

Hydrophobic Recovery of Polydimethylsiloxane Elastomer Exposed to Partial Electrical Discharge

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The hydrophobic recovery of polydimethylsiloxane elastomers was studied after exposure to partial electrical discharge. Silicone elastomers that were thoroughly extracted of free oligomeric impurities as well as those deliberately contaminated with low molecular weight (LMW) silicone fluids were used for these studies. Contact angle and X-ray photoelectron spectroscopy revealed that the recovery rates of the oxidized extracted samples are strongly influenced by the applied voltage, humidity, and aging condition. The recovery rates increase considerably as the applied voltage and the humidity during discharge increase. Remarkably, the oxidized samples stored under high vacuum (10^{-7} Torr) exhibit lower recovery rates than those aged in air. Free silicone fluid, when added to the elastomer, affects the recovery rate as well; however, significant recovery is seen even without any added fluid. These results imply that the LMW species that are formed *in situ* during electrical discharge are sufficient to cause the hydrophobic recovery of oxidized PDMS elastomers. © 2000 Academic Press

Key Words: polydimethylsiloxane; PDMS; partial electrical discharge; low molecular weight fluid effect; hydrophobic recovery.

I. INTRODUCTION

Silicone elastomers are currently replacing conventional inorganic ceramics in high-voltage applications because of their low weight, good electrical properties, and excellent water repellency (1).

When silicone elastomers are in service, the initial hydrophobic surface can be changed to a hydrophilic surface by partial electrical discharge or dry band arcing. The resultant hydrophilic polymer promotes spreading of water on its surface, which is conducive to the formation of dissoluble pollutants. As current leakage increases through that film, the evolved heat results in numerous local dry bands on the surface accompanied by water evaporation. Tracking or erosion may then occur as a consequence of the more severe arcing activity generated across the dry bands. This intense dry band arcing may also lead to surface degradation and finally flashover (2–4). It is therefore desirable to avoid spreading of water on the surface because it increases power dissipation from the overhead lines. It is well known that

the hydrophilic surface created by partial discharge regains its hydrophobicity partially or fully over time (5–8). This change from a hydrophilic surface to a hydrophobic surface, often referred to as “hydrophobic recovery,” is usually explained by the following mechanisms (9): (a) reorientation of polar groups from the surface to the bulk phase or reorientation of nonpolar groups from the bulk to the surface, (b) diffusion of LMW silicone fluid from the bulk to the surface, and (c) condensation of the surface hydroxyl groups.

Owen and co-workers (6) found that a thin wettable, brittle silica-like phase is formed after partial discharge of PDMS, which could prevent the reorientation of the polymer chains. These authors concluded that the diffusion of unoxidized LMW silicone fluid through the silica-like layer is an important mechanism of hydrophobic recovery (mechanism b). This process might be facilitated by cracks in the silica-like layer. They also suggested that a mild oxidation condition produces uncracked surfaces, with only a few of the polymer side groups oxidized. Since no crosslinking occurs, the chains in the network could reorient (mechanism a). The results of Owen and co-workers are consistent with those published recently by Hillborg and Gedde (7), who found that mechanically deformed specimens after partial discharge recover at a faster rate than the comparable untouched specimens. This observation is consistent with the presence of cracks in the thin silica-like layer allowing the migration of the LMW silicone fluid to the surface. Based on X-ray photoelectron spectroscopy (XPS) results obtained after partial discharge to silicone elastomers, Tóth *et al.* (10) suggested that the diffusion-controlled migration plays a more important role in the recovery process than the reorientation of the newly formed polar species from the surface toward the bulk. They concluded that roughly one-third of the recovery is due to the reorientation of the polymeric chains (mechanism a) and about two-thirds due to the diffusion of the LMW PDMS chains (mechanism b). On the other hand, using a combination of static secondary ion mass spectroscopy (SSIMS), XPS, and infrared spectroscopy on O^{18} plasma-modified PDMS rubber, Morra *et al.* (11) proposed that the hydrophobic recovery is due to burial of polar groups into the bulk (mechanism a) accompanied by the condensation of surface silanols and consequent cross-linking in the contact-angle-probed layer (mechanism c).

