

Novel Bi-Layer Conformal Coating for Reliability Without Hermeticity MEMS Encapsulation

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Abstract—A flexible, smooth, and low profile conformal coating was developed to accomplish the encapsulation of a micro-electromechanical system (MEMS) device that will be applied to sense the static pressure on aircraft during real flight testing. The encapsulant should be able to protect the MEMS device and the multichip module (MCM) from adverse environmental conditions, i.e., mechanical shock, temperature fluctuation, engine fuel and oil contamination, and moisture/mobile ion permeation. Presently, conventional packaging schemes for electronics cannot satisfy this specific outdoor application, and a new encapsulation combination has been designed in accord with the requirement of reliability without hermeticity (RWOH). A bi-layer structure was selected because of property limitations of a single material. Pliable elastomeric silicones, are typically flexible, water repellent, and abrasion resistant. The silicone encapsulant will be first applied to planarize the MEMS surface and function as durable dielectric insulation, stress-relief, and shock/vibration absorbers over a wide humidity/temperature range. To compensate for the deficiency of silicone on engine fuel/oil contamination, Parylene C is to be deposited afterward. This bi-layer coating can achieve excellent bulk properties, such as moisture and mobile ion barrier resistance, chemical compatibility, and electrical insulation characteristics. However, the poor adhesion of Parylene C to silicone greatly restricts its application. To address this problem, silane coupling agents were used as an adhesion promoter. Significant adhesion improvement was achieved by placing an interlayer silane coupling agent to provide interfacial bonding to the silicone elastomeric surface and the Parylene C film. Furthermore, a possible mechanism of adhesion enhancement will also be presented in this study.

Index Terms— Bi-layer conformal coating, micro-electromechanical system (MEMS), multichip module, Parylene C, reliability without hermeticity (RWOH), silane coupling agent, silicone elastomer.

I. INTRODUCTION

LONG-TERM, reliable protection of sensitive circuits and components is becoming more important in many of today's delicate and demanding electronic applications. The need for low profile, flexible, conformal and economical packaging in micro-electromechanical system (MEMS) based modular protection has resulted in a new conformal coating design exhibiting reliability without hermeticity (RWOH). General organic passivation materials such as polyimides, silicones, and epoxies have been used as RWOH encapsulants with reasonable success. However, these organic materials

are generally moisture permeable. Moisture diffusion rates through organic polymeric encapsulants vary with time. The rate depends on the type of material, the material thickness, and the length of diffusion time. Moisture on the surface of the device, in the presence of mobile ions such as Na^+ , K^+ , and Cl^- , will result in device failure [1]. To ensure RWOH, we propose to combine a low moisture permeable silicone elastomer coating with a thin film of Para-X-xylylene (Parylene C) to ensure long-term protection. The silicone elastomer is coated as a relatively thick layer (approximately 250 mil), primarily for protection of the delicate circuits and as a planarizing layer for the MEMS device. A thin layer of Parylene C (approximately 15–20 μm) is then deposited on top of the thick silicone elastomer layer. This novel combination provides an outstanding barrier to moisture and mobile ion (Na^+ , K^+ , Cl^-) permeation and an aerodynamically smooth surface for avionics applications. Furthermore, the low surface energy of Parylene C as a top coating can effectively prevent air borne particles [2] and jet fuel contamination of the underlying silicone coating.

A. Conformal Coating Materials

Silicones are typical pliable elastomeric materials, which can function as durable dielectric insulation, as barriers against environmental contamination and as stress relieving shock/vibration absorbers over a very wide humidity/temperature range. In this study, Dow Corning Q4-2817 and Dow Corning Hipec 3-6550 were selected as candidate elastomer, to function explicitly as a top profile planarizing agent.

Parylene is a conformal coating family that has high dielectric strength and excellent chemical resistance [3]. Currently, the Parylene family is composed of four types of commercial products: Parylene N, C, D, and F. Unlike conventional liquid coating, Parylene coatings are applied by vapor deposition in a vacuum chamber at ambient temperature. This unique method of vapor deposition can form 100% pinhole-free uniform coverage and has the ability to penetrate and coat very complex topographical geometry. Coating thickness with this deposition process is also easily controlled. In this study, Parylene C is selected as a suitable encapsulating candidate in terms of moisture permeation resistance, chemical resistance, deposition rate and cost consideration [4].

