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Multi-layered Poly-Dimethylsiloxane As A Non-Hermetic Packaging Material For Medical MEMS

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***Abstract*— Poly-dimethylsiloxane (PDMS) is an attractive material for packaging implantable biomedical microdevices owing to its biocompatibility, ease in application, and bio-friendly mechanical properties. Unfortunately, devices encapsulated solely by PDMS lack the longevity for use in chronic implant applications due to defect-related moisture penetration through the packaging layer caused by conventional deposition processes such as spin coating. This paper describes an effort to improve the performance of PDMS as a packaging material by constructing the encapsulant from multiple, thin roller casted layers of PDMS as a part of a polymeric multi-material package.**

I. Introduction

Implantable micro devices for monitoring or therapeutic functions are valuable medical instruments with expanding significance in next-generation healthcare systems. In the early 1960s, the size, weight and cost of the packaged systems ultimately limited the deployment of implantable devices to life-threatening cases only. With recent advances in micro and nano technologies, wireless powering and thin film rechargeable batteries, implantable systems are being miniaturized and expanded to many clinical fields. Such fields include cochlear implants for the deaf and visual prosthesis for the blind. Other examples include pain suppression devices, drug infusion systems, as well as epileptics and neural disorder treatment systems.

The next generation of implant systems will be small, compact, light, intelligent, sensitive, and less invasive. As such, they will be invaluable for real-time, chronic and continuous monitoring in patients. Device miniaturization and function convergence for microsystems developed specifically for research on human physiology using near "normal" animals could rapidly advance clinical healthcare, as well as pharmaceutical development. These microsystems, either implanted or surface attached, could also be a key technological step toward realizing personalized healthcare of the future. However, the high cost and long turnaround time in a traditional rigid-box hermetic package cannot meet the needs of new implants. The packaging is now the main bottleneck impeding the progress and implementation of these new medical microsystems and new innovative solutions.

Based on our extensive years of research experience in developing implantable electronic systems [1-4], we have observed that dip-coated and brush-coated polymeric layers, namely hysol epoxy and medical grade silicone, when used as sub-mm encapsulation layers for implants, generally fail in 1 to 6 days. Molded thick polymeric coatings (> mm in thickness) fair better but still fail in a few days to several weeks, and the performance is widely scattered. Examination of the encapsulants reveals that the failures are localized failures centered on defects and not a total breakdown of the material itself. From these observations, a multiple material thin film coating approach was proposed to meet the mechanical, vapor barrier, and biocompatibility requirements of MEMS implants [1, 4]. A hypothesis was also formulated that a non-hermetic polymer-based package technology with a usable lifetime can be developed if the localized, defect-related failure mechanisms can be prevented. To achieve successful non-hermetic packaging using polymeric materials, three distinct criteria must be met. They are: 1) the polymeric materials must maintain a high electrical resistivity when saturated with saline solution; 2) the device to be packaged must be free of any ionic contamination on its surface; and 3) the local defect density must be greatly reduced. As a part of the effort to develop a non-hermetic micropackage for implants, a study of the potential and limitation of multi-layer roller-casted medical grade PDMS which will serve as an outer layer of a multi-material polymeric package was investigated.

The first phase of this study investigated the efficacy of using multiple layers of the same polymeric material to minimize the effect of localized defects in the polymer. The basis for using a multilayered approach is illustrated in Fig. 1 which shows a simple cross-sectional schematic of a substrate coated with a 3-layer film. The multilayer technique decreases the probability of a defect causing failure by utilizing multiple roller coated thin film layers to overlap defects in previously deposited layers. If a defect

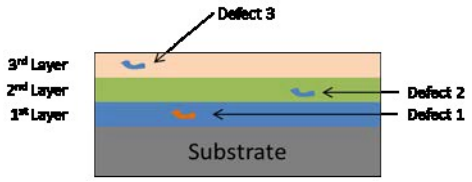

Fig. 1. Schematic of how multiple layers can overlap defects on individual layers to decrease the probability of failure.

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such as an air bubble is present in the first layer (the size of the defects is in the same order of the thickness of the layer), a second layer will overlap and cover that defect, thus decreasing the probability of the bubble causing a failure. A third layer further decreases the probability of a failure resulting from a through defect in the first and second layers which substantially decreases the overall probability of defect-related failure.

