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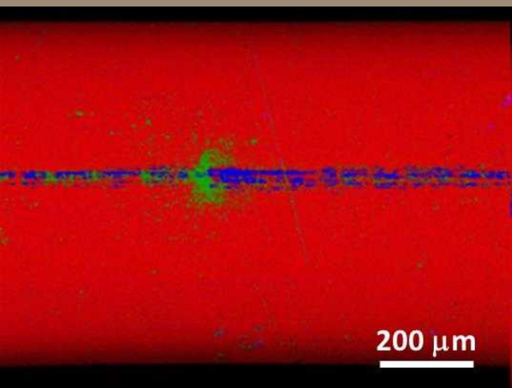

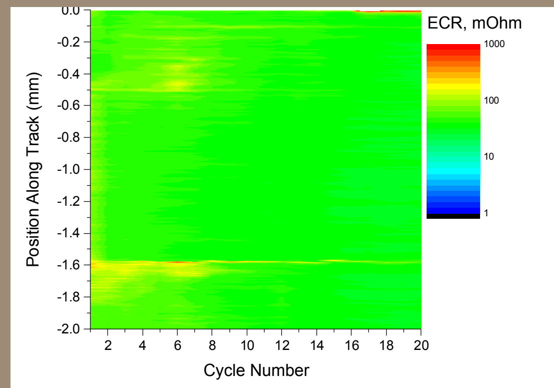

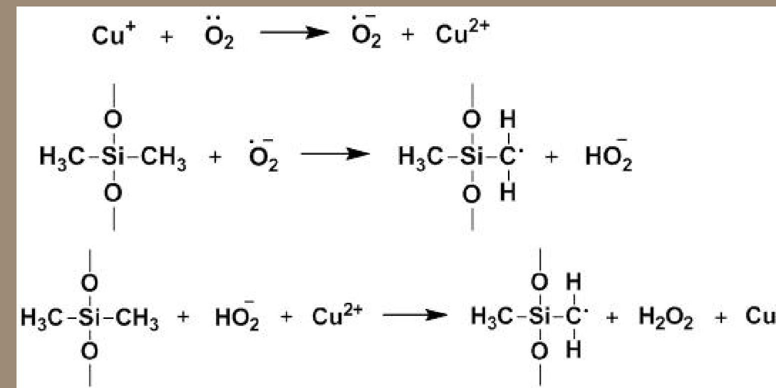

Electrical Behavior of Mechano-Chemical Deposits on Sliding Contacts

M.T. Dugger, B.L. Nation and R.S. Colbert

2016 STLE Annual Meeting, 16-19 May, Las Vegas, NV

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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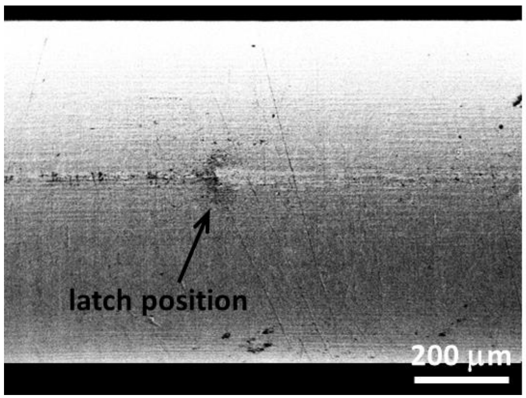
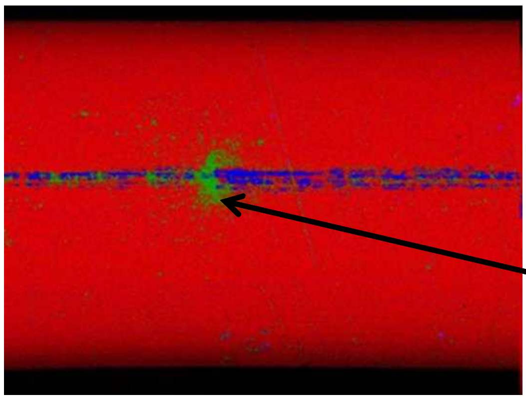
Deposits Are Detrimental to Electrical Contact Performance

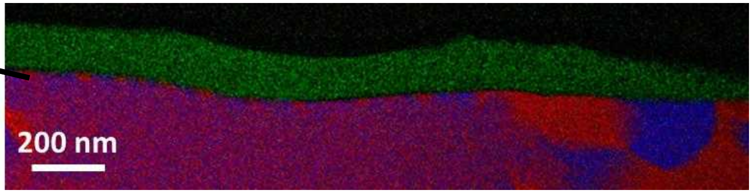
A fluid-damped accelerometer exhibits increased electrical contact resistance (ECR) after storage

- contacts are precipitation hardened Pd-Ag-Cu and Au-Cu-Pt electrical contact alloys (Deringer-Ney)
- the device is filled with 20 cSt PDMS (siloxane) fluid





Red = Paliney-7
Blue = Neyoro-G transfer
Green = Si-C-O deposit



EDS/AXSIA image of a focused Ion Beam (FIB) cross section through deposit

atomic %

	Pd	Ag	Cu	Pt	Ni	Zn	Au
Paliney-6	39.8	33.9	24.2	0.5	1.6	0.01	NA
Paliney-7	34.8	29.4	23.3	5.4	NA	1.6	5.4
Neyoro-G	NA	6.0	33	6.3	NA	2.2	52.5

Reaction of Organic Molecules at Sliding Surfaces is Well Known

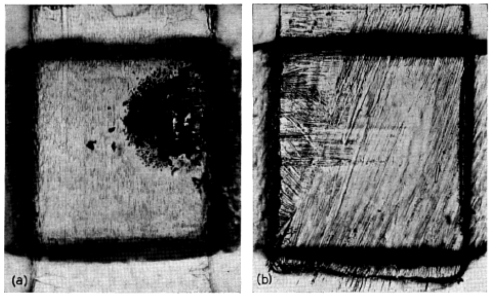
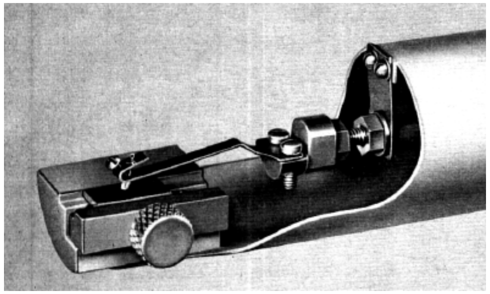


Fig. 1 — (a) Vinylite replica of a palladium relay contact, showing organic deposit formed from vapors of organic structural parts after 10^8 closures; (b) replica of a silver relay contact after 10^8 closures, showing complete absence of organic deposit. (Both magnified 75 times.)