Table I lists the normalized permeability of gases through polymer materials. The permeability of Parylene C is significantly lower than almost any other engineering plastic. Moisture permeability resistance of Parylene C is 40 times greater

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TABLE I
NORMALIZED PERMEABILITY OF GASES THROUGH POLYMER MATERIALS

Material	O ₂	N ₂	CO ₂	H ₂	H ₂ O
Parylene C	2.8	0.4	3	43	0.08
Parylene D	12	1.8	5	94	0.1
Parylene N	15	3	84	213	0.6
HDPE	73	17	228	n.d.	0.12
PS	138	23	400	n.d.	3.5
PTFE	223	133	n.d.	516	n.d.
LDPE	140	80	700	n.d.	0.6
PC	124	22	827	n.d.	1.5
FEP	295	126	657	381	0.16
Silicone	19000	n.d.	118000	17000	3

TABLE II
SWELLING CAUSED BY ORGANIC SOLVENTS AT ROOM TEMPERATURE

Solvents		% Swelling Parylene		
Class	Test Member	N	C	D
Alcohol	Isopropyl	0.3	0.1	0.1
Aliphatic Hydrocarbon	Iso-Octane	0.2	0.4	0.3
Amines	Pyridene	0.2	0.5	0.5
Aromatic Hydrocarbon	Xylene (mixed)	1.4	2.3	1.1
Chlorinated Aliphatic	Trichloroethylene	0.5	0.8	0.8
Chlorinated Aromatic	Chlorobenze	1.1	1.5	1.5
Chlorinated Aromatic	O-Dichlorobenze	0.2	3.0	1.8
"Freon"	Trichlorotrifluoroethane	0.2	0.2	0.2
Ketone	Acetone	0.3	0.9	0.4
Keton	2,4-Pentanedione	0.6	1.2	1.4

* Film strips were immersed in the test liquids for 90 minutes at room temperature and the thickness was re-measured by the IR method. In every case, equilibrium (no further thickness change) was reached before 90 minutes.

than that of silicone materials. Table II lists chemical compatibility results of Parylene films by film thickness measurements before and after immersion in different organic solvents for 90 min at room temperature [4]. Common solvents, especially those with similar chemical structures as Parylene, such as xylene, result in the most severe swelling of film thickness, which is still less than 3% of the original parylene film thickness. Parylene C shows excellent chemical resistance to aliphatic hydrocarbons such as octane, the primary component of engine fuel, only resulting in a swelling of 0.4%.

B. Interfacial Adhesion Promoters

In addition to material considerations, interfacial adhesion reliability between polyimide passivated Si-based substrate and silicone elastomer, silicone elastomer and Parylene C also play an important role in real-life applications. Also, the adhesion at various interfaces of the encapsulated MCM module must resist vibrations and air-stream impact during actual flight test.

Generally speaking, adhesion theory includes factors such as wet-out phenomena, weak boundary layers, polar adsorption, surface energies, and other effects. The predominant contributor to adhesion, however, is chemical bonding such as that introduced by organo-functional silane "coupling agents." These materials are used in very low concentration, but produce disproportionately large improvements in adhesion performance. Processing and fabrication conditions, which ensure the opportunity for interaction at both ends of the silane molecule, are essential to provide bonding on both sides of the interface [5], [6]. In this study, a total of 9 different functional silane coupling agents from Dow Corning Co. and Witco Corporation were used as candidate adhesion promoters. An appropriate mechanism is presented later in this study.

Since this is a long-term research project, reliability characterization and evaluation of the conformal coatings is still under study. This paper describes a preliminary aspect of the initial working results and reports a new bi-layer conformal coating design for similar potential applications.

