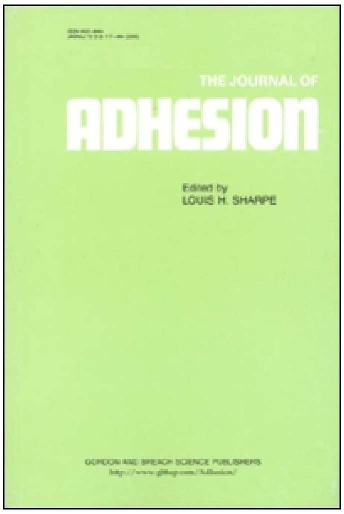

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Adhesion-Induced Interactions between Micron-Sized Zirconia or Carbon Spheres and Melamine-Cross-Linked Polyester Surfaces

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Adhesion-Induced Interactions between Micron-Sized Zirconia or Carbon Spheres and Melamine-Cross-Linked Polyester Surfaces

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The thermodynamic work of adhesion between micron-sized zirconia or carbon spheres and polyester–melamine surfaces was determined using the Johnson-Kendall-Roberts (JKR) relationship, after first validating the JKR approach for this system. The calculated works of adhesion for both zirconia and carbon were similar for any given substrate composition and were found to be approximately $35\,mJ/m^2$ *at low melamine concentrations, close to theoretical predictions. The apparent work of adhesion decreased with increasing melamine concentration, most likely due to the presence of a glassy melamine-rich surface layer, which is not representative of the bulk. The value found for low melamine concentration was assumed to be a true value for cocondensed polyester–melamine, and this was used to estimate surface modulus and the amount of excess melamine in the surface as a function of bulk composition.*

Keywords: Cross-linked polyesters; JKR; Particle adhesion; Sphere–surface interactions; Surface structure

INTRODUCTION

Adhesion of small particles to polymeric substrates is of both practical and fundamental interest. The concepts and understanding developed by the study of the fundamentals of small-particle interactions with polymers is important in such fields as xerography, where changes

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in particle adhesion are critical, and in the production of semiconductor devices, where resistance to particulate contamination is important. Also, the development of nonstick cooking utensils requires an understanding of the interaction between polymer surfaces and carbonaceous contaminants (*e.g.*, food).

Particles are attracted to substrates through certain types of interactions that create stresses between the materials. These stresses, in turn, create varying strains that may be elastic or plastic. Measurement of the interactions, and the mechanical response of the materials to these interactions, is necessary to understand the adhesion of particles on substrates.

Derjaguin and Bradley independently proposed the concept of adhesion-induced deformations between particles and substrates in the 1930s. Derjaguin [1] assumed that for a rigid particle in contact with an elastic substrate, the adhesion-induced contact radius a could be calculated from the Hertzian indentor model [Eq. (1)] with loading force F^0, particle radius R, Young's modulus E, and Poisson's ratio ν:

$$a^3 = \frac{3}{4}F^0\left[\frac{1-\nu^2}{E}\right]R. \tag{1}$$

Derjaguin's model, which took into account only elastic compression between the particle and the substrate, failed in some situations, and led Johnson, Kendall, and Roberts in 1971 to publish a refined model (referred to as JKR) [2], which is still used widely today. The JKR theory established that both tensile and compressive interactions contribute to the contact radius of the particle and the size of deformation.

The fundamental assumptions of the JKR theory are that the deformations are elastic, the contact radius is small compared with the particle radius, and all interactions are localized to within the contact region. The JKR equations [2] relate the contact radius a to the particle radius R, the thermodynamic work of adhesion W_A, any externally applied load P, Young's modulus E, and Poisson ratio ν, as shown in Eq. (2) for a rigid particle in contact with a substrate having a finite modulus:

$$a^3 = \frac{R}{K}\left\{P + 3W_A\pi R + \left[6W_A\pi RP + (3W_A\pi R)^2\right]^{1/2}\right\}, \tag{2}$$

where

$$K = \frac{4E}{3(1-\nu^2)}. \tag{3}$$

In the absence of an applied load, Eq. (2) reduces to Eq. (4):

