Evaluation and Characterization of Reliable Non-Hermetic Conformal Coatings for Microelectromechanical System (MEMS) Device Encapsulation

Jiali Wu, *Member, IEEE*, Randy T. Pike, C. P. Wong, *Fellow, IEEE*, Namsoo P. Kim, and Minas H. Tanielian

***Abstract*—The thrust of this project was to evaluate commercial conformal encapsulation candidates for low cost aerospace applications. The candidate conformal coatings evaluated in this study included silicone elastomers, epoxies, and Parylenes with bi-layer or tri-layer designs. Properties characterized in this study included mobile ion permeation and moisture ingress resistance, interfacial adhesion variation through thermal shock cycling and 85 °C/85% RH aging. Surface Insulation Resistance (SIR), Triple Track Resistance (TTR) and die shear strength were used for the corresponding electrical and physical property characterizations. Parylene F displayed excellent properties for environmental protection. Silicone elastomers displayed less resistance to the harsh environment as compared to the Parylene family (N, C, D types), but it could provide advantages for low residual stress applications. The change in adhesion strength between Parylene C and silicone elastomers after exposure to thermal shock cycling or 85 °C/85%RH aging for different time periods were conducted from die shear test in terms of the interfacial failure. SIR values of all the candidate materials after 1000 h exposure to 85 °C/85%RH, with 100 V dc for resistance measurement, range from 1×10^8–$1 \times 10^9 \Omega$. Leakage current values after 1000 h exposure to 85 °C/85%RH, 175 V bias, are in the range of 10^{-9} to 10^{-11} Amp. The bi- or tri-layer conformal coating combination investigated in this study showed significant promise for encapsulation of the microelectromechanical system (MEMS) devices.**

***Index Terms*—Conformal coatings, leakage current, mobile ion permeation, moisture ingress, surface insulation resistance.**

I. Introduction

RELIABLE nonhermetic conformal encapsulants have been widely applied since the 1970s [1]. Recent applications have been aimed at the automotive, communication and aerospace markets. The thrust of this study is to searching for a high performance nonhermetic conformal coating, which can protect the microelectromechanical system (MEMS) devices from adverse environment as well as mechanical stresses from internal or external due to the encapsulation process and real application. A primary focus to ensure the MEMS device operating reliability exposed to hostile environments is to use multilayer conformal coatings that exhibit complementary merits to meet the stringent requirements. Historically, reliable nonhermetic encapsulation materials are: high purity epoxies, room temperature vulcanized (RTV) silicones, fluorinated silicone based materials, fluorinated acrylics, polyurethane, Parylenes (poly para-xylyenes Types N, C, D, F), BCB and polyimides [2]–[10], []These materials can meet a broad variety of different aspects of environmental protection criteria.

The key specifications established by the aerospace industry for the prototype MEMS device required that the candidate commercial encapsulants should display excellent resistance to mobile ion permeation, high humidity, and constant thermal and mechanical properties within wide temperature extremes (-55°C–100°C). Low internal stress of the coating is also importance for guaranteeing the accurate static force sense of the piezopressure sensor. Additionally, a low profile (<2 mm) was necessary to ensure a proper fit for the device.

Typically, conformal coating applying methods include glob-top coating, spray coating, curtain coating, spin coating, and chemical vapor deposition (CVD). And one of the key issues for achieving high reliability with nonhermetic conformal encapsulants for microelectronic applications is pre-encapsulation cleaning [2]. Qualified pre-cleaning of the electric circuits can perform the best interfacial adhesion. The cleaning procedure adopted in this study based on the method developed by Wong and McBride [11].

Candidate nonhermetic conformal coatings exhibit chemical compatibility, an appropriate curing temperature, low residual stress, good adhesion, and good solvent resistance. A historical overview indicated that encapsulants meeting our targeted environmental protection requirements could be silicone elastomers and gels, epoxies, and poly-para-xylylenes (Type C and F) available from commercial market. However, their environmental protection performance for our particular application was not previously reported in the literatures. Therefore, it was our aim to select the high promising candidates and evaluate and characterize their required properties for MEMS device encapsulation.

Conformal coating candidate materials employed in this study include commercial epoxy resins, silicone elastomers and gels, Parylenes C and F type, and fluoroacrylics. Detailed test sample preparation, test setup and vehicle's configuration as well as series results will be presented in the following.

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J. Wu, R. T. Pike, and C. P. Wong are with the School of Materials Science and Engineering, Packaging Research Center, Georgia Institute of Technology, Atlanta, GA 30332-0245 USA.

N. Kim and M. Tanielian are with The Boeing Company, Seattle, WA 98124-2207 USA.

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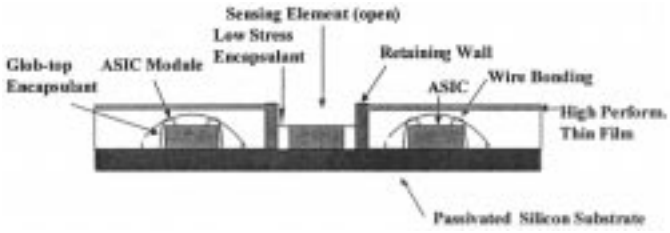


Fig. 1. Complex topographical schematic of prototype MEMS device.