II. EXPERIMENTAL

A. SIR Test Vehicle and Conformal Coating Material Selection

All the conformal coating materials applied in this project were initially evaluated by surface insulation resistance (SIR) measurements using an Alpha Metals SIR Ω meter (model 300) test instrument with custom copper/FR4 BellCore Y comb test vehicles. The 50.8 \times 50.8 mm SIR board surface areas were coated with candidate materials immediately after a special cleaning process [7]. After encapsulation, the coated test boards were fixed into a Delp. Corporation 16 pattern SIR rack. Initial measurements were made to verify that the needle type test pins penetrated the coatings and made contact with the copper comb pattern. Finally, the test fixture and samples were mounted in a blue M humid-flow combination temperature and humidity cabinet set at 85 $^{\circ}$ C/85% RH. All coatings would be continuously exposed to 85 $^{\circ}$ C/85% RH for 1000 h, with 100 V dc biasing.

The silicone candidates selected for this study were commercially available and the selection of coating materials was initially based on thermo-mechanical properties (under the condition of surviving temperature cycling from -55 to 125 $^{\circ}$ C) and level of mobile ionic impurities (<20 ppm each of Cl⁻, Na⁺, and K⁺). All the candidate coatings experienced SIR measurements for further screening.

The two silicone elastomers selected for SIR testing include:

- 1) Dow Corning Q4-2817 fluorosilicone gel;
- 2) Dow Corning Hipec 3-6550 silicone gel.

The moisture and mobile ion resistance of Parylene C with a 5 μ m coating thickness was also tested by SIR in this study.

B. Reliability Testing

1) Sample Preparation:

a) *Silicone elastomer coating:* The polyimide passivated Si-based substrates were cleaned using the same method as for SIR board treatment. Within 0.5 h after cleaning, the silicone materials were uniformly coated on the top of the

cleaned substrates using a spin coater (Specialty Coating Systems, Inc. Model 6708D). Curing procedure for the Dow Corning Q4-2817 was 100 °C for 30 min. While, for Dow Corning Hipec 3-6550 the curing procedure was 150 °C for 60 min.

b) Silane coupling agent Application: Direct pretreatment is the easiest and most satisfactory method to apply a silane to an inorganic surface. Two methods find extensive use.

- 1) Aqueous pretreatment, which is primarily used for fiberglass. Coating is accomplished by applying a dilute, aqueous solution of the silane, generally adjusted to pH 4 with acetic acid. The silane hydrolyzes and forms a layer of silanol molecules at the substrate surface. The solution, typically with a concentration of silane within 0.2–0.5%, will ordinarily dissipate onto the surface. This method is generally performed with high efficiency.
- 2) Nonaqueous blending, which is widely used for mineral fillers. The silane, which is undiluted or mixed with a spreading solvent, is added to the inorganic substrate and thoroughly dry blended at low shear to give a uniform coating. Dry blending offers a convenient and effective route to coupling agent treatment of particulate inorganics such as alumina, silica, and glass microbeads used as fillers in composites. A silane treatment level of between 0.5–1.0% is appropriate for most common minerals. The additive method is an alternate route commonly used to apply silane coupling agents. With this method, the silane is added to the polymeric phase from which it diffuses or migrates to the inorganic surface. Reaction occurs first by hydrolysis, and then by condensation to produce siloxane coupling linkages. Uniform treatment is dependent upon thorough blending of the mixture and filler. Typically, the silane is added at a concentration of 1.0% or less.

In regard to our unique bi-layer RWOH design, the three kinds of silane application methods discussed above were not appropriate. To address this problem, a new silane application method, named as vapor deposition, was used to directly deposit silane on the surface of the silicone elastomer. This method was selected to avoid film swelling and still produce a uniform adhesion promoter layer. Consequently, Parylene C is promptly polymerized on top of the silane while reactivity still sustains.

Prior to silane application, all the silicone coated substrates were treated with UV-ozone at 50 °C for 5 min. Immediately following treatment, the side with the silicone elastomer was exposed to the silane solutions. Silane solutions were then heated between 60–70 °C for 20 min to vaporize sufficient silane which was uniformly condensed on the entire surface of the silicone elastomers. An excess of silane will lead to a swelling in the silicone coating. After that, the samples were immediately placed into a Parylene deposition system, Lab Coater 2010, from Special Coating System Co. for Parylene C deposition.

c) Parylene C deposition: Parylene C deposition was conducted using the Gorham Method. The Parylene C coating process starts with the vaporization of the dimer at 150 °C,

