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Mechanically-Induced Degradation of Metallic Sliding Electrical Contacts in Silicone Fluid at Room Temperature

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***Abstract*— The degradation in electrical contact resistance of a contact pair sliding while submerged in silicone fluid has been investigated. While the contamination of electrical contacts by silicone vapors or migrating species at elevated temperature due to decomposition in electric arcs is well known, the present degradation mechanism appears to arise from chemical reactions in the silicone fluid at room temperature, catalyzed by the presence of the freshly-abraded metal surface. As a result of these reactions, a deposit containing Si, C and O forms in the vicinity of mechanical contact. The specific contact metals present and the availability of dissolved oxygen in the fluid have a dramatic influence on the quantity of reaction product formed. The chemistry of the deposit, proposed formation mechanisms, the impact on electrical contact resistance and mitigation strategies are discussed.**

Keywords—silicone; palladium; copper; friction polymer

I. INTRODUCTION

The understanding of the reaction of adsorbed species with metal surfaces to produce insulating films is still informed by the extensive work of Hermance and Egan [1], whose study demonstrated that Pt-group metals (most notably Pd) were very effective in forming adherent, insulating deposits from a vast array of different organic vapors. They found that as long as sufficient vapor pressure of the organic species was present, lowering the temperature (to -60ºC in some experiments) had no effect on deposit yield. They postulated that strong adsorption of organic molecules must strain them such that they become reactive and form higher molecular weight species, dubbed "friction polymer." Chaiken [2] concluded that charged particles emitted from rubbing surfaces must also play a role in the reaction process, since traditional catalysis inhibitors had no effect on deposit formation. Other metals in sliding contact with Pd also generate reaction products, except when Pd slides against Au, Ag, or alloys containing at least a third by weight of these metals [3], because a mechanically mixed layer forms at the interface that resists polymer formation. The reaction of liquid silicone with Pd contacts was implied [4] in an investigation of the effects of bulk fluid lubrication on contact fretting. While contact resistance increased slightly with silicone, the behavior was superior to that with unlubricated sliding. More reaction product formed when sliding in fluids, but the deposit was softer and less adherent so that it could be penetrated or displaced. This work did not explicitly state whether an insoluble deposit formed when Pd contacts slid in silicone.

The effects of silicone vapors on electrical contacts were subsequently studied extensively. Studies of the effect of adsorbed silicone on electrical contact performance [5-7] showed that as little as 10 ppm of silicone in the atmosphere was sufficient to degrade contact performance, and this degradation was attributed to the formation of SiO_2 and a "black product" in the contact resulting from thermal decomposition of the adsorbed silicone. More recently, it was found [8] that when operated in atmospheres containing ozone or amine with up to 1300 ppm of silicone vapor, the contact life of relays was prolonged compared to that in air due to the reactions between the silicone vapor and these species, which effectively reduced the amount of silicone adsorbed on contact surfaces.

The tribology literature is similarly replete with references to the interaction of silicone fluids with metal surfaces, particularly associated with their use as high temperature lubricants. As early as 1961 [9], means of delaying "gelation" of silicone fluid when exposed to iron, copper, and cerium in the temperature range of 200-300°C were investigated. In tests of lubrication of mild steel with alkylated silicones [10], researchers noted that a polymeric film formed and remained attached to the steel surfaces during tests at 120°C and above. In a landmark paper on the decomposition of silicones exposed to metal surfaces, [11] observed that the polymer reaction film formed at temperatures as low as 90°C, that it did not form on gold or platinum surfaces, and that it did not form if the metal and fluid were covered by nitrogen or argon gas. No film formation was observed below 90°C, and silicone decomposition was believed to be catalyzed by the metal oxide and involved attack of the highly ionic Si-C bond by O_2^- ions.