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Stewart and Urban (12) utilized photoacoustic Fourier transform infrared spectroscopy and dynamic mechanical analysis to understand the chemical composition of the surface and the mechanical properties of silicone elastomers after plasma treatment. They found that the surface crosslink density is not increased as a result of the highly energetic plasma environment but mostly new surface species are produced on the silicone elastomer with shorter chains. Other researchers (13–15) also reported the formation of these relatively short fragments by the scission of long polymer chains and proposed that the corresponding diffusion of the LMW species to the surface is responsible for hydrophobic recovery.

Even though there is a widespread belief that fluid migration from the bulk to the surface is a key mechanism for hydrophobic recovery (2, 7, 9, 10, 13–14), there are few papers that describe the effect of preexisting fluid in the bulk on the restructuring of the oxidized silicone elastomer. The purpose of this paper is to better understand the influence of the preexisting LMW silicone fluids on the hydrophobic recovery process of the oxidized silicone elastomer. We prepared silicone elastomers containing silicone fluids of low viscosity and then compared the recovery rates of these fluid-containing samples to those of the extracted samples after partial electrical discharge. Contact angle measurement and angle-resolved XPS were used to characterize the hydrophobic recovery of the PDMS elastomer on aging after exposure to partial discharge.

II. EXPERIMENTAL

1. Material

A two-component high-temperature vulcanized silicone elastomer (SYLGARD 184, Dow Corning Corp.) was used for this study. Sheets 1–2 mm thick were prepared by mixing thoroughly a hydrido-functional silicone crosslinker and a vinyl-terminated PDMS using a platinum catalyst at 75°C for 2 h. The unreacted oligomers were removed from the elastomer by 12 h of Soxhlet extraction with chloroform. In order to evaluate the influence of free PDMS fluid on the hydrophobic recovery, different sets of samples were prepared by adding methyl-terminated LMW silicone fluids (MW = 236, 770, and 3780, Gelest Inc.) to the compounds before curing. Fluid was added in the ratio of 5 weight parts to 100 weight parts of the base polymer, as this quantity of silicone fluid does not affect seriously the electrical and physical properties of the PDMS elastomer according to Dow Corning product specifications.

2. Exposure to Partial Electrical Discharge

High-voltage ac corona equipment (HIPOTRONICS, Model 750-5CTS B/S) with 50 kV of maximal voltage was employed to generate partial electrical discharge on the PDMS elastomer. Before the discharge, a sample was placed on a circular glass plate (diameter 1 cm, thickness 0.3 cm) with the gap between the electrode tip (diameter of 1 cm) and sample being 0.4 mm. The applied voltages were 6, 9, and 12 kV, and exposure time

to partial electrical discharge was 30 min for all samples. Discharge was carried out at $20 \pm 2\%$ of relative humidity, if not particularly specified. The oxidized specimens were carefully moved to the oven by holding the thin glass support in order to avoid any unnecessary deformation due to sample handling. The hydrophobic recovery of the oxidized samples was examined at 25, 60, 75, and 100°C for different amounts of time.

3. Analysis

The hydrophobic recovery of the oxidized samples was monitored by contact angles and angle-resolved XPS. Advancing contact angles were measured using a contact angle goniometer (Ramè–Hart, Inc., Model 100-00-115) following a method suggested by She *et al.* (16). The water drop was kept stationary by a needle and the substrate was moved by translating the stage horizontally at a fixed uniform velocity ($\sim 50 \mu\text{m/s}$). Advancing and receding contact angles were measured from the two opposite sides of the drop. All the angles reported are the averages of at least five measurements. Although both the advancing and receding contact angles were measured, only the advancing contact angles were used to monitor changes on the surface.

XPS analysis was done with a SCIENTA ESCA-300 instrument using AlK_{α} anode ($h\nu = 1486.6 \text{ eV}$) operated at 4.5 kW and 75 eV pass energy. Before the XPS spectra were taken, a sample stage was cooled down in order to minimize the removal of the LMW species from the elastomer. All spectra were taken at a 15° take-off angle to minimize the signal from the bulk. Data analysis was performed for C_{1s} , N_{1s} , O_{1s} , and Si_{2p} using SCIENTA's ESCA-300 analysis software package. An electron flood gun was used to neutralize the surface charge of the specimen. The binding energy of each atom was referenced to the binding energy of C 1s (285 eV).