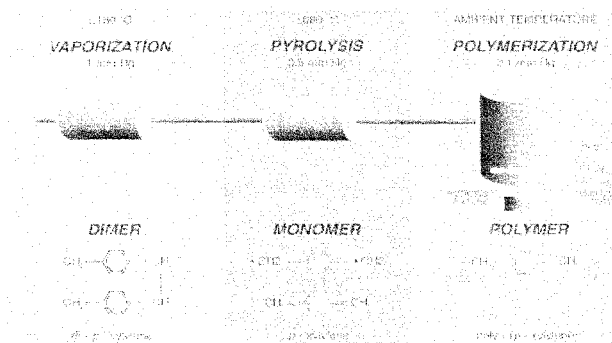


Fig. 1. Schematic graph of Parylene C deposition processes.

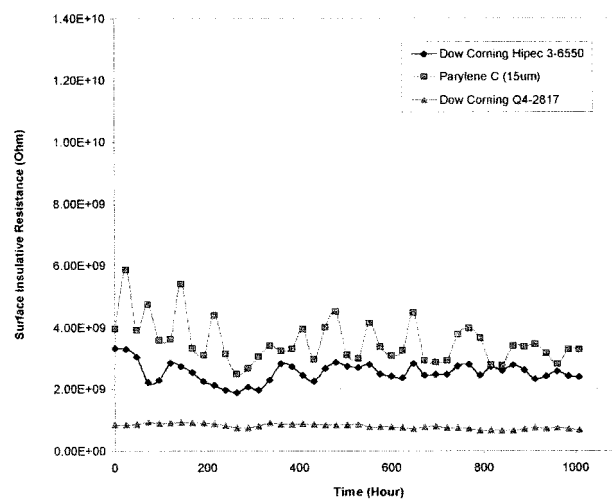



Fig. 2. Surface insulative resistance of Parylene C and silicone.

0.1 Torr. The resulting dimer gas is further elevated to 650 °C into the pyrolysis zone to obtain two methylene bonds which generate a stable para-xylylene. Finally, the monomer is sent to the ambient temperature deposition vacuum chamber where the para-xylylene polymerizes and coats on the surface of the silicone elastomers [8]. A Schematic graph of the Parylene C deposition process is illustrated in Fig. 1.

2) Reliability Testing: All the single layer or bilayer shear strength test samples were exposed to 85 °C/85% RH (relative humidity) chamber (Blue M, model no. AC-7602HA) for 1000 h and thermal shock chamber (thermotron environmental test chamber, ATS-320-DD-10-705-LN2) for 500 cycles. Thermal cycling was set from –55 to 125 °C. The time for each temperature ramp was 15 min, and the dwell period at either –55 or 125 °C was 15 min. The adhesion test interval for 85 °C/85% RH aging is 200 h, and for thermal stressing is 100 cycles.

3) Adhesion Strength Test: For the adhesion test, small dies with size of 2 × 2 mil were attached on top of the bi-layer conformal coating using an epoxy resin as an adhesive, which was dried at room temperature. Since the interfacial adhesion between die and Parylene C is strong enough that the delamination could only occur between the Parylene C and silicone elastomers. Shear strength test was performed with an adhesion analyzer (Royce Instruments System 552) at room

TABLE III
CANDIDATE SILANE COUPLING AGENTS FOR INTERFACIAL ADHESION ENHANCEMENT

Designation	Chemical Name	Chemical Formula
A-174	γ -Methacryloxypropyl trimethoxysilane	$\text{CH}_2=\text{C}(\text{CH}_3)\text{CO}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{Si}(\text{OCH}_3)_3$
A-1120	N- β -(Aminoethyl)- γ -amino propyltrimethoxysilane	$\text{H}_2\text{NCH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2\text{CH}_2\text{Si}(\text{OCH}_3)_3$
PS X554		$(\text{H}_2\text{NCH}_2)_2\text{Si}(\text{OCH}_2\text{CH}_3)_2$
Y-9669	N-Phenyl- γ -aminopropyl trimethoxysilane	 $\text{NHCH}_2\text{CH}_2\text{CH}_2\text{Si}(\text{OCH}_3)_3$
A-1102	γ -Aminopropyltriethylsilane	$\text{H}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{Si}(\text{OCH}_2\text{CH}_3)_3$
A-171	Vinyltrimethoxysilane	$\text{CH}_2=\text{CHSi}(\text{OCH}_3)_3$
A-151	Vinyltriethoxysilane	$\text{CH}_2=\text{CHSi}(\text{OCH}_2\text{CH}_3)_3$
A-172	Vinyl-tris-(2-methoxyethoxy)silane	$\text{CH}_2=\text{CHSi}(\text{OCH}_2\text{CH}_2\text{OCH}_3)_3$
A-2120	N- β -(aminoethyl)- γ -aminopropyl methyl dimethoxysilane	$\text{H}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{Si}(\text{CH}_3)_2\text{OSi}(\text{CH}_3)_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2$