The quantitative rationale for this concept is as follows. In an amorphous polymer body, the "universal" free volume fraction value is 2.5% [5] and therefore the total ratio of defects to body volume in a polymeric coating layer is at least 2.5%. For a multilayered encapsulant, if the randomly distributed defects (i.e., air bubbles) in each layer overlap to form *through defects* in the encapsulant then a localized failure will occur. Let γ be the probability to have such a defect in a thin film with thickness, d, area A, and the size of the bubble, (nd). Then the area of the bubble will be $(nd)^2$, the probability to have a defect in first layer is $\gamma_1 = \gamma$. The probability to have a defect in the second layer to overlap a defect in the first layer would be $\gamma_2 = \gamma_1 (\gamma/A)(nd)^2 = \gamma^2\{(nd)^2/A\}$. For a 3 layer film, the probability to have a bubble at the same location within area $(nd)^2$ would be $\gamma_3 = \gamma^3 \{(nd)^2/A\}^2$. If A = 10 mm^2, γ = 0.01, n=2, d = 0.05 mm, $(nd)^2 = 10^{-2}$ mm^2, then $\gamma^3 = 10^{-12}$ which is very small. Therefore the chance to have bubble-like through-defect failures will be greatly reduced. A more rigorous analysis [6, 7] yields similar results with a difference of n^m, where m is the number of the layers.

The multilayer process is a commonly used technique to improve the quality of encapsulating layers but, to the best of our knowledge, has not been used in conjunction with roller-cast PDMS. Several issues with conventional deposition processes for PDMS such as the introduction of air bubbles, folds, and inclusions has led to the use of roller-casting as a means of application.

II. PHASE 1

In the first phase of this investigation, we studied the lifetime of three different polymer-coated interdigitated electrodes (IDEs) fabricated on printed circuit boards (PCBs) exposed to saline at 40°C. Figure 2 shows the two test specimen designs included in this investigation. The inter-electrode resistance was measured periodically upon exposure. The initial resistance values were greater than $10^9\Omega$ and failure was declared when the resistance dropped to $10^8\ \Omega$. The coatings were applied using a brush or roller. Initial samples were encapsulated with a single layer of PDMS. These structures had a lifetime ranging from several hours to 6 days. Based on these results, all subsequent samples were coated with at least 3 distinct layers of the same material. Each layer was roller cast and a curing step was performed between each layer. The nominal thickness of each layer was estimated to be roughly 100 µm. For the same material multilayer studies, several polymeric films were tested. The Hysol epoxy and medical grade PDMS (Dow Corning MDX-4 4210) exhibited the longest lifetimes of all materials tested in 40°C saline, with a maximum

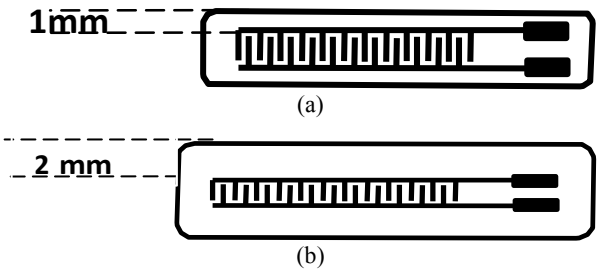

Fig. 2. PCB Test Board structures. (a) with a narrow edge, (b) with a wide edge.

lifetime ranging from 30 to 40 days for a single material with a 3 layer coating. The failure spots were small and difficult to locate even under a microscope. We found that by applying a potential of 10V across the IDEs on a failed device, a small current would flow and after many hours the copper electrodes would become blue, indicating that $Cu(OH)_2$ had deposited at the failure spot as a result of moisture-induced oxidation. Through optical inspection, we found that failures were associated with: 1) small air bubbles 2) foreign particles (dusts and fibers), 3) sharp spikes due to solder connections, 4) vertical steps in the substrate and other structures with a very small radius of curvature, and 5) residual ionic contamination of the substrate surface. To address issues related to particulate and ionic contamination, an initial substrate cleaning process was incorporated in subsequent coating processes and the coatings were applied under a particulate controlled hood [8]. A second set of experiments using 15 PCBs were made each coated with 3 layers of PDMS and tested in saline at 40ºC. Eight of the test structures were protected with a 175 µm-thick PDMS coating and had an average lifetime of 32 days. The maximum life time of the eight 175 µm-thick PDMS coated devices was 53 days, and 93 days for a 246 µm thick FP 4450 epoxy coated device [9].