The existing literature therefore demonstrates that organic species react on some metal surfaces during sliding, due to the catalytic effect of the metal surface and potentially charged particle emission, to form insoluble products that increase contact resistance. Silver, and to a lesser extent gold, resist deposit formation, while deposits form readily on Pd and other Pt-group metals. Silicone molecules adsorbed from the atmosphere can thermally decompose in arcing electrical contacts to form SiO_2 and carbonaceous films that increase electrical contact resistance. Polymerization of silicone fluids on some metal surfaces at much lower temperatures than electric arcs, but above room temperature, is associated with oxidative attack of the silicon-methyl bond to create radicals that polymerize to an insoluble reaction product. To date, the creation of insoluble reaction products on non-arcing electrical contacts sliding in bulk silicone fluid at room temperature has not been connected with electrical contact resistance degradation.

The present work describes a room temperature reaction between silicone fluid and some metal surfaces that proceeds after the creation of freshly abraded metal surface, and appears to be catalyzed by the presence of a metal oxide. Like the films formed by electric arcs or exposure to non-noble metals at 90°C and above, this film can significantly increase the electrical resistance between metal contacts.

Silicone fluid was chosen as a damping medium for an accelerometer due to its low viscosity index and its long term thermal and chemical stability. The accelerometer is required to operate at temperatures between -55°C and 85°C, and the low viscosity index reduces the performance variation over this temperature range. It employs electrical contacts mounted on an internal mass, which moves against a spring in response to the local acceleration field. The moving contacts engage stationary pins on either end of the device in a pin-socket contact configuration, such that normally closed contacts open, and normally open contacts close, to indicate whether the device has been exposed to acceleration. Once exposed to acceleration, the device latches in place to store that physical state. The contacts on the moving mass are therefore always in contact with stationary contacts on one end of the device or the other. During development and testing of this device, an optically black, low atomic number deposit formed on the electrical contact surfaces in and near the location of mechanical contact. This film was sufficiently abundant on some devices to result in elevated electrical contact resistance. While the deposit could be found along the entire sliding path between the electrical contact metals, devices exposed to mechanical vibration exhibited a "halo" of the deposit around the contact location that extended beyond the mechanical contact area. The present work describes efforts to understand the formation mechanism of this film, and strategies to mitigate its formation and impact on electrical contact resistance for contacts submerged in silicone fluid.

II. EXPERIMENTAL PROCEDURES

A. Materials

The sliding electrical contacts in question use precious metal alloys that can be heat treated to impart high mechanical strength. The stationary pins were fabricated from Paliney-6$^{®}$ and Paliney-7$^{®}$, while the moving leaf spring contacts were formed from Neyoro-G$^{®}$ [12]. The compositions of the alloys are shown in Table 1. The contact force between the springs and pins was nominally 88 mN per contact, provided by deflection of the Neyoro-G$^{®}$ springs when the contacts were engaged. As can be seen by the composition ranges, the Paliney$^{®}$ alloys contain a majority of Pd, Ag and Cu, with significant differences in Pt and Au content. The Neyoro-G$^{®}$ is a gold-based alloy containing Pt and Ag. All of the contact materials contain about 15 wt.% Cu.

TABLE 1. COMPOSITION OF ELECTRICAL CONTACT MATERIALS

	wt. %, min-max						
	Pd	**Ag**	**Cu**	**Pt**	**Ni**	**Zn**	**Au**
Paliney-6	43-45	37-39	15.5-16.5	0.8-1.2	0.8-1.2	0-0.01	NA
Paliney-7	34-36	29-31	13-15	9-11	NA	0.5-1.5	9-11
Neyoro-G	NA	4.0-5.0	13.5-15.5	8.0-9.0	NA	0.7-1.3	70.5-72.5

The silicone fluid used in this device was polydimethylsiloxane (PDMS) with 20 cSt viscosity at 25°C [13]. The accelerometers were filled with silicone fluid at room temperature, and a small air bubble about 10% of the free volume was included before the device was sealed, to allow for thermal expansion of the fluid when operated at elevated temperature.