$$a = \left(\frac{6W_A\pi}{K}\right)^{1/3} R^{2/3}. \tag{4}$$

The JKR model has been found to be most suitable for compliant, high-surface-energy materials. The equations predict that, in the absence of an applied load, the contact radius a varies with the 2/3 power of the particle radius R. DeMejo *et al.* [3] have validated this relationship for 10- to 100-μm-radius glass particles interacting with a soft polyurethane surface. Rimai *et al.* [4,5] further showed for the glass–polyurethane system that, for particles having a radius smaller than 5 μm, the 2/3 power relationship breaks down in favour of a 3/4 power relationship. However, if the mechanical properties of the materials are known for particles in the appropriate size range, and the deformation is elastic, the thermodynamic work of adhesion can be calculated using the JKR equation. For glass spheres of the appropriate size interacting with a compliant polyurethane surface, Rimai *et al.* [4] found the work of adhesion to be between 120 and 210 $\mathrm{mJ/m^2}$, in good agreement with DeMejo *et al.* [3], who calculated W_A to be 170 $\mathrm{mJ/m^2}$ for glass particles interacting with a polyurethane of different composition. More recently, however, Rimai *et al.* [5] found W_A values to be 47 $\mathrm{mJ/m^2}$ for 101-μm-radius glass particles interacting with a highly compliant polyurethane substrate.

In this article, we present the results of a study of the interaction between zirconia or carbon spheres and cross-linked polyester substrates of varying composition. Specifically, the study aims to determine the validity of the JKR approach for spherical particles interacting with cross-linked polyesters and to calculate the work of adhesion between spherical particles and polyesters as a function of polyester composition using the JKR treatment. A model is proposed that accounts for the work of adhesion values.

EXPERIMENTAL

Preparation of Zirconia Particles

Spherical zirconia particles with minimal surface structure were produced by spray pyrolysis of zirconium(IV) acetate, using a method adapted from Hook *et al.* [6] and Toikka *et al.* [7], as follows:

A solution of zirconium(IV) acetate in dilute acetic acid (15% w/w Zr, used as supplied by the Aldrich Chemical Company Inc., Milwaukee, WI, USA) was nebulised into a laboratory oven set at approximately 120°C using a small commercial perfume spray atomiser.

The mist produced by the perfume atomiser descended sufficiently slowly through the oven to allow surface tension to pull the particles into spherical shape as water and acetic acid were evaporated. The resulting white powder, solid zirconium(IV) acetate particles, was collected from the bottom of the oven [6]. The powder was placed in a Battersea fireclay crucible and slowly heated at 2 °C/min to 450°C in a muffle furnace over a 4-h period to evaporate any remaining acetic acid and water, and then the furnace temperature was rapidly increased to 800°C and held at this temperature for 4 h to promote the formation of tetragonal crystals of zirconium(IV) oxide [7].

The resultant zirconia was characterised by scanning electron microscopy (SEM) and X-ray diffraction (XRD).

Carbon Particles

Spherical glassy carbon particles, 10–40 μm in diameter, were obtained from Aldrich and used without further treatment.

Preparation of Polyester Surfaces

The hydroxy-functional polyester used throughout this investigation consisted of a 6:4 molar ratio mixture of isophthalic acid and adipic acid, copolymerised with a 9:1 mixture of neopentyl glycol and trimethylol propane. The ratio of acid to hydroxy functionality was adjusted to produce a hydroxy-functional polyester of average molecular weight (M_W) of 2500 and functionality of $\sim$3 hydroxy groups per molecule. The polyester was dissolved in ethyl ethoxypropionate at a concentration of 60 wt%.

Cross-linked coatings were formulated by varying the composition from 95:5 to 50:50 polyester/hexamethoxymethylmelamine (HMMM, Cyanamid Cymel 303, Cytek, W. Paterson, NJ, USA) cross-linker, on a mass percentage basis. Blocked acid catalyst, isopropylamine dodecyl benzene sulfonic acid (Cyanamid Cycat 600), was added at 2 wt% with respect to HMMM, and a small amount (0.2 wt%) of a polyether silicone surfactant (Byk Chemie BYK 306, BYK Chemie, Wesel, Germany) was added to improve film-forming ability. The viscosity of the liquid formulation was then adjusted by the addition of a hydrocarbon solvent (Exxon Mobil Chem., Houstou, TX, USA) Solvesso® 150 prior to coating.

