

Accelerated Testing Of Anodically Bonded Glass-Silicon Packages In Salt Water

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SUMMARY

This paper reports long-term accelerated test results of glass-silicon packages in aqueous solutions including phosphate buffered saline (PBS) and de-ionized water. An analysis of the dominant failure mode and the long term biocompatibility of the packages is also performed. Using the accelerated data at 85 and 95°C in saline with an Arrhenius model we calculate an activation energy of 1.26 eV and an expected lifetime of 177 years at 37°C in saline. After an analysis of the failed samples, we find that the main failure mechanism is the dissolution of the polysilicon layer at elevated temperatures in saline, which causes premature failure of the package. In animal models the package is consistently found to be biocompatible and robust after implantation periods of up to a year.

Keywords: Accelerated testing, Glass Package, Feedthroughs

INTRODUCTION

Packaging of electronic devices for use in harsh environments, such as the human body, presents a number of problems which are not a factor in most conventional applications [1]. Here the devices operate in an environment which is highly corrosive to many of the thin films used on the circuits. The requirements for the implantable devices that have to operate in the body for extended periods include: the package should stay hermetic for the duration of the implant, it should be chemically stable in order to prevent harmful side effects to the surrounding tissue, it should be mechanically durable and should be small enough to be implanted in volumes on the order of cubic millimeters.

Among some of the approaches that satisfy these requirements are the deposition of thin films, such as polymers, or using a protective shell, such as a titanium case, to seal the sensitive components. The main drawback of the former approach is that the encapsulating layer is typically only good for a short period. Some of the disadvantages of the latter approach are the difficulty of transferring signals in/out of the package and the inability to use RF telemetry to power the devices unless an antenna is placed outside of the package. In addition, both approaches typically require complex assembly procedures and can not always be miniaturized to accommodate many emerging applications. Our group has utilized glass capsules bonded to silicon substrates as a means to encapsulate implantable devices [1,2]. This package has been used in an implantable microstimulator for neuromuscular microstimulation, as shown in Figure 1. The telemetrically operated microstimulator utilizes a solenoid coil as an antenna

to receive an RF signal from an external transmitter. An integrated circuit chip, located under the coil, picks up the RF signal, generates a constant supply voltage and stores it on a hybrid chip capacitor, demodulates the carrier to extract control commands transmitted to the implant, and delivers a constant current pulse for a specified period of time to the biological tissue surrounding it. The receiver coil, the capacitor, and the integrated circuit CMOS chip are all protected from biological tissue using the glass capsule. The stimulating electrodes that are in contact with the tissue are outside the glass capsule, located at the two ends of the support silicon substrate, and are connected to the electronic circuitry using feedthrough lines supported on the silicon substrate. The microstimulator is required to operate in the body for a period of 40 years, and the package should provide a hermetic environment for the circuitry, the coil, and the capacitor during this time.

Obtaining a detailed understanding of the stability and hermeticity of this type of package in harsh environments is important for many applications involving biomedical and

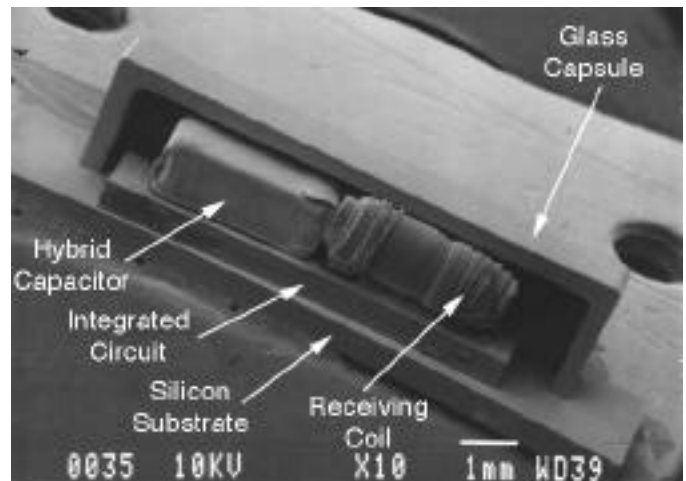


Figure 1: SEM micrograph of an implantable microstimulator.

microelectromechanical systems (MEMS). In this paper we will present results from long-term accelerated testing of these glass-silicon packages in salt water and DI water solutions, and will discuss the biocompatibility of these packages based on long-term tests in animal models. First, a short description of the fabrication process for the silicon substrate and the glass capsule is provided. Next, accelerated test results and biological testing in animal models are presented. Finally, the paper provides some concluding remarks.