B. Diagnostics

The base diagnostic technique used to detect material transfer and deposit formation was scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS). For analysis of low atomic number deposits, EDS maps were acquired with a beam energy of 5 kV to improve surface sensitivity. EDS maps were processed with a multivariate analysis algorithm (Automated eXpert Spectral Image Analysis, or AXSIA) to identify the spectral signatures and spatial maps of the species present [14]. Transmission electron microscopy (TEM) was employed to further investigate the thickness and composition of low atomic number deposits on contact surfaces. Electron transparent sections were extracted from deposits on electrical contact surfaces using a focused ion beam (FIB), and imaged in the TEM with EDS. The AXSIA analysis was also performed on EDS spectral data from the TEM sections.

The effects of contact parameters on electrical contact resistance (ECR) for these materials were investigated using a custom electrical contact pin-on-flat "ECR tribometer." In this device, a 1.6 mm radius hemispherically-tipped pin of one contact metal was loaded and slid against a polished flat of another contact metal. The flat sample was mounted in a fluid cup that held approximately 5 ml of the silicone fluid so that the sliding interface was submerged during testing. Both surfaces were polished to a 20 nm Ra surface finish using metallographic techniques, and then cleaned ultrasonically in acetone and alcohol, then dried in flowing nitrogen. Four-wire electrical contact resistance measurements could be made between pin and flat using a Keithley model 2401 source meter. ECR measurements were made in 4-wire mode with 20 mA DC sourced current and 1.1 V DC open circuit voltage limit. Since vibration appeared to be a critical factor in deposit formation, the ECR tribometer was modified so that a small speaker mounted to the base could be energized (148 Hz) to intentionally induce vibration and intermittent contact between the pin and flat during sliding. With vibration off, smooth sliding occurred with constant contact between the pin and flat. The entire device was housed in a glovebox to permit control of the test atmosphere. Tests were performed in laboratory air (8-40% relative humidity) or in dry nitrogen (<10 ppm O_2 and <200 ppm H_2O). The tests used an applied load of 50±5 mN between the pin and flat, a sliding speed of 1 to 3 mm/s, and testing proceeded as follows. During initial investigation of conditions required to form the deposit, wear tracks 1 to 2 mm long were formed in air while varying the presence of simultaneous mechanical vibration and electrical current. Subsequent tests were performed to investigate the influence of the deposit on ECR. In these tests, a 1 mm long wear track was first created using vibration between pin and flat. Then, without induced mechanical vibration, the length of the wear track was extended by 0.5 mm on either side and multiple sliding passes were applied with the addition of a probe current to evaluate deposit buildup as a function of ECR throughout the entire length of the original wear track. In this way, the ECR was measured as the pin traversed the previous wear track created with vibration including the effects of multiple wear passes over the deposit.

Reactions between metals and silicone during mechanical contact alone were further investigated using a "tumble test." In this test, a polyethylene bottle of 200 ml volume was filled approximately 20% full with commercially pure copper or 440C stainless steel balls 3 mm in diameter. Silicone fluid was added to the bottle until the balls were just submerged. The bottle was then sealed and made to tumble end over end lengthwise in a rotary tumbler at 6 rev/min. At this speed the balls would fall from one end of the bottle to the other as it rotated, created a high rate of mechanical contact events between the metal balls in the presence of the silicone fluid.

Analysis of the silicone fluid from accelerometer, pin-on-flat, and the tumble test was performed using nuclear magnetic resonance (NMR) spectroscopy. ^{29}Si solution NMR was performed to determine the average molecular weight (MW) using end group analysis. ^{1}H solution NMR was used for the determination of water concentration and organic impurities. ^{1}H and ^{13}C solution NMR of the reaction product extracted from the tumble test was performed by centrifuging the silicone fluid to concentrate any solids, removing the bulk silicone fluid, and then dissolving the deposit in $CDCl_3$ (deuterated chloroform) prior to analysis. The NMR spectra were obtained on a Bruker Avance 500 instrument at 500 MHz, using standard experimental conditions.