III. RESULTS AND DISCUSSION

1. Recovery Rates of The Extracted Samples

(a) *Effect of storage in air.* Water-in-air advancing angles (θ_a) were measured as a function of aging time after a sample was exposed to partial electrical discharge. Water drops completely spread on the surface in all cases immediately after the discharge. For the samples before discharge, contact angle hysteresis decreased from 28° to 8° as the free LMW silicone fluid was added to the compound (Table 1).

TABLE 1
Contact Angles of Water on Silicone Elastomer Containing Different Amounts of Silicone Fluid before Partial Discharge

Sample	Advancing angle (θ_a)	Receding angle (θ_r)	Contact angle hysteresis
Extracted	112	84	28
5% fluid (MW = 3780)	110	95	15
5% fluid (MW = 770)	109	101	8
5% fluid (MW = 236)	105	97	8

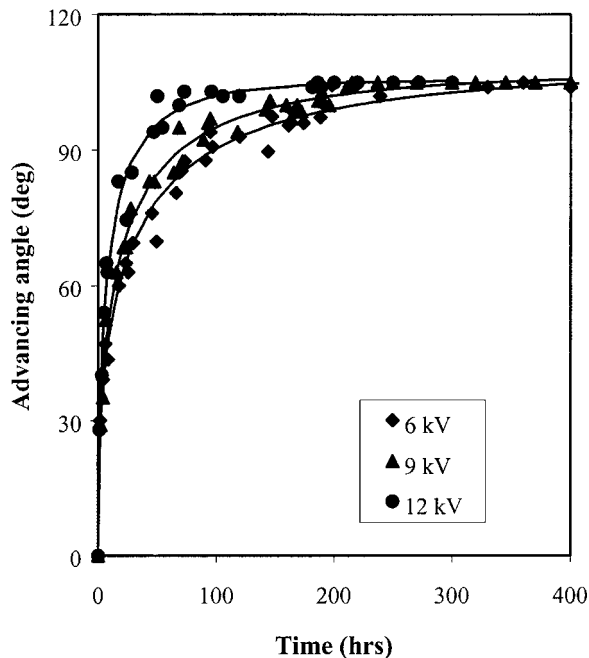


FIG. 1. Plot of advancing contact angle versus aging time for the PDMS elastomers that were extracted in chloroform and exposed to partial discharges of different intensities. The samples were aged in air at 25°C.

Figure 1 shows the advancing contact angles (θ_a) of water measured at the aging temperature of 25°C for the extracted samples. The dependence of advancing angle on aging time can best be expressed (Fig. 2) by a stretched exponential function having the form shown in Eq. [1]. The stretched exponential

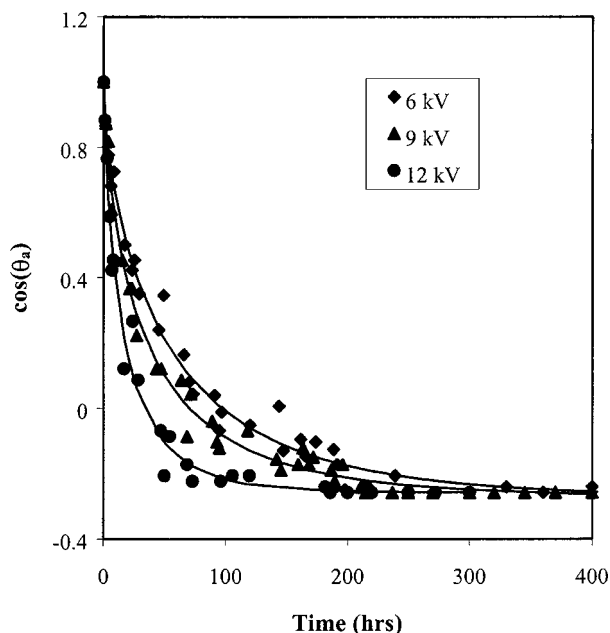


FIG. 2. The cosines of the advancing contact angles (data shown in Fig. 1) are plotted as a function of aging time. The data fit nicely a stretched exponential function, which is indicative of a diffusional process occurring at the surface.

