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Outgassing Species in Optoelectronic Packages

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The microelectronics community has long been plagued with the problem of moisture formation and out-gassing of various fixed and organic gaseous species into the device cavity. It is now very apparent that the optoelectronic packaging community is having the same problems only made more complex by the use of inadequate test methods, unproven materials and misconceptions in the supply and user industries. A test protocol is provided that addresses these issues, which allows the optoelectronic community to improve device quality and reliability.

A complete quantitative analysis of the internal gases of a package cavity can not only verify elevated internal moisture in suspected moisture-related failures but can also provide invaluable information on the source of moisture and other potentially corrosive gases as well as information on the processing history of the device. The most widely accepted method for evaluating internal gas content is Residual Gas Analysis (RGA) via mass spectrometry. The mass spectrometry method for RGA involves the ionization and separation of gas molecules as they flow from the package cavity followed by a measurement of their relative abundance as a function of their mass-to-charge (m/e) ratio.

Military Standard 883, Method 1018^1 , developed in the mid-1970's, was primarily utilized by the microelectronics industry for determining water vapor content on a pass/fail limit basis. In developing the internal water vapor content criteria for MIL-STD-883, the moisture specification of 5,000 ppmv was chosen to eliminate gross corrosion in integrated circuits and hybrids. Specifically, the 5000 ppmv limit corresponds to a dew point of – 2.37°C. This limit assumes that if a package is sealed at 1 atmosphere and contained less than 5000 ppmv moisture content, no liquid condensation will occur.

Today, the telecommunications industry has broadened the scope of the test and is also focusing on other important gases. Telcordia[™] general specification GR-1221-CORE (passives) and GR-468-CORE (actives) specify the reliability assurance practices for optoelectronic devices used in telecommunications^{2, 3}. Specifically, laser pump modules have experienced failures caused by organic impurities. Such failures have led to increased testing of materials used within the package cavity to study the link between adhesive reliability and component reliability.

A major advantage of the mass spectrometry method of analysis is that in addition to moisture all other gases present in the package can be measured⁴. The other gases typically include N2, O2, Ar, CO2, H2, methane and ammonia. In addition, a host of other process residuals (e.g., isopropyl alcohol, acetone trichloroethylene and tetrahydrofuran) and leak test residuals (helium and fluorocarbons) can also be detected. Therefore, an overall view of the internal package atmosphere may be achieved, yielding useful information on the device's processing history and an indication of other potentially harmful gases other than moisture. Helium backfilling has become a common technique in the telecommunication industry mainly to eliminate the pressurization step during fine leak and to eliminate the de-adsorption of helium from fibber coating. Monitoring helium backfill

percentages on a routine basis insures adequate mixing and circulation in the seal enclosure, determines sensitivity limits for fine leak detection and more importantly is used for leak detection, by RGA, after environmental testing. Hydrogen out-gassing and subsequent reduction of oxides after seal can raise the moisture content above specific limits. Hydrogen out-gassing may also contribute to die level metal hydride formation and metal absorption phenomenon, thereby affecting the performance of the semiconductor. Identification of organic out-gassing species is among the most important RGA results for the optoelectronic industry. A new technique, developed at ORS, Inc., utilizes the RGA technique in tandem with GC/MS. The separation capability and low ppb sensitivity of GC/MS allows the manufacturers to identify multiple out-gassing compounds not traditionally measured by RGA alone.

Although the Military Standard 883, Method 1018 is routinely cited in the telecom specifications, not all the elements of the test conditions may be appropriate. Test requirements regarding pre-bake and test conditions at 100°C may not be suitable for many components utilizing certain fiber materials, low temperature solders, polymeric adhesives, plastic encapsulated components and proprietary solvents. Unlike microelectronic packages, typical fiber optic modules are only rated to 85°C.

A second consideration to be made when discussing the adoption of military specifications by the optoelectronic community is the insufficiency in testing that is created. Optoelectronic packages have environmental issues that military specifications never addressed (and vice versa). The use of Military Standard 883, Method 5011, for example, in screening adhesives for optoelectronic packages, is a good start. However, out-gassing species, even in the lowest concentrations shown acceptable to high reliability military parts, could prove fatal to the highly sensitive surfaces of the lens and fibers in the Optoelectronic package.

Out-gassing species and moisture generation in all microelectronic packages have created a very complex problem. It is now recognized that residual moisture within the cavity of a device is increased by internal chemical reactions between base metals, plating and other packaging materials. It has been demonstrated that hydrogen dissolved within the package alloys or plating easily permeates into a package cavity and commences reduction processes of all surface oxides (metal) within the cavity⁵. Furthermore, organic compounds that are integral to the plating process can also desorb into the cavity. This source of organics is in addition to any residual organic compounds from adhesives, gel coatings, particle getters, etc.