III. RESULTS AND DISCUSSION

A. Pin-Socket Contacts

A section from one of the stationary pins extracted from the accelerometer is shown in Fig. 1. The figure shows transfer of gold and copper from the Neyoro-G® spring contact down the axis of the pin, and a deposit containing Si, C and O in and around the wear track. The deposit extends in a wide halo around the position where the device was latched. A FIB cross-section of the deposit and near-surface metal was extracted from a region in the halo, outside but adjacent to the wear track. Fig. 2 shows the EDS spectral image from the TEM section, and again indicates a deposit containing Si, C and O on the pin surface. This section indicates that the deposit can be over 100 nm thick in some locations.

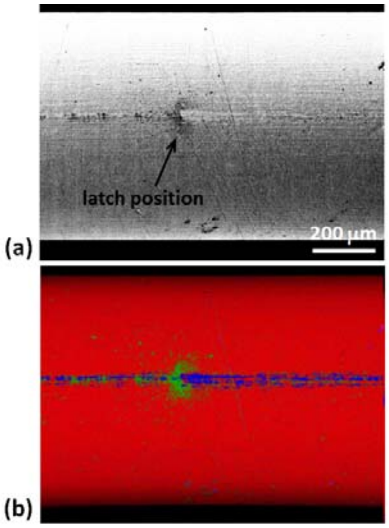

Fig. 1. Backscattered SEM image (a) and AXSIA image of the EDS maps (b) for a Paliney-7 pin contacted by a Neyoro-G spring contact. In the AXSIA image, red = Paliney-7 matrix, blue = transferred Au and Cu from the Neyoro-G, and green = a Si, C, O deposit.

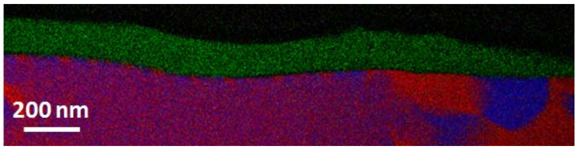

Fig. 2. AXSIA-processed EDS image from the TEM, of a FIB section through the deposit in a halo. The deposit is over 100 nm thick in some locations. In the AXSIA image red and blue represent phases in the Paliney-7 pin, and green = Si, C, O.

B. Pin-on-Flat Tests

The sliding contacts in the accelerometer were replicated in the ECR tribometer in an effort to understand the formation mechanism of deposits. In this case, a Neyoro-G® pin was slid over the Paliney-7® flat while submerged in fluid. Since the thermal decomposition of silicone fluid in electric arcs is well known, the initial impetus for inducing vibration between the pin and the flat was to generate intermittent contact, expecting that contact make-and-break arcs would result in the formation of SiO_2 and black deposits on the contact areas as reported in the literature [5-7]. Fig. 3 shows backscattered SEM images of the terminus of the wear tracks from tests with current and vibration applied simultaneously, current without vibration, and vibration without current. These experiments showed formation of a black (low atomic number) deposit when vibration was present. A small amount of deposit was formed with current and no vibration, but the quantity of deposit formed when vibration was present without current clearly demonstrates that current was not required for the deposit to form. The amplitude of vibration at the pin-flat interface is not known, but one explanation for the ability of vibration to produce deposits from silicone fluid is that high sliding speed at the pin-flat interface creates surface asperity temperatures

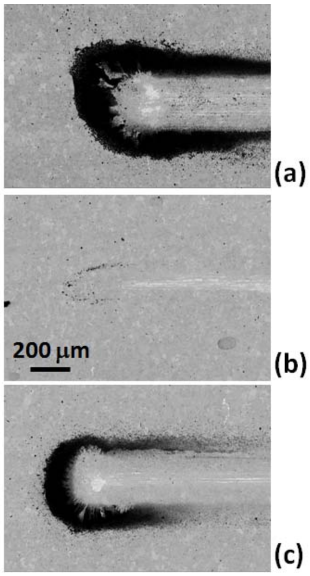

Fig. 3. Trailing edge of wear tracks between a Neyoro-G pin and a Paliney-7 flat at 49 mN applied load. Track (a) was with current and vibration, track (b) with current only, and track (c) with vibration only.