When taking plastic replicas of fielded contacts, organic deposit trapped on replica

Developed high frequency oscillatory tribometer to produce deposit by sliding



H.W. Hermance and T.F. Egan, Bell System Tech. J. **37** (1958) pp. 739-776

- estimated conversion of *1 ML per pass* of adsorbed benzene into polymer
- *thermal decomposition ruled out*; invoked "…straining and deformation of the molecules…" to cause reactions

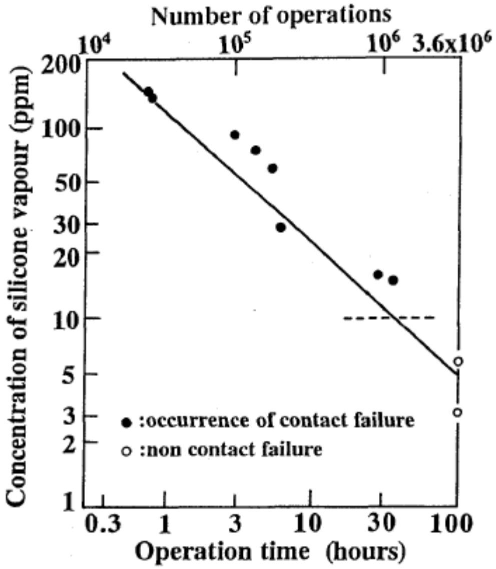
TABLE VII — FRICTIONAL POLYMER FROM VARIOUS ORGANIC COMPOUNDS*

(Pd/Pd, 120 Cycles, 30-gm Load, 170-Micron Stroke, 4 × 10^6 Wipes)

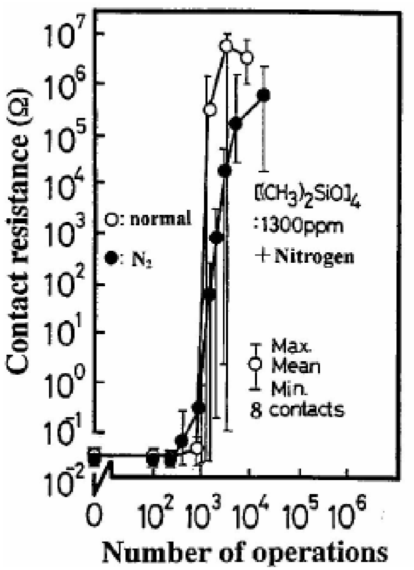
Group A — No Polymer Formed	
Methane } down to −60°C	Formaldehyde
Ethane } down to −60°C	Carbon tetrachloride
Propane	Methylamine
Methyl alcohol	Ethylene diamine
Group B — Trace to 5 Micrograms	
Propane (−60°C)	Ethyl alcohol
Butane (down to −60°C)	Ethyl chloride
Ethylene	Ethyl ether
Group C — 5 to 19 Micrograms	
Pentane	Propylamine
Hexane	Butylamine
Propene	Amylamine
Butene	Hexylamine
Butadiene	Triethylamine
Propanol	Pentadecafluorotriethylamine
Butanol	Butylchloride
Pentanol	Hexachlorbutadiene
n-Hexanol	n-Nitrobutane
2-Hexanol	Cyclohexanol
Acetaldehyde	Benzyl alcohol
Propionaldehyde	Aniline
Acetone	Hydroxybenzyl alcohol
Methylethylketone	Monochlorbenzene
2-Butanone	Thiophene
2,5-Hexanedione	Pyridine
Acetonitrile	Dioxane
Butyronitrile	Phenol
Hexanenitrile	Morpholine
Hexachlorbenzene	o-Cresol
Group E — 20 to 40 Micrograms	
Acetylene	Cyclohexane
Benzene	Crotonaldehyde
Toluene	Butyraldehyde
Xylene	Cyclohexanone
Naphthalene	5-Hexen-2-one
Cyclohexene	Limonene
Group F — Over 40 Micrograms	
Acrolein	Styrene
Acrylonitrile	Benzaldehyde

* The highest grade chemicals commercially available were used without further purification.

Electrical Contact Degradation by Silicones



T. Tamai and M. Aramata, Proc. IEEE Holm Conference on Electrical Contacts, Pittsburgh, PA, 27-29 Sept. 1993



T. Tamai, Proc. IEEE Holm Conference on Electrical Contacts, Montreal, QC, Canada, 25-27 Sept. 2006, pp. 26-31

- Adsorbed silicone vapors ***thermally decompose*** during make-break arcs and result in ECR increase in micro-relays
- Operating in nitrogen has no effect on this degradation mechanism

