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Adhesion and Moisture Barrier Characteristics of Roller-Cast Polydimethylsiloxane Encapsulants for Implantable Microsystems

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Abstract

Poly-dimethylsiloxane (PDMS) is a highly attractive polymer to encapsulate implantable micro-electromechanical systems (MEMS) due to its biocompatibility, simplicity in processing, and low Young's Modulus. However, conventional deposition processes introduce defects that prevent the use of PDMS as a long term packaging material. In order to address these issues, we have developed a novel roller-casting process for depositing PDMS. This paper presents the findings of a study to simultaneously investigate the adhesion properties and moisture barrier characteristics of roller-cast, multilayered PDMS encapsulants subjected to testing in saline at 85°C.

I. Introduction

Today, there is significant interest in developing mechanically-flexible MEMS for implantable medical applications mainly due to the decreasing feature size of such devices, low cost, and ease of implantation of these systems. Micro-electromechanical systems utilize microfabrication techniques to create functional microscale structures for sensing and control of their local environment. Sensing and actuation functions of MEMS require direct interaction and contact with the local surrounding environment while the control electronics such as the power source require complete isolation, thus making the packaging of MEMS much more complex than that for integrated circuits [1].

Current packaging technologies for implantable MEMS are limited by material selection, size, robustness, biocompatibility, and lifetime. Currently, packaging options for implantable MEMS are significantly limited. Metallic or ceramic hermetic packaging enclosures guarantee long device lifetime, but limit miniaturization, mechanical flexibility, and are also associated with high costs. The hard surfaces of ceramic and metallic packaging also can introduce negative side effects to the host of the implantable system. Polymeric encapsulations offer mechanical flexibility, but are not suited for long-term implantation. Increasing the lifetime and robustness of a polymer-based packaging technology provides many potential benefits: an increased protection against the environment, increased usable lifetime of the device, and increase implant lifetime. Poly-dimethylsiloxane (PDMS) is of particular interest for microsystem packaging because of its ease of use, low cost, biocompatibility, and low Young's modulus. Unfortunately, traditional deposition processes for PDMS, such as spin coating and dip coating, give rise to defects that eventually lead to catastrophic failure of the implanted system. To address this issue, we have developed a novel, roller casting method to deposit PDMS-based encapsulants. Proof-of-concept studies

have shown promising results, yielding an encapsulation that lasted 38 days in saline at 40°C and 13 hours in saline at 85°C [2]. This study extends our previous work by simultaneously examining the moisture barrier characteristics and adhesion properties of multi-layered roller-cast PDMS subjected to accelerated lifetime testing.

II. Experimental Design

Accelerated lifetime testing (ALT) is an experimental method used to decrease the time for a failure to occur on a component, analyze the mechanism of failure, and formulate a solution to prevent or decrease the probability of that failure occurring. Several techniques have been developed for accelerated testing, all of which can be grouped into three main categories: mechanical tests, electrical tests, and environmental tests [3].

Environmental testing was chosen since the main concern for this study is to develop and test packaging materials that will provide excellent protection against ion carrying bodily fluids and moisture. Environmental accelerating testing subjects samples to simultaneously high temperatures and high humidity to induce failures. In this study, test coupons containing eight interdigitated electrodes (IDEs) were subjected to soaking in saline solution at 40°C and 85°C at a relative humidity (RH) of 100%.

Test coupons contacting eight IDEs were fabricated on PC boards and subjected to a rigorous multi-step cleaning procedure based on ultrasonic agitation. The preencapsulation cleaning process is considered to be one of the most critical processing steps in device packaging [4]. A schematic of a test coupon containing the 8 IDEs can be seen in Fig. 1.

Coupons were subjected to one of the following surface treatments: (1) untreated (no surface treatment), (2) oxygen plasma treated, or (3) coated with Dow Corning P5200 adhesion promoter, after which PDMS was deposited by roller casting using a method detailed elsewhere [1]. Each surface treated sample was divided into groups. A description of each group with the corresponding amount of samples and IDEs tested can be seen in Table I.

