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Separation modes in microcontacts identified by the rate dependence of the pull-off force

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We report the observation of two distinct modes of rate-dependent behavior during contact cycling tests. One is a higher pull-off force at low cycling rates and the other is a higher pull-off force at high cycling rates. Subsequent investigation of these contacts using scanning electron microscopy (SEM) demonstrates that these two rate-dependent modes can be related to brittle and ductile separation modes. The former behavior is indicative of brittle separation, whereas the latter accompanies ductile separation. Thus by monitoring the rate dependence of the pull-off force, the type of separation mode can be identified during cycling without interrupting the test to perform SEM. © 2008 American Institute of Physics. [DOI: 10.1063/1.2967855]

Operation of a microelectromechanical system switch requires closing and opening of the microcontacts over many billions of contact cycles.¹ During this process the contact resistance and contact adhesion change, often leading to fail-to-close or fail-to-open switch failures. In order to study the evolution of microcontacts, we developed a scanning probe microscope (SPM) based contact test station.² Contact testing was performed between hemispherical contact bumps (microfabricated on very stiff SPM cantilevers) and planar substrates. Both the bumps and substrates were coated with the contact test material. Using this contact tester, we have studied different contact materials³ and contact adhesion.⁴

Microcontacts may separate in brittle or ductile modes.⁵ In the brittle mode [Fig. 1(a)], rupture occurs abruptly with little, if any, plastic deformation. In the ductile mode [Fig. 1(b)], rupture occurs more gradually with significant plastic deformation. In previous studies,²⁻⁴ the type of separation mode (ductile or brittle) which occurred could only be identified by scanning electron microscopy (SEM) observation and not during the cycling test.

Depending on the separation mode, the surface morphology of the microcontacts can change, eventually leading to large changes in the pull-off force (i.e., the force needed to separate the contacts).^{4,5} In this paper, we report our observation that the magnitude of the pull-off force depends on the cycling rate, and that this rate dependence is related to the separation mode. This behavior allows the separation mode to be identified during testing without SEM observation.⁶

To study the rate dependence of the pull-off force, a 250 nm gold thin film was sputtered on the hemispherical bump (15 μm radius of curvature) of an especially fabricated very stiff test cantilever ($K \cong 10^4$ N/m) and also on the mating planar substrate. The tests were performed in laboratory air with a relative humidity of 30%–40%. The cycling tests use a small piezoactuator in addition to those in the SPM to achieve a high cycling rate. The pull-off force was measured using the SPM.

The pull-off forces at two cycling rates (0.5 and 300 Hz) were compared by suddenly switching from one frequency to

the other. The maximum contact force was controlled at 200 μN during this cycling test. After testing, the samples were inspected in the SEM. We observed two distinct modes of rate-dependent behavior of the pull-off force. One mode exhibits a higher pull-off force at low cycling rates (HFLR). The other mode exhibits a higher pull-off force at high cycling rates (HFHR). SEM observation shows plastically flattened features [Fig. 1(a)] at the surface of the contact in the HFLR mode. However, in the HFHR mode, ductile necks and/or signs of material transfer are observed [Fig. 1(b)].

We then investigated the effect of the maximum load on the rate-related pull-off force. For one load sweep, the maximum load was increased from zero to 250 μN , and then decreased back to zero. The load sweep was performed at 0.5 Hz, and then followed by a 300 Hz test. The test results are shown in Fig. 2. In both the HFLR and HFHR modes, the pull-off force increases with an increase in the loading force. However in the HFLR mode, as shown in Fig. 2(a), a larger pull-off force is measured at 0.5 Hz than at 300 Hz. The difference of the pull-off force between the two rates increases from 20 μN at a maximum loading of 150 μN , to 60 μN at a loading of 250 μN .

It is noted from Fig. 2(b) that for a loading force less than 200 μN in the HFHR mode, the pull-off forces measured at the two rates (0.5 and 300 Hz) are almost the same. However, at a maximum loading force greater than 200 μN , the pull-off force measured at 300 Hz becomes larger than that measured at 0.5 Hz. For a loading force of 250 μN , there is a 40 μN difference in the pull-off force between the two cycling rates. It is also noted that the increase of the pull-off force is more consistent in the HFLR mode than in the HFHR mode.

We also studied the rate-dependent pull-off force properties by sweeping the cycling rate from 0.5 to 1000 Hz. During this test, the maximum loading force was controlled at 200 μN . During the rate sweep, 13 different cycling rates were tested. At each cycling rate, the sample was cycled for 30 s for pull-off force measurement. In the HFLR mode the pull-off force tended to decrease only at rates above 100 Hz (Fig. 3).