Electrical Contact Nanotribometer

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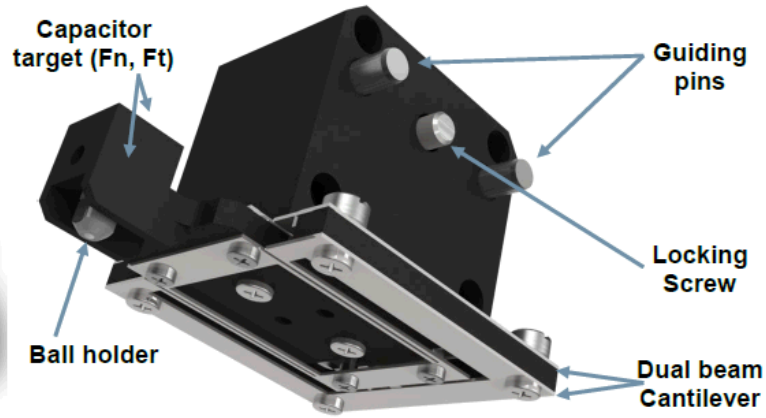
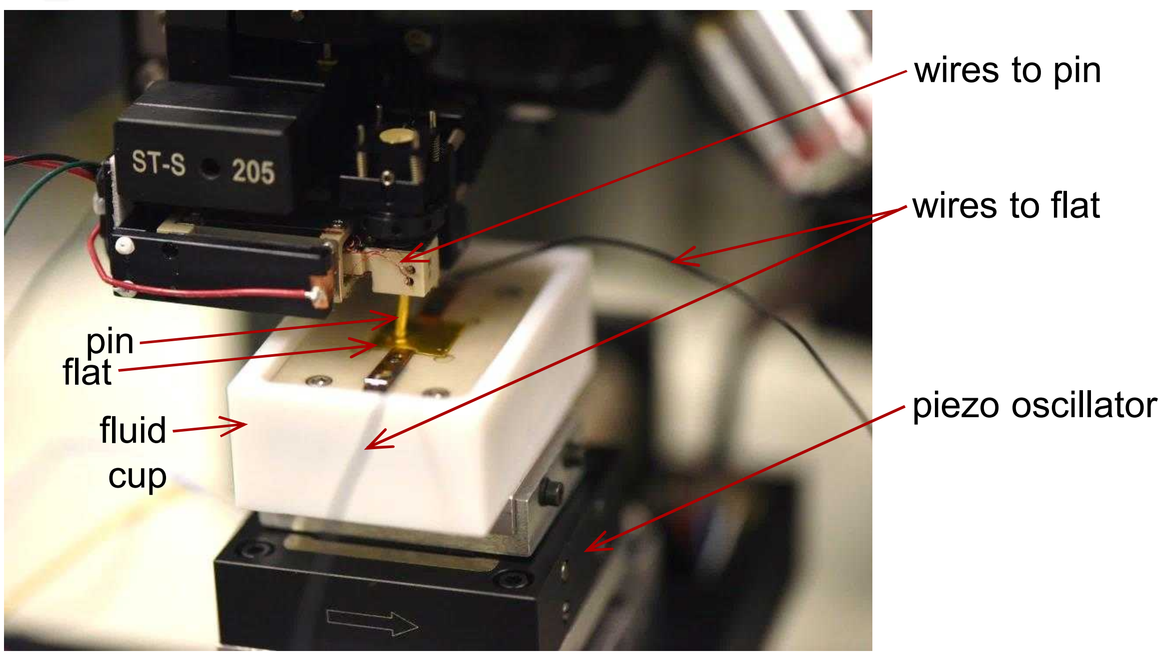
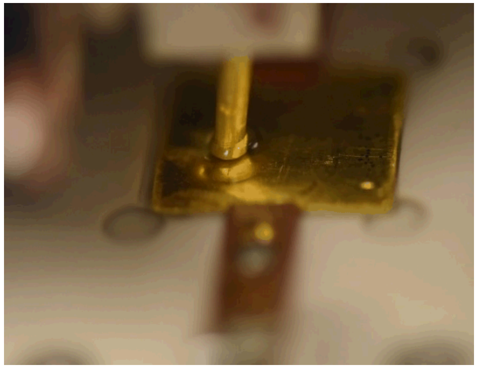


Nanotribometer modified for 4-wire ECR measurement

Test parameters:

- N = 50 mN
- pin radius = 1.6 mm
- track length = 1 to 2 mm
- fluid volume = 5 mL
- i = 10 mA, 1 V open circuit

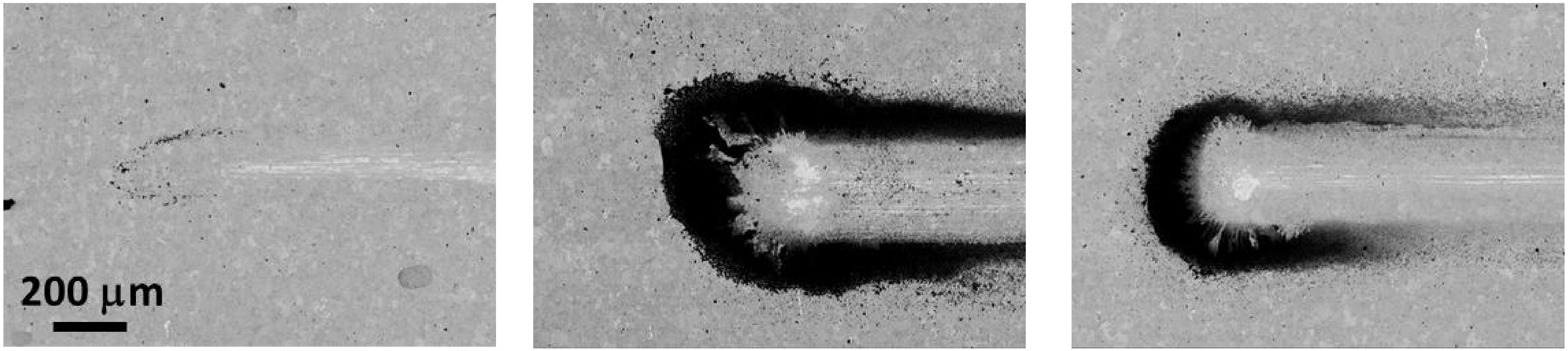




Pin-on-Flat Tests

BSE images after 100 cycles

thermal decomposition ? X No!



current + no vibration | current + vibration | no current + vibration

Initial experiments focused on the role of contact parameters in deposit formation

- current alone produced gold transfer, but very little deposit
- suspected make-break arcs during movement, leading to thermal decomposition of silicone

Deposit is created during mechanical contact of the metals, with little or no influence of current