The surface treatments are applied just after cleaning the substrate a just prior to PDMS deposition. The oxygen plasma surface treatment used a RF power, time, and pressure of 25 W, 25 s, and 900 milliTor, respectively. Cleaning, surface treatments, roller-casting, and all of the other processing steps were performed in a class 100 cleanroom facility as foreign materials and particulates have been seen to decrease the lifetime of polymer based packaging [5]. Dow Corning MDX4-4210 Biomedical Grade Elastomer was also prepared strictly following the processing steps prescribed by the manufacturer. A 1:10 mixture of elastomer to curing catalyst is mixed and degassed for 30 minutes to remove all of the visible bubbles.

A systematic approach was developed to apply the PDMS film uniformly to the IDE substrate by roller casting. The sample area was much larger than the roller length; therefore the samples were split into four column-like regions as shown in Fig. 2.

Beginning with Column #1, the roller was placed at the bottom edge of the board after dipped into a tray containing PDMS. The roller was moved from the bottom edge to the top edge of the sample completing one stroke. Each column receives 100 strokes at a rate of 1.5 strokes per second. A force equivalent to 250 to 300 grams was applied by the roller during the coating procedure. This process is repeated until all of the remaining columns (2, 3, and 4) have received a coating of PDMS. The coatings were cured at 70°C under vacuum for 1 hour. The process is repeated two more times to obtain a total of three layers before being subjected to accelerated lifetime testing.

A data acquisition system was designed and fabricated to continuously monitor the leakage current of 144 samples soaked in saline at 40 and 85°C until failure. Fig. 3 contains a schematic of the accelerated lifetime testing system.

Failure was defined when the surface insulation resistance dropped below 100 MΩ. National Instruments Labview™ 7.0 was used to control the switching of 16:1 multiplexers, acquire the temperature of the controlled water baths, and measure surface insulation resistance measurements of each test coupon. A vertical flow valve was installed to maintain a constant water level due to the fast evaporation rate of the saline solution at 85°C.

Pull off tests were performed on samples in accelerated testing at 85°C after electrical failure was observed. Samples were also subjected to pull off testing prior to soak testing under accelerated conditions at 85°C. Dollies were glued to the surface of the PDMS with Dow Corning RTV adhesive. Prior to mounting the dollies on the surface of the samples and applying the adhesive, Dow Corning adhesion promoter was applied to the surface of the dollies and left over-night to cure. Once the adhesion promoter cures, Dow Corning RTV adhesive is applied to the surface of the dollies and glued to the surface of the sample. After the adhesive reaches a full cure, the edges around the stud were cut to ensure the force of the pull-off is solely in the vertical plane. Dollies were pulled off solely in the vertical direction using a pull tester. Fig. 4 contains a photograph of the pull tester.

III. Results and Discussion

All of the test coupons treated with the adhesion promoter failed immediately upon immersion into the 85°C saline solution. This may have been due to the high sensitivity of the adhesion promoter to moisture. Approximately 20% of the oxygen/plasma samples also had immediate failures. Samples that received no treatment exhibited no immediate failures, and also exhibited the longest lifetimes when compared to the lifetimes of the oxygen/plasma samples. Samples were observed under microscope before the commencement of the accelerated lifetime study to ensure there were no particulates found in between the PDMS layers and also the PDMS-substrate interface. Fig. 5 and Fig. 6 show average surface insulation resistance versus time for representative untreated and plasma treated samples, respectively. The maximum lifetime of both the untreated and oxygen plasma samples was 1008 hours (~ 42 days). This is the longest reported accelerated lifetime study at 85°C for roller-casted multilayered PDMS encapsulants.

For the untreated and plasma treated samples, voids were seen to form after resistance failure presumably due to electrolysis-related reactions. Liquid was found inside of the voids when they were probed with a pin, indicating that in these regions, significant moisture either permeated through the PDMS film, a failure occurred at the edge of the test specimens, or moisture penetrated along the interface between the PDMS and the electrical feed-throughs. Fig. 7 contains photographs of an untreated sample before soak testing, an untreated sample with voids after failure, and an oxygen/plasma sample with voids. Voids were seen to initially form at or near the electrode structures and eventually expand over time to span across several trace-lines.