STRUCTURE AND FABRICATION

Although the silicon-glass package in this study is used to encapsulate the microstimulator from aqueous environments [2], this same packaging technique could also be used in other applications that require operation in harsh environments and need multiple sealed feedthroughs. Figure 2 shows the overall structure of the glass package, the silicon substrate, and electrical feedthroughs. A #7740 Pyrex glass capsule is electrostatically bonded to a silicon support substrate at a temperature of 320°C using a voltage of ~2000V, thus creating a permanent seal at the interface of these materials [1]. The glass capsule can be either fabricated by custom molding, or batch fabricated using ultrasonic machining of a glass wafer [1], and can have any shape or dimensions. It has a polished top surface and can be easily bonded to a silicon substrate, as shown in Figure 1. The cavity created by the glass capsule and the silicon substrate can house all of the components, such as IC chips, capacitors, inductors, that need to be protected from the outside environment. This packaging technology also allows the formation of several hundred feedthroughs on the silicon substrate. These polysilicon feedthroughs are protected using deposited thin films, as shown in Figure 2. The ability to form integrated, sealed feedthroughs is one of the most important features of this packaging approach and is instrumental to many future microinstrumentation systems where multiple sensors and actuators have to be interconnected with their interface electronics. The glass capsule is bonded to a top polysilicon layer deposited on top of the feedthroughs. Since the glass capsule is bonded directly to this top polysilicon layer, bonding temperature can be reduced to 320°C to prevent damage to hybrid components.

The fabrication sequence for the silicon substrate and the feedthrough lines begins by growing a thick layer of thermal oxide (1 μ m) to isolate the feedthrough lines from the substrate. A layer of LPCVD polysilicon (1 μ m) is next deposited at 570°C by pyrolyzing silane (SiH₄) and doped by diffusing Phosphorus using POCl₃ liquid source at 950°C for 30 minutes. The resulting sheet resistance of the feedthrough lines is ~10 Ω /square. The polysilicon is next patterned using reactive ion etching (RIE) to produce vertical sidewalls required for planarization. A combination of 2000Å low-temperature oxide (LTO) (deposited at 420°C) and 2 μ m of phosphosilicate glass (PSG) is next deposited on top of these polysilicon leads. The LTO improves the adhesion of PSG to the polysilicon lines. The PSG is next reflowed at 1100°C in steam for 2 hours for planarization of the top surface. The PSG is then removed everywhere except the bonding region. The PSG and the layers beneath are encapsulated with a layer of stress compensated 3000Å/1500Å/3000Å LPCVD oxide/nitride/oxide sandwich. Next the dielectrics are removed everywhere outside of the device region down to the silicon substrate. A layer of fine grain polysilicon is deposited using the same recipe as the one used for the feedthrough lines. This layer is lightly doped using a POCl₃ diffusion furnace at 950°C for 30 minutes. This doping reduces the sheet resistance and preserves the surface quality of this top polysilicon layer. This top

polysilicon layer also seals the oxide/nitride/oxide sandwich and prevents its exposure to moisture hence blocking water diffusion through these layers into the package. It is next patterned to define the bonding region over the feedthrough lines. Next, contacts are opened and two metalization steps follow to form stimulating electrodes and interdigitated comb structures that serve as dew point sensors. Finally the wafer is diced and cleaned in hot TCE/Acetone/IPA/DI water prior to bonding. The substrates are next bonded to a glass capsule (Corning #7740) using anodic bonding forming a hermetic seal. Substrate fabrication requires a total of 6 masking steps.

