Elimination of Moisture in Adhesive Sealed Packages

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INTRODUCTION

The technology, which is the subject of this paper, represents the convergence of two long-term programs involving different marketplaces and different product requirements, but with a common concern—moisture barrier. In one case, the project involves the packaging of vision die and the second one involves the packaging microwave die. Both product types require packaging in a cavity package. From a commercial perspective, both programs have been ongoing since 1984. In that year an adhesive product was introduced for the purpose of sealing both and CCD and microwave packages. Most of the packages were and continue and to be ceramic.

During the intervening seventeen years between product introduction and the present, more than twenty million packages have been sealed using the basic package adhesive formulation which is the model for his study.

In late 2000 some difficulties were reported with the performance of a microwave die. Initially it was thought that this problem might be related to potential out-gassing of adhesives during the sealing process. However, this proved to be an incorrect assumption. Actually, the problem was traced to the intrusion of moisture during storage of the sealed devises prior to their use. The die themselves have proven to be substantially more moisture sensitive than the prior technology. It was at this point that the two programs converged since substantial progress had been made in the development of hydrophobic, low ionic formulations designed specifically for vision packages. It is these new formulations and the improvement of process controls that have lead to a reliable solution to the moisture intrusion problem.

The FET moisture intrusion problem represented approximately 15% of the sealed devices. The answer was found in the sealing process. Inadequate temperature and pressure controls in sealing fixtures resulted in a reduced cross section of the sealed footprint. This inconsistency did not effect yield performance as measured by gross or fine leak but it certainly had a major effect on the moisture vapor transmission. The problem was corrected by the combination of a change to an isothermal seal process system plus a more hydrophobic sealing adhesive.

This paper provides a summary of the steps taken in the solution of the reported moisture intrusion problem. Because of its long history of successful performance, the model adhesive formulation compound is used as a comparator to determine if new hydrophobic formulations do indeed outperform materials which have been in successful use for a long time.

DISCUSSION

Interpretation of the data developed is based on the observation of the change in the dew point of the dry air atmosphere in the package cavity. This is done by sealing ceramic circuit covers on cover glass used normally for vision package sealing. It permits the visual observation of the package interior without disturbing the seal. By using this technique it is possible to make observations of the rate of moisture intrusion under various atmosphere conditions. It also permits the observation of moisture egress during periods of low exterior moisture content. Moisture will travel in both directions across the package seal depending upon which side of the seal has the higher concentration. It also allows for the observation of the initiation period during which the sealed membrane becomes moisture saturated prior to the passage of moisture from outside the cavity to the cavity interior.

Testing procedures used for the evaluation of product reliability typically involve the continuous exposure of a package under test to conditions of high moisture content and high temperature. Relationship, if any, between the accelerated testing and reality are unknown. The variables are so many and varied in seal processing, configuration, thickness, etc. that such conclusions would be highly suspect at best. As an example, the thickness cross section of the seal membrane will have a major influence on the rate of moisture penetration. In the case of a FET package, the adhesive thickness cross section between leads is as much eight times greater than the thickness cross section over the leads. It is highly doubtful that any theoretical assumptions based on measurements of consistent membrane thickness would relate directly to the performance of such a package. In addition, the width of the seal footprint has a major influence on the rate of moisture penetration. It was shown in this study that this seal width can vary by fifty percent or more from package to package in a typical multiple fixture sealing process.

At the beginning of this study the test specimens were prepared using a twenty-five-position aluminum fixture. Even though the yield percentage of a sealing process was 100% as measured by gross leak testing, the testing results at various moisture and temperature conditions was so variable that no conclusions could be drawn on the data and the whole program was started over. The scatter was caused by variations in the width of the seal footprint. This is believed to be caused by a combination of variables, including pressure differences in spring to spring in the sealing fixture, variations in the thermal profile as a result of the mass of the fixture itself, and the uncontrolled time variable from the moment the pressure is applied to the temperature at the moment of isolation of the cavity environment.

B-staged adhesives are semi-solid at room temperature and they will cold flow. When spring pressured is applied at room temperature, within minutes, the sealant will begin to compress and flow between the leads. This will directly influence the moment when the air inside the package is captured by the adhesive during the thermal profile, having a significant influence on the pressure inside the cavity during the heat-up cycle. As the air and the package temperature increases during the thermal profile, the pressure of the confined air also rises. At the same time, the viscosity of the adhesive is lowered. This gives rise to the potential that the rising air pressure will cause a bulge in the seal cross section, or even the initiation of a potential blow out or pinhole. It was very difficult to detect such anomalies in a package system which is not transparent. This phenomenon has no measurable effect on the yield performance on gross leak. But the effective reduction in the width of the seal footprint has a major effect on the moisture transmission rates.