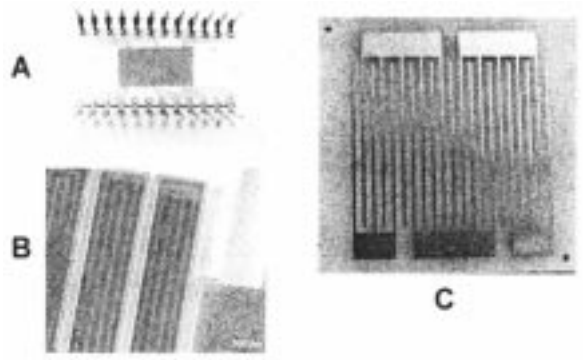


Fig. 2. Sample test vehicles for environmental testing. (a) Triple track resistor test vehicle—TiPdAu metallization + 75 μm spacing. (b) Scanning electron microscope (SEM) picture of metal tracks of triple track test vehicle. (c) Modified Bellcore Y comb pattern—Cu metallization + 2 mm spacing.

II. Experimental

A. Prototype MEMS Device

The design specifications of the prototype MEMS device consisted of a 20.32 mm × 20.32 mm (equals to 0.8 in × 0.8 in) square multilayer structure with a complex topographical surface (Fig. 1). The base substrate was polyimide passivated silicon. Copper metallization was employed by an MCM-D process. Discrete devices, analog and digital application specific integrated circuits (ASICs) were integrated onto the substrate with a pick-and-place approach. Interconnection was accomplished with a variety of methods including wire bonding, beam lead attach, electrically conductive adhesives, and tape automated bonding (TAB).

B. Moisture Ingress, Mobile Ion Permeation, and Adhesion Test Vehicles

In order to determine the conformal coating environmental protection, resistance to moisture ingress and mobile ion permeation into the prototype device, unique test vehicles were selected to characterize the encapsulants nonhermetic sealing properties (Fig. 2). Fig. 2(a) is the triple track test (TTT) vehicle used for monitor resistance to moisture ingress and mobile ion permeation of the coatings. It consists of a set of three meandering parallel conductor lines, which are made of Ta_2N metallization with 75 μm line spacing. And TiPdAu contacts metallized on an Al_2O_3 substrate. Fig. 2(b) is a SEM image of metal tracks of TTT vehicle.

The test vehicle used for evaluating surface insulation resistance [Fig. 2(c)] was modified Bellcore Y SIR test board. These vehicles consisted of a 80 mm × 80 mm (equals to $2in \times 2in$) FR4 substrate with a five-probe pad copper metallized comb pattern with 2 mm line separation.

Die shear adhesion testing was conducted with 25.4 mm × 25.4 mm (equals to $1in \times 1in$) monolayer and bilayer conformally coated test vehicles as substrate and 2 mm × 2 mm (equals to 80 mil × 80 mil) silicon die as tiles. These vehicles consist of a sandwich test structure that includes a substrate of polyimide passivated silicon, a middle layer of the conformal coating materials (silicone, or silicone and parylene C, or epoxy and silicone) and a top layer of tiles passiviated with polyimide. Each die shear adhesion data was an average of ten samples.

C. Pre-encapsulation Cleaning Technique

The preparation of silicon substrates, tiles, SIR and TTT test vehicles included the following sequential steps to insure cleanliness:

1) 5 min soak in Bioact® EC-7R™ terpene organic solvent;
2) 5 min soak in terpene during ultrasonic cleaning;
3) 5 min soak in isopropyl alcohol;
4) 5 min soak in isopropyl alcohol during ultrasonic cleaning;
5) 5 min soak in distilled water during ultrasonic cleaning;
6) 3 time rinses with distilled water;
7) 2 min soak in 50 ppm surfactant;
8) 5 min rinse with distilled water;
9) 120 °C bake at 28 mm Hg for 30 min;
10) UV-Ozone treatment at 50 °C for 5 min with a Samco Model UV-1 dry stripper.

D. Test Vehicle Encapsulation

Liquid epoxy and silicone coatings were spin coated on substrates with a Specialty Coating Systems (SCS), Model P6708D coater. Parylene C and Parylene F films were deposited by the Gorham Method. Parylene C films were deposited with a SCS model PDS 2010 coater. Parylene F films were deposited with a unique vacuum deposition system at SCS. The candidate commercial encapsulants evaluated in this study were cured according to manufacturer specifications.

FP4450 and *FP4460* are single component thermal cure epoxy resins supplied by Dexter Hysol. These epoxies were cured in air at 150 °C for 30 min.

Q1-4939 and *DC1-4207* are two components (base resin and curing agent) thermal cure silicone encapsulants supplied by Dow Corning. Q1-4939 encapsulated samples were cured in air at 150 °C for 60 min. DC1-4207 samples were cured at 60 °C for 30 min.