The formulations were coated onto aluminium panels using a no.32 stainless steel drawdown bar, then baked in a ventilated air oven at 300°C until the metal substrate reached a temperature of 245°C (referred to as the peak metal temperature, PMT), quenched in water, and air dried. For DMA measurements, the samples were coated onto silicone-release-coated aluminium panels for ease of removal from the metal substrate and cured in the same way. The nominal dry film thickness for all formulations was 20 μm.

SEM Sample Preparation

Samples of cross-linked polyesters of 1 cm^2 on aluminium were sprinkled with either zirconia or carbon spheres and placed in an air oven at 50°C for 7 days, sputter-coated with gold, and stored in a desiccator until required for SEM analysis.

Analytical Techniques

The creep moduli of the cross-linked polyesters were obtained using a TA Instruments Q800 Dynamic Mechanical Analyser (TA Instruments, New Castle, DE, USA) fitted with a thin film tension clamp. In a typical experiment, a sample of polyester film 30 mm long and 5 mm wide was removed from the silicone-release-coated aluminium substrate, placed in the clamp, and heated to 80°C for 2 h to remove any existing thermal stress and residual solvent, before being equilibrated at 50°C, all with minimum load applied. After equilibration, a load of 30 mN was applied for 1 h, and the resultant deformation recorded. The load was then removed, and the deformation of the sample monitored for a further 30 min to ensure complete recovery of the original dimensions. Data from any samples that did not fully recover after removal of the load were discarded. From the applied creep stress and the resultant creep strain, the creep modulus for 1 h at 50°C was calculated.

SEM was conducted using a Leica Stereoscan 440 scanning electron microscope (Leica Cambridge Ltd., Cambridge, England). Images were obtained by observing particles tangential to the substrate surface. That is, the sample in the SEM chamber was almost vertical. In this way both particle radii and contact patch radii could be measured with ease.

XRD analysis of the zirconia was carried out using a Philips PW 1730 X-ray generator (Philips, Eindhoven, Holland).

RESULTS AND DISCUSSION

Structure and Morphology of Zirconia Spheres

SEM examination of a sintered zirconia sample showed the presence of some spherical particles, although a large amount of irregularly shaped material was also present. The sample was therefore subsequently sieved through a 64-μm sieve. Optical microscopic and

SEM examination of the resultant material showed mostly spherical particles with a minimum of surface asperities (as reported previously by Hook *et al.* [6]), in the diameter range 10–60 µm.

XRD analysis (Figure 1) showed that the sieved sample consisted of approximately 70% tetragonal and 30% monoclinic zirconia, with a high degree of purity and crystallinity.

SEM Observation of Carbon Spheres

Although the quoted nominal diameter range of the carbon spheres as received was 10–40 µm, in practice the particles were found by SEM to be in the range 12–30 µm.

Spherical Particles on Surfaces

Typical SEM micrographs of zirconia and carbon on polyester surfaces are shown in Figures 2 and 3, respectively. Particles are truly spherical, and the polyester surfaces are essentially featureless. Where a particle was found to be covered with extraneous material (more common for carbon particles), it was not used in the calculations.