- related to the creation of freshly-exposed metal surface by sliding

$^1H/^{13}C$ NMR Analysis of the Reaction Product

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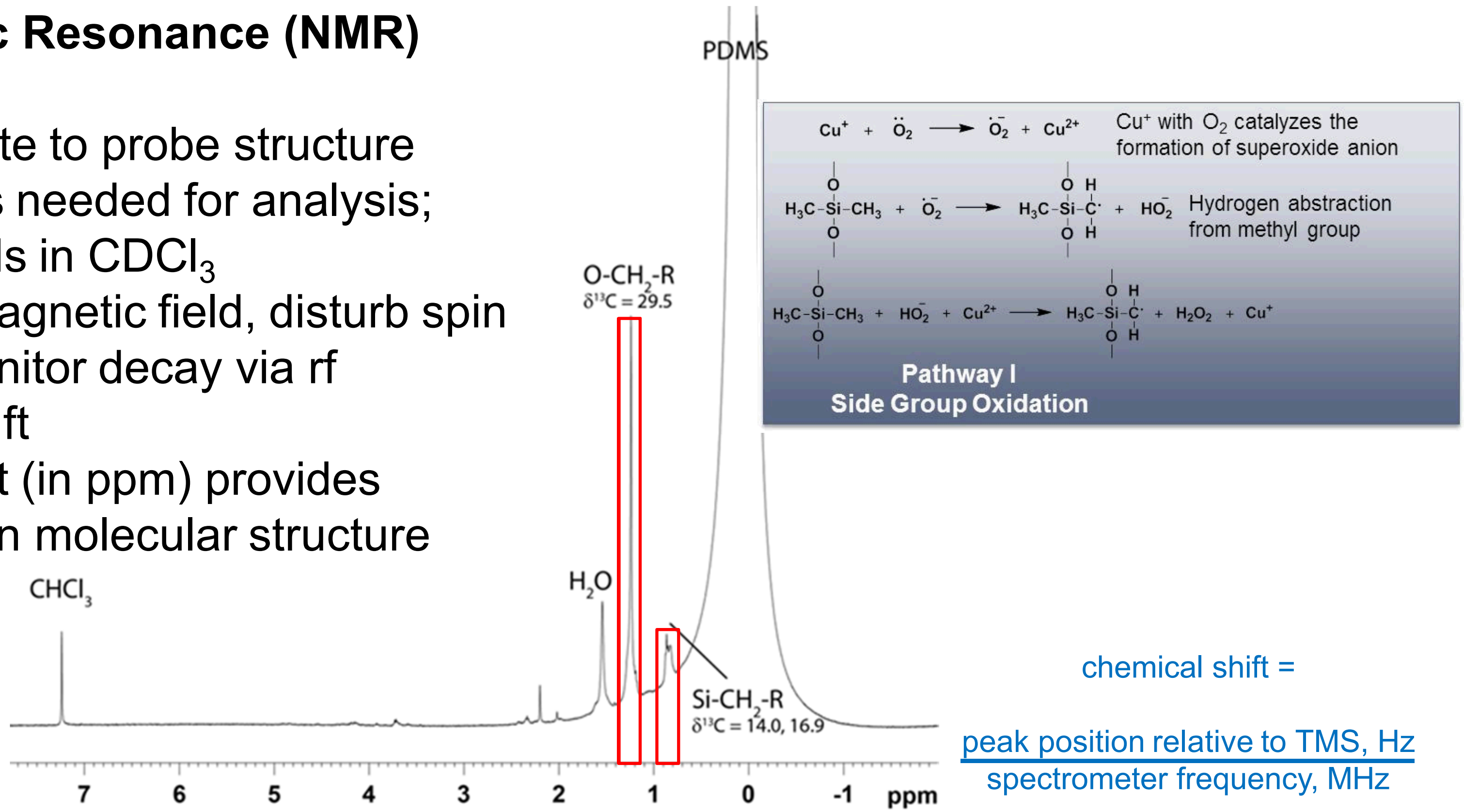
Nuclear Magnetic Resonance (NMR) Spectroscopy

- uses spin state to probe structure
- mg quantities needed for analysis; dissolve solids in $CDCl_3$
- in a strong magnetic field, disturb spin state and monitor decay via rf frequency shift
- chemical shift (in ppm) provides information on molecular structure



$$\text{chemical shift} = \frac{\text{peak position relative to TMS, Hz}}{\text{spectrometer frequency, MHz}}$$

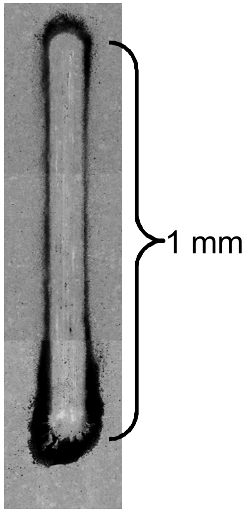
- **Product consistent with alkyl radical formation by metal catalysis**
- **Methyl side group oxidation, and free radical condensation is the dominant degradation pathway**

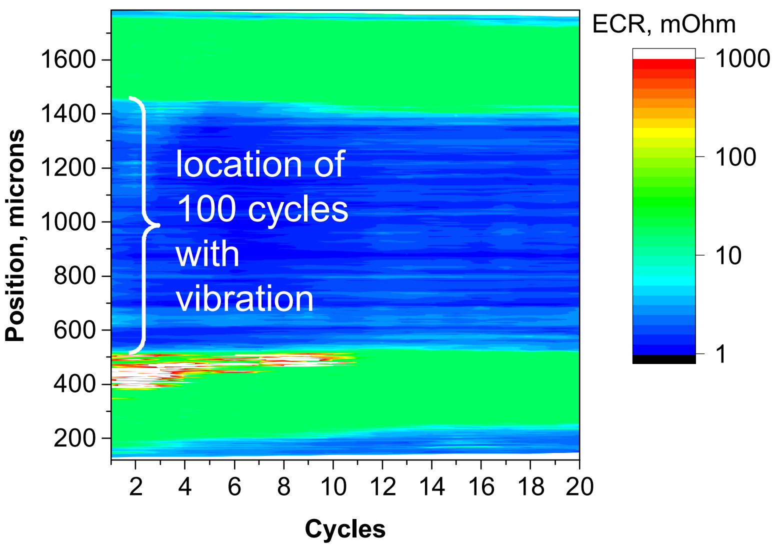
Relating Deposit to ECR Change

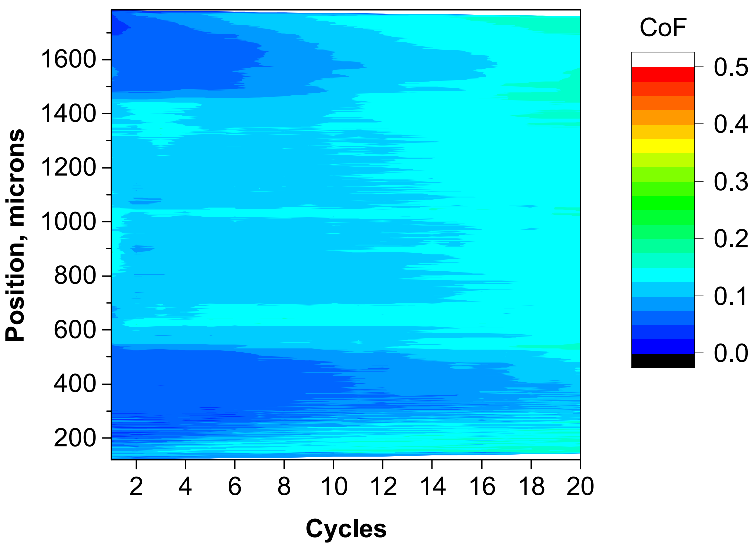
Procedure:

