SAN02000-0474C

Presented at the 23rd Annual Meeting of Adhesion Society, Myrtle Beach, SC, February 23, 2000

Assessment of Surface Contamination with Contact Mechanics

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INTRODUCTION

We are particularly interested in the work of adhesion measurements as a means to facilitate our understanding of the adhesive failure mechanisms for systems containing encapsulated and bonded components. Of the several issues under investigation, one is the effect of organic contamination on the adhesive strength for several types of polymer/metal interface combinations. The specific question that we are trying to address is at what level of contamination does adhesive strength decrease. The use of contact mechanics, the JKR method, is a good approach for studying this question. Another approach being studied is the use of interfacial facture mechanics [1]. The model contaminant is hexadecane – non-polar, medium molecular weight hydrocarbon fluid. We choose hexadecane because it replicates typical machining fluids, is nonreactive with Al surfaces, and should not dissolve readily into the adhesive systems of interest. The application of a uniform, controllable and reproducible hexadecane layer on Al surfaces has proven to be difficult [2]. A primary concern is whether studies of model systems can be extended to systems of technological interest.

The JKR theory is a continuum mechanics model of contact between two solid spheres that was developed by Johnson, Kendall and Roberts [3]. The JKR theory is an extension of Hertzian contact theory [4] and attributes the additional increase in the contact area between a soft elastomeric hemisphere to adhesive forces between the two surfaces. The JKR theory allows a direct estimate of the surface free energy of interface as well as the work of adhesion (Wa) between solids. Early studies performed in this laboratory involved the determination of Wa between silicone (PDMS) and Al surfaces in order to establish the potential adhesive failure mechanisms. However, the JKR studies using commercial based PDMS was fraught with difficulty that were attributed to the additives used in commercial PDMS systems. We could not discriminate hydrogen-bonding effects between Al_2O_3 and hydroxyl groups in the PDMS, and other possible bonding mechanisms. A model PDMS elastomer and polymer treatments were developed for studying solid surfaces by measuring the degree of self-adhesion hysteresis [5] as indicator of surface properties.

The goal of this work is to measure the adhesion between PDMS/Al surfaces – contaminated and two cleaning techniques. A custom-made JKR apparatus is used to determine the amount of hysteresis and Wa.

EXPERIMENTAL

Preparation of PDMS Lens

Model networks of poly(dimethylsiloxane) were synthesized by reacting vinyl-capped poly(dimethylsiloxane) with tetrakis (dimethylsiloxy) silane in the presence of a platinum catalyst. The use of the platinum – divinyltetramethyldisiloxane complex allowed the polymerization to proceed at room temperature without the formation of by-products. All reactants were used as received from Gelest Inc.

The procedure used to synthesize the PDMS networks is as follows. First, tetrakis (dimethylsiloxy) silane was added using an Eppendorf volumetric dispenser. With the amount of cross-linker fixed, a 0.75 stoichiometric amount of vinyl-capped PDMS (6000 g/mol) was added with a 3 ml syringe. The contents were then mixed thoroughly and stored in a freezer at –20 C. Once the temperature was low enough to slow the reaction, approximately 150 ppm of platinum – divinyltetramethyldisiloxane complex was worked into the system. Hemispheres, which are used as the JKR lens, were prepared using an oiler (medium, DL-32). Drops of the mixture were placed on tridecaflro-1,1,2,2-tetrahydroctyl trichlorosilane treated glass surface.

DISCLAIMER

Substrate preparation

Initial studies with polished Al 6061-T6 with the average surface roughness, (as determined by Dektak Surface Profilometer) measured 250 - 850 Å and proved to be too rough for determining film thickness of the hexadecane. Also the JKR measurement proved unreliable because the roughness interfered with the contact area measurements. All the data reported here were made by sputtering 400 nm of Al onto glass slides. The average roughness was 30 Å. As measured by ellipsometry, the average hexadecane thickness was 10-20 Å. Two cleaning procedures were: 1) acetone rinse followed by plasma clean; 2) acetone rinse followed by chromic-sulfuric clean/etch and HPLC grade water rinse. A clean N_2 stream removed any residual fluid.

JKR studies

A hemispherical lens is mounted into the JKR apparatus. Measurements were made at 30 - 60 %RH. Loading and unloading measurements were done with an incremental change in displacement of 3 μm per step. The work of adhesion was determined by recording the contact radius as a function of contact load and using the JKR equation to fit the data:

$$a^3 = (R/K)\{P + 3\pi WR + [6\pi WRP + (3\pi WR)2]^{1/2}\} \quad (1)$$

Where a is the contact radius, R is the effective radius of the lenses, K is the material property, P is the contact load, and W is the work of adhesion.

RESULTS AND DISCUSSION

A plot of the experimental data in Figure 1 shows no hysteresis or negative tensile forces, thus an ideal JKR plot, for two PMDS hemispheres [5] and therefore allows accurate calculation of Wa from Equation 1. In studying systems that have strong adhesive forces the curves can be difficult to interpret because of their nonlinearity. Inconsistent JKR data was obtained from sputtered samples. We found some form of cleaning was necessary. Rinsing the sputtered Al samples with acetone after deposition left a visible film. Surface analysis of the residue gave inconclusive results. Deploying aggressive cleaning resolves the residue issue and gave consistent results. In Figure 2 a small amount of hysteresis and pull-off force is seen. This is consistent with the increase in surface reactivity from the expected above the sputtered Al after the chromic-sulfuric acid clean. However, in Figure 3 where the surface is contaminated with hexadecane, the large hysteresis and pull-off force are inconsistent with what would be expected. The surface energy of the PDMS was measured to be 22 mJ m^{-2} and is mostly non polar. Thus the expectation is that the non-polar interactions give a low hysteresis and pull-off force. The results from the plasma-cleaned surface are as expected. In Figure 4 large hysteresis and pull-off forces for the plasma cleaned surface are seen. A substantial decrease with hexadecane contaminate is seen in Figure 5. The results from Figures 4 and 5 are contrary to Figures 2 and 3. The source of this discrepancy is under study. In Figure 4 the adhesive strength can be determined from the pull off force from rapid drop off in contact area at constant tensile force. We are attempting to measure the crack growth at constant tensile forces from these samples.