Figure 3 shows the SEM view of a finished glass-silicon package. The glass package is 2.8mm tall and wide, and 8mm long. The walls are 300 μ m in thickness, and the bonding surface is polished. Figure 4 shows a close-up view of the bonding region with its flat surface, the stimulating electrode, and the feedthrough lines connecting the electrode to the area inside the bonding region.

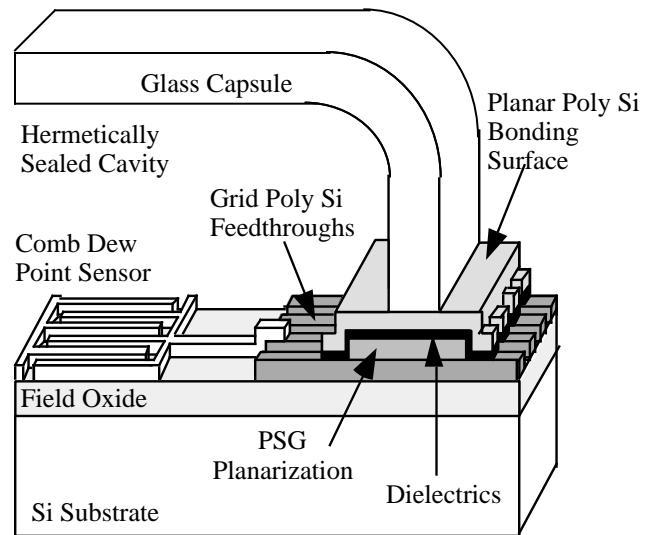


Figure 2: The structure of the hermetic package with planarized polysilicon feedthroughs.

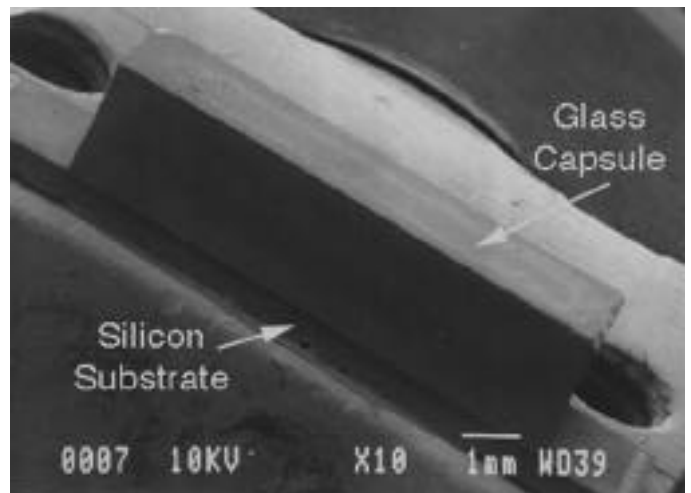


Figure 3: SEM micrograph of the complete package.

ACCELERATED TESTING

The bonded samples are placed in small jars containing the testing solutions (phosphate buffered saline or DI water) at two accelerated temperatures of 85°C and 95°C. Every few days, the packages are cooled down to room temperature, cleaned with DI water, and are tested for room temperature condensation both visually with an optical microscope and electrically with integrated dew point sensors. With visual inspection one can see the presence of moisture inside the glass capsule. For the electrical tests we monitor the impedance of the dew point sensors with the pads that are outside the package. Any condensation of moisture on the outer surface of the dew point sensor is detected by a large change in the impedance magnitude and phase; the package is considered as failed at that point. Package failure for these studies is defined to be condensation at room temperature, which corresponds to less than 5% relative humidity (RH) at a soaking temperature of 95°C, and less than 10% RH at a soaking temperature of 85°C. Ultrasonically machined glass capsules with their polished top surfaces allow one to directly monitor the bonding surface for leakage paths.