1. Measure CoF and ECR along 2 mm track for 3 cycles with no vibration
   a. 88 mN (9 gf) contact force
   b. 20 mA DC current, with 1.1 V open circuit limit
2. Perform 100 cycles sliding with vibration on central 1 mm
   a. 50 μm displacement amplitude orthogonal to sliding direction, 100 Hz
3. Measure CoF and ECR along 2 mm track for 20 cycles with no vibration

initial wear track with vibration

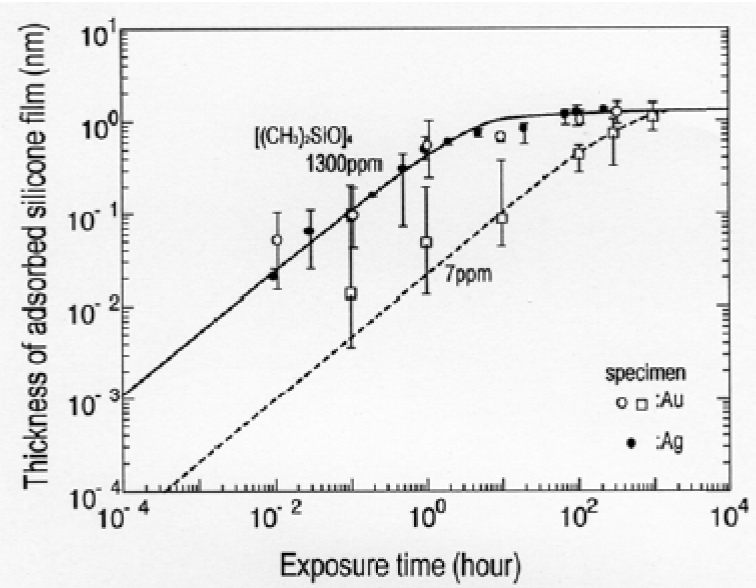




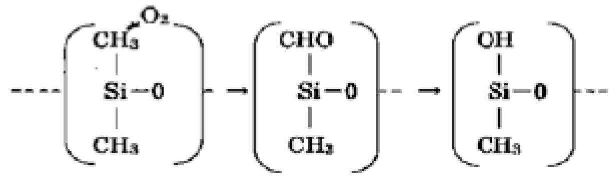


Increased ECR is observed when pin slides over deposit

Tamai proposed oxidation and crosslinking of silicones

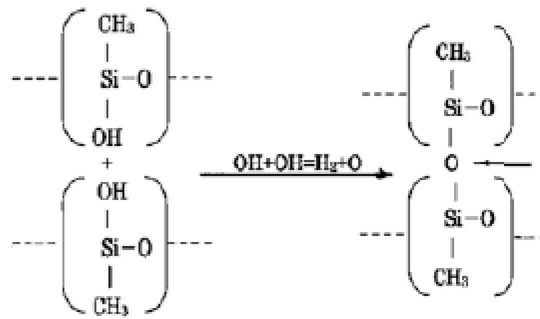


T. Tamai, "Adsorption of silicone vapor on the contact surface and its effect on contact failure of micro relays," IEICE Trans. Electronics, vol. E83-C, no.9 (2000) pp.1402-1408



$CH_3+O_2=CHO+H_2O$, $CHO+O_2=OH+CO_2$

(a) Oxidation process of CH_3

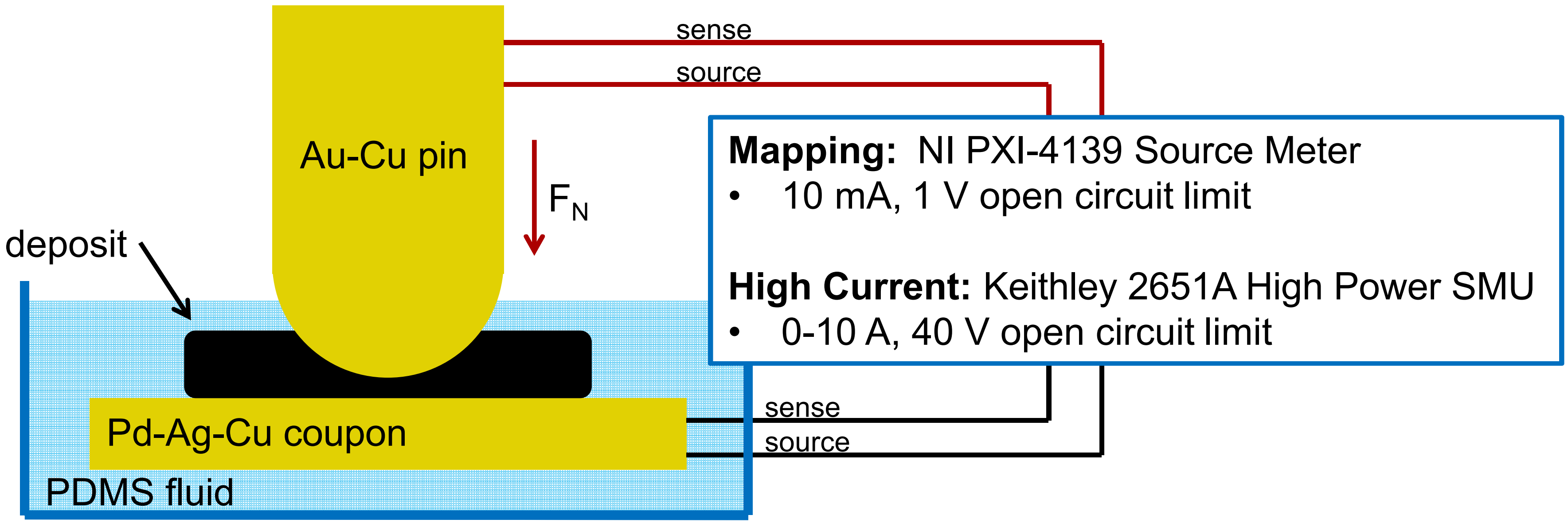


(b) Vulcanization between molecules D_4

T. Tamai, S. Sawada, Y. Hattori, "Manifold Decomposition Processes of Silicone Vapor and Electrical Contact Failure," Proc. International Conference on Electrical Contacts 2012, Beijing, China