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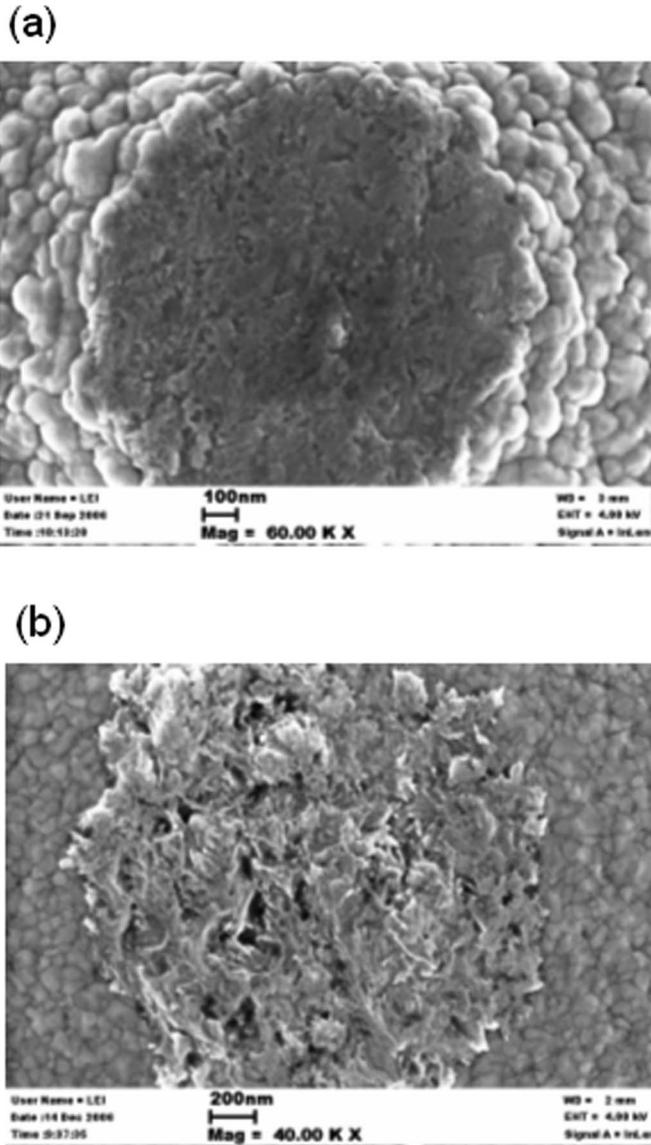


FIG. 1. SEM micrographs of gold contact bumps subjected to a maximum loading force of 200 μN . (a) Brittle separation. (b) Ductile separation.

The observed HFLR behavior could be explained by the contact time and load effects on the interfacial bond strength. For brittle separation, the magnitude of the pull-off force depends on the bond strength at the interface. The longer the time in contact and the greater the force, the stronger the bond formation is, which in turn leads to a higher separation force.

The kinetics of forming or destroying the interposing surface film could contribute to the observed HFLR rate-dependent features. Since gold is hydrophilic and the tests were performed in room air, such surface film effects could be due to the meniscus force effect. The experiments by Szoszkiewicz⁷ have shown that at 37% relative humidity, the mean meniscus nucleation time is 4.2 ms at room temperature. For contact times less than the mean nucleation time, the number of menisci formed will be reduced. The longer the time in contact, the more menisci can develop, leading to a larger meniscus force. Our rate sweeping experiment shows that the pull-off force drops when the cycling rate is greater than 100 Hz. This may indicate that the equilibrium time for meniscus condensation is about 5 ms, which is close

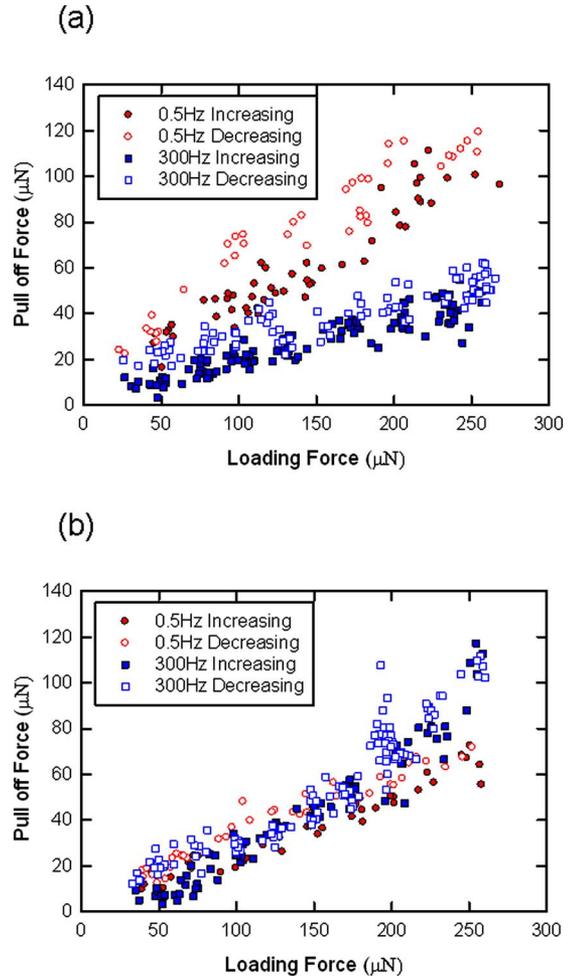


FIG. 2. (Color online) Rate-dependent pull-off force with (a) HFLR and (b) HFHR.

