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Study of Moisture Transport in Silicone Gel for IGBT Modules

K. Zhang, G. Schlottig, E. Mengotti, O. Quittard, F. Iannuzzo

Abstract – In this paper, an original study on moisture absorption and desorption inside silicone gel for power modules is presented. Silicone gel from two suppliers has been cured at different conditions and exposed to a defined humid environment for a defined amount of time. The mass of gel, the relative humidity, and the temperature at a certain depth beneath the gel surface have been measured, and the moisture diffusion coefficients inside the specimens have been calculated. For the two materials we observed significantly-different changes in gel mass, but similar humidity levels deep inside the gel. The influence of curing and bake-out conditions as well as the difference in absorption and transport in the materials have also been discussed.

1 Introduction

Silicone gel has been commonly used as the encapsulant of the most industrial power modules. Foremost, it provides electrical insulation between conductors [1], but it also acts as a barrier against contaminants and moisture ingress to the active chip and wire bonding area. In outdoor applications like high-speed trains or solar power plants, the power semiconductor devices face harsh work conditions, including high-humidity environments. The moisture penetrates the silicone gel and can induce failure. So far, varied humidity-related failure mechanisms have been studied. In [2], investigations on the influence of humidity and temperature on the electric breakdown strength and the electrical aging performance of silicone gel showed that the breakdown voltage can reduce by more than 50% compared to normal ambient condition. Another moisture-induced failure is the semiconductor's BEOL layer corrosion, which is a long-term aging effect. By applying high voltage at the presence of moisture, the chip edge passivation corrodes, which will cause blocking-capability degradation of the device and finally lead to device failure [3].

Although passivation layers play a crucial role in protecting the devices from humidity, the silicone gel can have a significant influence on the humidity resistance performance of the power modules. To evaluate and compare the gel functionality among different manufacturers, the humidity-, temperature response of silicone gel is needed to perform a thorough analysis of possible stressors. By conducting dedicated tests, the moisture transfer coefficient of the silicone gel can be worked out, used to study moisture transport in gel-filled power modules, compare the humidity-resistance performance for different gels, and ultimately used for a reliable prediction of humidity-related degradation.

2 Experimental procedures

2.1 Silicone gels

We used two-component silicone gels from two manufacturers, both intended for power module encapsulation. The gels were mixed at room temperature using a vacuum chamber with mixing setup to prevent air-filled bubbles, inaccurate proportioning or mixing, which affects the resulting gel purity and properties. The degassing process is very important as the air-filled bubbles' existence reduce the voltage blocking capability as well as the moisture resistance capability of the silicone gel.

Although the cured silicone gel is a stable material, stresses

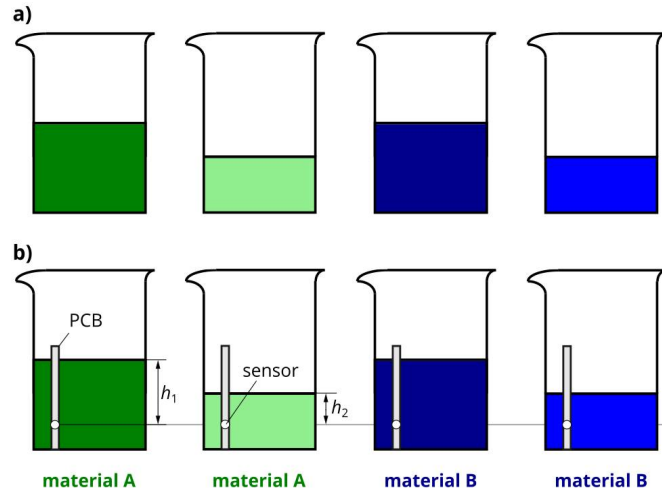


Figure 1. The set of test specimens: Silicone-gel materials from two different manufacturers, A (green) and B (blue), were tested in two groups: (a) as a bulk material for gravimetric measurements, (b) with rH & T sensors in them. Darker color corresponds to a thicker layer of silicone gel.

occurring to power modules in real life have a significant impact on chemical and physical properties of them, thermal stability being one of them. Time and temperature can affect the durability of the gels in high-temperature applications. The higher the temperature, the shorter the time the material will stay functional. Overtime heating or too high temperature during the curing process may influence the gel's lifetime. During the preparation procedure of silicone gel, a thermal oven is usually used to heat to speed up the silicone gel's curing procedure, the heat time is usually from minutes to hours based on the recommendation of the silicone-gel manufacturers. The adopted curing condition is shown in Table I.