high enough to cause thermal decomposition of the fluid. A pure gold pin and flat subjected to identical experiments did not exhibit the formation of low atomic number deposits in the vicinity of the wear track, suggesting that the reactions are not due to thermal decomposition of the fluid, nor to shear-induced degradation of the fluid at high shear rates at the interface. Rather, it appears that the deposit is formed by processes similar to those responsible for the reaction of organic vapors on contact surfaces to form friction polymer, described in the literature [1].

The composition of the worn area on the Paliney-7® flat was examined with EDS and AXSIA image analysis, and the results are shown in Fig. 4. Transfer of gold and copper can be seen near the center of the wear track. Surrounding this area of transfer, in the area outside the mechanically deformed area, the low atomic number deposit was found to be composed of silicon, carbon and oxygen. This deposit composition is very similar to that on the accelerometer pin surfaces shown in Fig. 1. Unlike the literature reports of gold-based alloys transferring to palladium surfaces creating an interface that resists deposit formation [3], the gold and copper transfer in this case did not suppress silicone degradation.

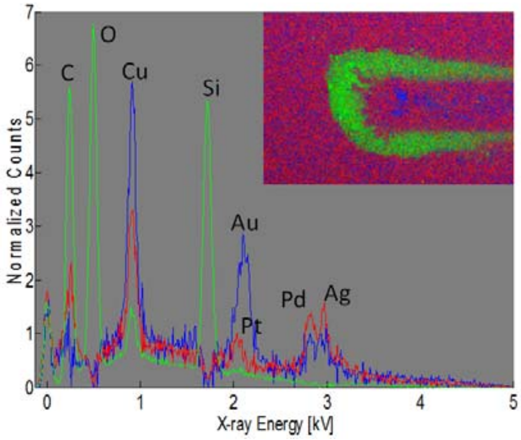

Fig. 4. AXSIA EDS spectral image from a wear track formed by a Neyoro-G pin sliding on Paliney-7 with vibration and current present. Spectral image components correspond to: red=Paliney-7 coupon, blue=transfer of Au and Cu from Neyoro-G, and green=Si, C, O reaction product.

To investigate the impact of the deposit formed at Neyoro-G® and Paliney-7® contacts on electrical contact resistance, tests were conducted in the ECR tribometer over a 1 mm long wear track with vibration and no current. In previous tests these conditions produced deposit on the Paliney-7® surface, like that shown in Fig. 3. Following this deposit generation step, electrical contact resistance measurement was made over a 2 mm long track that included the previous worn area, as described in the experimental section. Fig. 5 displays the contact resistance measured over the 2 mm long wear track as a function of repeated cycles of sliding over this track. The figure shows that ECR is elevated at the ends of the wear track previously created with vibration. This is consistent with previous literature reports of ECR increase at the ends of sliding paths, due to the accumulation of deposits by mechanical wiping in these areas [15]. The figure also indicates that repeated sliding of the pin over the wear track, under conditions that do not rapidly form the deposit, result in the contact resistance returning to background levels. In the test illustrated, the deposit is worn through after approximately 15 cycles of sliding over the same wear track.

C. *Deposit Generation and Characterization*

NMR spectroscopy on samples of the bulk fluids from both the accelerometer tests, and the pin-on-flat tests, revealed no significant differences in the silicone fluid before and after the sliding experiments. The recovered commercial 20 cSt PDMS fluid contained a hydroxyl-terminated and a minor cross-linked chlorinated silicone species in concentrations of up to a few hundred ppm. Discussion with the supplier revealed that these are likely residues from the manufacture of the silicone fluid. No additional degradation products from reaction of the fluid with the metal surfaces in the presence of dissolved oxygen were found.