Substrate Creep Modulus

As discussed previously, the JKR relationship is appropriate for a purely elastic system, and a substrate modulus value is used in the

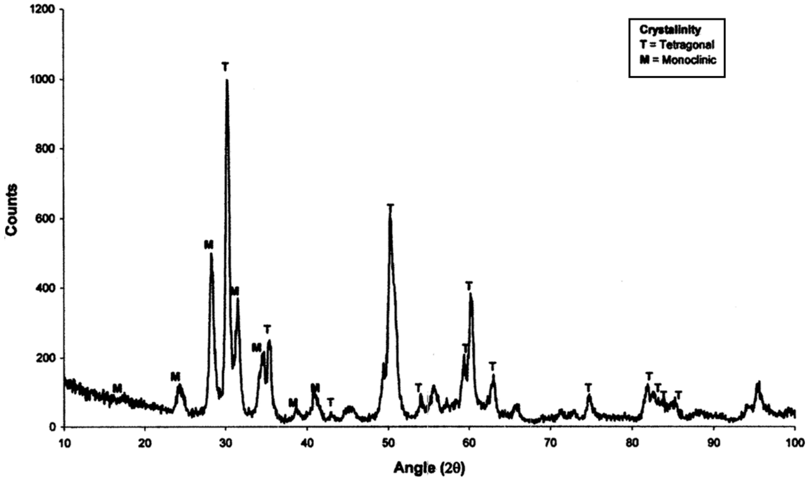

FIGURE 1 XRD analysis of sieved zirconia spheres.

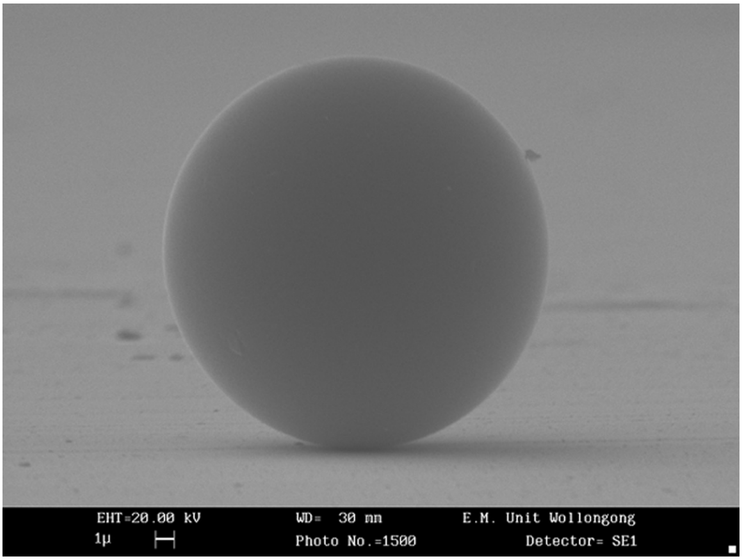

FIGURE 2 SEM image of a zirconia particle on a polyester surface.

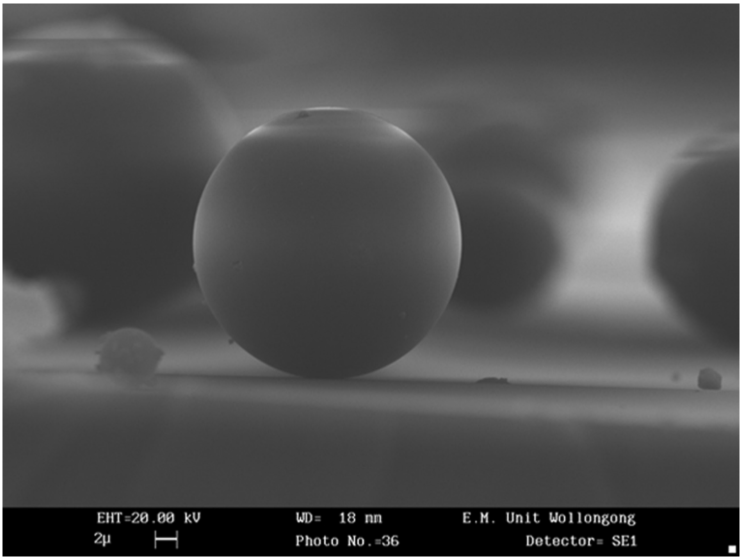

FIGURE 3 SEM image of a carbon particle on a polyester surface.

calculation of the work of adhesion. Rimai *et al.* [4] obtained W_A values for glass particles on polyurethane surfaces, using Young's modulus in the calculation. The system used here is viscoelastic, and so the questions arise whether the JKR relations can be used, and if so, what is the appropriate modulus. Because the contact problem is one of changing boundary conditions, the viscoelastic correspondence principle cannot be used, and so, in principle, the problem is very complicated. Because the contact size only increases in our experiment (rather than increases then decreases), and because the polyester material creeps rapidly for a short time and then its creep modulus becomes fairly constant after about 10 min under load at 50°C, the 1-h creep modulus is used. This approach is a simplified version of the approach used by Falsafi *et al.* [8] applicable to the zero load situation.

