

HARSH ENVIRONMENTS AND VOLATILES IN SEALED ENCLOSURES

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ABSTRACT

Functionality of many types of microelectronic, optoelectronic, medical, and micro- and nanomachine devices depends on protection afforded by a hermetically sealed enclosure. But certain volatiles in enclosure headspace can create a unique, harsh mini-environment for sealed devices. External thermal, mechanical, or chemically harsh environments of post-seal testing or field service can induce, or aggravate, already harsh internal mini-environments.

Depending on type of device, vapors, adsorbates, or condensates of moisture, hydrogen, oxygen, hydrocarbons, ammonia, and other volatiles can deteriorate or destroy device function. Devices can be susceptible to failure mechanisms such as corrosion, electrical leakage or instability, dendritic growth, fogging, stiction, jammed moving parts, etc. caused by these species. In vacuum-sealed enclosures minuscule amounts of any volatile deteriorate vacuum quality and degrade devices that depend on ultralow headspace gas pressure.

Undesired volatiles can be impurities in blanket gas used during seal. They can volatilize from materials within the enclosure during post-seal thermal excursions. They can enter through leak paths caused by poor sealing processes or post-seal thermal or mechanical stresses of harsh environments that compromise seal integrity. This paper identifies the types and sources of volatiles that threaten device function, summarizes failure mechanisms that volatiles can cause, reviews data from mass spectrometric analysis of headspace gas composition, and discusses material and process considerations for controlling headspace gas composition.

Key words: Hermetic packages, hermeticity, moisture, volatiles, corrosion.

INTRODUCTION

Hermetic Packaging

With advantages of low cost and high-volume processing plastic encapsulation rapidly supplanted hermetics, and twenty-five years ago it appeared that hermetic packaging would become practically obsolete for microelectronic devices. But the hermetic style has maintained a “niche” role in packaging electronics for high reliability and/or certain harsh environment field service applications. In addition, there are many new types of devices whose optical or mechanical function simply can not be encapsulated in plastic.

Most early microelectronics were sealed in hermetic packages, especially those in military and aerospace applications. As early as the 1960’s users and manufacturers recognized that corrosion of device metallization caused by moisture trapped in, outgassing into, or leaking into packages was a major reliability problem¹. Reliability and costs of both military and aerospace programs were significantly affected²⁻³.

Mass spectrometric analyses of hermetics originally focused on gaining knowledge and control of package moisture levels to prevent device corrosion. With accumulation of device operation, associated reliability information, and mass spectrometry data, additional failure mechanisms due to both moisture and other volatile species emerged. Need for this information grew with development of optoelectronic and micromechanical devices/systems in enclosures; clearly the gaseous headspace of hermetics could constitute a unique harsh mini-environment for encapsulated devices. It was also clear that harsh external environments of the entire package assembly, especially elevated temperatures and temperature cycling below dewpoint during testing or field service, could aggravate harshness of the headspace mini-environment.

Measuring Gases in Sealed Enclosures

To understand the magnitude of moisture issues, reliability scientists and engineers adapted mass spectrometry to measure moisture in package headspace. This involved puncturing a hole in the package (usually in the lid) and evacuating headspace gases into a quadrupole mass spectrometer for quantitative analysis, per Figure 1. The methodology was formalized in the military specification system⁴ and is the analysis method of choice today.

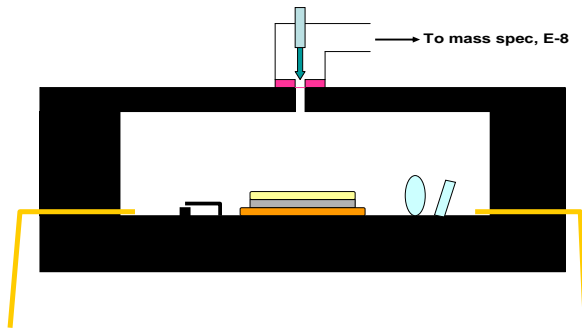


Figure 1. Sampling Headspace of a Hermetic Package

Early sealed-package analyses focused on water vapor as analyte, but revealed other volatiles for which failure mechanisms emerged as package technologies matured. With the cost-driven and widespread adoption of organic adhesives to mount or protectively coat components, mass spectrometry revealed these products often sourced volatiles that created chemically harsh internal environments.

