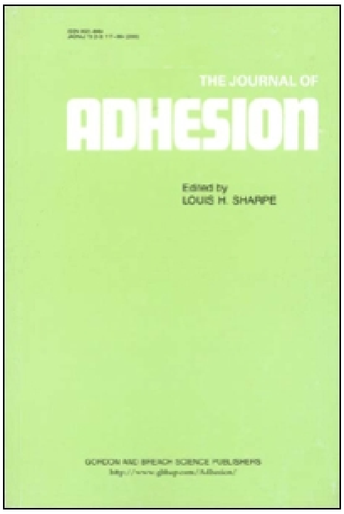


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# Adhesion of Micron Sized Coal Particles to a Massive Coal Substrate

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The force of adhesion between 2–5 $\mu$m coal particles and a coal substrate was measured using an ultracentrifuge. The repeatability of the data was about $\pm 8$ percent. The data indicate that neither the orientation of the substrate surface with respect to its bedding planes nor an initial positive charge of several hundred elementary units significantly affects the force of adhesion. However, the force of adhesion on cleaved substrates is about 40 percent higher than on those which were mechanically polished. The force needed to remove 50 percent of the particles was about 2 millidynes.

## INTRODUCTION

The measurement of the force of adhesion between 1–10 $\mu$m coal particles and a massive flat coal substrate presents a formidable problem. Such particles are at the limit of optical resolution and may adhere with forces $10^5$ times their weight. Furthermore, the irregular shape of coal particles and the inhomogenous chemical composition of the particles as well as the substrate make theoretical calculations approximate at best. Nevertheless, an understanding of the factors that affect the force of adhesion in this system is of considerable importance to the coal industry.

The chemistry and structure[1] of naturally occuring coal is exceedingly complex and varies from source to source such as to prevent any simple classification in the scientific sense. Coal is best described as a combustible

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sedimentary rock formed from the remains of living organisms. Most generally coal is a macroscopically structureless system consisting essentially of a matrix of carbon and hydrogen linked in an amorphous organic system with some small regions of graphitic carbon. The impurity level of components other than coal can exceed five percent. As a consequence, the characterzation of coal substrates or coal dust beyond presenting the geographic source of the material and/or coal rank is of little consequence. The coal used in the following studies originated from the Pittsburgh seam and is generally classified as bituminous.

The purpose of this investigation was to develop a technique for the measurement of the force of adhesion between coal particles and a massive coal substrate and to investigate some of the factors that influence this force.

## THEORY

The most important mechanisms which establish the force of adhesion are chemical forces,[2] electrostatic forces,[3] Van der Waals forces,[3] and the surface tension forces of adsorbed liquid films.[4]

The force of adhesion between a metallic spherical particle and an infinite flat substrate due to electrostatic charges was given by Krupp[3] as:

$$F_{el} = \frac{Q^2}{16\pi\varepsilon_0\left[\gamma + \frac{1}{2}\ln\frac{2R}{Z}\right]^2 RZ} \tag{1}$$

where:

$Q$ = charge on particle

$\varepsilon_0$ = permeability of free space

$\gamma$ = Euler's constant = 0.577

$R$ = radius of particle

$Z$ = distance between the substrate and the surface of the particle

In the derivation of Eq. (1), Krupp assumed that the substrate was at ground potential. Table 1 shows the electrostatic contributions to the force of adhesion for various sizes of coal particles and the number of charges per particle. If the particle is an insulator and the charges do not lie entirely in the surface layer, the electrostatic force of adhesion will depend on the exact charge distribution within the bulk and will, in any case, be reduced by perhaps one or two orders of magnitude.

The electrostatic charges on the coal particles originate through ion-particle impact, fracture processes or by particle-particle collision[5]. Although positive charges of several hundred elementary units have been measured on natural coal particles,[6] the distribution of these charges on the particles is not clearly defined and the resulting force of adhesion cannot be calculated.