These organic species and the in-situ generation of moisture from reduction processes caused by the desorbing hydrogen are not yet fully explained on a theoretical basis. The fact that hydrogen is retained by (and desorbs from) ferrous and other alloys has long been known, as well as the fact that the plating processes yield hydrogen. The movement of these gases and the subsequent chemical reactions that occur have been reported as early as 1944, when Ellingham published his first "thermodynamics diagram" depicting metal oxide reductions^{6, 7}. Others, as of late, recognize that basic thermodynamics predicts the moisture generation in device cavities⁸. These assessments do not address the added and most important factor of chemical catalysis. One should not overlook the fact that a significant number of chemical elements are being utilized in optoelectronic packages that are also used extensively in various chemical processes as catalysts!

Fortunately, not all packaging designs and materials of construction lend themselves to the above problematical scenarios. Unfortunately, it has been shown that the contributing factors can, and will, come and go on a lot-to-lot basis. Hence, it is imperative to identify those factors for moisture generation (and organic out-gassing) early in the product life cycle. This has been successfully done by the authors in a newly developed analytical protocol. The essentials of RGA as discussed above are employed, and the individual

elements from which the device is fabricated, are assessed on a lot-to-lot basis until they can be shown to be non-contributors. As shown in Table 1, base kovar alloy, normally manufactured in hydrogen-enhanced environments, can be ultimately finished so as to be essentially hydrogen free. A mixed lot variation is also indicated.

	Control	S/N 1	S/N 2	S/N 3	S/N 1A	S/N 2A	S/N 3A
Plated Lot #1	133	433	430	2189	445	544	2660
Plated Lot #2	144	234	309	266	255	222	218
Abraded Lot #1	xxx	2588	3329	3090	296	130	138
Abraded Lot #2	220	275	250	XXX	122	137	ххх

Table 1: Variation of Hydrogen Content (ppmv) in Package Elements

Control and S/N 1, 2 & 3: Thermally treated 50 hours @ 150°C S/N 1A, 2A & 3A: Original samples thermally treated for additional 50 hours @ 150°C

As noted above, the protocol utilized in our work concentrates on analyzing the individual elements of the package to ascertain that they will remain hydrogen free. Without process control and monitoring of assets one will only invite disaster at some time in the future. As shown in Figure 1, a lid manufacturer has delivered two separate lots of product, each with its own level of residual hydrogen. The upper curve will ultimately contaminate the device cavity with unacceptable levels of hydrogen and moisture, whereas the lower curve can yield a functional, reliable finished product.



Figure 1: Hydrogen Content Variations Between Lots

Once acceptable levels of process and material residuals are demonstrated to be minimal and maintainable, it is strongly recommended that future production lots be protected by proper documentation and the requirements being stipulated in the individual purchase orders and specifications. Naturally, these requirements should be periodically verified via independent assessments. In conclusion, it has been clearly shown that residual hydrogen and organic species can out-gas into the cavity of an optoelectronic package, the net result being the in-situ generation of moisture and/or the condensation of the organic species on optical surfaces. These problems can be significantly reduced, even eliminated, via a protocol recently developed by the authors. But even after solution of the problem, the optoelectronic manufacturer will have to remain vigilant to the potential of recurrence until the supply industries respond positively to optoelectronic package needs.

Biographies:

Philipp wh Schuessler is an analytical chemist with B.S. and M.S. degrees from the Rochester Institute of Technology. He has spent over 35 years in support of the defense industries manufacturers, IBM/FSD, Loral, & Lockheed Martin. Simultaneously he was an active member of the standards community, JEDEC – the Joint Electronic Devices Engineering Council, for 20 years. He chaired over a dozen task groups, including those for Test Methods 1018, 5011 & 5013, and also served as secretary to JC13.5 for 10 years. He has published over 45 technical papers and holds 20 published invention disclosures and four published patents. He is presently a consultant for the microelectronic devices industries. He may be contacted at (518) 239- 4534.

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References:

1) Department of Defense, Mil-Std-883, "Test Method Standard – Microcircuits", Rev E, 1996

2) Telcordia Technologies, Inc, GR-1221-CORE, Issue 2, January 1999

3) Telcordia Technologies, Inc, GR-468-CORE, Issue 1, January 1998

4) ORS White Paper - "Commercial Practice: Internal Vapor Analysis of Microelectronic Devices", 1996, Oneida Research Services, Whitesboro, NY

5) Schuessler, P. & Gonya, S. "Hydrogen Desorption from Base and Processed Packaging Alloy", Rome Laboratory/NIST Workshop on Moisture, April 2,'93

6) Ellingham, H.J.T., "Reducibility of Oxides and Sulfides in Metallurgical Processes", J. Soc. Chemical Industry, Transactions and Communications, May - 1944

7) Gaskill, D.R. "Introduction to Metallurgical Thermodynamics", Hemisphere Publishing, NY, 1981,2nd edition

8) Greenhouse. "Hermiticity of Electronic Packages", William Andrew Publishing, Norwich, NY, 2000

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