Volatility behavior of package materials can be studied for engineering and process development/improvement purposes by baking samples in sealed ampoules under controlled conditions. Ampoules are broken and the volatiles released for mass spectrometric analysis. If a clean piece of metal foil is included in the ampule, it can be removed after cooling and analyzed by surface methods like reflectance infrared and SEM/EDX for condensable molecular compounds and salts.

SOURCES OF GASES IN HERMETIC ENCLOSURES

Figure 2 is an idealized example of a package with various components such as a MEMS device, an adhesive-attached chip with conformal coating, and optical components. No enclosure is exactly like this but the figure represents several kinds of components and displays the several mechanisms by which their encapsulating headspace can become a harsh environment.

Fill Gas (“A” in Figure 2)

Fill gas for sealing can contain impurities from its source, from piping delivering it to the seal chamber, or from loss of seal integrity of the chamber itself. Gases introduced this way include moisture, oxygen, and volatile organics.

Package Materials (“B1” in Figure 2)

Metals, ceramics, and platings are also rich sources of vola-

tiles, especially moisture and hydrogen (see Figure 3).

Assembly Materials (“B2” in Figure 2)

Component attachment adhesives, conformal coatings, and related materials outgas many types of volatiles, including moisture, organics, ammonia, and other volatile reactants.

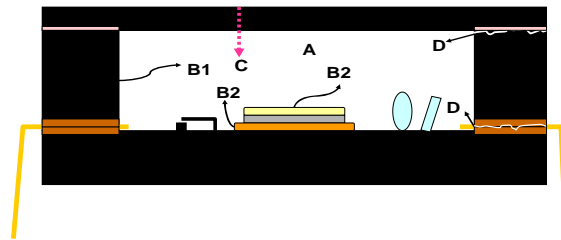


Figure 2. Sources of Volatiles Within Sealed Packages

Permeable Materials (“C” in Figure 2)

Any permeable material such as polymer lid seals or membranes allow air and moisture to diffuse into headspace.

Leaks (“D” in Figure 2)

Loss of package seal integrity allows ingress of moisture and oxygen through leak paths into the package. Loss of integrity can occur (1) in the vicinity of welds or other joints attaching the lid to the package body, (2) along interfaces between package body and lead feedthroughs, (3) along the interface between glass and metal in glass-to-metal seals, (4) through cracks in seal glass that connect interior headspace to the exterior environment, and/or (5) through cracks in the package body.

Leak defects may be present from time of package seal due to poor handling or other deficiencies, poor control of materials and process, and can be induced by temperature excursions, cycling, or mechanical vibration or shock during testing or field service. Leaks are often blamed for many moisture-related failures. In today’s hermetic packaging, the greatest impact from leaks is on vacuum-sealed devices where even the slightest ingress of volatiles adds gas molecules and degrades vacuum quality.

HEADSPACE GAS COMPOSITIONS

Gas compositions of sealed headspaces can be complex. Mass spectrometry provides both survey capability to identify volatiles and, with proper calibration, quantitative capability to identify how much of each species is present.

Mass spectrometric data often enable sources of unwanted volatiles to be identified, with root cause identification of device failure modes caused by headspace gas. Composition of natural air in Table 1 is the benchmark for interpreting mass spectrometric data from sealed packages. Except for moisture, four major and minor components of natural air, covering four orders of magnitude of concentration, are fixed in concentration and are the starting point for understanding headspace gas composition.

Table 1. Chemical Composition of Natural Air

Species	Concentration, v%
Nitrogen	78.08
Oxygen	20.95
Argon	0.934 (9340ppmv)
Carbon Dioxide	0.038 (380 ppmv)
Moisture	≈0.1-3.0*
All others	<<0.01†

*varies with humidity, temperature, and pressure.