TABLE I

Force of adhesion between a graphite substrate

| Spherical particle diameter μm | $F^a_{VdW}$ | $F^a_{el}$ under condition of no. of charges/particle[b] | | | | $F^a_{ST}$ | |
|---|---|---|---|---|---|---|---|
| | | 10 | $10^2$ | $10^3$ | $10^4$ | $\rho = 5$ | $\rho = 10$ |
| 1 | 14 | .00011 | .011 | 1.1 | 110 | 1.8 | .9 |
| 5 | 72 | .000023 | .0023 | .23 | 23 | 9.0 | 4.5 |
| 10 | 140 | .000011 | .0011 | .11 | 11 | 18 | |

[a] Force in millidynes
[b] Z = 4Å

$$\rho = \frac{\text{Particle diameter}}{\text{Diameter of water film}}$$



The Van der Waals force of attraction, resulting from the instantaneous dipole fluctuations present in matter, was given by Krupp[3] as:

$$F_{VdW} = \frac{h\bar{\omega}R}{8\pi^2} \tag{2}$$

where:

$Z$ = Distance from the surface of the particle to the substrate
$R$ = Radius of the particle
$h\bar{\omega}$ = Lifshitz constant

The equation applies to a spherical particle near a flat substrate. The Lifshitz constant, $h\bar{\omega}$, is a material property related to the frequency dependence of the dielectric constant of the material and was given by Krupp as 6.9 eV for diamond and 7.2 eV for graphite. The range of this variable is between about 1 and 10 eV.

The force between a particle and substrate in the presence of extensive adsorbed water films due to the creation of a meniscus between the particle and the substrate was given by reference 4.

$$F_{ST} = 4\pi\gamma R \tag{3}$$

where $\gamma$ is the surface tension of water (72 dynes/cm) and $R$ is the radius of the water meniscus. The equation assumes that the particle is a smooth sphere whose radius is much larger than the radius of the ring of water.

Table 1 also indicates the magnitude of force due to surface tension under the conditions where the radius of the particle is ten times and five times the radius of the ring. An adsorbed layer of water of even one or two monolayers in thickness shields the Van der Waal's force which will reduce the attraction due to these interactions by about an order of magnitude.

The force of adhesion due to direct chemical bonding along an interface cannot simply be estimated due to a complete lack of understanding of the chemistry of coal surfaces. One might assume, however, that such bonding, if it occured, would develop forces of attraction which far exceed those presented above: a case which was not observed in experiment.

## EXPERIMENTAL

Krupp[3] and Corn[7] present critical reviews assessing the various techniques for measuring the attractive forces between various microparticles and substrates. Among these are the use of the microbalance,[7] pendulum,[8] aerodynamics,[9] impact[10] and a centrifuge.[11,12,13]

The latter appears to provide the most reliable and nearly absolute data available. In the case of the modern ultracentrifuge the test samples can be hermetically sealed from the ambient conditions and the temperature controlled. The force tending to fracture the particle from the substrate is given by:

$$F = \rho V a_c \tag{4}$$

where $\rho$ is the mass density which was usually taken as 2.0 gm/cc, $V$ is the volume of the particle which was estimated as an average considering the particle size distribution and $a_c$ is the centrifugal acceleration due to the rotating system.

In the following experiments an International B-60 centrifuge with an SB283 rotor capable of attaining 280,000 g's of centrifugal acceleration was used. The variable angle titanium buckets as shown in Figure 1 were used to support the samples in the centrifuge. The buckets were constructed such that as the speed of rotation increased, the bucket moved from the rest vertical position to a horizontal position. The 1.3 cm OD titanium bucket maintained the ambient environment around the sample by an "O" ring seal. The titanium bucket also contained a thin-walled plastic test tube (1.1 × 10 cm) which supported an aluminum framework that in turn held the bulk coal substrate about 0.5 cm above the electron microscope grid. The aluminum support was designed to withstand the high forces and yet not overload the centrifuge itself. The coal substrates (0.5 × 0.5 cm) were carefully mounted on the aluminum support using an epoxy adhesive. Coal particles were