The same processes were used to package several 2-channel micropower telemetry units. A schematic of one micropowered telemetry unit can be seen in Figure 3. Each unit was packaged with two layers of two materials (FP4450 + PDMS) with an approximate total thickness of 250 µm. These units were evaluated in saline at 40°C. The #10 telemetry unit had a lifetime of 73 days. Failure was defined when the current consumption of the device increased from its initial value of 0.610 µA to 0.657 µA. Figure 4 shows the consumed current versus time for this device [8, 9]. After reaching the failure point, the device was retrieved from the saline solution, cleaned and dried. Upon retesting, the device returned to its original operating status with respect to consumed current. Interestingly, this result shows that the lifetime of the packaged telemetry unit is smaller than but almost equal to the sum of the lifetimes of the two materials that constitute the multilayered package (FP4450 + PDMS).

From the Phase I experiment several conclusions can be made: 1) the hypothesis that life time can be extended by the packaging technique is verified, 2) the multiple material package lifetime is almost the sum of each material layer, and 3) multiple layers of the same material, the thin film

roller casting technique, and the multi-material approach for a long term micro-package of implants are all worthy of further investigation.

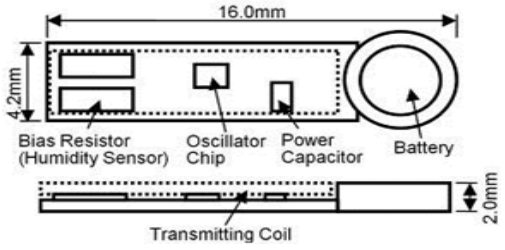

Fig.3. (top) Schematic diagram of the telemetry unit used to evaluate the PDMS-based package developed in Phase 1 [8], and (bottom) photograph of telemetry unit.

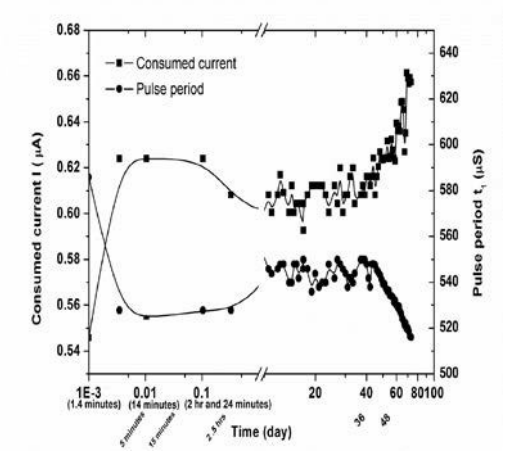

Fig. 4. Consumed power versus time for the packaged telemetry device shown in Fig. 3.

III. PHASE 2

In order to improve the lifetime of PDMS packaging and to decrease the overall thickness of the encapsulant, a second phase of this investigation was recently initiated utilizing an improved pre-encapsulation technique and roller casting process. A principal objective of Phase 2 was to investigate the efficacy of the improved roller casting process in conjunction with the multilayered technique for encapsulants that consist only of PDMS and have an overall thickness less than 100 μm [10]. Such encapsulants would be advantageous for mechanically flexible microsystems (such as neural interfaces) where mechanical compliance and low form factor are highly desired.

In this phase of the investigation, eight test coupons containing eight IDEs fabricated on PC boards were first subjected to a multi-step ultrasonic cleaning procedure. Samples were coated using Dow Corning MDX4-4210 Biomedical Grade Elastomer by roller casting immediately after the cleaning and/or surface treatment. Each IDE received 100 roller strokes at a rate of 1.5 strokes/s and a force equivalent of 250 to 300 g. Samples were cured under vacuum at 70°C. The roller casting process was repeated twice more with a curing step performed between each process. The third coating was cured for 16 hours. Film thicknesses ranged from 40 to 70 μm.