Table I-Silicone gels

Material	T_{cure}	t_{cure}	T_{dry}	t_{dry}	Sensor	Depth
A	125	150	125	24+4	Yes	30,40
A	125	150	125	24+4	No	30,40
B	150	240	125	24+4	Yes	30,40
B	150	240	125	24+4	No	30,40

2.2 Test specimens

Test specimens were built in 70mm diameter borosilicate glass beakers (Duran, 95mm high). Figure 1 shows a schematic of all specimens. One group of beakers was filled with two kinds of gel at two different heights, and equipped with humidity sensors inside. Another group of beakers served as the reference specimens without sensors. They have been prepared for humidity-monitoring- and mass-change measurement during



Figure 2. Climatic chamber contains the test specimens.

moisture absorption (85°C/85% rH) and desorption tests (125°C /0% rH).

According to our own experience, gel's datasheet recommendations about mix ratios of the two parts forming the gel material are used as an important reference, but the actual composition is finely tuned in the specific manufacturing process. Therefore, the preparation recipes adopted for gel A and gel B are based on our real-application experience, specifically about mix ratio, curing temperature and curing time.

The beakers for the gel specimens are made of chemically- and high-temperature stable glass. The gel samples have only one surface exposed to the open environment, which makes the moisture-transport process happening through this top surface only. A sensor to measure the relative humidity and temperature inside the gel has been vertically placed into the beaker. The sensor location is measured from the gel surface down to the active point of the sensor module. In real applications of silicone gel, more specifically in our case, the power modules, the silicone gel layer inside the plastic housing would not be as thick as the here-used sensor embedding depth. However, in order to learn the result when silicone gel is exposed at a high-humidity environment for a long time and check if the moisture could ingress into that deep, the aforementioned depth was chosen.

To drive away the residual moisture inside the gel before tests started and to provide a comparable initial state of the specimens, a dry-bake procedure (MSL standard procedure) was performed after curing cool-down and intermediate storage of the gel specimens.

2.3 Climatic chamber and humidity sensor

A state-of-art climatic chamber was used to expose the specimen to humidity. Temperature, relative humidity, and test time can be pre-set to realize the desired test profile. However, if the test profile includes temperature change, the relative humidity inside the chamber will drop drastically during the temperature transient as the humidity supply will turn off at each temperature change. This is only needed to be taken care of if the rapid temperature change is involved within the test profile.

The sensor SENSIRION SHT31 Smart Gadget Development Kit was chosen to monitor both the relative humidity inside the chamber and for specimens. The capacitive SHT31 sensor is constructed with an integrated polymer cover, which effectively protects the sensor opening from direct contact with the silicone gel and thus allows us an embedded use. It gives $\pm 2\%$ rH accuracy (0-100% rH) and $\pm 0.2^\circ\text{C}$ accuracy (0-90°C) which was not re-calibrated for the experiments here

described [4]. To provide a Bluetooth communication, the sensor is supplied on a PCB board. This PCB is designed such that the humidity sensor is attached on a floating island which only has very narrow ligaments connecting to the main part. This structure surrounds the sensor almost entirely with silicone gel and excludes local interface-diffusion components, which could help to reduce the moisture absorption influence of PCB when highly accurate relative humidity and temperature parameter is desired. To power the gadget, additional wires from outside of the humidity chamber were soldered to the PCB.

The test profile of the humidity tests is shown in Table II.