Figure 4 shows the creep modulus of the polyesters, observed at 50°C, as a function of HMMM cross-linker concentration. The typical error in the creep modulus measurements (not shown on the figure) was found to be ± 10%. As expected, there is a generally increasing trend in modulus with increasing cross-linker concentration.

Validation of JKR Approach

In the absence of an applied load and assuming a purely elastic response to adhesion-induced stresses, the JKR adhesion theory [2] predicts a 2/3-power relationship between the contact radius a and the particle radius R for an ideal rigid spherical particle interacting

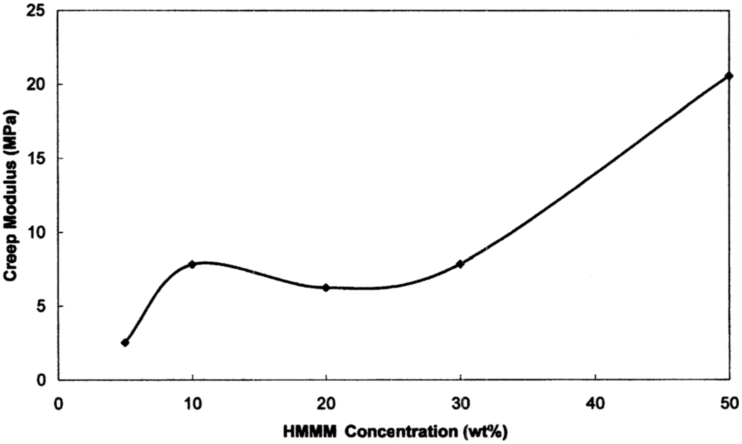

FIGURE 4 One-hour creep modulus as a function of substrate composition.

with a flat deformable surface [9], according to Eq. (4) and described previously. Thus a plot of log a *vs* log R should yield a straight line of slope 2/3.

SEM observation of fractured zirconia spheres revealed that they were hollow, with an approximate wall thickness of 1.6 µm. The force (due to gravity) of a 14.6-µm-radius zirconia particle was calculated to be 2.1×10^{-10} N, which is considered to be negligible; therefore the zero-applied-load assumption is valid.

Figure 5 shows a series of log a *vs.* log R plots for zirconia spheres interacting with polyesters of varying cross-linker (HMMM) concentrations. The least squares line of best fit in all cases was found to have a slope of 0.67 ± 0.02, with correlation coefficients typically between 0.75 and 0.85, demonstrating that for spherical particles in the size range used, the JKR equation [Eq. (4)] is valid.

It was not possible to validate carbon-particle interaction with the polyesters in the same way, because the size range of particles was much narrower than for zirconia. It is fair to assume, however, that the JKR interaction parameters will be similar for carbon and zirconia; therefore the approach is assumed to be valid for carbon also.

Work of Adhesion

The work of adhesion, W_A, for carbon and zirconia particles interacting with cross-linked polyesters, based on calculations using Eq. (4), is shown in Figure 6 as a function of polyester composition (wt% HMMM). It can be clearly seen that within the bounds of experimental