DC1-2577, *DC1-2620*, *DC1-3140*, and *DC1-3145* Clear are single component room temperature vulcanizing silicone encapsulants supplied by Dow Corning. Curing of these coatings was accelerated in air at 100 °C for 30 min.

Parylene C (poly (chloro-para-xylylene)) and *Parylene F* (poly (difluoro-xylylene)) were originally developed by Union Carbide. These materials were conformally coated onto test vehicles at Specialty Coating Systems, Inc. using a three-step vacuum deposition process.

Fluorad FC-720 and *FC-725* are fluoroacrylic conformal coatings in butyl acetate supplied by 3M Specialty Chemicals Division. The coatings were cured at 100 °C for 30 min.

E. Environmental Stressing

Device operating conditions were simulated with temperature humidity aging and thermal shock cycling. Temperature humidity aging was conducted at 85 °C/85%RH with 175 V bias for 1000 h in a Blue M Humid-Flow combination temperature and humidity cabinet. Thermal shock cycling was performed with a Blue M Model test chamber with 1 h temperature cycling from −55 °C to 125 °C with 15 min dwelling at each temperature extreme (Mil. Spec 1014, Step B) for a total of 500 cycle.

F. Die Shear Testing

Die shear measurements were performed with a Royce Instruments System 552, 100 K adhesion analyzer. Test vehicles subjected to 85 °C/85%RH for accelerating aging first and then were measured every 200 h for a total duration of 1000 h. Samples subjected to thermal shock cycling were measured every 100 cycle for a total of 500 cycle.

G. Surface Insulation Resistance and Leakage Current Measurements

To characterize the conformal coating resistance to moisture ingress and mobile ion permeation, TTT and SIR test vehicles were used. *In-situ* SIR measurements were conducted with an Alpha Metals, Model 300 Sirometer, at 85 °C/85%RH with 100 V dc for resistance measurement for 1000 h. *In-situ* leakage current and resistance measurements were recorded at 85 °C/85%RH, 175 V dc bias, for 500 h with a Keithley Model 82 005 multiplexer, Hewlett Packard 34 401A multimeter, Hewlett Packard Harrison 6207B DC Power Supply, Keithley Model 616 Electrometer, Dana 4700 Multimeter, and National Instruments LabView 5.0 software for automation.

III. Results and Discussion

The initial selection of an effective protective coating for the prototype MEMS device was focused on identifying two candidate material/encapsulants. The first candidate encapsulant was applied to the surface of the epoxy encapsulated ASICs to provide environmental protection and a planar surface of the microelectronic devices and interconnects, at the same time the internal stress caused by the coating will not affect the accurate measurement of the piezoelectric pressure sensor. The second candidate encapsulant was deposited in the central part of the device to protect the sensing element and beam leads from the corrosion due to the mobile ion permeation and moisture ingress. This candidate encapsulant required extremely low stress to prevent the device and beam lead shifting during operation.

A survey conducted to identify potential commercially available candidates indicated low stress silicones have historically shown good performance in harsh and hostile environments. Furthermore, a thin film of Parylene deposited on top of the silicone will enhance its organic contamination [12]. Electrical testing methods including SIR and triple track testing methods were selected as a fast and efficient screening method for qualifying potential candidate materials.

TABLE I
Surface Insulative Resistance
Results (85 °C/85% RH, 100 V dc Applied on Every 6 h)

Selected SIR Data	
Material	**Average 1000 Hour Resistance (GΩ)**
DH FP 4450	3.61
Dow Corning 3-6550	3.0
Parylene C 15 μm	2.85
Dow Corning Q1-4939 (10:1)	.77
3M FC-725	.0064
3M FC-722	.0026

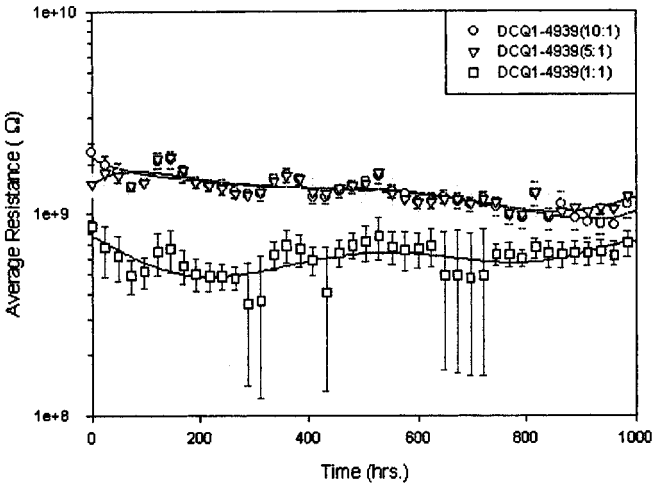


Fig. 3. Surface insulative resistance result for low tress silicone elastomers and gels (85 °C/85%RH, 100 V dc applied on every 6 h for resistance measurement).