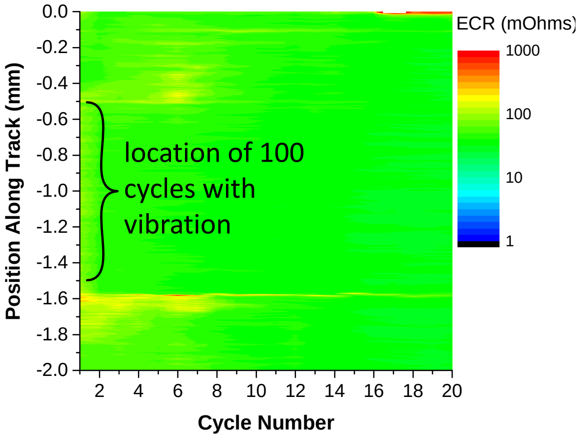

Fig. 5. Electrical contact resistance for a Neyoro-G pin sliding over a Paliney-7 flat, after 100 cycles with vibration and no current on the central 1 mm of the track.

Since the material transferred from the Neyoro-G pin to the wear track did not suppress the reaction of the surface with silicone fluid, it was suspected that copper in the deposit was responsible for the enhanced reactivity of silicone. In addition, a method was desired to produce large (mg) quantities of the reaction product for chemical analysis to yield information about the chemical pathways responsible for deposit formation. Since mechanical contact alone was responsible for deposit formation, and copper in the transfer layer was thought to catalyze deposit formation, a high frequency of copper-on-copper contact in silicone fluid was expected to result in significant quantities of the product for analysis. This led to the development of the "tumble test" described above. Both copper and stainless steel balls were tumbled in silicone fluid for 12 hours. Within just two hours, the silicone fluid in the bottle containing the copper balls began to darken, primarily due to abraded metallic particles. After 12 hours of mechanical contact, the bottles appeared as shown in Fig. 6. The silicone fluid in the bottle containing stainless steel balls had not visibly changed, but fluid in the bottle containing copper balls had turned visibly black (Fig. 6). Samples of the fluid from each bottle were spun in a centrifuge to separate solid products from the fluid. This resulted in solids collecting in the bottom of the centrifuged ampule of fluid tumbled with copper balls, as shown at the bottom of Fig. 6. The ^{29}Si NMR spectroscopy of the liquid phase, extracted from above the solids from both ampules, reveals there are distinct but only small changes in the silicone fluid following tumbling with both the stainless steel or copper balls. Most notable was the presence of Si-O-Si cross-linking revealed in the ^{29}Si NMR by the presence of new Q_3 silicon species (CH_3SiO_3-) observed between $\delta \sim -65$ and -67 ppm. No solid deposits were collected by centrifugation from the fluid tumbled with the steel balls. This further supports the conclusion that interface temperature during contact, and shear gradients in the fluid layer, do not cause the solid deposit formation. The centrifuged solids in the fluid tumbled with copper balls, however, consisted of a layer of copper particles at the bottom of the ampule, with a layer of black deposit on top of the copper particles. The black deposit was primarily in the form of solid particles that were not soluble in the silicone fluid. A sample of this solid was carefully removed and dissolved in $CDCl_3$ so that it could be analyzed by NMR.

Fig. 6. Bottles from the tumble test containing stainless steel balls (left) and copper balls (right), after tumbling for 12 hours. Samples of the fluid were centrifuged in the vials below to extract solid particles for analysis.

The results of $^1H/^{13}C$ NMR spectroscopy on the black deposit are shown in Fig. 7. In addition to the large peak corresponding to the base PDMS methyl groups, two peaks were found that correspond to cross-linked products resulting from the participation of methyl radicals in the fluid. The presence of radical induced cross-links in the reaction product is consistent with a reaction mechanism that involves oxidative degradation of the PDMS molecule. This can occur as direct oxidation of the silicon atom perhaps via involvement of superoxide anions (oxygen activation with Cu^+) or free radical attack (hydrogen abstraction) on the methyl side group, peroxidation and follow-up oxidative chemistry. Radical condensation and associated cross-linking would then lead to the formation of a high molecular weight insoluble product, as observed on contacts. More importantly, since no large concentration of cross-linked species occurs in the bulk silicone fluid (also supported by GPC analysis which could not detect molecular weight broadening), the degradation process must be very heterogeneous in nature and chemically very efficient within a local domain. Extensive cross-linking occurs directly at the Cu metal surface and is proposed to result in the contact film formation. Additional chemical characterization is underway.