Twenty eight glass-silicon packages have been subjected to accelerated testing in saline solution, 20 packages have been tested in DI water, and 14 packages have been implanted in animals. Table 1 shows a summary of our results from soak tests in phosphate buffered saline. Of the 17 samples tested in saline at 85°C, the average lifetime was 116 days and the longest lasting sample survived for a period of 321 days. Moreover, from the 11 samples at 95°C, the average lifetime turned out to be 38 days and the longest package survived for 70 days. Some of these packages failed prematurely, which upon further inspection was attributed to surface nonuniformities, particulates residing on the bonding surface, and glass to silicon misalignment. The packages that failed after an extended period of time did so primarily because of the dissolution of the polysilicon bonding layer in saline at higher temperatures. This will be explained in more detail later.

Moisture penetration into the packages can be modeled with an Arrhenius relationship and the mean time to failure (MTTF) can be modeled as $MTTF = A \exp(-Q/kT)$. In order to predict the lifetime of the package at body temperature (37°C) one needs to determine MTTF and the activation energy (Q) for the process. Using the data at 85 and 95°C, we have calculated an activation energy of 1.26 eV and an expected lifetime of 177 years at 37°C in saline. Note that these values take into account the dissolution of the top polysilicon layer at higher temperatures, and are believed to be worst case values.

In addition to the soak tests in saline we have also soaked a set of 20 samples in deionized water. Of the 10 samples tested in DI water at 85°C, the average lifetime was 396 days with the longest lasting sample surviving for 506 days, whereas out of the other 10 samples at 95°C the average lifetime turned was 136 days with the longest surviving for 484 days. Table 2 shows a summary of our tests in DI water. Using this data, we have calculated an activation energy of 1.22 eV and an expected lifetime of 485 years at 37°C.

We have also bonded a set of 6 samples as a control group and have soaked them in saline at room temperature. Table 3 below summarizes these results. After 2 years of testing all of the samples, except for one, are still soaking and show no signs of moisture. The one failed sample was lost due to mishandling. These room temperature tests are important because they provide data under normal conditions and can be used as a control group to verify the integrity of the calculated MTTF under accelerated conditions.

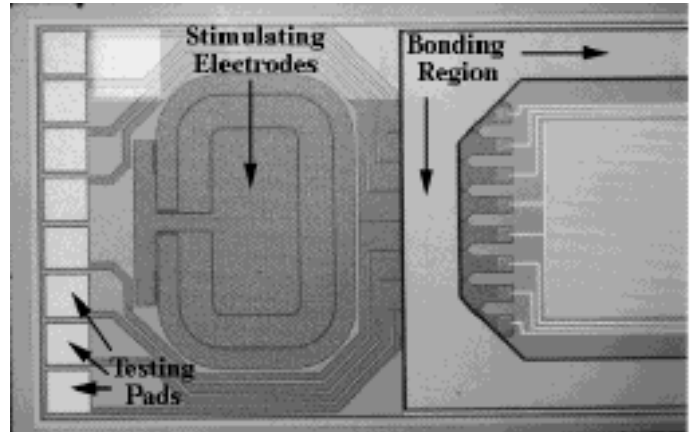


Figure 4: A close-up view of the bonding region, the stimulating electrode, and the polysilicon feedthrough lines.

Table 1. Accelerated testing results in saline.

Soaking Temperature	85 °C	95 °C
Sample size	17	11
Samples failed in 1 day	3	5
Longest lasting package	321 days	70 days
Average lifetime (MTTF)	116 days	38 days
Calculated lifetime @ 37°C	177 yrs.	177 yrs.

Table 2. Accelerated testing results in DI water.

Soaking Temperature	85 °C	95 °C
Sample size	10	10
Samples failed in 1 day	2	1
Longest lasting package	506 days	484 days
Average lifetime (MTTF)	396 days	136 days
Calculated lifetime @ 37°C	485 yrs.	485 yrs.

Table 3. Summary of soak test results in saline at 23°C.