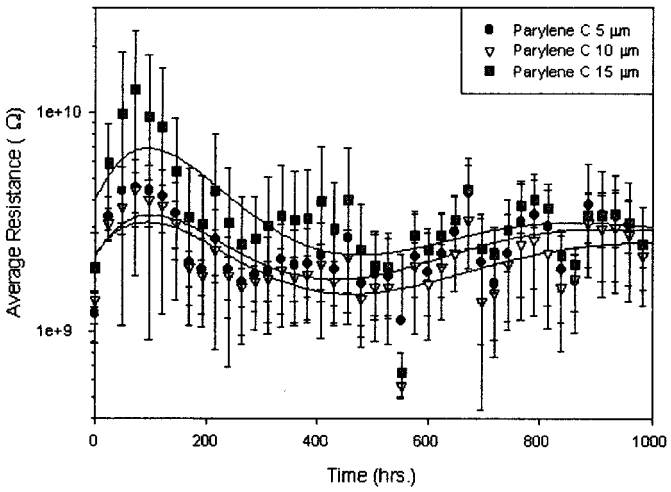


Fig. 4. SIR results for varying film thickness of Parylene C (85 °C/85%RH, 100 V dc applied on every 6 h for resistance measurement).

A. Surface Insulation Resistance (SIR) Measurements

SIR test was performed in 85 °C/85%RH to provide electrical property evaluation for screening candidate encapsulants. The SIRs of the candidates were measured every 6 h under 100 V dc. Two test vehicles were coated with Dow Corning Hipec 3-6550

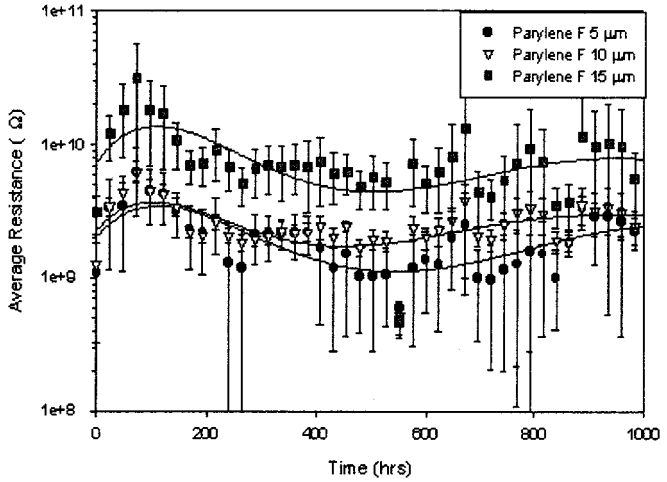


Fig. 5. SIR results for varying film thickness of Parylene F. (85 °C/85%RH, 100 V dc applied on every 6 h for resistance measurement).

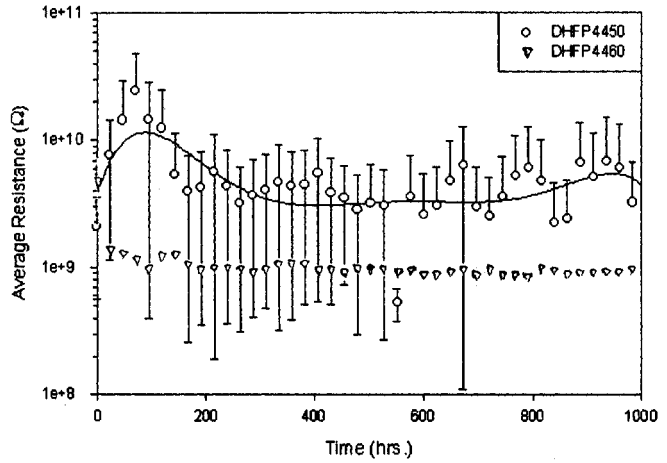


Fig. 6. SIR results for commercial epoxy encapsulants (85 °C/85%RH, 100 V dc applied on every 6 h for resistance measurement).

silicone for standardization and to monitor chamber cleanliness. The DC 3-6550 had an initial resistance range of 2.8×10^9–$4.1 \times 10^9\ \Omega$ and completed 1000 h of testing in the range of 2.7×10^9 to $3.5 \times 10^9\ \Omega$ (Table I). SIR measurements of the low stress Q1-4939 indicated that the silicone gel of 10:1 provided the best environmental protection (Fig. 3).

Samples coated with 5 μm, 10 μm, and 15 μm films of Parylene C and F performed well with resistance value beginning between 1.0×10^9 and $5.0 \times 10^9\ \Omega$ and finishing 1000 h in the same range (Figs. 4, 5). Parylene F coatings show a higher degree of environmental protection than their Parylene C counterparts. No visible surface corrosion was observed on the Parylene F coated comb patterns after 1000 h aging.

SIR measurements of epoxy glob-top encapsulants surveyed indicated that the FP4450 afforded a higher degree of protection than the FP4460 (Fig. 6). The thickness of the epoxy encapsulants was in excess of 76.2 μm (equals to 3 mils). The FP4450 showed better environmental protection properties than FP4460 and SIR results of $0.5 \times 10^9\ \Omega$ to $3.0 \times 10^9\ \Omega$ initially and $0.01 \times 10^9\ \Omega$ to $6.0 \times 10^9\ \Omega$ after 1000 h.