TABLE 2
Half Recovery Times of The Extracted, Oxidized PDMS Samples

Aging temperature (°C)	Applied voltage		
	6 kV (h)	9 kV (h)	12 kV (h)
25	33.4	20.4	10.5
60	6.4	4.1	2.9
75	3.4	3.3	1.4
100	1.3	1.5	0.9

function gives better agreement with the experimental data than the simple exponential function suggested by Chatelier and co-workers (17, 18),

$$\cos(\theta_a) = a + b \exp(-ct^m), \quad [1]$$

in which a , b , c , and m are constants.

From Eq. [1], the half recovery time ($t_{1/2}$), which is the time required for the surface to reconstruct halfway, was calculated (19). The recovery rates are highly dependent on the applied voltage at low aging temperature, whereas the influence of voltage on the hydrophobic recovery becomes less significant at aging temperatures above 60°C (Table 2). Hillborg and Gedde (7) reported similar results that the recovery rate increases with increasing corona exposure time. These results can be explained if we assume that the LMW fragments are created as a result of partial discharge and these species affect the recovery of hydrophobicity. If lower molecular weight species are formed by chain scissions at higher voltages, it follows that the initial recovery rates would increase due to the migration of these LMW species to the surface. Note that the formation of LMW species by chain scission has been observed in many other nonsilicone corona/plasma applications (20–23).

(b) *Effect of storage in high vacuum.* Other researchers (7, 24) have reported an effect of the aging medium on the hydrophobic recovery. For example, storage in liquid nitrogen suppresses the hydrophobicity recovery of the oxidized samples, but unfortunately no experimental data were provided for the extracted samples. Hillborg and Gedde (7) found that the oxidized samples that were quenched in liquid nitrogen show a faster hydrophobic recovery rate due to cracking of the silica-like phase. Presumably, fluid migrates more readily through a crack rather than through an intact silica-like layer. On the other hand, Tóth *et al.* (10) found that the solvent-extracted samples also exhibit a measurable hydrophobic recovery, suggesting a possible role of reorientation of the oxidized polymer chains in the recovery process. According to our studies, the extracted samples exhibit a significant hydrophobic recovery. These experimental data can be explained neither by the reorientation of the polymer chains nor by the migration of preexisting free PDMS chains. In a separate experiment, we monitored the contact angles on the oxidized, extracted samples upon storage in high vacuum (10^{-7} Torr). Our results show that the recovery rates of the

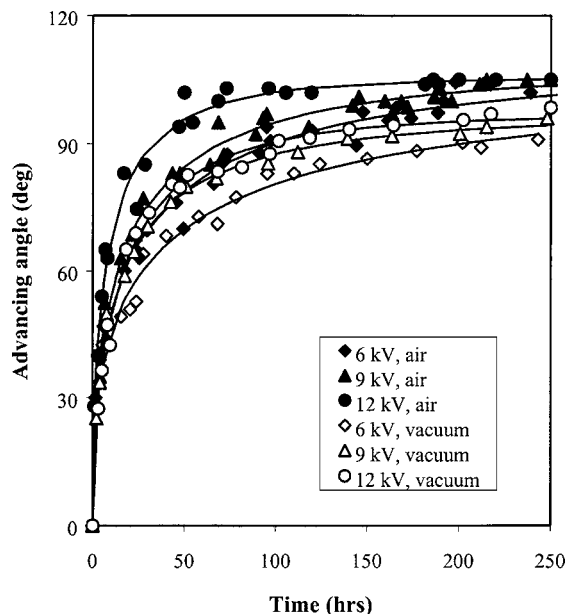


FIG. 3. The hydrophobic recovery rates of the oxidized PDMS elastomers decrease when the samples are stored in vacuum.

extracted samples stored in high vacuum decrease moderately compared to those stored in air (Fig. 3), suggesting that small volatile species are removed from the oxidized PDMS surfaces. It may also be argued that the atmospheric water participates in the recovery process by enhancing the diffusion rates of LMW species trapped in the silica-like layer. Control experiments performed in both dry and water-saturated nitrogen environments, however, show that the recovery rates of these samples are similar to each other and to those aged in air. These experiments performed under controlled environments also preclude the possibility that the recovery process is due to airborne organic contaminations. Hence it is likely that the slow recovery in high vacuum is due to the removal of some of the lowest molecular weight species from the topmost surface (2–3 nm) that are created *in situ* during exposure of the polymer to partial electrical discharge.