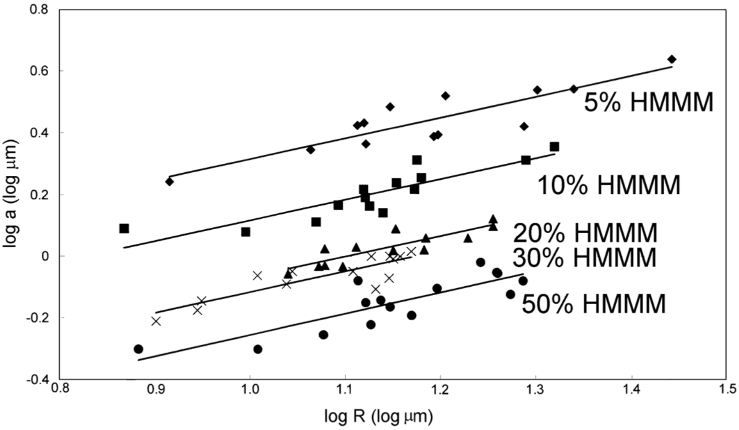

FIGURE 5 Validation of JKR approach.

error, the interaction of both carbon and zirconia particles with the polyester was the same, and W_A decreases with increasing HMMM content.

Note that in the evaluation of W_A, a Poisson's ratio ν of 0.5 was used. Previous studies [10,11] have established that the glass-transition temperatures of these substrates vary between 10 and 30°C, depending on the measurement method used. Therefore, this value of Poisson's ratio is believed to be correct, because at the temperature of the study the substrates would be expected to display elastomeric behaviour.

The work of adhesion is related to the surface energy of the particle γ_1, the polyester surface γ_2, and their combined interfacial energy γ_{12}, according to Eq. (5) [4]:

$$\mathrm{W_A} = \gamma_1 + \gamma_2 - \gamma_{12}. \tag{5}$$

Srividya *et al.* [12,13], in their work on deposition of carbon/fluorine films on stainless steel in an argon plasma, found the surface energy of amorphous carbon films in the absence of fluorine to be between 44 and 52 mJ/m^2, depending on the hydrocarbon used for deposition. The surface energy of zirconia was found by Hao *et al.* [14] to be 52 mJ/m^2. Given the similarity of the reported surface energies for the two particulate materials [12–14], it is no surprise that the work of adhesion for the two systems was the same for each substrate composition.

The surprising feature of the results is that the calculated work of adhesion decreases with increasing cross-linker (HMMM) concentration in the polyester. Rimai *et al.* [4], in their work with glass

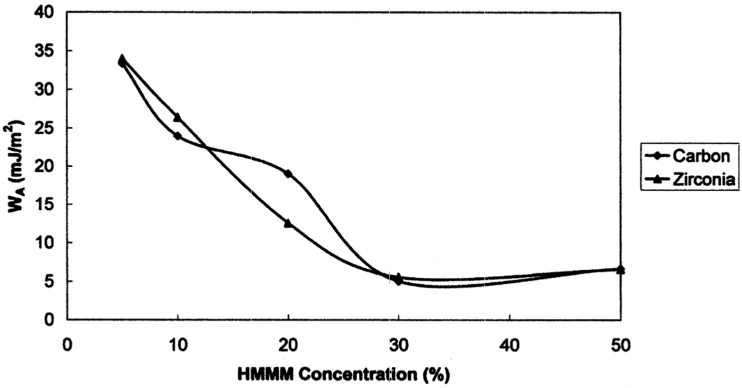

FIGURE 6 Apparent work of adhesion as a function of substrate composition.

spheres on polyurethane surfaces using JKR treatment, found the work of adhesion to be approximately 170 mJ/m^2. Their subsequent value of 47 mJ/m^2 [5] for larger glass spheres is similar to that found in the present study at the lowest cross-linker concentration (5 wt% HMMM, 34 mJ/m^2). As the HMMM concentration increases, the W_A value seems to be excessively low when compared with the results of previous experimental research [3,4,15].

The decrease in W_A cannot be attributed to a change in the equilibrium solid surface energy of the polyester substrate. Lukey *et al.* [10] recently showed that although there was some scatter in the data, the surface energy of the polyester does not show any discernible trend as a function of HMMM concentration.