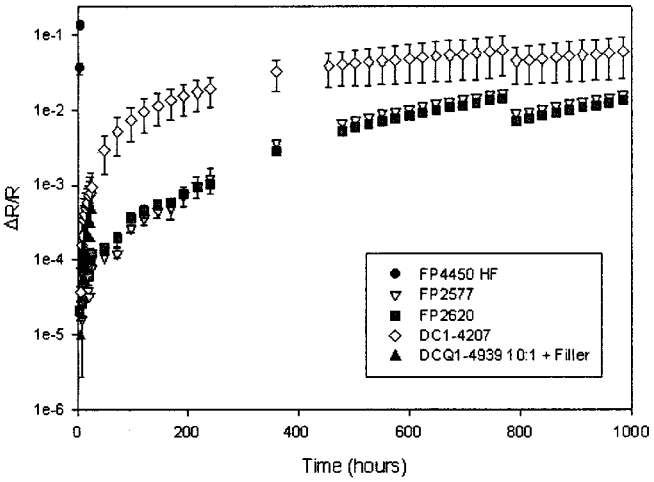


Fig. 7. *In-situ* triple track resistance measurements for candidate encapsulants. (85 °C/85%RH, 175 V dc bias).

TABLE II
TRIPLE TRACK RESISTANCE CHANGE DATA AFTER 1000 h THB AGING

Selected Triple Track Data	
Material	**Average 1000 Hour ΔR/R**
Parylene F 15 µm	.000166
Parylene C 15 µm	.00055
Dow Corning 3-6550	.0485
Dow Corning 1-4207	.061
Dow Corning Q1-4939 (10:1+ Filler)	Failed
Dexter Hysol FP 4450	Failed
Dexter Hysol FP 4651	Failed
Bare Chip	Failed

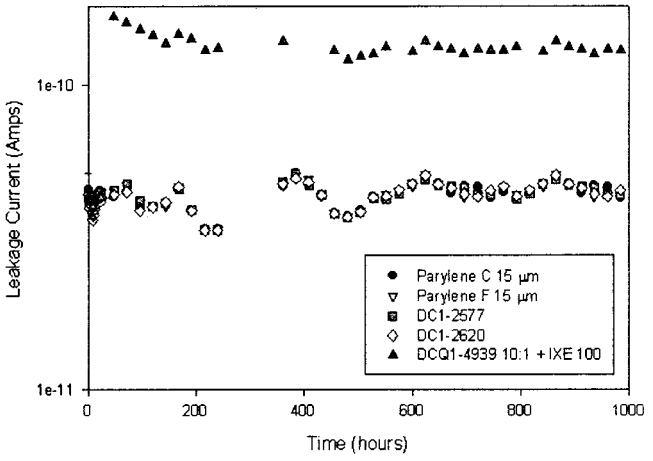
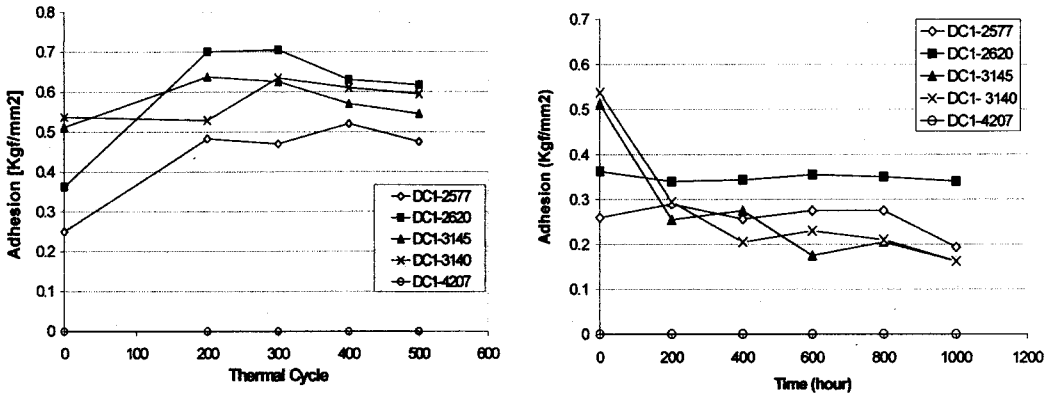


Fig. 8. Triple track leakage current data on the selected conformal coating candidates (85 °C/85%RH, 175 V dc bias).

In comparison, results from the 3M FC-722 and FC-725 began between 0.0021×10^9 and $0.0056 \times 10^9\ \Omega$. After 1000 h, the range had shifted to span from 0.0024×10^9 to $0.0029 \times 10^9\ \Omega$ for the FC-722 and from 0.0071×10^9 and $0.0058 \times 10^9\ \Omega$ for the FC 725. These results indicate an inferior resistance to moisture ingress. So these two materials were discounted from further qualification testing (Table I).