Table II-Test profile

STEP	Item	T (°C)	rH (%)	Time (h)
STEP 0	Bakeout	125	0	30
STEP 1	Absorption	85	85	300
STEP 2	Cooling	NA	NA	12
STEP 3	Desorption	125	0	24

3 Results and discussion

3.1 Moisture Absorption

In order to determine the dry weight before the experiments start, specimens were baked out at 125°C for 30 hours. During STEP 1, relative humidity and mass uptake were measured continuously as the specimens were exposed to 85°C/85% rH conditions in the climatic chamber. To measure accurate weight change, an electronic precision scale was used with a resolution of 0.1mg. All specimens were periodically weighed during the STEP 1 phase. The moisture mass uptake of specimens at the time t was calculated using the following equation:

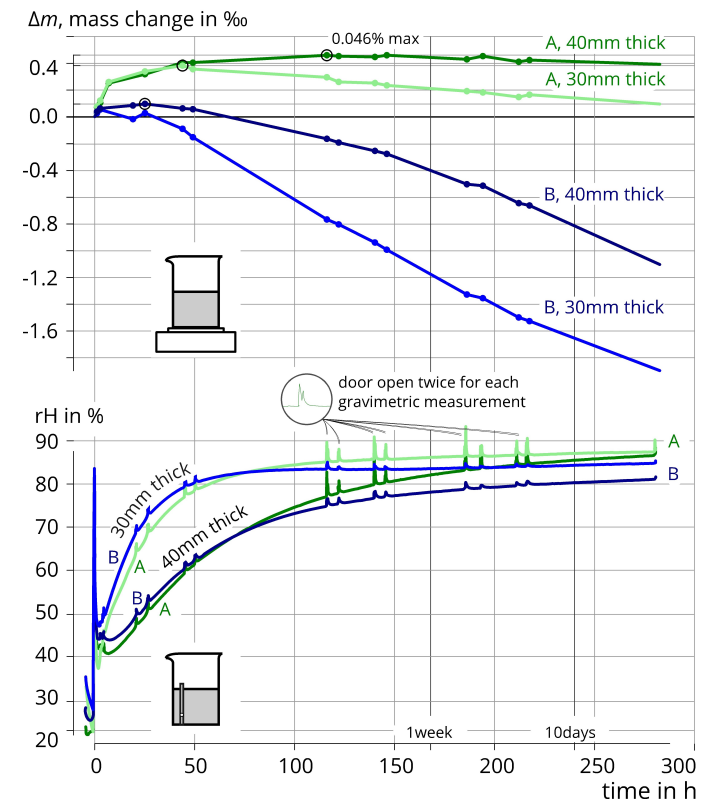


Figure 3. Experiments on moisture uptake of silicone gel for different specimens at 85°C/85% rH.