Gamage *et al.* [11,16] recently described the self-condensation of the HMMM (melamine cross-linker) present in polyester–melamine clearcoats. At low (10 wt% HMMM and less) melamine concentrations, the material composition was found to be relatively uniform; however, at high melamine concentrations, excess melamine cross-linker segregates to the surface of the film [16] and forms a self-condensed phase. The rate of segregation is controlled by the difference in the polyester–melamine surface energies and the ratio of the concentration of bulk and surface melamine [16]. This self-condensed melamine–melamine network has been shown [11] to have a higher *Tg* than the cocondensed polyester–melamine network phase; thus, at higher melamine contents, particles would interact with an increasingly glassy surface.

The main reason for the reduction in the apparent work of adhesion at high melamine concentrations is believed to be connected with the calculation itself. The JKR equation [Eq. (4)] includes a parameter K that is proportional to the modulus. Combining Eqs. (3) and (4) gives Eq. (6), which shows that for given contact and particle radii, W_A is proportional to modulus E:

$$E = \frac{9\pi(1-\nu^2)W_A}{2(a^3/R^2)}. \tag{6}$$

As the melamine content increases, the creep modulus of the bulk material increases, as shown in Figure 1. However, because of the increased concentration of surface melamine, the modulus experienced by the particle would actually be considerably higher than the measured modulus. If the actual surface modulus, which unfortunately cannot be accurately measured, were used in the equation, the calculated W_A would be expected to be considerably higher. Indeed, this change in apparent W_A may be a means of indirectly determining the surface modulus, as follows.

Previous studies [11,16] have suggested that at low HMMM concentrations the polymer is fairly homogeneous and there is little surface segregation of HMMM. Given that the surface energy of the polyester does not change significantly with composition [10], it is fair to assume that the *actual* work of adhesion across the entire composition range may be similar to that found for the 5 wt% HMMM substrate (34 mJ/m^2).

A slightly different interpretation of Eq. (6) suggests that for constant W_A, E is inversely proportional to a^3/R^2. Therefore, using the particle contact information obtained from SEM and assuming W_A to be constant at 34 mJ/m^2, E becomes the modulus experienced by the particle (*i.e.*, the surface modulus). This calculated surface modulus at 50°C is shown in Figure 7 as a function of melamine concentration. The values quoted are the average of the zirconia and carbon results, because these have been previously shown to be equivalent. It can be seen that the calculated surface modulus increases markedly with increasing HMMM concentration.

A second possible reason for the reduction of the apparent work of adhesion is that, as the HMMM content is increased, the surface becomes increasingly glassy, due to the concentration at the surface of the self-condensed HMMM network, as described previously. The lower adhesion comes from reduced real area of contact because the asperities are less deformable.

It is possible to speculate even further and to use this information to estimate the composition of the surface. To do this, a number of

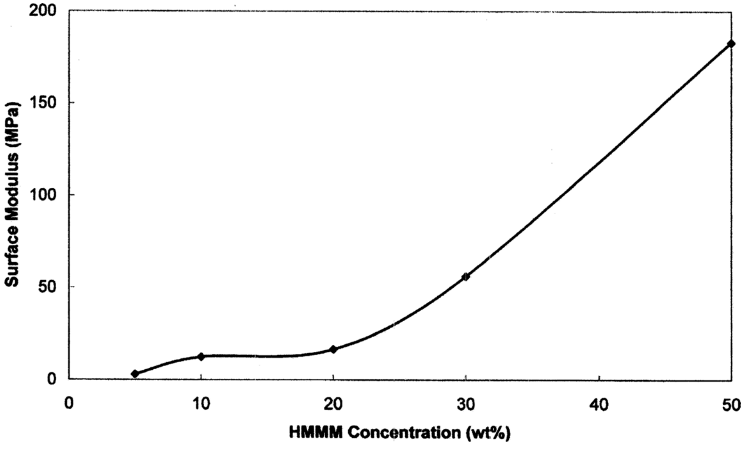

FIGURE 7 Calculated surface modulus at 50°C as a function of substrate composition.