(a) (b)

Fig. 9. Adhesion changed with aging time for monolayer silicone elastomers on polyimid passiviated Si substrates: (a) thermal shock (-55 to 125 °C) cycling and (b) temperature/humidity (85 °C/85%RH) aging.

B. Triple Track Test (TTT) Measurements

TTT was employed to screen potential candidates in terms of their environmental protection property. For qualification screening of potential candidate materials, the TTT vehicles as shown in Fig. 3(a) was selected. Resistance measurements were recorded along each metal track in the serpentine pattern. The change in resistance would prove to be the primary factor in determining the failure of test vehicles during triple track measurements. A chip failure was defined as any drastic change in the resistance of the triple track test vehicle. In general, resistance values were seen to slowly increase over time due to corrosion of the Ta_2N metallization pattern. When a vehicle failed, the slope of the change-in-resistance plot would rapidly increase. Once initiated, the failure process occurred in less than 24 h. Chips that did not fail within the initial 24 h could still be examined for relative merit. The epoxy glob-top encapsulants revealed their inherent weakness in a high humidity environment, resulting from the presence of epichlorohydrin in the base agent that increased the presence of chloride ion contaminants. Epoxies typically failed quickly, like the FP4450 HF shown in Fig. 7, which failed within 48 h. Silicone elastomers all showed similar trends, but the DC1-4207 was clearly inferior to DC1-2620 or DC1-2577. The shift seen in the plot around 750 h is due to system irregularities. In the case of Parylenes, 15 μm coatings showed similar excellent moisture resistance protection results, as indicated in Table II.

The leakage current values do not provide convenient answers as trip track resistance. The noise floor of the testing apparatus was found to be roughly 4.0×10^{-11} A as established by the silicone elastomers and Parylenes in Fig. 8. The data does indicate that the silicones and Parylenes show excellent resistance to moisture and mobile ion permeation. The actual leakage current values were $<4.0 \times 10^{-11}$ A, indicating that the triple track test chips were still protected. Chips that had suffered mobile ion permeation would experience leakage current values that would rise above the noise floor. This would differentiate the good and bad conformal coatings. Samples such as Hipec Q1-4939 10:1 mixed with some filler generated bad results.

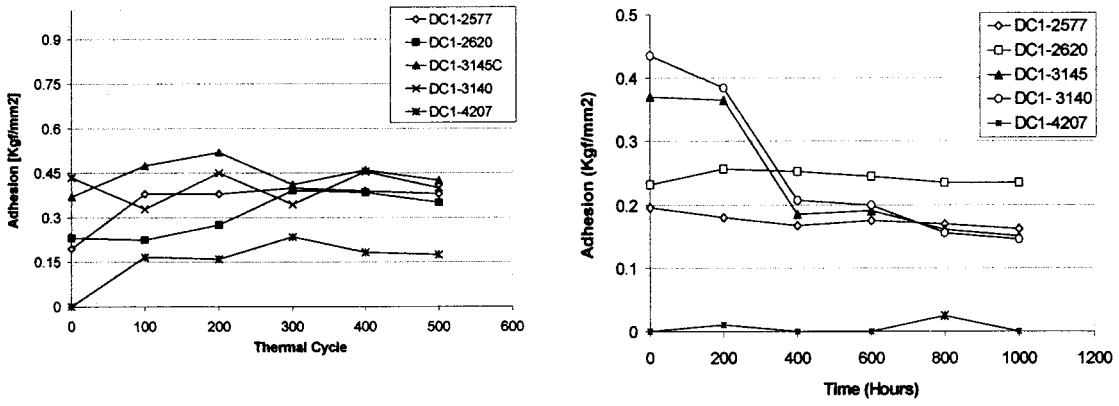
C. Long Term Hostile Environmental Conditioning for Die Shear Testing

Preparation of single-layer and bi-layer coating samples had been done for evaluating the long-term adhesion reliability. The single-layer and bi-layer coated test vehicles were subjected to thermal shock cycling and temperature humidity aging with 100 cycles and 200 h intervals, respectively.

Samples subjected to these two testing protocol consisted of five single-layer, 15 bi-layer combination coatings. The single-layer samples include the following silicone elastomers: DC1-2577, DC1-2620, DC1-4207, DC1-3140, and DC1-3145. All silicone samples were coated on polyimide passivated Si substrates. The 10 bi-layer combination coatings included the above silicone elastomers coated with Parylene C using two novel adhesion promoters to serve as adhesion promoters. The additional five bi-layer coatings were combinations of FP4460 and the above five silicones. FP4460 was selected as a base layer due to the known reliable property for the ASICS glob-top protection. Bi-layer combinations were selected to mimic the nonhermetic coating approach for the MEMS device. Results from the thermal shock cycling and temperature humidity aging testing indicate that the DC 1-2620 has the strongest adhesion for a single-layer design. For bi-layer designs, DC1-3145 silicone in combination with Parylene C showed the highest adhesive strength. Alternatively, DC 1-2620 with FP 4460 epoxy showed the best strength in comparison to other silicone candidates used with epoxy. Results from these investigations are outlined in Figs. 9–12.