A strict and meticulous handling process of all samples was adopted as contamination was not only found in the environment but also any surface that samples came close to and in contact with. Prior to sample cleaning and roller casting PDMS all of the surfaces, surrounding areas, and tools used to prepare the test coupons were wiped with IPA to decrease the probability of introducing contamination and particulates on the test coupon's surface. The test coupons were also prepared in a class 100 clean room to further reduce the probability of a failure by particulates.

A data acquisition system was designed and constructed to continuously monitor the leakage current of the samples soaked in saline at both 40°C and 85°C. The system was designed to determine average surface insulation resistance as a function of time by monitoring the leakage current between the adjacent electrodes. Typically the IDE's exhibited surface insulation resistance in the 10 GΩ range prior to emersion in the saline bath. Failure of the encapsulant was defined when the average surface insulation resistance dropped below 100 MΩ.

Samples were evaluated using conventional accelerated lifetime soak testing in saline solution [11]. Leakage current was continuously monitored for 32 samples soaked in saline at 40°C and 32 samples soaked at 85°C. Fig. 5 shows the surface insulation resistance vs. time for a representative sample while Table 1 summarizes the results at 85°C. The maximum lifetime of samples subjected to testing at 85°C was 42 days.

TABLE 1. RESULTS OF ACCELERATED LIFETIME STUDY AT 85°C.

Days of field usage	Number of failed plasma treated devices	Number of failed untreated devices
10	8	11
20	13	9
30	0	4
40	1	3
50	1	5

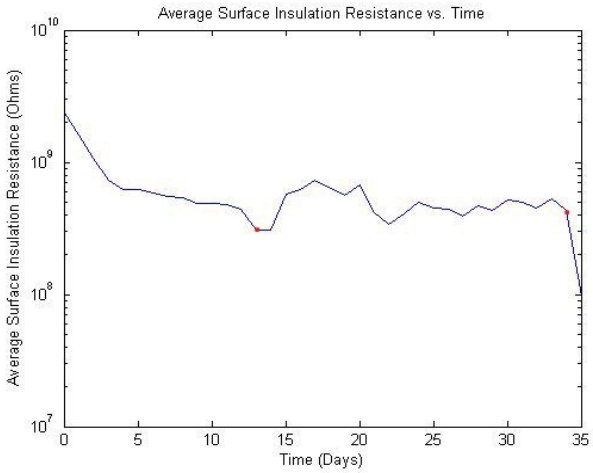

Fig. 5. Average surface insulation resistance vs. time for a sample subjected to accelerated lifetime testing at 85°C in saline. This particular encapsulant failed after 35 days of testing.

After failure, samples exposed to accelerated lifetime tests at

85ºC were examined for evidence of coating failure. Fig. 6 is a photograph of a test coupon after electrical failure. The photograph shows that for this sample, electrical failure is associated with voids that formed between the PDMS coating and the PCB substrate. It is likely that these voids are a result of hydrogen gas formation caused by electrolysis of moisture that penetrated the coating. Fig. 7 shows surface insulation resistance versus time from one of the 32 samples

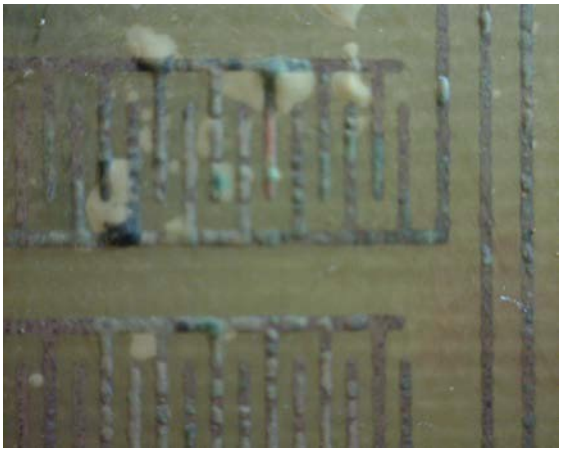

Fig. 6. Optical photograph of an IDE after testing at 85ºC showing a defect that led to device failure.

subjected to the 40ºC soak tests. No failures have been observed for any samples in this testing group after 228 days. To the best of our knowledge, this represents the longest successful accelerated lifetime test for PDMS-based packages, especially in sub 100 µm thickness range. Using an activation energy of 0.79eV calculated using an acceleration ratio of 38.8 [9], the estimated lifetime of the roller-cast PDMS encapsulant is 5.5 years at physiological temperature.