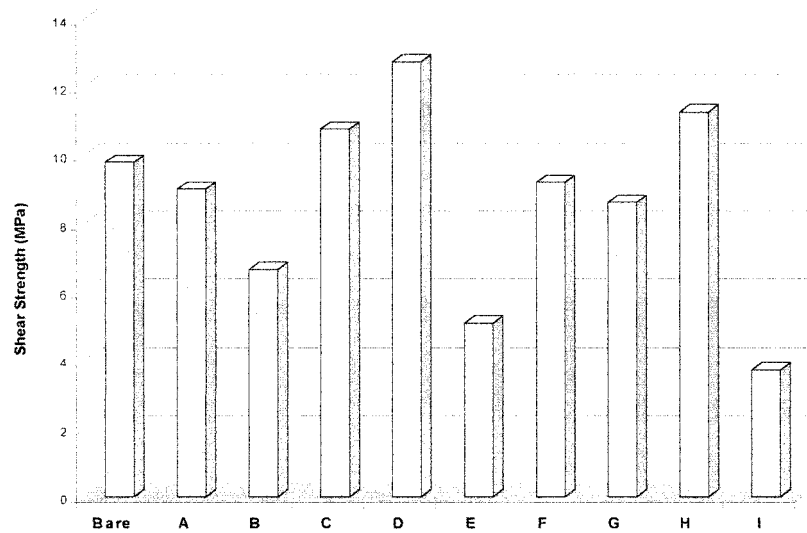


Fig. 3. Shear strength between Parylene C and Dow Corning Q4-2817 elastomer with various silane coupling agents after sample preparation.

temperature. Testing was conducted at a speed of 0.1 mm/s (0.004 in/s) with a vertical offset of 49 μm (0.002 in).

III. RESULTS AND DISCUSSION

SIR measurement results in Fig. 2 indicate that the Dow Corning Hipec 3-6550, Q4-2817 and Parylene C conformal coatings pass the aerospace specification limit of $1\text{E}08 \Omega$. The surface insulation resistance shows a slowly decreasing trend during the testing period. This can be attributed to moisture

and mobile ion ingress facilitating mobile ion migration from cathode to anode during biasing, which will result in resistance decrease.

To improve the adhesion strength between the silicone elastomer and Parylene C, nine different functional silane coupling agents were selected as adhesion promoter candidates between Parylene C and Silicone Elastomer based on chemical covalent relation between silane organic functional groups and Parylene C chemical structure. Chemical structures of all nine silane coupling agents are listed in Table III.

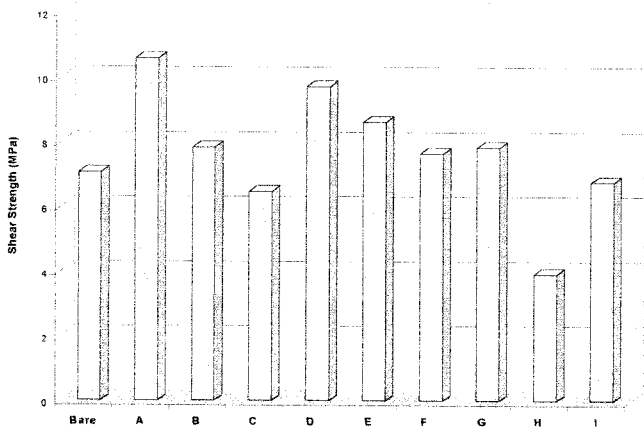


Fig. 4. Shear strength between Parylene C and Dow Corning Hipec 3-6550 elastomer with various silane coupling agents after sample preparation.