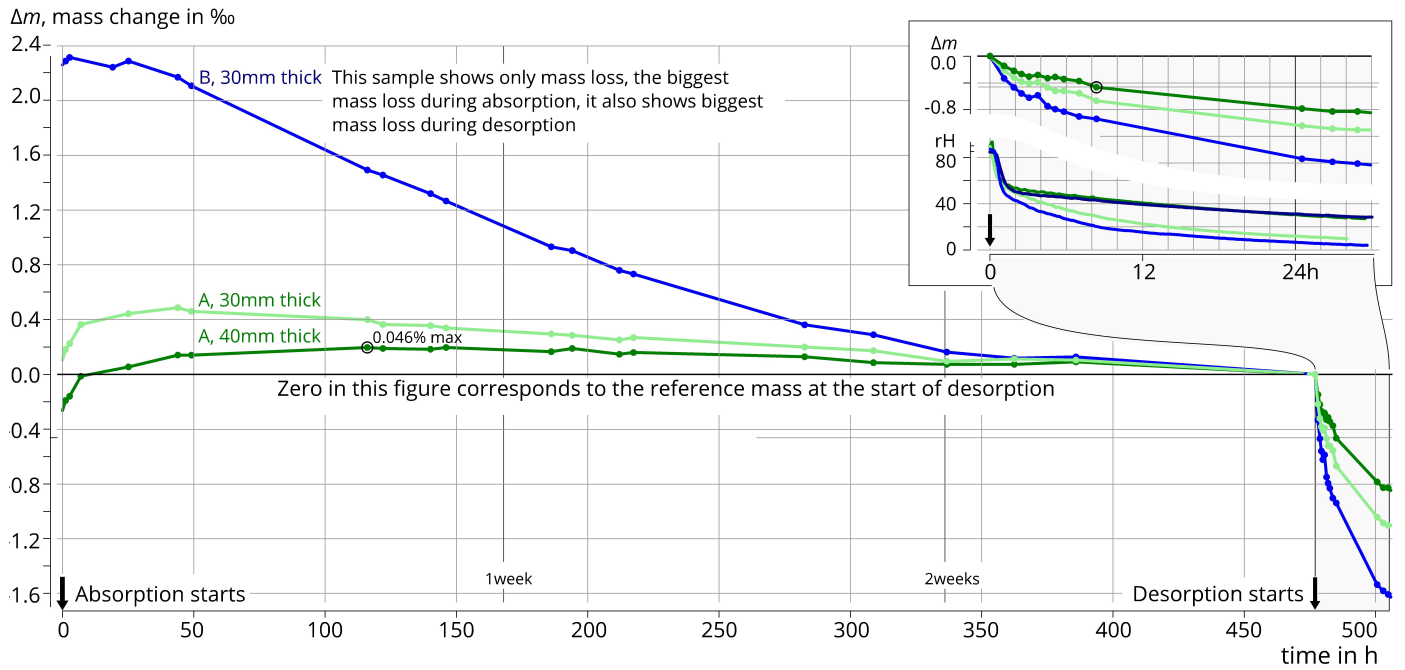


Figure 4. Mass change over time inside silicone gel specimens during moisture absorption and desorption test.

$$\text{Mass Uptake (\%)} = \frac{m(t) - m_{Dry}}{m_{Dry}} \times 100 \quad (1)$$

where $m(t)$ is the specimens' weight at measuring time t and m_{Dry} is the dry weight before experiments start.

The results of moisture absorption at 85°C/85% rH condition from gravimetric measurement (dotted line) and humidity sensors (solid line) are shown in Figure 3.

The relative humidity change among different samples shows a significant difference in the time they take to reach a high level of humidity, such as 80% rH inside the gel, and with it an appreciable moisture-resistance performance between the two materials A and B. The rH signals show an earlier and steeper increase in humidity in the thinner gel samples initially, the geometry dominates the results. After 4 days material B generally shows a slower rate of rH increase compared to material A. After 10 days material B also shows a lower saturation level, independently from the here tested geometry. Note that material A reaches a relative humidity higher than the chamber condition. This suggests to run additional experiments for the in-gel situation, especially for looking at the dew point on power semiconductor surfaces. The spikes shown in Figure 3 are due to the chamber's door opening to allow the gravimetric measurements.

The gravimetric measurements show that only material A gel mass increased throughout the first 50 hours. Afterward, mass change was not so obvious and showed a slightly decreasing trend. For material B, no mass uptake is seen at the beginning. On the contrary, it started to lose mass right after the beginning of the absorption test and continues in a long-time duration. To exclude two additional influences, we repeated the experiment: The few hours of ambient-condition storage after curing followed by bakeout might have concealed an initial, rapid moisture uptake under ambient conditions. Also, the bakeout temperature duration might have started to degrade the material already, or to represent a form of post-process-cure, masking the moisture absorption of interest entirely. Therefore, both an immediate bakeout without possible moisture uptake

time and moisture exposure without prior bakeout were carried out. However, the same behaviour was observed at the beginning of the test for material B, albeit with some difference in mass change.