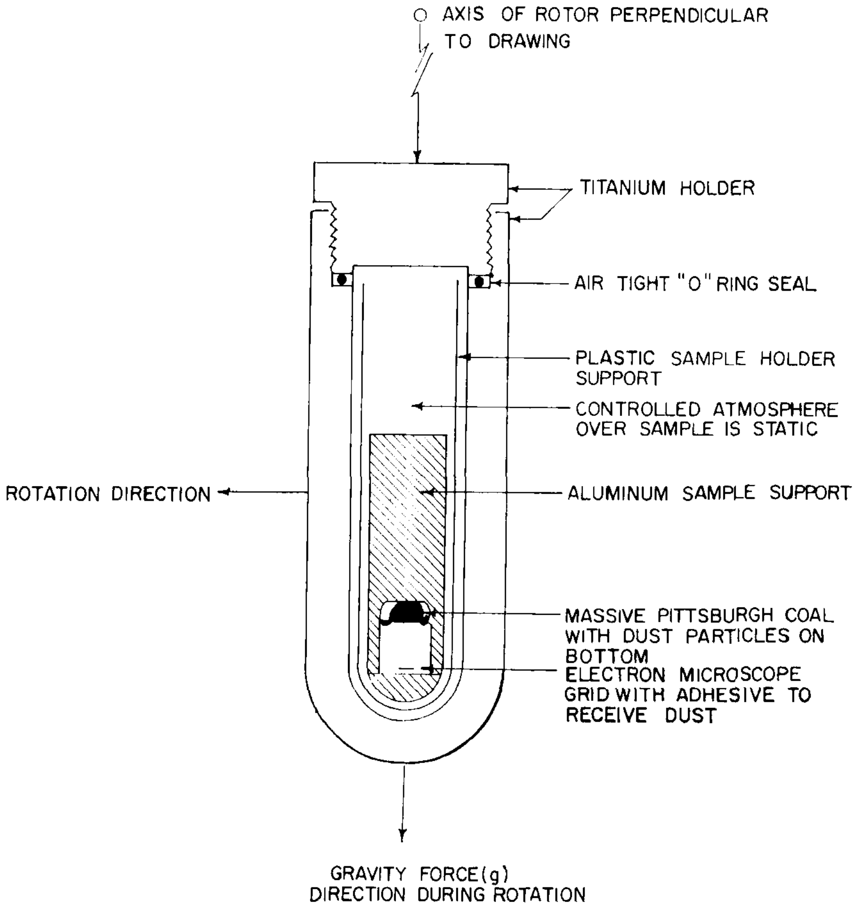


FIGURE 1 Centrifuge capsule.

allowed to settle on the substrate. The substrate and holder were then placed inside the centrifuge and exposed to accelerations of up to 280,000 g's in small increments. After each increment of acceleration, the electron microscope grid was examined and the number of particles that was removed by that increment of acceleration was counted in the electron microscope. Thereafter a clean grid was placed in the system and the next higher increment of acceleration was studied. The percentage of particles remaining on the substrate after each increment of acceleration was plotted versus the force encountered during that interval of acceleration.

All substrates were cut from a block of Pittsburgh coal such that their surfaces were either parallel or perpendicular to the bedding planes of the coal block. The surface of the substrates were either cleaved by means of a sharp blade or mechanically polished. The polished substrates were cut with a water cooled cut-off wheel, dried and then dry polished by standard metallographic techniques with 00 grade emery paper. Scanning electron micrographs were prepared by the Bureau of Mines, Pittsburgh, which indicated that both surfaces were very rough with surface steps many times larger than the test particles. At 1000X magnification the surfaces by the two preparation techniques were indistinguishable by optical means.

The substrates were first cleaned by exposing the surface to a high velocity jet of filtered compressed air. The substrate and holders were then placed in the centrifuge and accelerated to 100,000 g's to remove any additional particles. Ideally, the substrates would be accelerated to just below their fracture strength, but this strength varied from substrate to substrate and could not be known without destroying the substrate.

Coal particles were placed on the substrate in a known and repeatable way and in known concentrations utilizing the apparatus shown in Figure 2. The coal particles used in this study were supplied by Donaldson Co., Minneapolis