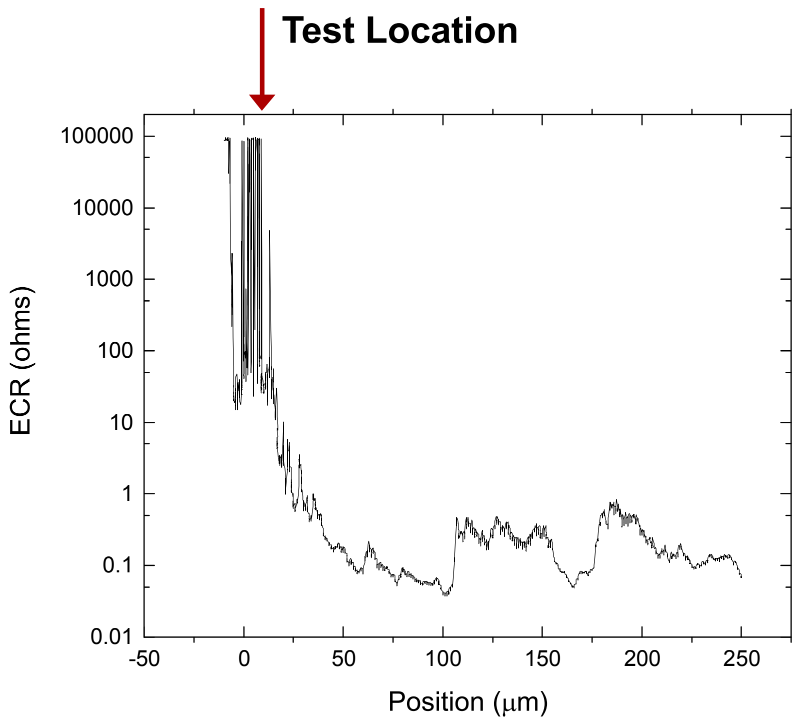
- crosslinking of siloxane due to CH_3 oxidation at "moderate" temperature

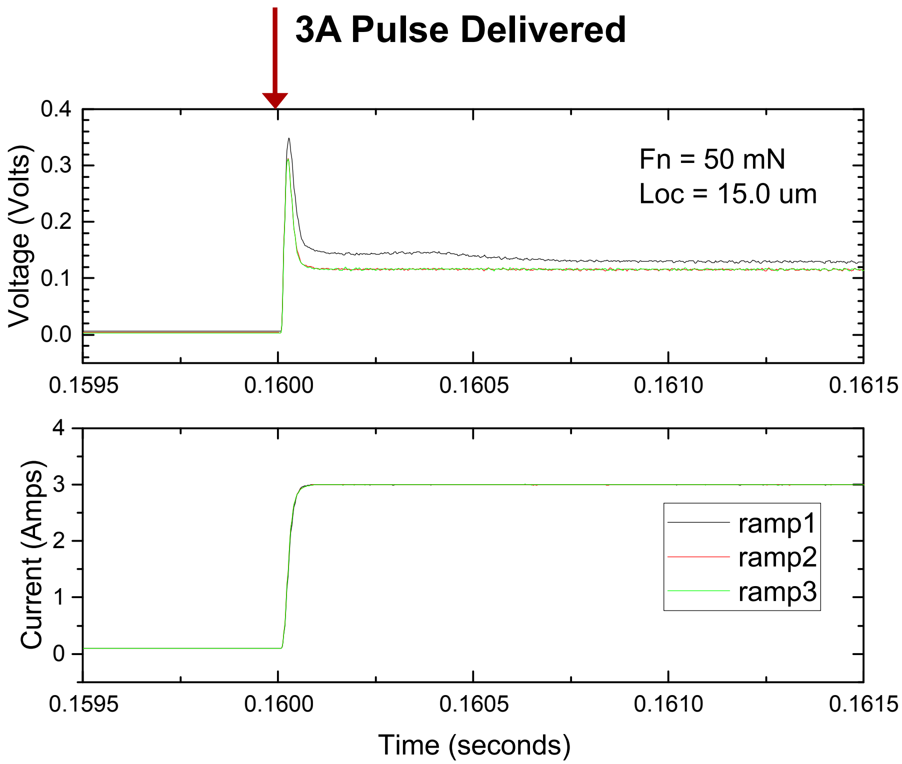
Probing Mechanical/Electrical Behavior



- map ECR near deposit using low probe current
- select location, deliver current pulses or ramps
- determine ECR versus force by ramping load on pin