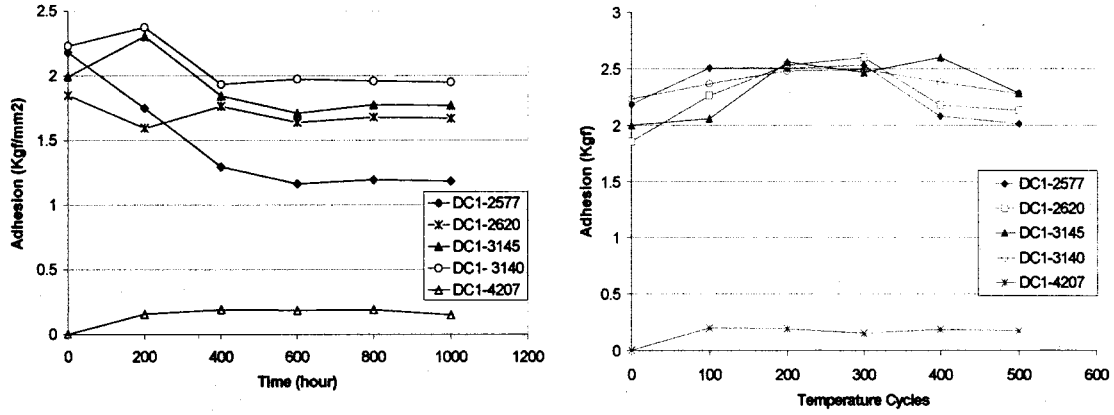
Fig. 9 shows that the DC1-2620 conducts the best adhesion between silicones and polyimide passiviated Si substrates. Adhesion between silicone candidates and PPS substrates initially increase after exposure to thermal shock cycling, however, after a certain stable period, the samples started to decrease due to the CTE mismatch. While adhesion variation after exposure to 85 °C/85% RH shows continuous decreasing with the aging time, which might be caused by the diffusion or release of the oligmers escaped from silicone bulky matrix.

Adhesion for bi-layer of silicone elastomers and FP4460 changed with aging time is displayed in Fig. 10. The adhesion



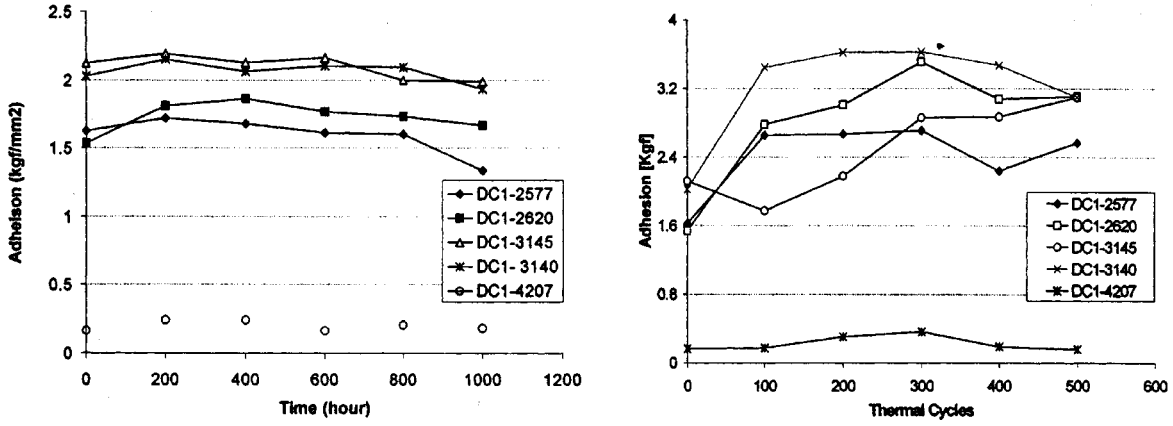
(a) (b)

Fig. 10. Adhesion between silicone elastomer and FP4460 epoxy changed with aging time for bilayer conformal coating system: (a) thermal shock (−55 to 125 °C) cycling and (b) temperature/humidity (85 °C/85%RH) aging.



(a) (b)

Fig. 11. Adhesion between silicone elastomer and Parylene C changed with aging time for bi-layer system with Adhesion promoter AP-B: (a) temperature/humidity (85 °C/85%RH) aging and (b) thermal shock (−55 to 125 °C) cycling.



(a) (b)

Fig. 12. Adhesion between silicone elastomer and Parylene C changed with aging time for bi-layer system with Adhesion promoter AP-A; (a) Temperature/Humidity (85 °C/85%RH) aging; (b) Thermal Shock (−55 to 125 °C) cycling.

continuously decreases when the samples were exposed to 85 °C/85%RH atmosphere. While the adhesion show a slight change for samples during the course of thermal shock cycling testing, but not as much as that of single-layer silicone on the polyimide passiviated Si substrate.

Adhesion variations for bi-layer of silicone elastomer and Parylene C after 85 °C/85%RH aging and thermal shock cycling are illustrated in Figs. 11, 12. After the 85 °C/85%RH aging, adhesion for both of AP-A and AP-B bi-layer samples tended to decrease continuously. While samples exposed to thermal shock

cycling remained their adhesion. This result indicates that the AP-A is a more effective adhesion promoter to the interfacial adhesion.