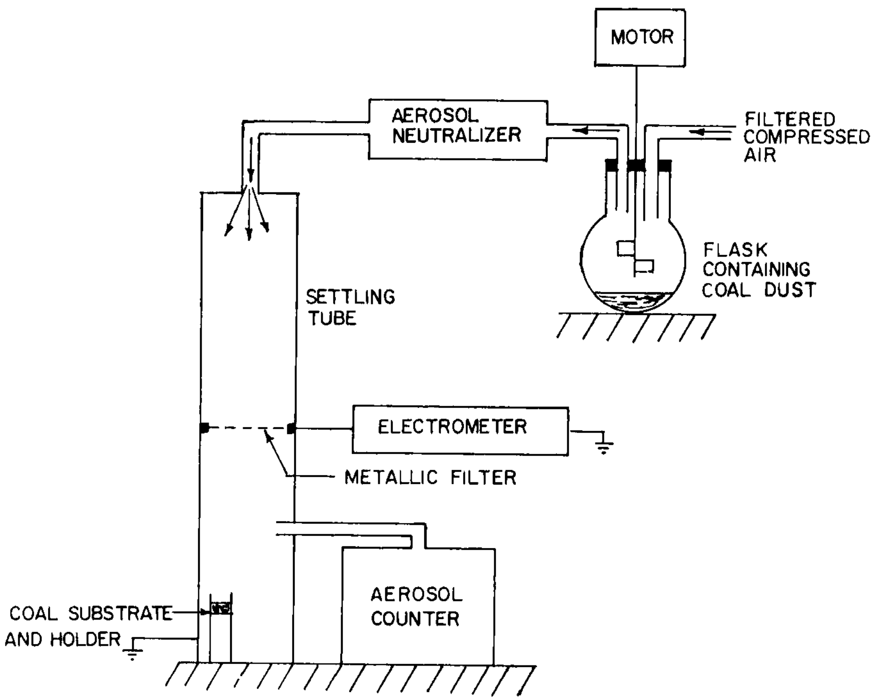


FIGURE 2 Particle settling apparatus.

(code number of 284C) and had a mass mean diameter 5.4 $\mu$m. These particles have been thoroughly characterized by de Merville.[6] The particles were placed in the flask and stirred by a motor driven brass propeller. A stream of filtered air then carried a small quantity of the particles out of the flask. The air stream and corresponding high velocity gradient have the desirable effect of breaking up particle agglomerates. The stream of particles could then be either passed through an electrostatic charge neutralizer (Thermosystems, Inc., St. Paul, Model 3012) or introduced directly into the 6 cm OD by 1.2 m settling tube and permitted to settle under the action of gravity. Elimination of the charge on the particles permitted an evaluation of the effect of electrostatic charge on the force of adhesion. The particle concentration was measured by a Bausch and Lomb 40-1A Aerosol Counter and the charge carried by the falling aerosol was measured in a Faraday cage by the Cary 301 electrometer also described by de Merville.[6]

A clean electron microscope grid was placed in the bottom of the settling tube adjacent to the coal substrates and after a mass of particles was allowed to settle, this grid was removed and examined to determine the concentration of particles. In order to minimize particle-particle interaction on the surface, the minimum distance between particles on the substrate was not less than two particle diameter, i.e. 6 $\mu$m, the substrates were recleaned and the dust deposition was repeated.

If too few particles were settled on the microscope grid, representative results would not be obtained. A minimum of one particle per 500 $\mu m^2$ was used and at least 50,000 $\mu m^2$ were examined on each substrate before each increment of acceleration. If the density of particles was lower than one particle per 500 $\mu m^2$, the grid was placed back in the settling tube and more dust was deposited.

The relative humidity was 20 percent at a temperature of 70°F in all experiments. Figure 3 is an electron micrograph of the particles used in this experiment.

## RESULTS AND DISCUSSION

Experiments were performed on 11 different substrates in which those numbered 1–6 were processed and stepped through increments of acceleration three separate times each before it was decided to spin them fast enough for destruction. Substrates 7–11 were used in only one sequence. Figure 4 shows the results from three sequences run on substrate number 2 and three sequences run on substrate number 6.

The rather narrow band of data scatter shown in Figure 4 was somewhat surprising in that coal is not a homogeneous material and the particles were