†hydrogen, helium, and other trace gases. Any that occur in packages comes from processes or materials and not air.

“HARSH” VOLATILES IN ENCLOSURES

Moisture

Water as condensate, adsorbate, or vapor has a profound impact on reliability of all types of encapsulated devices. As condensate it promotes metal corrosion. As adsorbate it promotes surface electrical leakage (and corrosion), electrical instability, or dendritic metal growth. As vapor its molecules raise internal gas pressures from levels intended to provide an evacuated surrounding.

Three monolayers of adsorbed water are the threshold condition to support electrical leakage across a surface⁵. The threshold condition for adsorbing three monolayers of water molecules on a surface from an enclosed space is 5000 parts per million by volume (ppmv, = 0.5 volume percent) water vapor⁵. The dewpoint temperature at which condensate forms in an enclosed space at 1 atmosphere pressure and 5000ppmv water vapor is -2°C (just below freezing).

The convergence of these facts led to adopting 5000ppmv as the maximum allowed moisture content in sealed microelectronic devices. This limit applies today to military electronics and is the “default” moisture limit for any sealed device where other knowledge-based guidelines are lacking.

Besides corrosion and surface electrical leakage, moisture condensate fogs mirrors and lenses of electrooptical devices⁶. Light scattering by even the slightest amount of condensate disrupts device function even if electrical function is not affected. Any optical device at or below dewpoint is susceptible.

MEMs devices with moving parts like gears or lever arms can fail due to stiction (static friction), an adhesive force between surfaces in contact. Four major kinds of stiction include capillary forces, hydrogen bridging, electrostatic forces, and Van der Waals forces. Capillary forces and hydrogen bridging occur due to capillary action of condensed liquid water “gluing” micromechanical moving parts of devices to one another. Adsorbed water molecules promote chemical bond formation via hydrogen bridging, similarly bonding moving parts together. Depending on the microphysics of contacting surfaces and part design, stiction can occur at exceedingly low levels of relative humidity⁷. In such cases, complying to even a 5000ppmv water vapor maximum is inadequate and much lower moisture levels must often be achieved to insure reliable MEMs operation.

Table 2 provides some examples of actual gas analysis results for IC devices pertaining to need for moisture control. Sample A is an example of how sealed enclosure gas compositions ought to look. Samples B, C, D, and E are examples of actual devices which failed due to excessive package moisture.

Table 2. Moisture in Sealed Enclosures. Results in All Tables Expressed in Volume Percent (1v% = 10,000ppmv)

Species	A	B	C	D	E
N ₂	99.80	93.90	96.00	53.50	76.10
O ₂	<0.01	3.55	<0.01	<0.01	21.00
Ar	<0.01	1.15	<0.01	0.13	0.93
CO ₂	0.01	0.32	0.01	2.59	0.08
H ₂ O	0.02	1.04	1.50	39.00	1.46
H ₂	0.19	0.03	0.15	<0.01	<0.01
He	<0.01	<0.01	2.33	<0.01	0.40
HC ^a	<0.01	<0.01	<0.01	<0.01	<0.01
Methanol	<0.01	<0.01	<0.01	3.62	<0.01
2-Meth ^b	<0.01	<0.01	<0.01	0.29	<0.01

a. HC = Hydrocarbon Compounds

b. 2-Meth = 2-Methoxy ethanol

Sample A is a large power management hybrid used on International Space Station. Pre-launch to ISS, these packages harbored no harsh environment due to moisture or other volatiles and the headspace was not degraded by harsh temperature cycle testing. Hybrid devices like these have been flying successfully on ISS for more than five years.

Sample B is a flatpack device for an automotive application that failed due to a lone silver dendrite that shorted adjacent pins. The package contained enough moisture to support metal dendritic growth. The moisture appears to be humidity from residual air in the package seal gas.

Sample C is a flatpack-enclosed IC that failed due to aluminum corrosion. Moisture at 1.5v% corresponds to a dewpoint of +6°C, adequate for moisture to condense on surfaces during cold dwell times of temperature cycling and initiate metal corrosion. Oxygen and argon are absent so air is not present. Moisture is strictly from material outgassing.