IV. Conclusion

In this investigation, preliminary results have been outlined for candidate coatings that show promise as effective nonhermetic encapsulants for MEMS device applications. Comparison of SIR and TTT results indicate that the silicone elastomers and Parylenes type F and C display excellent resistance to moisture ingress and mobile ion permeation and are viable moisture and mobile ion permeation barrier candidates. The 10:1 ratio (part A: part B) of Q1-4939 is an excellent candidate for applications requiring a low stress encapsulant that also provides good environmental protection. The epoxy based FP4450 and FP4460 epoxy encapsulants exhibit excellent adhesion and fair resistance to moisture ingress, which make them become attractive glob-top candidates. Silicone elastomers show significant promise as a planarizing top-layer that will afford additional environmental protection for enhanced high-reliability applications. Results indicate that the fluoroacrylic coatings evaluated in this study were inappropriate for encapsulation of MEMS devices. Results from additional in-depth investigations including military specification environmental protection qualification testing is of significant interest and will be further examined in this program.

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Jiali Wu (M'99) received the B.S. degree in chemistry from Zhejiang University, China, and the M.S. and Ph.D. degrees in chemistry from the Shanghai Institute of Metallurgy, Chinese Academy of Sciences, Shanghai, China.

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 has been with the Electronics Packaging Research Group, School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, as a Postdoctoral Fellow since 1997. She is currently working on polymer development for the application of various electronics packaging, mainly including multilayer conformal coatings for reliability without hermeticity encapsulation, reworkable high temperature adhesives for advanced MCM-D processing, underfills, and parylene interfacial adhesion enhancement on polymeric passiviated substrates.

Randy T. Pike, photograph and biography not available at the time of publication.

C. P. Wong (SM'87–F'92) received the B.S. degree in chemistry from Purdue University, West Lafayette, IN, and the Ph.D. degree in organic/inorganic chemistry from Pennsylvania State University, University Park.

He was a Postdoc Fellow at Stanford University, Stanford, CA. He spent 19 years with AT&T Bell Labs and was elected a Bell Labs Fellow in 1992. He is a Regents' Professor with the School of Materials Science and Engineering and a Research Director with the Packaging Research Center, Georgia Institute of Technology, Atlanta. He holds over 45 U.S. patents, numerous international patents, and over 240 technical papers in the related area. His research interests lie in the fields of polymerica materials, reaction mechanism, IC encapsulation, hermetic equivalent plastic packaging, electronic packaging processes, interfacial adhesions, PWB, SMT assembly, and component reliability.

Dr. Wong received the AT&T Bell Laboratories Distinguished Technical Staff Award in 1987, the AT&T Bell Labs Fellow Award in 1992, the IEEE Components Packaging and Manufacturing Technology (CPMT) Society Outstanding and Best Paper Awards in 1990, 1991, 1994, 1996, and 1998, the IEEE Technical Activities Board Distinguished Award in 1994, the 1995 IEEE CPMT Society's Outstanding Sustained Technical Contribution Award, the 1999 Georgia Tech's Outstanding Faculty of the Year Award, the Georgia Tech Sigma Xi Faculty Best Research Paper Award in 2000, and the University Press (London, U.K.) Award of Excellence in 2000. He is a member of the National Academy of Engineering, a Fellow of AT&T Bell Labs, and was the General Chairman of the 41st Electronic Components and Technology Conference in 1991. He served as Technical Vice President (1990 and 1991) and President (1992 and 1993) of the IEEE CPMT Society.

Namsoo P. Kim received the B.S. degree from Seoul National University, Seoul, Korea, in 1972, and the M.S. and Ph.D. degrees in material science and engineering from the University of California, Los Angeles, in 1979 and 1981, respectively.

After graduation, he worked at American Microsystems, Inc. (AMI), Santa Clara, CA, as Manager of the Packaging and Materials Department. His research involved material issues for silicon integrated circuit fabrication and packaging technologues. In 1986, he joined Boeing where he is Associate Technical Fellow in the Department of Information, Communication, Sensor & Electronics Technologies, Boeing Phantom Works, Seattle, WA. He is Technical Principal Investigator for various packaging research programs utilizing emerging component technologies including micro-electro-mechanical system (MEMS), CSPs, and flip chips. His past research programs included application of thin and thick film materials for multichip modules and photovoltaic devices. He is an author or co-author of over 40 journal and conference papers and three U.S. and international patents.

Minas H. Tanielian received the B.S., M.S., and Ph.D. degrees from the University of Chicago, Chicago, IL, in 1974, 1975, and 1981, respectively, all in solid state physics.

He is the Manager of the Sensor and Microsystems Technology Organization, Boeing Phantom Works, Seattle, WA. His interests span a variety of technologies and applied physics topics such as thin film materials, electronic devices, photonics, and microsystem integration. Current activities relate to smart sensors, multisensor networks, and various microsystem applications.