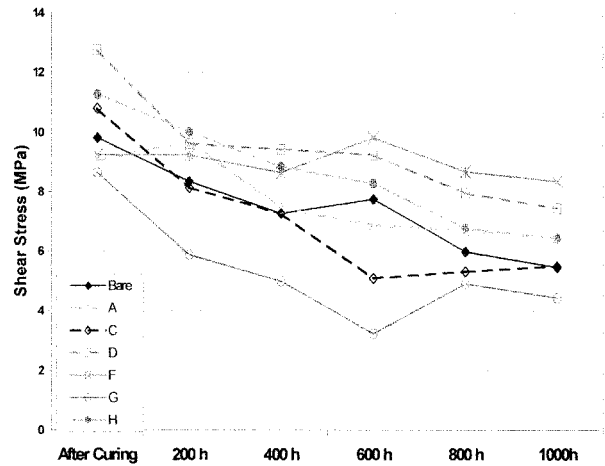


Fig. 6. Shear strength between Parylene C and Dow Corning Q4-2817 elastomer with various silane coupling agents after exposure to 85 °C/85% RH stressing.

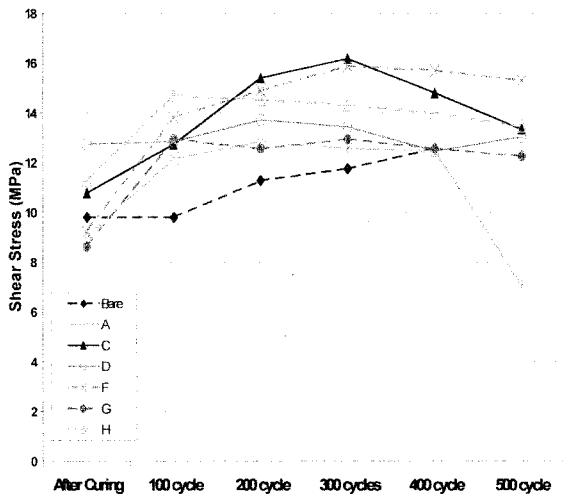


Fig. 5. Shear strength between Parylene C and Dow Corning Q4-2817 elastomer with various silane coupling agents after exposure to thermal shock aging.

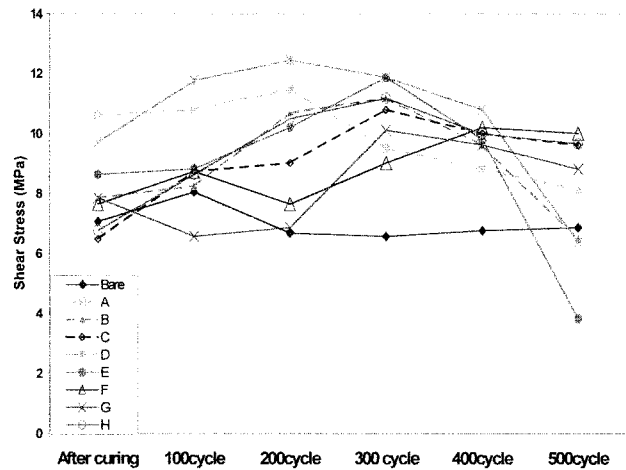


Fig. 7. Shear strength between Parylene C and Dow Corning Hipec 3-6550 elastomer with various silane coupling agents after exposure to thermal shock aging.

Shear strengths between Parylene C and Dow Corning Q4-2817 after sample preparation using various silane coupling agents as adhesion promoters are illustrated in Fig. 3. Results indicate that shear strengths obviously increase using silane coupling agents C, D, and H, and shear strengths decrease using silane coupling agents A, F, and G. Shear strengths greatly decrease using silane coupling agent B, E, and I. Therefore, samples with silane coupling agents A, C, D, F, G, and H require further reliability testing. Shear strengths between Parylene C and Dow Corning Hipec 3-2817 after sample preparation are shown in Fig. 4. Results indicate that shear strengths increase using silane coupling agents A, B, D, E, F, and G. Shear strengths decrease using silane coupling agents C and I, and Shear strengths greatly decrease using silane coupling agent H. Subsequently, samples with silane coupling agents A, B, C, D, E, F, G, and I will be subjected to further reliability testing.