Possible explanations of the silicone gel mass loss during the moisture absorption test are:

- Some error is to be expected from the gravimetric measurements of humidity absorption in silicone gel. We attempted to measure uptake in 1mm thick samples, but the resolution of the precision scale proved insufficient. However, additional uncertainty is added from the time it takes to bring samples from the curing chamber to the humidity exposure (setup-dependent), from the time it takes to do the gravimetric measurement (difference in exposing the mass-samples to exposing the sensor-samples to humidity), from time required between dry-back and humidification. Also, the sample temperature itself impacts the scale precision, more so for samples with higher thermal mass.
- Material B contains adhesion-promoting additives. The heat exposure of material B during cure, but especially the humidity exposure could drive some additives away out of the gel. A chemical reaction between some of the water molecules with some of the additives leads to volatile products, which leads to a mass loss. The in-gel relative-humidity data shows that the absorption is a parallel process, but for material B the net mass change is a mass loss. The manufacturer confirmed that this magnitude of mass loss can be reached and repeated in experiments. The thinner samples lose mass earlier and also to a greater extent, despite more additives being at disposal in the thicker samples. This can be explained, because although (1) more moisture can travel into the thicker material layer, (2) only the very same absolute surface is available for evaporation of volatile compounds, independently of thickness. Thus, the ratio changes, and the thicker material will have less net loss compared to absorbed humidity.
- The loss of mass due to a chemical reaction with the in-

bound water is temperature dependent – an aspect we did not study by various exposure temperatures. Others have reported on this aspect for polyester with an onset at 80°C [5].

3.2 Moisture Desorption

After the absorption test, the chamber was powered off and the samples were left inside with the door closed for an entire night (STEP 2). Expectedly, as the temperature inside the chamber decreased gradually after reaching in-gel values beyond 80% rH, the embedded sensors reached rH =100% due to reaching the dewpoint.

The subsequent desorption step (STEP 3) is shown in Figure 4. Both relative humidity and gel mass decreased faster than the moisture mass was absorbed at 85°C: The maximum absorbed mass of 0.046% in the thick material A after 5 days yielded in 8h of desorption. While one material B sample could not be continued, the remaining sample, that showed only mass loss during absorption, also shows the biggest mass loss during desorption, For all samples, the 24h desorption mass loss is greater than their mass gain in absorption, despite substantial curing time, the initial dry-bake, and in addition to mass loss exhibited already during the absorption phase. During the test, some cracks were observed inside material B, forming from the bottom of the beakers, which could have happened due to the intrinsic toughness and adhesion properties of silicone gel. However, the cracks disappeared after samples were taken out to room conditions. The well-known self-priming property of silicone gel might explain the self-healing of the observed cracks.

3.3 Moisture diffusion coefficients calculation

The diffusion coefficient is a fundamental parameter in describing the migration of moisture in silicone gel during the moisture absorption or desorption procedure.

Mass transport in silicone gel can be represented as a diffusion process according to Fick's second law, which assumes that the moisture flux is directly proportional to the concentration gradient in a material. Moisture diffusion in silicone gel can be assumed to be one-dimensional, in the direction of the insulation thickness, in our case, is the direction and shortest distance for moisture transport from the gel surface to the humidity sensor. The governing equation for a one-dimensional diffusion in a polymer sheet of thickness L is described by:

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2} \quad (2)$$

where C is the moisture concentration (kg/m³), D is the moisture diffusion coefficient (m²/s), t (s) is time and x (m) is the axis along the concentration gradient.

Using the initial boundary conditions for C:

$$\begin{aligned} C &= C_0, t = 0 \\ C &= C_{eq}, x=L, t \geq 0 \\ \frac{\partial C}{\partial x} &= 0, x=0, t \geq 0 \end{aligned} \quad (3)$$

Here C₀ is the initial moisture content, C_{eq} is the equilibrium moisture concentration, x is the distance from the rH sensor, L is the thickness of the bulk film.

