to achieve higher transfer efficiency and better removability. Utilizing this knowledge of toner adhesion property, we can improve the quality of resulting images by arbitrarily modifying the surface roughness of the toner particles. Compared with other adhesion characterization techniques, the current method has several advantages: (i) it measures the adhesion properties of individual particles rather than average properties of a group of particles; (ii) it requires no attachment of a particle to the tip of a probe and is thus nondestructive for the particles; and (iii) no particle diameter data is required for determining the work of adhesion between the particle and the substrate.

Table I. Summary of the experimental results of the work of adhesion measurements of three set of toner particles on silicon substrate.

Particle type	Particle No.	Particle diameter (μm)	Average particle diameter (μm)	Pre-rolling stiffness (N/m)	Average pre-rolling stiffness (N/m)	Work of adhesion ($\mathbf{mJ/m^2}$)	Average work of adhesion ($\mathbf{mJ/m^2}$)
Uncoated	1	8.0	9.1 ± 1.1	0.500	0.37 ± 0.19	26.5	19.7 ± 10.4
	2	10.5		0.413		21.9	
	3	10.5		0.195		10.4	
	4	8.9		0.255		13.5	
	5	9.5		0.097		5.1	
	6	9.5		0.162		8.6	
	7	9.1		0.642		34.1	
	8	8.1		0.535		28.4	
	9	7.5		0.535		28.4	
Uncoated	1	6.7	6.0 ± 0.4	0.586	0.429 ± 0.171	31.1	22.8 ± 9.1
	2	5.8		0.512		27.2	
	3	6.0		0.368		19.5	
	4	6.4		0.368		19.5	
	5	5.7		0.172		9.1	
	6	5.4		0.702		37.3	
	7	6.2		0.269		14.3	
	8	5.7		0.455		24.2	
Coated (50% SAC)	1	6.1	6.0 ± 0.3	0.230	0.075 ± 0.070	12.2	4.0 ± 3.7
	2	6.1		0.044		2.3	
	3	5.6		0.066		3.5	
	4	6.0		0.066		3.5	
	5	6.3		0.022		1.2	
	6	5.7		0.024		1.3	
	7	6.2		0.027		1.4	
	8	5.6		0.117		6.2	

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