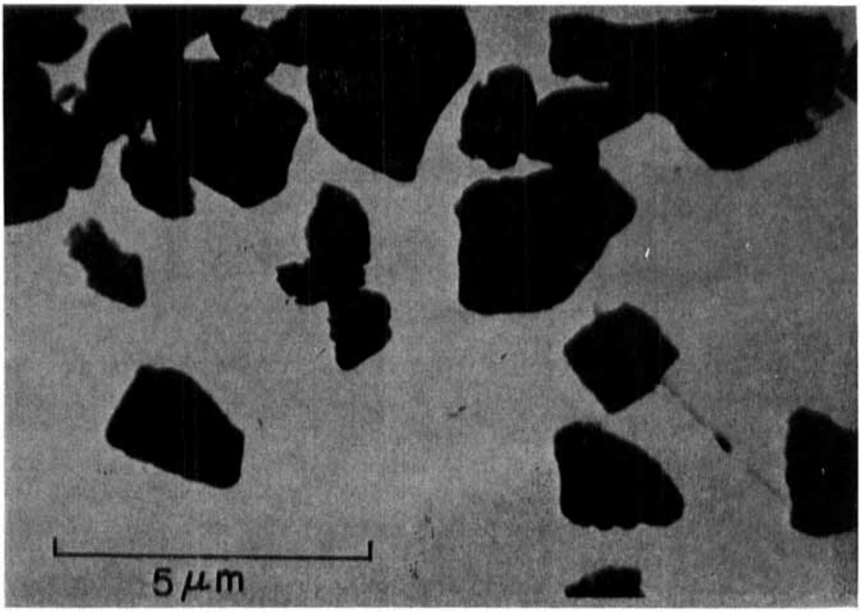


FIGURE 3 Donaldson 284C coal dust particles.



most irregularly shaped. The average accuracy in the first 15 sequences was about ±8 percent of the number of particles removed per given force.

Some substrates were cut such that their surface was parallel to the bedding planes of the bulk coal and others were cut perpendicular to the planes. A comparison of the data from both substrates indicated that there was no significant difference in the force of particle adhesion. This result seems reasonable because of the extreme inhomogenity of coal in both orientations.

The presence of electric charges on the particles and substrate prior to contact was also observed to cause no significant difference in the force of particle adhesion. It is evident from this observation that $F_{el}$ due to electrostatic charges, does not make a significant contribution to the overall adhesive force.

A comparison of the force of particulate adhesion on cleaved substrates and on mechanically polished substrates is shown in Figure 5. The force of adhesion between particles and naturally cleaved surfaces is about 40 percent higher than that for the mechanically polished surfaces. This seems to disagree with most investigators[16,18] who have found that particles adhere more strongly to smooth substrates. In their experiments, however, the surface asperities were always much smaller than the particles. The surface irregularities in this experiment were about 50 to 200 times the particle

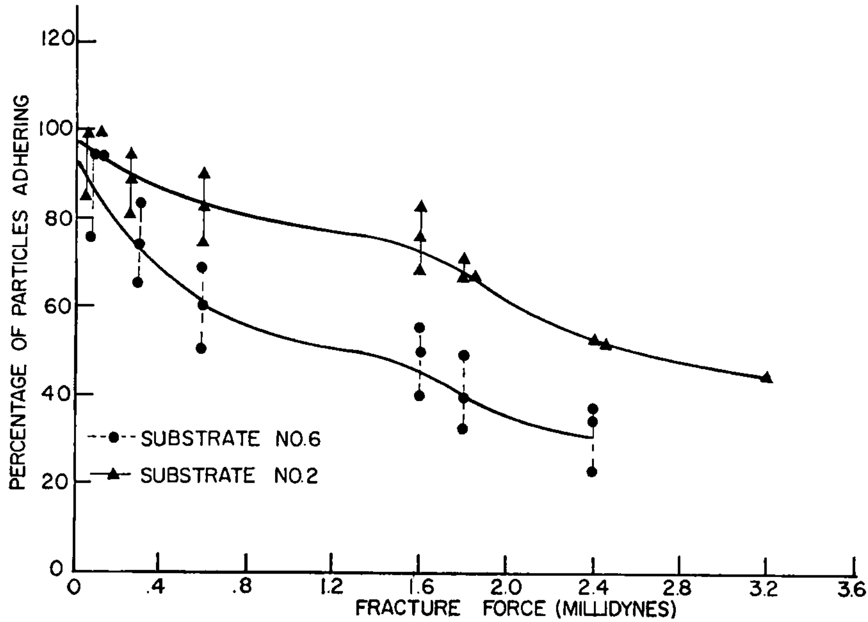


FIGURE 4 Typical range of data for three runs on each of two different coal substrates