to the meniscus nucleation time observed by Szoszkiewicz.

Furthermore, we can verify that in the HFLR mode the measured difference in pull-off force is of the same magnitude as the meniscus force effect. It was shown that the force difference between the high cycling rate and low cycling rate is in the range of 20–60 μN . For a water thin film between a hemispherical bump and a flat surface, the meniscus force can be estimated by

$$F_m = 4\pi R \gamma_{lv} \cos \theta, \tag{1}$$

where γ_{lv} is the surface tension of the water film equal to 72.7 dyn/cm. Since there is an intrinsic layer of water thin

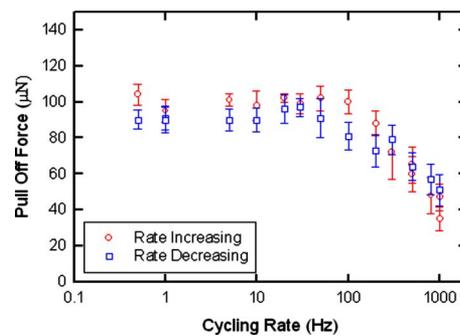


FIG. 3. (Color online) Cycling rate test of rate-dependent pull off force with HFLR.

film on a gold surface, the contact angle is chosen as zero. Using an effective radius of curvature of $45\ \mu\text{m}$ (estimated using the plastic deformation model of Kogut and Etsion⁸), the meniscus force can be calculated to be about $40\ \mu\text{N}$, which is approximately the measured force difference between 0.5 and 300 Hz. Thus both the transition time and the magnitude of the force suggest that the HFLR effect could be due to a meniscus force effect in brittle separation.

Unlike brittle separation, ductile separation is associated with bulk material stretching and nanostructure neck formation. Such nanostructure separation could activate viscous effects in the gold. Normally for inelastic deformation in a metal, time-independent yield flow activated by nucleation of dislocations is the dominant deformation mechanism. However, when the structure scale reduces to the nanoscale, the dislocation sources are largely reduced resulting in a time-dependent creep flow. In creep flow, diffusion is a dominant mass transport mechanism. The low barrier height for atom diffusion in the nanostructure allows atoms to move fast on the surface. Surface tension driven diffusion has been observed in the separation of Au/Au (Ref. 9) and Pb/Au (Ref. 10) nanojunctions. Even though it is unrealistic to assume the nanojunction is a liquid, it is believed that at the last stage of the separation (when the neck is narrowing down to several nanometer), the effect of viscosity in the junction is very important.¹¹ Such viscous effects could be the reason for observed HFHR features, which gives a greater pull-off force at a high cycling rate.

In summary, two rate-dependent pull-off force modes have been observed in gold microcontacts. One is a HFLR and the other is a HFHR. We found that each of these rate-dependent modes can be related to a particular separation mode, i.e., brittle or ductile. For HFLR behavior, the rate dependence may be due to surface effects during brittle separation. The longer contact time at lower cycling rates can promote the development of stronger bonds leading to a larger pull-off force between the contacts. Since all these tests were performed in room air with 30%–40% humidity, such surface effects could be due to meniscus formation in the contacts. We found that the measured force and rate de-

pendence are consistent with the characteristics of meniscus condensation.

For HFHR behavior the rate effects could be due to viscosity in the ductile separation. During ductile separation there are nanotips drawn out of the surfaces. Surface tension driven diffusion in these nanostructures can then cause viscous effects during separation. The viscous effects can lead to a larger pull-off force at a higher unloading rate. In general, once ductile separation has been identified during the cycling test, the surface is found to be damaged. SEM inspection shows that the contact surfaces in HFHR mode are rough, with significant wear and sharp peaks which are characteristic of ductile separation.

The significance of this work is the identification of the rate-dependence of the pull-off forces in metal contacts and its relation to the separation modes. By monitoring rate-dependent features during a cycling test, the separation mode can be identified *in situ* without the need for SEM observation. Further work in this area may allow the prediction of ductile and brittle separation modes based on materials, roughness, and cycling conditions.

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