In this study, observations were made at different temperature conditions: 85/85, 65/85, 50/85, and 37/85. Table 1 shows the water vapor concentration at various conditions tested in this study. Because of the ease of observation of the dew point inside each package, it is practical to make multiple measurements on a single package by returning the package to the humidity environment. Therefore, observations can be made on the timing interval for the adhesive cross section to become moisture saturated by measuring the initial detection of moisture condensation at 10º C. All of the test specimens have dew points less than 5º C as sealed. After the initiation time was observed, the time interval for the dew point increase from 10º C to 25º C was determined. This gives a direct indication of the rate of moisture penetration at the various test temperatures. Finally, specimens with a dew point of 25º C were removed from the moisture environment and placed in a desiccator jar to determine the rate of egress of the moisture.

We know both intuitively and from the data developed, that moisture flows in both directions across the package seal. If the driving force for moisture flow is from the inside out of the package, the moisture vapor concentration in a package will be reduced until it approaches equilibrium with the outside environment

Regardless of the conditions used to drive moisture into the packages, when the dew point inside the package reached 25ºC. exposure to still air in a desiccator jar at 22ºC for 14 days would reduce the dew point back to 15ºC. In addition, baking the moisture out of the package on a heater block for 24 hours at 150ºC reduces the dew point back to the original value. Re-exposing the same packages, which were dried out, duplicated both the initiation time and the transmission rates measured in the initial exposure. One set of packages exposed to 65ºC and 90% RH. Was cycled through the wet/dry cycle three times and the results on the third try were the same as the first.

CONCLUSIONS

1.) To achieve consistent and predictable results, the sealing process conditions must be controlled. In this study, the control was achieved by using an iso-thermal sealing system. However it is achieved, the seal must provide a consistent width of the leak path for moisture in order to yield consistent and predictable results.

2.) The data provides base line performance information on an adhesive system which has been used successfully for a wide of variety of applications for a period of seventeen years. This is the " model" for comparison to improved systems.

3.) An improved sealing system was compared to the "model" system and found to have substantially more longevity as measured by the time factor to reach a dew point of 25º C inside the package.

4.) Analysis of the comparative data of the two formulations tested show that different conclusions would be drawn from tests at different temperatures. The "new" material out performed the "old" material by a factor approaching 10 at the 85/85 condition. However, as the temperature is reduced, the difference is not as spectacular, although still a factor of two. This is believed to be due to two factors. Firstly, the "wet" Tg of the old material is approximately 70ºC while the comparable wet Tg of the new material is approximately 105ºC. It is technically unsound to test a plastic material for a performance property above the Tg.

5.) Analysis of these data predict devices sealed with these or similar polymer adhesives will function indefinitely if high humidity exposure is interspersed with low humidity or the package service temperature is significantly above the ambient room temperature. Actually, prediction is a misnomer since 17 years of historical performance trumps accelerated aging tests.

6.) Microwave power die typically operate at temperatures in the range of 100 to 160ºC. When the power is ON, the driving force for moisture is inside out.

7.) It is advisable to maximize the functional width of the package seal to minimize the moisture intrusion potential. These data were developed at a width of 0.55mm.

PROCEDURES AND DATA

SEALING SYSTEMS:

Initially, a twenty-five position, aluminum, three plate sealing fixture was used. Each site in the fixture was fitted with a coil spring designed to produce 1 Kg. of force to each package. Clamping force is provided with bolts and wing nuts in the four corners. The middle plate locates the package body. Sealing instructions call for a 1 hour heat soak in a forced air oven pre-heated to 160ºC. No heat ramp cycle or time between loading and oven cycling is specified. Cooling is required before sealed part removal.

ISOTHERMAL PROCESS

The standard ITS sealing system was used. It involves thermostatically controlled top and bottom platens. The two package components (top and bottom) are pre-heated to the cure condition before pressure is applied allowing for pressure equalization before the sealing force is applied. The force is applied with independent pneumatic pistons for each package location. Because the inside and outside pressure are equal during sealing there is a constant seal width and thickness.

HUMIDITY CHAMBERS

Standard commercial humidity cabinets were used Wet bulb/dry bulb temperatures indicated that the humidity was in the range of 85 to 90 % at all four temperatures tested.