Sample D is a metal package with epoxy used to seal the lid. The epoxy outgassed volatiles as shown by the organic compounds, accompanied by huge amounts of water, during post-seal temperature excursions. Oxygen is absent so air is not present and the moisture is far higher than simple humidity ingress accounts for. The epoxy was inadequately cured pre-seal, allowing high post seal temperatures to create a harsh environment in the package.

Sample E is an external operating housing of a medical diagnostic device sealed in air. The moisture content is due to natural relative humidity of the fill air. At 1.46v% there is concern for useful product lifetime since there is potential for device corrosion, as in Sample C, or instability at that concentration. Sealing under nitrogen or dry air is preferred.

Oxygen

Early work with solder-attached components revealed a failure mode in which oxygen in headspace penetrated microcracks or pinholes in solder exposed at the bond line around the periphery of the component and oxidized interior surfaces. This condition induced differences in coefficients of thermal expansion and stresses between the package and the mounted component during thermal cycling, weakening the solder joint and the strength of component attachment⁸. The potential for this mechanism led to the only other MIL Spec-imposed limit for a volatile in hermetic enclosures. MIL PRF 19500 limits oxygen to 2000 ppmv⁹. Oxygen can be present because of air impurity in inert gas used for sealing, as an outgassed by-product from decomposing organics, or via air ingress through post-seal leaks, to create a harsh mini-environment for solder-mounted components, as shown in Table 3.

Table 3. Oxygen Causing Lift of Mounted Component

Species	A	B
N ₂	95.78	92.97
O ₂	<0.01	1.33
Ar	<0.01	0.14
CO ₂	<0.01	<0.01
H ₂ O	<0.01	0.35
H ₂	4.33	0.15
He	<0.01	5.23
HC ^a	<0.01	<0.01

Samples A and B are power devices in a metal package. Unit A functioned normally, but the solder-attached chip in Unit B lifted from substrate. Failure analysis confirmed that surfaces of the solder separations were heavily oxidized. Argon and residual oxygen suggest air ingress through a leak. Failure analysis showed seal glass separated from a feedthrough pin allowing a small pathway for air ingress.

Hydrogen

Hydrogen is exceedingly mobile. It diffuses rapidly to buried interlevel dielectric interfaces in device structure where it collects to create nonuniform charge distributions and threshold voltage shift. Depending on design and geometry, it can threaten dependable function of MOS devices and compound (e.g. gallium arsenide) devices¹⁰⁻¹¹.

Hydrogen reacts with metal oxides producing water vapor as a by-product. The physical chemistry is described in Ellingham diagrams, which plot Gibbs free energy change (ΔG) for metal oxidation reactions versus temperature¹². Depending on chemistry, surface areas of oxide, and amounts of hydrogen, amounts of water vapor produced can be significant. Since the free energy of the reaction is temperature-dependent high temperature environments can drive the reaction and raise moisture levels in packages.

In sealed system-level housings enclosing a variety of components under air, hydrogen accumulated from metals or batteries creates a dangerous harsh environment if its

concentration reaches the lower explosive limit in air of 4% and a component produces a spark discharge.

Most headspace hydrogen in electronic packages is sourced from outgassing of plating or the ferrous alloys of package piece parts themselves, such as Kovar (Fe-Ni-Co). This material can release large amounts of hydrogen to headspace over time and temperature. Figure 3 plots hydrogen accumulation in two different Kovar metal packages over time.