Current Pulses on Contaminated Contacts





- initial voltage spike ~ 350 mV as current rises
- ECR stabilizes at ~50 mΩ

Current Sweeps on Contaminated Contacts

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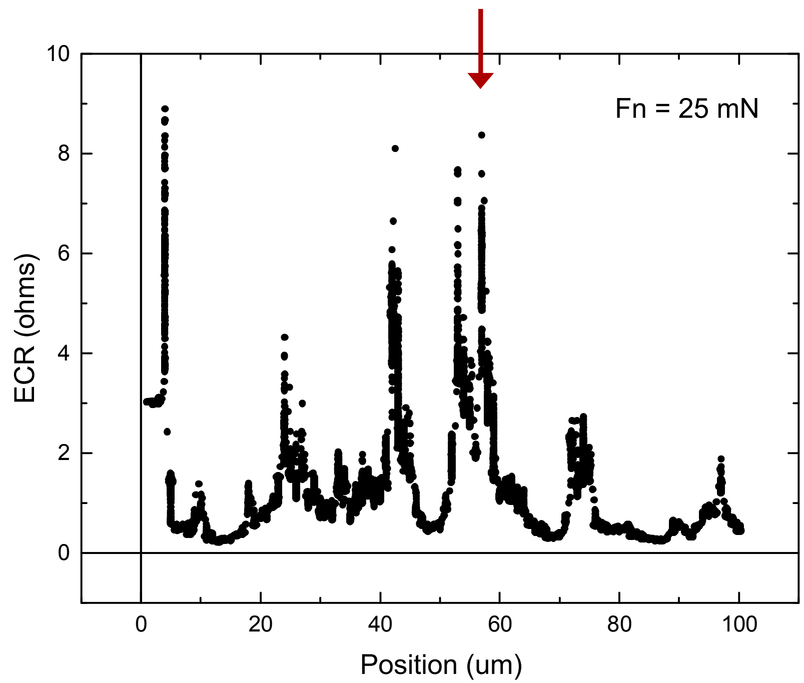
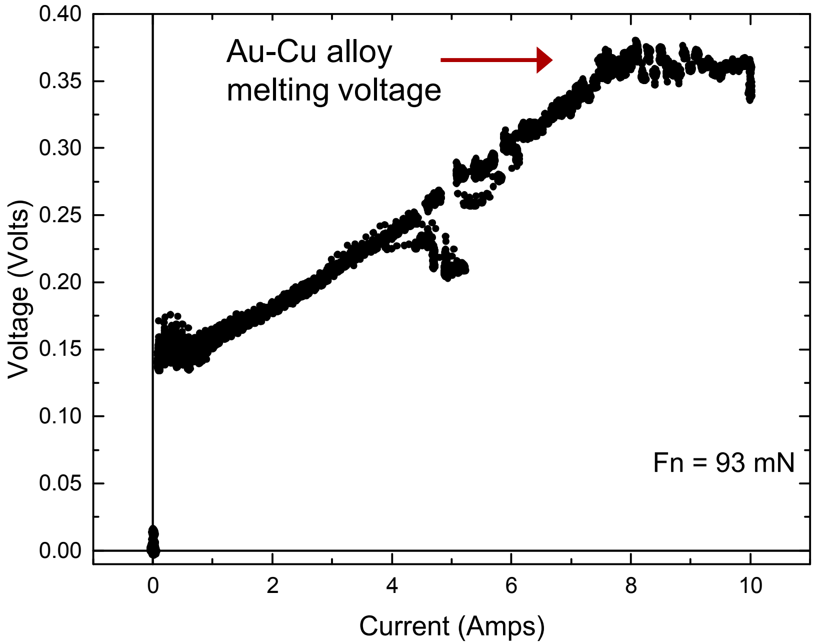


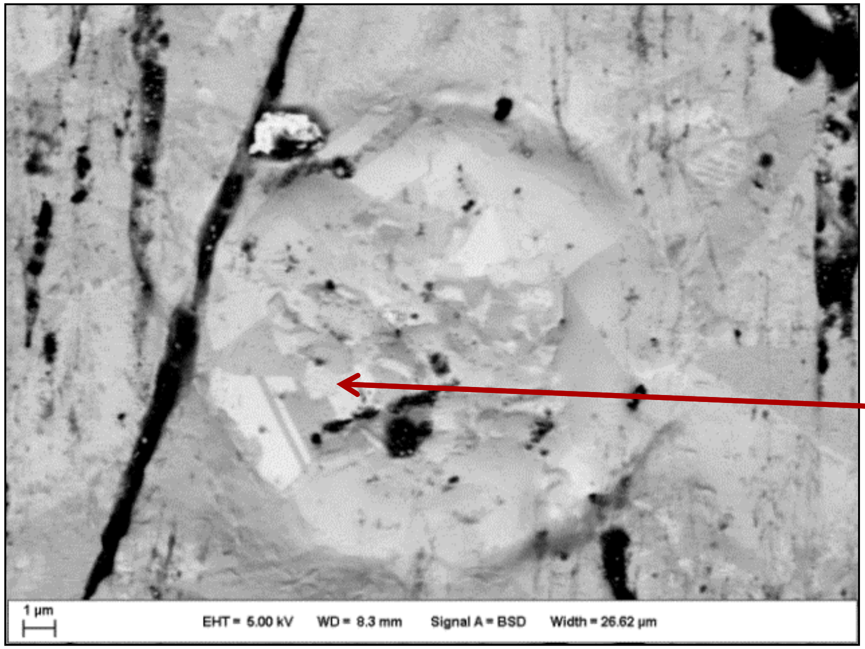


- slower current sweeps reveal an abrupt turn-on of current
- dielectric breakdown of deposit

Effect of Current on Contact Alloys

Resistance decreases after high current flow

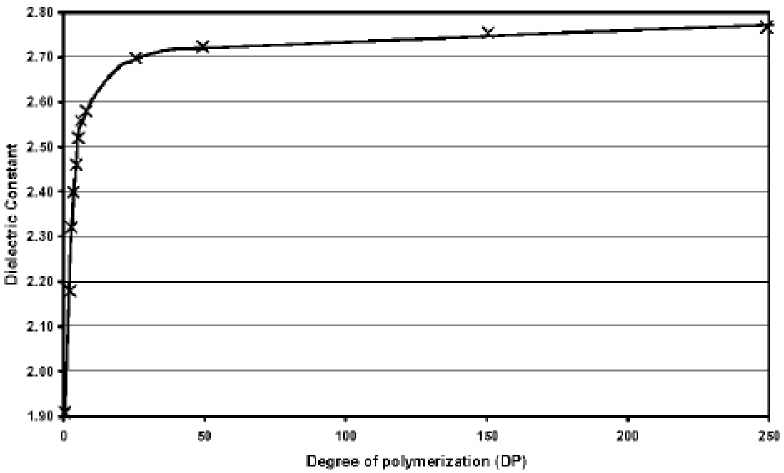
- dielectric breakdown of deposit
- localized softening/melting of contact materials



Backscattered electron channeling contrast image of Pd-Ag-Cu alloy after high current exposure

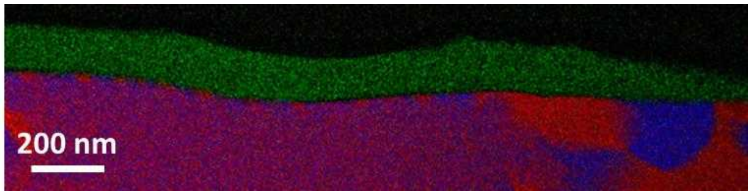
Local heating induces microstructural changes in the electrical contact alloy

Dielectric Strength of Silicones

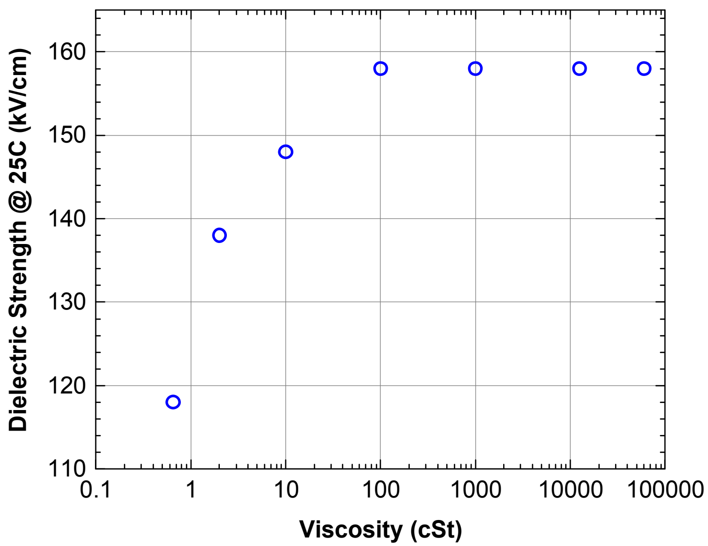


Baker, E. B.; Barry, A. J.; Hunter, M. J. *Ind. & Eng. Chem.* Nov 1946, *38*; 1117-1120.