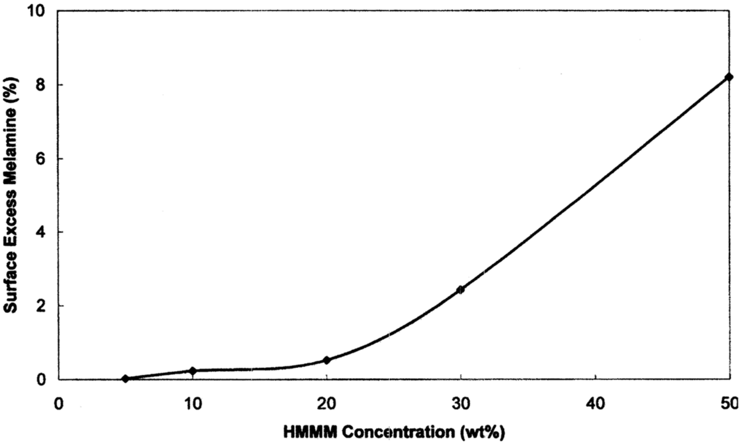

FIGURE 8 Calculated surface excess melamine as a function of substrate composition.

assumptions need to be made. First, we assume that the measured creep modulus at a given composition is that of the cocondensed polyester–melamine matrix (Figure 1). Second, that the self-condensed melamine–melamine phase at the surface is in a glassy state. This latter assumption is not unreasonable, as it has been found that the *Tg* of the self-condensed melamine phase can be as high as 70°C [11]. Third, we assume the modulus of the glassy self-condensed phase to be 2 GPa. Again, this is not unreasonable, because the glassy modulus of a cross-linked polymer is typically between 1 and 3 GPa. By treating the surface as a composite material comprising two phases of different moduli as a first approximation, we can apply the simple rule of mixtures to calculate the volume fraction of self-condensed surface excess melamine, as in Eq. (7):

$$E_s = E_m A_m + E_b(1 - A_m), \tag{7}$$

where E_s = calculated modulus of the surface (Figure 7), E_m = modulus of the self-condensed melamine phase (estimated at 2 GPa), E_b = measured modulus of bulk material (Figure 1), and A_m = volume fraction of self-condensed melamine phase.

The surface excess melamine, calculated on this basis, is shown in Figure 8 as a function of bulk composition. It can be seen that at bulk compositions of more than about 20 wt% HMMM there is a major increase in the melamine concentration at the surface. This is consistent with previously reported data [10,16], which showed by XPS that the nitrogen content of the surface (which can only arise from melamine) was considerably greater at high melamine concentrations than that which would be predicted from the bulk composition assuming homogeneous distribution of components. In an earlier work [10], the C–N/C=O ratio (a measure of the surface melamine content) was 0.72 for 20% HMMM, consistent with a homogeneous distribution of melamine, whereas at 50% HMMM the ratio was 3.42, compared with 1.81 assuming a homogeneous distribution, indicating considerable surface segregation of melamine at this composition. At 30% HMMM, the respective ratios were 1.00 and 0.72, indicating a small but significant amount of surface segregation.

CONCLUSIONS

The average slope of the log–log plots of contact radius as a function of particle radius was found to be 0.67, which validates the JKR adhesion theory [Eq. (4)] for the particle sizes and polyester–melamine substrate compositions used. This result is consistent with the assumption that an elastic response to the adhesion-induced stresses between zirconia or carbon particles and polyester substrates is present for particles of 10–60 µm.

The calculated W_A values for both zirconia and glassy carbon spheres interacting with cross-linked polyester surfaces were similar for any given substrate composition, consistent with the similar surface energies for the two materials.

At low (5 wt%) melamine concentration, W_A was found to be $34\,\mathrm{mJ/m^2}$. This value lies close to the values previously determined [4,5] for glass particles interacting with compliant polyurethane substrates.

At higher melamine concentrations, the experimentally calculated W_A results are lower than expected. The W_A values found are attributed to melamine segregation within the polymer. High cross-linker concentrations encourage the formation of a self-condensed, melamine-rich, glassy, high-modulus surface layer, which is responsible for the low particle adhesion [10]. Assuming the actual W_A to remain unchanged across the substrate composition range allows the estimation of a surface modulus, which can be further used to estimate the surface composition.

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