A general analytical solution to the Eq. (2) for the moisture absorption is given by Crank [6]:

$$\frac{C(x,t)-C_0}{C_{eq}-C_0} = 1 - \frac{4}{\pi} \sum_{n=0}^{\infty} \frac{(-1)^n}{2n+1} \exp \left[\frac{-D(2n+1)^2 \pi^2}{L^2} t \right] \cos \left[\frac{(2n+1)\pi}{L} x \right] \quad (4)$$

Usually, it will be assumed that the weight gain of the polymer during the sorption experiment corresponds to the weight of moisture ingress into the polymer. The amount of moisture m_t (i.e. mass gain) that has entered the polymer sheet at time t is obtained after integration of the moisture concentration C(x) over its thickness L:

$$m_t = \int_0^L (C - C_0) dx \quad (5)$$

The experiments presented in this study were performed with silicone gel into a glass recipient in which the moisture ingress will take place only from one side. The moisture absorption will thus be described by the one-side diffusion model:

$$\frac{m(t)-m_0}{m_{eq}-m_0} = \frac{C(t)-C_0}{C_{eq}-C_0} = 1 - \frac{4}{\pi} \sum_{n=0}^{\infty} \frac{(-1)^n}{2n+1} \exp \left[\frac{-D(2n+1)^2 \pi^2}{L^2} t \right] \quad (6)$$

where m_t is the moisture absorbed at time t whereas m₀ and m_{eq} are the initial and equilibrium mass gain, respectively.

The initial phase (m_t / m_{sat} < 0.6) of the moisture uptake is a Fickian process controlled by the concentration gradient where Eq. (6) can be simplified as follows [7]:

$$\frac{m(t)-m_0}{m_{eq}-m_0} = \left(\frac{4D}{\pi L^2} \right)^{\frac{1}{2}} t^{\frac{1}{2}} \quad (7)$$

Using Eq. (7), the diffusion coefficient is usually extracted from the initial slope of the m_t / m_{eq} versus sqrt time plot.

For isothermal conditions, the changes in the relative humidity are directly proportional to the changes in the moisture concentration (i.e. absolute humidity). In this case Eq. (7) can be rewritten through:

$$\frac{m(t)-m_0}{m_{eq}-m_0} = \frac{rH(t)-rH_0}{rH_{eq}-rH_0} = \left(\frac{4D}{\pi L^2} \right)^{\frac{1}{2}} t^{\frac{1}{2}} \quad (8)$$

where rH_t is the relative humidity at time t whereas rH₀ and rH_{eq} are the initial and equilibrium relative humidity, respectively. As for Eq. (8), the diffusion coefficient will be thus

extracted from the initial slope of the fraction of rH_t / rH_{eq} as a function of the square root of time.

By applying the aforementioned calculation method, the moisture diffusion coefficients have been worked out from different specimens, shown in Table III. As it can be seen from Figure 4, due to the mass loss trend in the absorption test, the model to calculate the diffusion coefficient doesn't apply as-is. The mass gain is superimposed with a mass loss that probably also contains some of the gained water molecule mass. Thus, the saturation period cannot be read from the gravimetric measurement result for some specimens, and the model needs to be adjusted.

Table III-Diffusion Coefficients Calculation

$D (m^2/s)$	A_1	A_2	B_1	B_2
$d (m)$	4.00E-02	3.00E-02	4.00E-02	3.00E-02
rH_t / rH_{eq}	1.15E-09	1.02E-09	1.52E-09	2.29E-09
m_t / m_{eq}	1.39E-09	N/A	N/A	N/A

4 Conclusion and outlook

The increase of humidity deep inside the gels at relatively low effective moisture absorption is in line with the common understanding of the typical behavior of silicone gels. The fact that this can be masked by relative and effective mass loss, suggests to measure both the mass change and the in-gel relative humidity in parallel during such moisture studies.

Additionally, the following should be checked:

- The temperature dependency of mass loss should be studied, e.g. by adding a 30°C/60% rH condition. Answering this should guide whether this effect can be ignored and other conditions should be used to study moisture transport, or whether a more sophisticated model should be used to calculate the diffusion coefficients.
- An extended 1w heat exposure should isolate mass changes without moisture-induced reactions.
- Interface diffusion may play a role and should be looked at in a dedicated control experiment with increased interfacial diffusion path.
- Absorption measurements with sensors at various heights could confirm the coefficient calculation.
- A setup with a sensor placed in an enclosure behind the bulk material of interest could bring the sensor back to its intended in-atmosphere use.

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