Shear strength results of samples submitted to thermal cycling are shown in Fig. 5 after 100 cycles. Shear strength

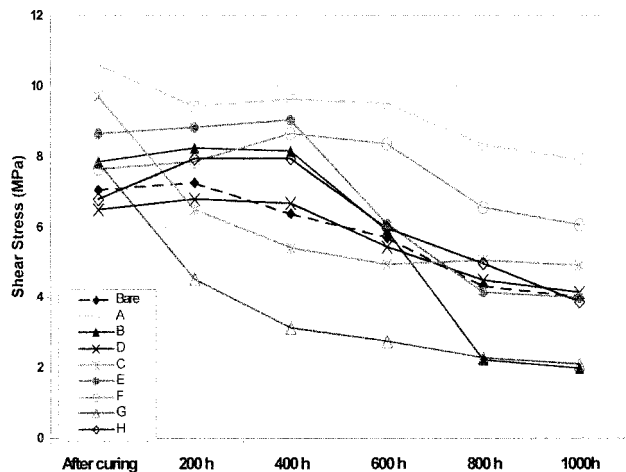


Fig. 8. Shear strength between Parylene C and Dow Corning Hipec 3-6550 elastomer with various silane coupling agents after exposure to 85 °C/85% RH stressing.

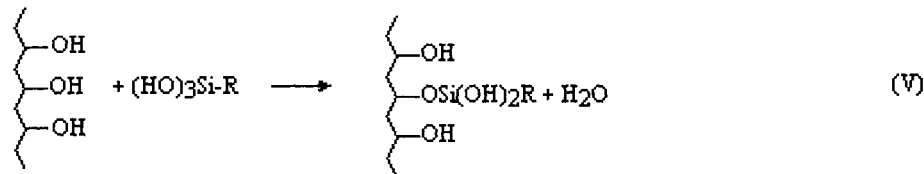
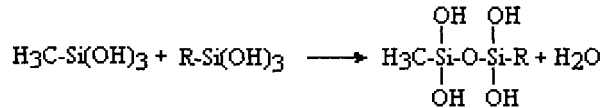
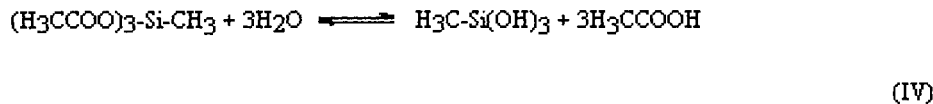
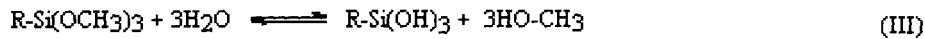
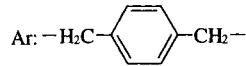
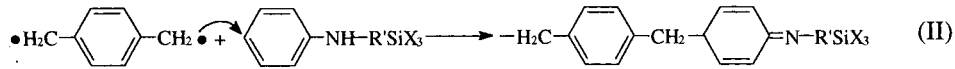
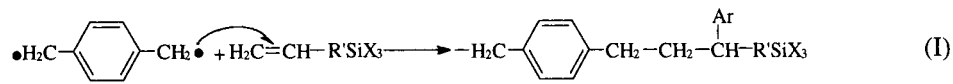


Fig. 9. Equations (I)-(V).

results between Parylene C and Dow Corning Q4-2817 after 85 °C/85% RH aging are shown in Fig. 6 at 200 h intervals. From Fig. 5, it is observed that the shear strength of most samples increases after exposure to thermal cycling for 100 cycles. The possible reason for this is that the intrinsic cross-linking reaction of the silicone elastomer is maintained under thermal cycling during this period, and some cyclic oligomers, such as D3, D4, and D5, etc. are continuously diffused out. As a result, the modulus increase of the silicone elastomer leads to a shear strength increase. However, after sample exposure to thermal cycling for a considerable time, CTE mismatch between Parylene C (35–40 ppm) and silicone elastomer (300–500 ppm) intensified and causes microdelamination to occur at some interfacial sites, which finally become the failure centers during shear testing and the interfacial adhesion is lowered. While shear strengths for most samples continuously decrease under 85 °C/85% RH aging due to the modulus of silicone elastomer reduction as shown in Fig. 6. This situation may be the result of two factors. One possible reason is that when the bilayer sample is kept under 85 °C for an extended time, some