The result of angle-resolved XPS also supports the proposal that the LMW species, created during partial discharge, migrate to the surface. The binding energies of O_{1s} , C_{1s} , and Si_{2p} of untreated PDMS elastomer were found at 532.8, 285, and 102.5 eV, respectively, similar to literature values (11, 15, 25). The binding energy of Si_{2p} is shifted to 103.9 eV after the discharge, which is consistent with the formation of an inorganic silica-like phase associated with Si atoms bonded to more than two oxygen atoms (26). On the treated samples, new C_{1s} peaks corresponding to oxidized carbon at 286.9 eV (C–O), 287.9 eV (C=O), and 289.3 eV (OC=O) were detected (27–29). No nitrogen peak was detected in the spectra. Table 3 summarizes the atomic composition of silicon, carbon, and oxygen observed at two different take-off angles, 15° and 90° , on an elastomeric surface that was exposed to a discharge voltage of 6 kV. The spectra

taken at a low take-off angle (15°) carry more surface-sensitive information than those taken at high take-off angle (90°). The fact that the concentration of silicon in higher oxidation state (i.e., SiO_2) is enhanced at 90° compared to that at 15° is consistent with the scenario that the upper surface of the elastomer is covered with a thin more organic-like layer. Examination of the atomic compositions indicates that the oxidized carbon species are present deeper in the surface layer as their intensity is more attenuated at 15° as expected. The concentration of unoxidized carbon species at the 15° take-off angle is, however, considerably higher than that in the spectra taken at 90° , which suggests that the upper surface of the oxidized polymer is covered with a carbonaceous species. In conclusion, therefore, it can be said that the surface of the oxidized PDMS is covered with a thin layer of unoxidized methyl silicone, which, in all likelihood, arises from diffusion from the subsurface region to the exterior of the surface during the time (2 h) the samples were oxidized and transferred to the XPS chamber. This carbonaceous species must be a component of dimethylsiloxane, as the concentration of Si at lower oxidation state is found to be higher at the 15° take-off angle than at the 90° angle.

2. Effect of Humidity And Temperature on The Recovery Rate

(a) *Humidity.* Figure 4 shows that the samples oxidized at high humidity exhibit a faster recovery rate than those at low humidity. The effect is particularly pronounced in the 15–20% humidity range. Water-in-air, a reactant in such an oxidation reaction, must contribute significantly to the production of LMW species. The results suggest that the LMW species are created in ample amount when the humidity is $\geq 20\%$, strongly implying that water participates in the chain scission reaction of siloxanes.

(b) *Aging temperature.* The recovery rates for the extracted samples increase as the aging temperature increases from 25 to 100°C . Activation energies (E_a), obtained from the Arrhenius plots, are 40, 32, and 31 kJ/mol at discharge voltages of 6, 9, and 12 kV, respectively (Fig. 5). The decrease in the activation energy from 40 to 31 kJ/mol with increasing discharge voltage is qualitatively consistent with the observations of Hillborg

TABLE 3
Atomic Composition of the Extracted, Oxidized Sample
Exposed to 6 kV

Element	Take-off angle	
	15° (%)	90° (%)
C(unoxidized)	31.9	12.9
C(oxidized)	1.8	6.3
Si(unoxidized)	14.3	4.6
Si(oxidized)	15.6	25.2
O	36.4	51.0

Note. Data were taken 2 h after discharge. The atomic compositions of the unoxidized sample are as follows: C (45.3%), Si (30.0%), and O (24.7%).