- dielectric strength rapidly saturates with increased polymerization (viscosity) to 158 kV/cm
- suggests ~10 nm deposit thickness
- consistent with TEM observations

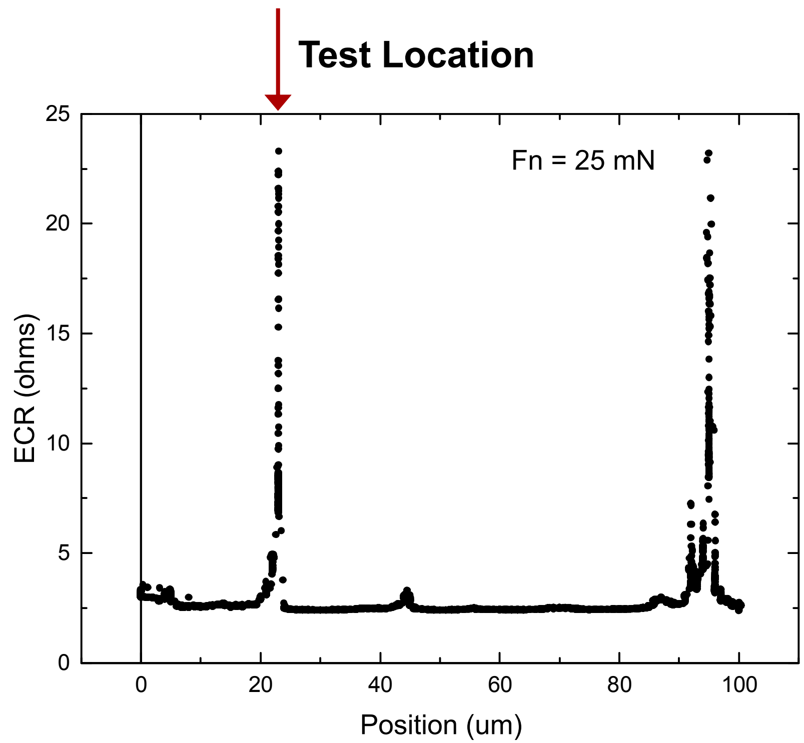


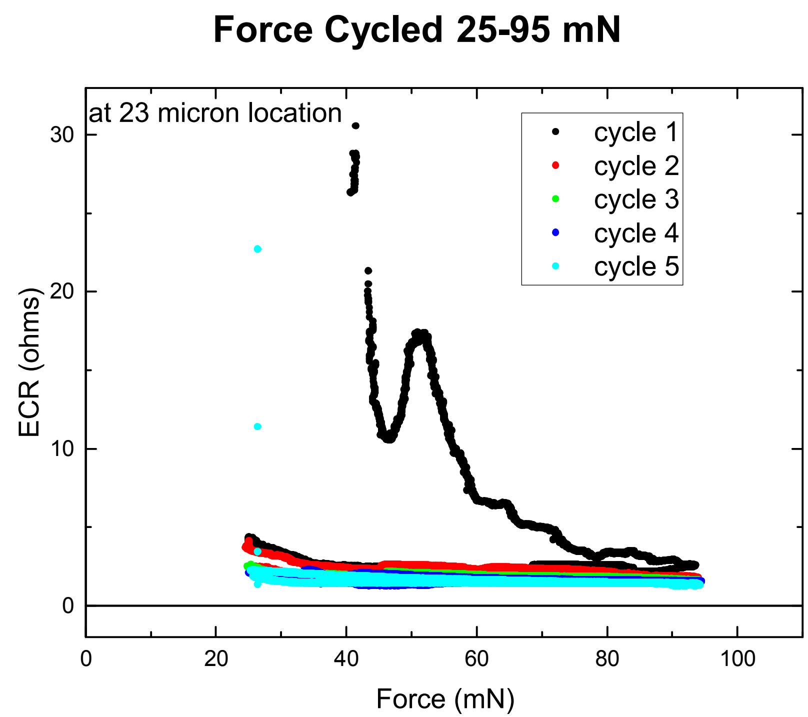
EDS/AXSIA image of a Focused Ion Beam (FIB) section through deposit (green)



Dow Corning Corp., Midland, Mich.
Data Sheets 22-931A-90, 22-926D-93, 22-927B-90, 22-928E-94, 22-929A-90, 22-930A-90

Force Sweeps on Contaminated Contacts





- first force cycle exhibits elevated ECR at low contact force
- subsequent cycles exhibit reduced ECR, indicative of mechanical displacement of deposit

Conclusions

Silicone fluid can degrade at room temperature, catalyzed by fresh metal oxide surfaces

- Cu-containing alloys require only mechanical agitation to continually displace passivating surface films and expose surface oxide
- deposit increases electrical contact resistance by orders of magnitude
- methyl side group oxidation is the dominant silicone reaction mechanism

Conduction mechanism of thick deposits

- dielectric breakdown of polymerized film
- joule heating to soften the deposit and contact alloy, changing the contact surface
- at sufficient force, mechanical contact can displace the deposit and establish low ECR conditions

Future Work

1. Understand the influence of aging on mechanical and electrical behavior of deposits
   - Does crosslinking in films continue over long duration, changing film properties?
   - Determine the kinetics of any film property changes
2. Determine the reactivity of various surfaces for deposit formation

Acknowledgments

Sandia Colleagues:

- Nic Argibay for electrical contact discussions
- Todd Alam for NMR
- Mat Celina for discussions on polymer degradation
- Bonnie McKenzie and Amy Allen for SEM and EDS

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