low molecular silicone cyclics may be continuously generated and trapped in the cross-linked network as plasticizers, since the high moisture pressure intuitively prohibits the cyclics outward diffusion. Another possible reason is that the moisture may cause the siloxane to inversely hydrolyze into silanols at 85 °C. The moisture came from permeation of moisture in 85% RH atmosphere through the Parylene C sheared (removed) areas during prior die shear testing.

From analysis of the results from Figs. 5 and 6, it can be concluded that the best adhesion promoters for the Parylene C and Dow Corning Q4-2817 combination are silane coupling agents D and F.

Shear strength results of the Parylene C and Hipec 3-6550 bi-layer combination after different intervals of thermal cycling and 85 °C/85% RH aging are shown in Figs. 7 and 8, respectively. The shear strength trends are the same as those between Parylene C and Dow Corning Q4-2817. It can be observed that the best two silane coupling agents for Parylene C and Dow Corning Hipec 3-6550 are silane coupling agent A and F as confirmed by adhesion reliability testing.

IV. MECHANISM FOR SILANE COUPLING AGENTS AS ADHESION PROMOTERS

Silane coupling agents are a family of organosilicon monomers, which are characterized by the formula: $R-SiX_3$. In this formula, R is an organofunctional group attached to silicon in a hydrolytically stable manner, and X designates hydrolyzable groups that are converted to silanol groups by hydrolysis.

Among these nine silanes, the ones that contain a vinyl, methacryloxypropyl, or phenyl amino-functional group at one end, and a methoxy-functional group at another end will function as the best three adhesion promoters.

These silanes chemically bond to the methylene linkage of Parylene C through radical-initiated polymerization. The possible bonding mechanisms are shown in (I) and (II) in Fig. 9. The di-para-xylylene radical that comes from Parylene C thermal pyrolysis maintains high reactivity before polymerization. This free radical will readily attack the $c = c$ double bond according to Markownikoff's free radical mechanism [9].

For the anilino group, since the amino function group is an electron-donating group and its lone electron-pair on the nitrogen atom in the P orbit will conjugate with an electron " π cloud" above or below the benzene rings. The electron-density on the phenyl ring will be higher than that of the normal condition, which will make the benzene ring more active. As a result, the xylylene free radical group will selectively bond to the carbon atom according to the steric site under the allow of Markownikoff Law.

On the other end, since methoxy is the smallest and active group bonded to the Si atom, it is easily hydrolyzed into silanol [see (III) in Fig. 9]. Since there are components such as silica filler and methyltriacetoxysilane in Dow Corning Q4-2817 and Hipec 3-6550, the silanol group would condense with the methyltriacetoxysilane hydrolyzed silanol group or directly bond to the surface of the silica [see (4) and (5), respectively].

V. CONCLUSION

A novel bilayer conformal coating for MEMS device encapsulation has been designed in accordance with the requirement of RWOH. Parylene C is a promising conformal coating for aerospace application. Adhesion between Parylene C and silicone elastomer has been intensified by applying silane coupling agents at the interface as adhesion promoters. The adhesion promoted coupling reliability has also been investigated by die shear testing after thermal shock stressing and 85°C/85% RH aging. Test results indicate that the silane species with a vinyl or anilino group at one end and a methoxy group at the other end will show the best adhesion enhancement. A proper mechanism was also presented.

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From 1991 to 1997, her major research work focused on microsensor fabrication and application in electrochemistry field and die bonding with Au/In bi-alloy isothermal solidification technique. She joined the Electronics Packaging Research Group, School of Materials Science and Engineering, Packaging Research Center, Georgia Institute of Technology, Atlanta, as a Postdoctoral Fellow, in 1997. She is currently working on multilayer conformal coatings for reliability without hermeticity, reworkable high temperature adhesives for MCM-D processing, parylene application, and its adhesion enhancement on polymeric substrates using silane as coupling agents.

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