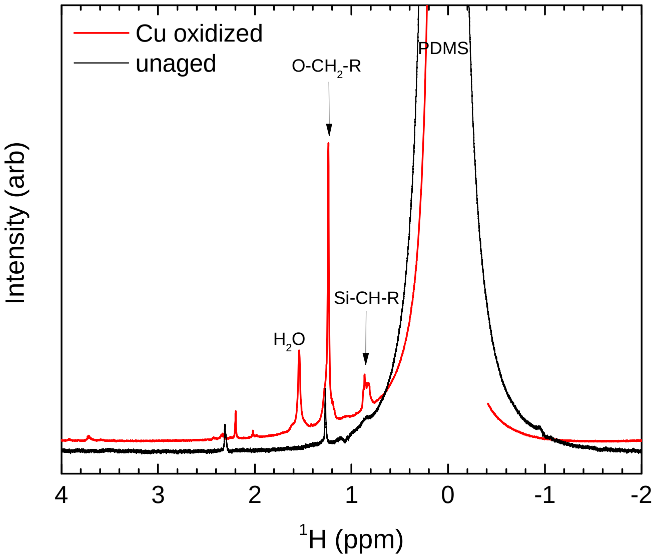

Fig. 7. ^{1}H NMR of the black deposit created by mechanical contact of copper balls in the tumble test. Peaks to the left of the main PDMS peak (arrows) indicate the formation of radical-induced cross-linked species in the silicone fluid.

IV. CONCLUSIONS

Sliding of electrical contact alloys containing Pd, Ag, Cu and Au operating submerged in silicone fluids have been investigated. A deposit containing silicon, carbon and oxygen is formed on contact surfaces after sliding. The deposit was reproducibly created in the laboratory by the mechanical activation of the sliding surfaces, and did not require the presence of current or electric arcs to form. This behavior is similar to the formation of friction polymers on palladium surfaces from organic vapors reported in the literature [1], although this reaction mechanism for bulk silicone fluids with electrical contact surfaces has not previously been reported. Contrary to literature reports of friction polymer formation from organic vapors, however, having one of the contacting bodies high in gold content transfer to the palladium alloy surface did not suppress the reaction product formation with the silicone fluid. This may be due to the presence of copper in the transferred material. The reaction product appears to collect outside the wear track, but is probably formed inside the wear track and displaced through mechanical action to the sides and ends of the mechanically abraded area. Measurements of electrical contact resistance on deposits previously created on flat coupons indicate that the contact resistance can be orders of magnitude higher on the ends of the sliding path covered by deposits, compared to the bare metal surface.

The hypothesis that copper in the transfer layer can induce reactions in the silicone fluid was verified by tumbling copper balls in the presence of silicone fluid. Mechanical contact of the copper surfaces produced a black, insoluble reaction product in the silicone fluid. Extraction and NMR analysis of this deposit revealed the presence of radical-induced cross-linked degradation products in the PDMS, consistent with oxidative degradation of the fluid. The NMR data suggest that these radicals condense and cross-link to form the insoluble reaction product at the metal surface. From a chemical point of view, oxidative PDMS degradation occurs in small domains, presumably where the presence of copper catalysis (different oxidation states), dissolved oxygen and PDMS facilitate highly localized favorable reaction conditions. This may also involve additional free radicals introduced by mechanical friction. No reaction product was observed on pure gold surfaces subjected to sliding contact, which suggests that deposit formation from sliding contacts in silicone fluid can be mitigated by plating contact surfaces with a thin layer of gold, so that sliding takes place at a gold-gold interface.

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