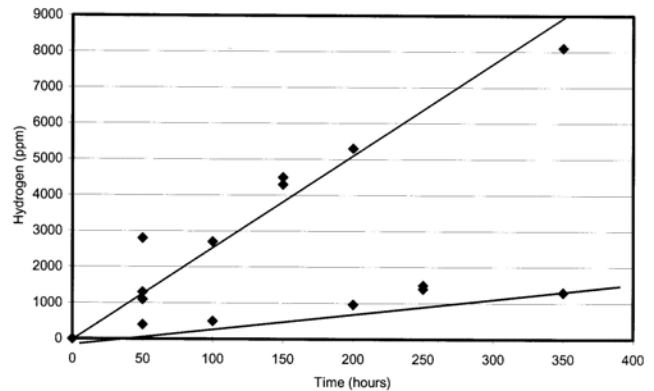


Figure 3. Hydrogen Accumulation in Kovar Packages

Table 4 identifies harsh environments due to hydrogen in two different applications. Sample A1 is a pin-grid array with 4.33v% hydrogen whose devices had threshold voltage instability, while those in Sample A2 with minimal hydrogen were stable. Sample B is a large air-sealed marine navigation aid enclosure which accumulated 12.50v% hydrogen in its air fill due to battery leakage. Such an enclosure is vulnerable to explosion if an arc or spark discharge occurs.

Table 4. Hydrogen Causing Threshold Voltage Shift; Hydrogen Exceeding LEL in Air-sealed Navigation Aid

Species	A1	A2	B
N ₂	95.41	99.46	64.79
O ₂	<0.01	<0.01	17.08
Ar	<0.01	<0.01	0.84
CO ₂	0.21	0.22	1.12
H ₂ O	0.06	0.31	3.75
H ₂	4.33	<0.01	12.50
He	<0.01	<0.01	<0.01

Ammonia

Some epoxy adhesives used for component attach contain 2-cyanoguanidine, C₂H₄N₄, (dicyandiamide), a curing agent. If such adhesive is not absolutely fully cured pre-seal, and carefully baked post-seal to volatilize curing by-products, elevated post-seal temperatures will outgas the by-products into headspace. Ammonia, NH₃, is a decomposition by-product of C₂H₄N₄.

If an enclosure contains volatile ammonia with high moisture, and condensation occurs, NH₃ will dissolve in the

condensate forming ammonium hydroxide (NH₄OH), raising condensate pH, and raising chemical attack potential.

Even when moisture content complies to the 0.5v% limit, ammonia in vapor phase has been shown to promote precious metal dendrite growth. Table 5 is an example of a unit which experienced an electrical short due to a silver dendrite bridging adjacent bond wires¹³. Failure modes involving ammonia are easily prevented by avoiding adhesives with 2-cyanoguanidine curing agents.

Table 5. Ammonia in Hybrid Package

Species	Silver dendrites	Dendrite-free
N ₂	95.37	99.37
O ₂	0.04	0.03
Ar	0.03	0.04
CO ₂	0.34	0.15
H ₂ O	0.19	0.35
H ₂	0.06	0.08
He	<0.01	<0.01
HC ^a	0.09	<0.01
NH ₃	2.57	<0.01
CH ₃ OH	1.31	<0.01

Organics (Hydrocarbons).

Volatile hydrocarbon compounds create a harsh internal environment by condensing or adsorbing onto critical surfaces. Condensates can cause electrical leakage, fog optics, and jam or clog MEMs gears, cantilevers, and other micromechanical parts by accumulating micro-amounts on surfaces. Dozens of different volatile organic compounds have been detected in sealed enclosures of various types.

Table 6 is an example of volatiles posing a fogging danger to surfaces in a device containing optical components, as condensate of one or more of the three hydrocarbons could fog mirrors or lenses. This internal environment was created by post-seal outgassing of organic adhesive, which also added carbon dioxide and water vapor to an otherwise benign headspace filled with nitrogen and helium.

Table 6. Volatile Hydrocarbons in Sealed Optical Device

Species	Telecom Optical Device
N ₂	85.10
O ₂	<0.01
Ar	<0.01
CO ₂	0.58
H ₂ O	0.47
H ₂	<0.01
He	13.30
Methyl ethyl ketone	0.14
Methanol	0.18
Tetrahydrofuran	0.09

MEMs devices require ultrapure gaseous headspace to function properly. Table 7 shows gas composition of a MEMs with jammed gears. The gas composition appears quite benign. But failure analysis of this device revealed

tiny crystals on the gear teeth. Gas chromatography/mass spectrometry identified the deposits as behenonitrile, [CH₃(CH₂)₂₀]C≡N, and stearonitrile, [CH₃(CH₂)₁₆]C≡N, whose deposits jammed the gear teeth causing device failure¹⁴. Chemical analysis showed these compounds in the device mount materia. Normally 0.18v% of volatile hydrocarbon would not be a concern in most hermetics, but that level can be a major concern for MEMs.