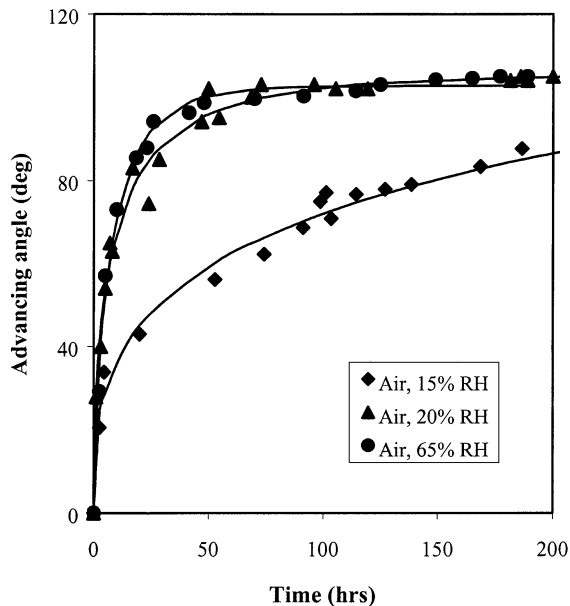


FIG. 4. The effect of humidity on the recovery rates of the extracted samples subjected to 12 kV. Recovery rate is significantly affected by humidity in the range of 15 to 20%, but remains unchanged above 20% humidity.

and Gedde (7), who reported that the activation energy decreases from 57 to 36 kJ/mol as the discharge time is increased from 0.3 h to 200 h. Presumably these activation energies (E_a) correspond to the diffusion of species contributing to hydrophobic recovery. As either the discharge voltage or discharge time

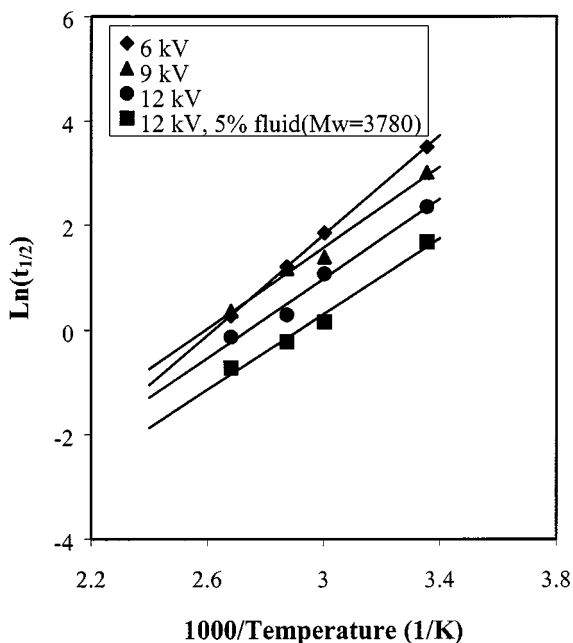


FIG. 5. Arrhenius plots of half recovery time ($t_{1/2}$) for the PDMS elastomers that were exposed to electrical discharges of different intensities. All the PDMS samples were extracted before discharge treatment except the one that contained 5% of free silicone fluid (MW = 3780).

TABLE 4
Half Recovery Times of The Fluid-Containing Samples Aged at 25°C

Discharge voltage (kV)	Molecular weight of added fluid			
	236 (h)	770 (h)	3780 (h)	No fluid (h)
12	2.6	3.5	5.4	10.5
6	4.3	9.4	11.3	33.4

is increased, lower molecular weight species of lower E_a are formed.

3. Effect of The Preexisting LMW Silicone Fluid in The Bulk

The oxidized samples containing silicone fluids exhibit a faster recovery than the extracted samples for all the discharge conditions (Table 4). At the aging temperature of 25°C and the discharge voltage of 12 kV, preexisting PDMS fluid has a less significant effect on the restructuring of the oxidized PDMS elastomers (Fig. 6). However, as the molecular weight of PDMS fluid is decreased from 3780 to 236 g/mol or the discharge voltage is decreased, unreacted silicone fluid in the bulk has a greater effect on the recovery process. These results are consistent with the following scenario. At high voltage, enough LMW species are formed and dominate the recovery process, with the added fluid having no major effect. At low discharge voltage, there is less chain scission and thus the added fluid somewhat