ACKNOWLEDGEMENT

This work was performed at Sandia National Laboratories, which is supported by the United States Department of Energy under contract number DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy. We thank Michael L. Thomas for taking the ellipsometry measurements and Catharine H. Sifford for determining surface roughness. Also, we thank Carol Jones Adkins and Rachel Giunta for helpful discussions.

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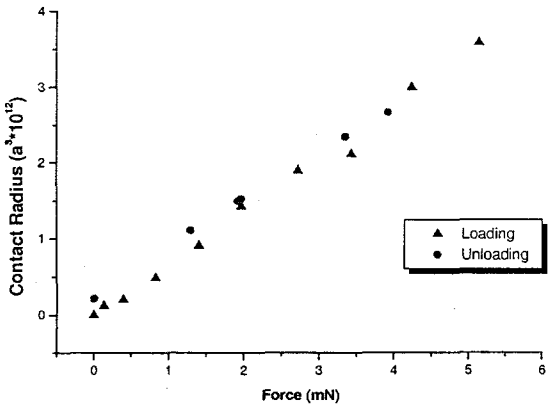

Figure 1 - JKR Results of Two PDMS Hemispheres – Wa = 44.02, K= 1.06×10^6

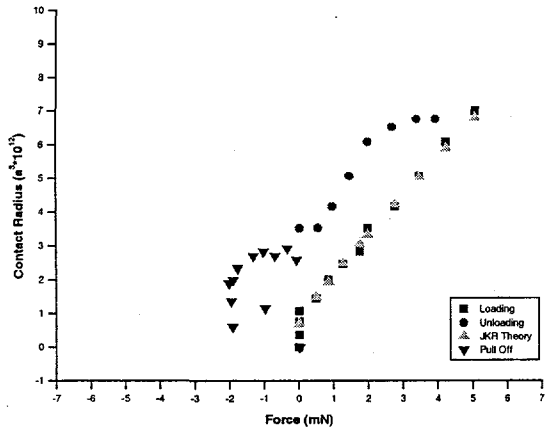

Figure 2 - Sputtered Al, Cleaned with Chromic-Sulfuric Acid

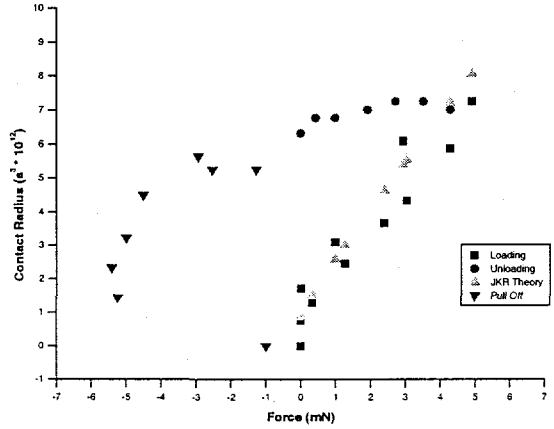

Figure 3 - Sputtered Al, Cleaned with Chromic-Sulfuric Acid and Hexadecane Contaminated

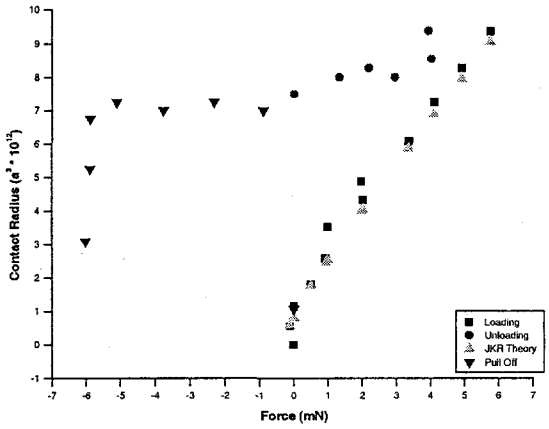

Figure 4 - Sputtered Al Plasma Cleaned

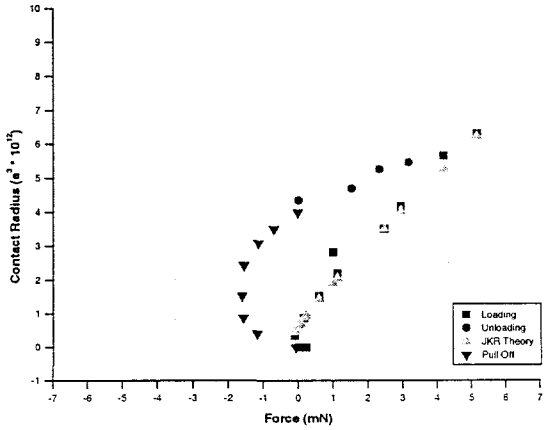

Figure 5 - Sputtered Al Plasma Cleaned and Hexadecane Contaminated