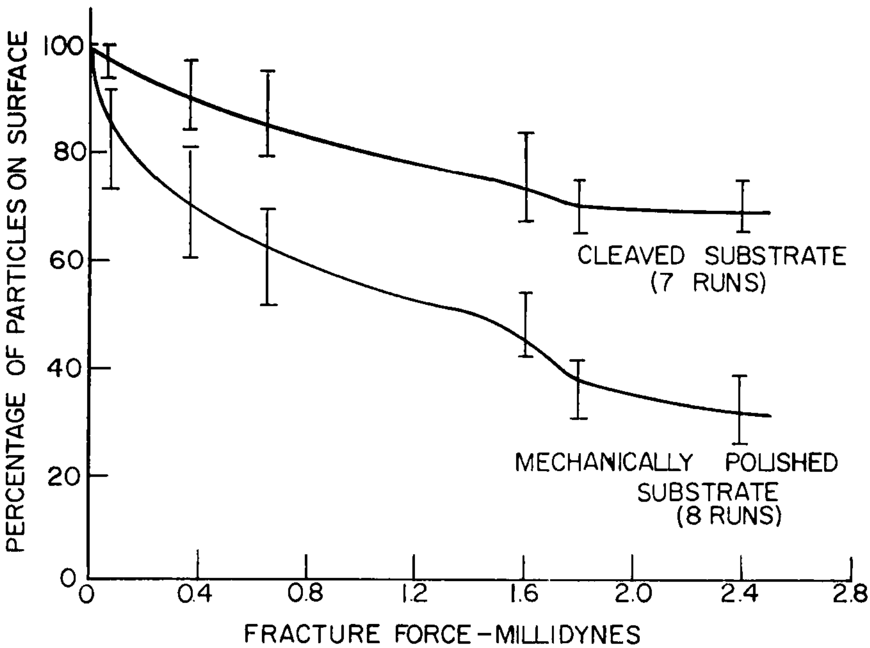


FIGURE 5 Force of adhesion between 3 $\mu$m coal particles and various coal substrates.

diameter on rough substrates and about three to five times the particle diameter on the smooth substrates. In both cases the surface irregularities were larger than the particles.

Table II shows the acceleration at which each substrate fractured and the percentage of particles still adhering at this force. It is evident that a large fraction of the particles on all substrates adhere with a force greater than the fracture strength of coal.

TABLE II
Summary of substrate failure

| Substrate | Largest number of g's | Percent of particles still adhering |
|---|---|---|
| 1 | 200,000 | 50 |
| 2 | 150,000 | 45 |
| 3 | 100,000 | 60 |
| 5 | 150,000 | 72 |
| 6 | 150,000 | 35 |
| 7 | 150,000 | 50 |
| 8 | 150,000 | 70 |
| 9 | 100,000 | 60 |
| 10 | 100,000 | 60 |
| 11 | 100,000 | 85 |
| 12 | 150,000 | 40 |



There are two potentially serious sources of error with the centrifuge technique. First, if the particles are deposited on the substrate too densely, a large number of the particles will fall on top of each other and the measured force will be the force of adhesion between two particles rather than the force of adhesion between a particle and a substrate. This did not prove to be a problem because the maximum density to which particles were deposited was $0.014/\mu m^2$, which gives a probability of only .08 that two particles touch. The other potential source of error arises from counting as particles fragments of the substrate that are removed by the applied force. Two factors tend to minimize this problem. First, the Donaldson 284C coal dust that was used in this experiment had a small size distribution (2 $\mu$m to 5 $\mu$m) and any particle observed beyond this range was not counted. Secondly, the coal substrates always failed catastropically which covered the microscope grid with an easily visible layer of coal fragments.

Finally, it should be pointed out that the observed force of adhesion was about 1/20 of the calculated Van der Waals force as shown in Table I. The Van der Waals force of attraction in Table I was calculated under the following assumptions, none of which are true: adhering bodies are graphite; the particle is a perfect sphere; the substrate is a flat surface; the distance of closest approach between the particle and substrate is 4Å and no adsorbed water film was present at the particle substrate interface. The latter is particularly critical since real surfaces always adsorb several layers of water vapor under ambient conditions. As noted earlier, this layer of adsorbed water could reduce the Van der Waals force by a factor of 10. It is difficult to determine to what degree the other assumptions might effect the force of adhesion in this system.

## CONCLUSIONS

This investigation demonstrated that the force of adhesion between 2 to 5 $\mu$m coal particles and a coal substrate can be measured with an ultracentrifuge. Repeated tests under identical experimental conditions show that the force can be measured to an accuracy of about $\pm 8$ percent.

The data indicate that neither the orientation of the substrate surface with respect to its bedding planes nor an initial positive charge of several hundred elementary units significantly affects the force of adhesion. However, the force of adhesion on cleaved substrates is about 40 percent higher than on those which were mechanically polished. The force needed to remove 50 percent of the particles was about 2 millidynes.



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