Pull strength tests were performed on samples after failing resistance testing. Table II summarizes the pull strength of all samples before and after immersion testing. The pull strength of all the samples decreased after immersion testing. The highest pull strengths were observed for samples with the adhesion promoter while the untreated and plasma treated samples exhibited comparable pull strengths. The decrease in bonding strength suggests that the bonding between the packaging material-substrate interfaces must be strong to achieve a longer lifetime and must also maintain a high adhesion throughout implantation.

Furthermore, this suggests that not only the material properties, such as the rate of moisture permeation of the polymeric packaging material, but also the interface properties are of great importance to successful encapsulation. Untreated samples were seen to have longer lifetimes and higher bonding strength than oxygen plasma treated samples suggesting that higher bonding strength of packaging materials will provide a longer lifetime and better reliability. Voids were only seen after electrical failure.

Test coupons evaluated at 40°C have recently failed after 10 months of testing, representing the longest successful accelerated lifetime test for PDMS-based packages. From the accelerated lifetime data, encapsulant lifetime at physiological temperature is estimated to be ~ 5 yrs. Large voids were also seen to occur in samples at 40°C after failure.

IV. Conclusion

The adhesion of all the samples subjected to 85°C in saline solution was seen to decrease after electrical failure. Samples treated with Dow Corning P5200 adhesion promoter failed immediately but had the highest pull off strengths. Untreated samples were seen to have the longest lifetimes and also higher pull off strengths than that of oxygen/plasma treated samples. A decrease in pull off strength was observed after electrical failure. Voids were observed after failure indicating that the durability and lifetime of the adhesion is of great importance. Samples tested at 40°C failed after 10 months. To the best of our knowledge this is the longest recorded accelerated lifetime study for PDMS encapsulants.

References

1. Lachhman, S. MS Thesis. Case Western Reserve University; Cleveland, OH: 2012.
2. Zhang, R. MS Thesis. Case Western Reserve University; Cleveland, OH: 2011.
3. Ulrich, RK.; Brown, WD. Advanced Electronic Packaging (IEEE Press Series on Microelectronic Systems). 2. Wiley-IEEE Press; 2006.
4. Wong CP, McBride R. Preencapsulation cleaning methods and control for microelectronic packaging. IEEE Transactions on Components Packaging and Manufacturing Technology Part A. 1994; 17
5. Bu, L.; Cong, P.; Kuo, H.; Ye, X.; Ko, W. Micro package of short term wireless implantable microfabricated systems. Annual International Conf. of the IEEE Eng. in Med. and Bio. Society; 2009.

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Fig. 2.
Schematic of the four imaginary columns used for reference while the coating procedure is performed.

Fig. 3.
Schematic of the data acquisition system designed for the accelerated lifetime of PDMS based encapsulants at 40 and 85°C.

Fig. 4.

A photograph of the pull tester used to measure the adhesion of 85°C accelerated lifetime study samples before and after soak testing.

Fig. 5. Average surface insulation resistance vs. time for an untreated sample in saline at 85°C. Lifetime ~ 28 days.

Fig. 6. Average surface insulation resistance vs. time for an O₂ plasma treated sample in saline at 85°C. Lifetime ~ 15 days.

(a)

(b)

(c)

Fig. 7. Photographs of IDE's: (a) untreated sample before soak testing, (b) untreated sample after soak testing showing voids, (c) plasma treated sample after soak testing showing voids.

TABLE I

GROUPS OF EACH SET OF DEVICES WITH THE TYPE OF SURFACE TREATMENT ADMINISTERED.

Group	Number of IDEs	Surface Treatment	Temperature
A	32	None	40°C
B	32	None	85°C
C	32	Dow Corning P5200 Adhesion Promoter	85°C
D	32	Oxygen/Plasma Surface Treatment	85°C

Table II

Pull strength before and after soak testing at 85°C in saline.

Unsoaked	Plasma Treated (N)	Untreated (N)	Adhesion Promoter (N)
1	73.4	83.8	150.1
2	74.8	95.3	140.4
3	73.2	75.8	145.7
4	40.5	85.7	144.6
After Soaking			
1	12.5	38.5	55.4
2	25.2	14	57.9
3	5.5	38.9	63.7
4	18.8	37.3	55.1