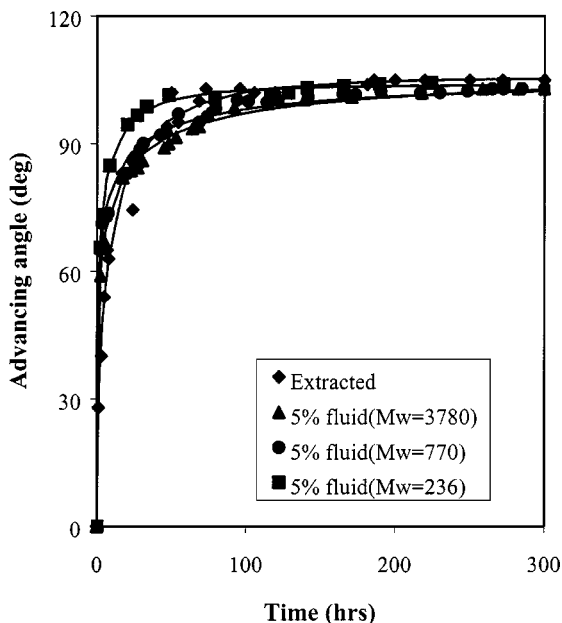


FIG. 6. Comparison of hydrophobic recovery rates of the extracted and the fluid-containing PDMS elastomers at 25°C after being exposed to a discharge voltage of 12 kV. Note that the recovery rate is the highest when the molecular weight of the added fluid is the lowest, which indicates that the diffusion of species from the bulk to the surface plays an important role in the recovery process.

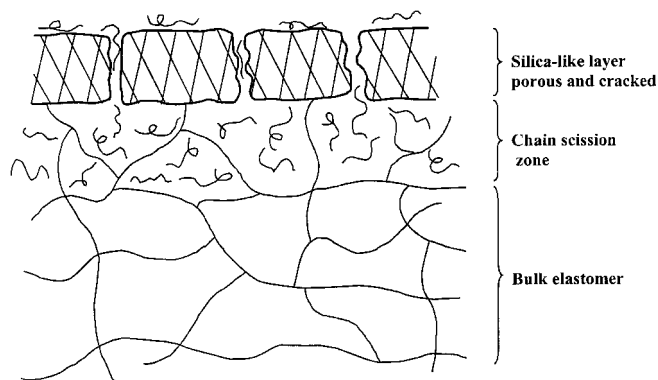


FIG. 7. A schematic showing different zones that are produced on a silicone elastomer as a result of exposure to partial electrical discharge. The uppermost surface is affected mostly, which converts to a silica-like layer. Beneath the silica-like layer, it is envisaged that the scission of polymer chain occurs. The silica-like layer is probably porous or cracked, through which the *in situ* produced LMW species migrate and adsorb at the air-polymer interface. If free fluid is present in the network, it also contributes to the recovery process.

compensates for the lack of production of LMW species. As expected, the lower molecular weight fluid has a greater effect on the recovery process than the higher molecular weight fluid because the LMW fluid diffuses faster than the high molecular weight fluid.

The recovery rates of the fluid-containing samples are not, however, vastly faster than those of the extracted samples, suggesting that fluid migration from the bulk to the surface is not the necessary mechanism for the hydrophobic recovery process. Since the reorientation of polar groups can be ruled out in our case and the recovery process is slowed down at high vacuum, the LMW species created by partial discharge must play a major role on the hydrophobic recovery of the oxidized PDMS elastomers. The evidence, so far, suggests that the source of these LMW species is very close to the surface. The overall picture is as follows. The uppermost surface of the elastomer is converted to a silica-like layer when exposed to electrical discharge. In the zone beneath the silica layer, significant chain scission occurs producing the LMW species. These LMW species eventually migrate to the surface through the porous or cracked silica-like layer, and thus the surface regains its hydrophobicity (Fig. 7). Free fluid, present in the network, undoubtedly aids the recovery process.

IV. CONCLUSION

Designed experiments have been carried out to investigate the key factors that contribute to the hydrophobic recovery of PDMS elastomers exposed to partial discharge. Studies of the hydrophobic recovery rates under various environments provide strong evidence that the *in situ* formation of the LMW species and its subsequent migration to the surface constitute the major mechanism of the hydrophobic recovery. Preexisting free silicone fluid, nonetheless, plays a role in the recovery process when

it is present in the network, but its effect is secondary to that of the *in situ* produced LMW species.

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