Soaking Temperature	23 °C
Sample size	6
Samples failed in 1 day	1
Samples under test	4
Longest lasting package	770 days
Average lifetime (MTTF)	641 days

An analysis of the failure modes for these packages has been conducted both with a SEM and an optical microscope. We have found that for the tests in saline, the dissolution of the top polysilicon layer is the dominant cause of failure. In all of the packages, the top polysilicon layer dissolves over time leading to the appearance of a leakage path across the bonding region, as shown in Figure 5. We have also measured the approximate dissolution rate for polysilicon in DI water to be 0.15 μ m/day and 0.05 μ m/day at 95°C and 85°C respectively, and for polysilicon in saline to be 1.6 μ m/day and 0.6 μ m/day at 95°C and 85°C. When we inspected the samples that are soaking at room temperature in saline, we did not observe any dissolution of polysilicon and hence did not experience any dissolution related failures within the past 2 years. These observations suggest that dissolution related failures are temperature dependent and hence are not significant at body temperature. The dissolution of polysilicon can be slowed down by coating it with a silicone coating. Coated and uncoated samples were soaked in saline at 95°C, and as shown in Table 4, the mean time to failure (MTTF) of the packages increased from 38 days for uncoated samples to 239 days for coated samples. Due to the slower rate of silicon dissolution in DI water, the samples tested in DI water have lasted longer than those tested in saline. In both cases, the samples in 85°C tests have longer lifetimes confirming the strong dependence of dissolution rate on temperature. Moreover, we expect that the actual dissolution rate at body temperature is significantly lower hence the devices should last at least 177 years in the body. In order to more accurately predict the MTTF of these packages at body temperature, one needs to prevent the dissolution of polysilicon at higher temperatures. Note that this dissolution is an artifact of the testing process and not of the structure.

BIOCOMPATIBILITY TESTING

Fourteen packages have been subjected to biocompatibility studies. After bonding, the packages are sterilized and implanted into animals. These packages have been implanted for different periods of time as follows: 1 month (7 samples), 2 months (4 samples), 6 months (1 sample), 9 months (1 sample) and 12 months (1 sample). After their implant period the devices are harvested, and the tissue surrounding the devices has been sectioned and analyzed. In all of these tests, the devices were covered with healthy tissue indicating that they did not cause any damage to the living tissue. The histology results from the implants show that the devices are biocompatible and robust and also remained hermetic for the duration of the implants. There was no sign of attack on any of the materials used to fabricate the package and there was no sign of dissolution. A more detailed description of the histology results will be reported in the future.

CONCLUSIONS

A silicon-glass package developed for an implantable microstimulator has been extensively tested in saline and

deionized water solutions under accelerated testing conditions. Applying an Arrhenius model to our accelerated results in saline, we have calculated an activation energy of 1.26 eV and found a lifetime of 177 years at the body temperature in saline. One of the main failure modes for the package has been found to be the dissolution of polysilicon in saline at these elevated temperatures. The application of a silicone coating has increased the mean time to failure of the device by a factor of 7 at 95°C. The in-vivo tests which have continued for up to a year, show that the package is biocompatible and rugged.

Table 4: Comparison of coated and uncoated samples soaked in saline at 95°C.

Sample treatment	Coated	Uncoated
Sample size	3	6
Longest lasting package	311 days	70 days
Average lifetime (MTTF)	239 days	38 days

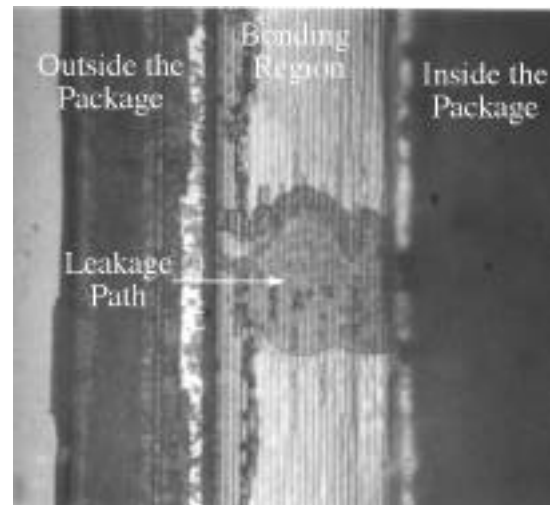


Figure 5: Optical photograph showing a leakage path.

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