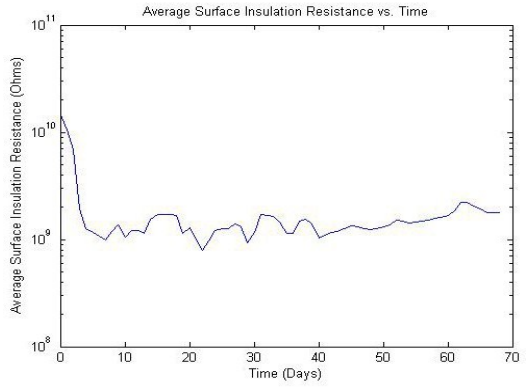

Figure 7: Average surface insulation resistance vs. time for a sample subjected to accelerated lifetime testing at 40ºC in saline. This sample is still under test after 228 days.

IV. Conclusion

The roller casting multilayer technique has shown to be an excellent encapsulating technique for packaging implantable micro-electromechanical systems. Accelerated lifetime tests in saline at 85ºC indicate that a PDMS-based tri-layer has potential for use in long-term (2 to 5 years) implant applications, an observation that is supported by tests at 40ºC in saline. At the very least, the roller casting technique appears to be suitable for packaging microdevices for fundamental medical research. The simplicity and lower cost and time make the evaluation of non-hermetic packaging of implant systems a worthy subject for further study.

Current research is focusing on a multi-material package consisting of Parylene C as a vapor barrier, epoxy to serve as a mechanical protection layer, and PDMS as a biocompatible outer coating to reduce any negative side effects while in a body environment. The interfaces between each material as well as the interface between the substrate and packaging materials are currently under investigation. Current studies investigate the effectiveness of several adhesion promoting and surface treatments on the lifetime of multi-material packaging.

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References

[1] W.H.Ko and T. Spear, “Packaging Materials and Techniques for Implantable Instruments”, IEEE Eng. Med. And Biol. Magazine, 2, March 1983, pp.22-38, 1983.

[2] C.D. Fung, P.W. Cheung, W.H.Ko, and D.G. Fleming (Eds.) Micromachining and Micropackaging of Transducers, Elsevier, Armsterdam, 1985.

[3] W.H. Ko, M.R. Neuman, and K.Y. Lin, "Body Reaction of Implant Packaging Materials," Biomaterials, Editor: L. Stuart, Plenum Press, New York, 10:55-65 (1969).

[4] W.H. Ko,” Packaging of Microfabricated Devices and Systems”, Materials Chemistry and Physics, 42, pp.169-175, Elsevier (1995).

[5] A. Asano and K. Takegoshi, “Free volume study of amorphous polymers detected by solid-state 13C NMR linewidth experiments”, J. Chem. Phys. 115, 8665 (2001).

[6] É.É. Bronshtein, B.S. Va, “Probability method of assessing the quality of multilayer coatings” Chemical and Petroleum Engineering, 1989. 25(1): p. 42-45.

[7] V.A. Golovin, “Multilayer Protective Polymeric Coatings:Providing for Their Continuity”, Protection of Metals, 2007. 43(7): p. 679-688.

[8] L. Bu, P. Cong, H. Kuo, X. Yeh, and W. Ko, ”Micro-package of Short Term Wireless Implantable Microfabricated Systems”, IEEE EMBS, EMBC 2009. Ann. Int. Conf. Proc., Paper SaET7.4, pp. 6395-6399, (2009).

[9] R. Zhang “The Study of MEMS Acoustic Sensor for Totally Implantable Hearing-Aid System and Micropackage Technology for Implantable Devices”, M.S. Thesis, Case Western Reserve University, Cleveland, OH, 8/2011.

[10] S. B Lachhman, “Roller-Cast Poly-Dimethylsiloxane as a Non-Hermetic Encapsulant for MEMS Packaging”, M.S. Thesis, Case Western Reserve University, Cleveland, OH, 1/2012.

[11] R. K. Ulrich, W. D. Brown, “Advanced Electronic Packaging: 2nd Edition”, Wiley-IEEE Press 2006.