DEW POINT TEST

The apparatus consists of a brass heater block controlled at 110 + or – 1ºC and a variable aluminum chill plate controlled at settings ranging from 0 to 25ºC in 5º increments with accuracy of + or – 1ºC of the set temperature. Dew point was determined by placing the room temperature package to be tested glass face down on the heater block for 10 seconds followed by transfer to the chill plate in less than 2 seconds for a 7 second exposure. After the 7 second exposure, the specimen was turned glass face up to a stand set with 30º incidence to a light source. Appearance of condensation on the inside surface of the cover glass is the dew point indicator.

MATERIALS TESTED

Some typical data comparing the standard “old” and “new” formulations are shown in Table 1. The sealing and cure cycles for both materials was a one hour exposure at 165ºC after the initial 6 minute ITS sealing cycle at 165ºC.
The materials were applied to black ceramic (92 % Alumina) circuit covers which have a wall thickness of 0.55mm. The adhesives were applied to the lid seal surface at a thickness of 0.1mm. The cover glass used for the opposite seal surface is standard 0.5mm borosilicate glass used normally for vision packages. After sealing, the average increase in overall height of the assembly was 0.03mm and this is identified as the bond line thickness.

In the initial test series using a sealing fixture, moisture intrusion was observed in many of the assembled test units in a few days at both the 85 and 65ºC test conditions. This was unexpected. Visual inspection of the seals found that the assemblies which showed premature intrusion has effective seal widths which were 50% or less than the ceramic wall thickness. This is due to pressure development in the seal cavity during cure. All of these specimens were discarded and the ITS sealing system was used to seal the replacement set. Both sets had 100% yield using gross leak, but all of the ITS specimens had a consistent 0.55 mm seal width. All of the data points presented are averages of 6 specimens.

Tabulation of the data for both the old and new materials are shown in tables 3 and 4 respectively. The data show that the new material appears to be 10x better than the older, proven product at the 85/85 condition. However, at 35/85 the comparison is closer to 2/1. This is due to the difference in Tg between the two materials as well as the molecular structure. The 85/85 condition is inappropriate for the older material because it is above the product’s wet Tg value. Even 65/85 is marginal as an accelerated condition for this product.

Two sets of 6 test specimens of each material were exposed at the 65/85 condition until the inside dew point was 25ºC. One set of each was placed in a desiccator jar with fresh molecular serves as the desiccant. The standard material showed a reduction in dew point of 5ºC in 8 days while the new material required 14 days for the dew point reduction to 20ºC. Only a single set of each was subjected to oven conditioning at 150ºC for 12 hours (overnight). In this case, both materials showed dew points of 15ºC. This was done to point out that moisture also escapes from the package interior if the moisture driving force is in the inside out direction. The data are shown in table 5.

Table 1
Parts Per Million Moisture Vapor by Volume

Condition	PPM
85ºC/85%RH	570,000
65ºC/85%RH	245,000
50ºC/85%RH	123,333
35ºC/85%RH	55,000
25ºC/Dew Point	31,111 1
20ºC/Dew Point	20,333 3
15ºC/Dew Point	17,369
10ºC/Dew Point	10,632
5ºC/Dew Point	8,666

Table 2
Package Adhesive Sealant

	1984 Formulation	New Formulation
Lab Shear Strength	Over 4,000 psi	Over 4,000 psi
Tg by DCS (dry)	100ºC	145ºC
Tg by DSC (Saturated)	68ºC	110ºC
CTE (below Tg)	30 ppm/ºC	30 ppm/ºC
Extractable Chlorides	<50 ppm	<25 ppm

Table 3
1984 Formulation

Dew Point	Exposure Temperature @ 85%RH			
	85	65	50	35
10	*	*	9	39
15	*	0.5	20	63
20	*	1	31	86
25	0.5	2	43	141

* Less than 8 hours

Table 4
1984 Formulation

Dew Point	Exposure Temperature @ 85%RH			
	85	65	50	35
10	6	9	19	63
15	7	11	43	95
20	7	12	68	166
25	8	14	81	285

Table 5
Wet/Dry Cycles
@ 65ºC/85RH

Dew Point	1984					
	0	1	2	0	1	2
10	*	*	*	9	8	9
15	0.5	0.5	0.5	11	12	12
20	*	1.5	1	12	13	13
25	2	2.5	2	14	15	15

Dried 16 hours @ 150ºC