Table 7. Mechanically Jammed MEMs Device

Species	Device
N ₂	95.90
O ₂	<0.01
Ar	0.05
CO ₂	3.48
H ₂ O	0.07
H ₂	0.26
He	<0.01
Hydrocarbon	0.18

Carbon Dioxide and Argon

Data in Tables 2, 4, 5, 6, and 7 show that carbon dioxide can occur in amounts much higher than normal ambient air (0.038v%) in some enclosures. This is always due to decomposition by poorly cured, inadequately baked organic materials used in packages. Carbon dioxide is soluble in water, producing carbonic acid, a weak acid which slightly lowers pH of aqueous condensate that may otherwise be close to neutral. Some corrosion mechanisms might be advanced by increased condensate acidity due to carbon dioxide dissolved in condensate. This chemistry was postulated in early failure analyses of corroded nichrome resistors¹⁵, but the role of carbon dioxide if any in actual corrosion failures has never been directly confirmed.

Argon is used as fill gas for arc lamps because it supports arc discharge. As an inert it can also used as fill gas for sealed devices. Unless needed for arc lamp applications, manufacturers should be careful not to exceed about 50v% argon fill, as any arc or electrostatic discharge in argon-enriched headspace could damage devices.

UNIQUE HARSH ENVIRONMENTS

Vacuum

Vacuum can be harsh, or it can be benign, depending on application. Devices like quartz crystal oscillators and MEMs structures often depend on a greatly reduced number of molecules in vacuum-sealed package headspaces for a benign environment in which to operate reliably. Any increase in volatile atomic or molecular species can degrade electrical function or cause mechanical stiction. Vacuum compromise occurs by any or all of the mechanisms of Figure 2 that increase internal pressure of an initially vacuum-sealed unit. A small increase can be significant. Moisture is a special concern as it is hard to pump from vacuum, yet volatilizes readily from materials under vacuum. Table 8 is an example of headspace vacuum compromised by moisture outgassing. Vacuum level in the failed device became "only" 4.7x worse than the good

device, but that was enough additional water molecules to create a “harsh environment” leading to gear stiction.

Table 8. Vacuum Compromise in a MEMs Enclosure

Species	Good MEMs Device	Failed MEMs Device
Ion source pressure	3.0E-8 torr	1.4E-7 torr
N ₂	81.20	41.9
O ₂	<0.01	<0.01
Ar	1.86	1.44
CO ₂	7.17	6.23
H ₂ O	<0.01	49.30
H ₂	9.75	1.13

Air, Of Necessity

Most applications use inert, dry headspace fill gas. But in some applications using inert fill is not physically possible or cost effective, e.g. Table 2 Sample E and Table 4 Sample B. Another example is enclosure headspace in well logging tools, which necessarily begins as ambient air with its humidity, a harsh environment from inception. “Harshness” can worsen with long field-service times and high temperature. Components require protective coatings, yet coatings can be sources of harsh volatiles. Table 9 is a study of an exemplar coating. Material was tested in an ampule sealed under pure nitrogen for baseline study. Outgassing into nitrogen in the sealed ampule at 250°C for 1 hour yielded moisture, hydrogen, and volatile organics from the material. Ambient surrounding the material in a tool was then analyzed post-field service. Oxygen from air is consumed by oxidizing metal surfaces of the enclosure, but is replaced by increased levels of moisture, carbon dioxide, and hydrocarbons from material reactions. High-integrity impermeable coatings free of physical defects are essential to protect devices from tool ambients. Coating must be thoroughly de-gassed to minimize its volatiles. It must adhere intimately to all surfaces so its own volatiles do not collect in interfacial voids as a localized harsh environment.

Table 9. Outgassing in Data Logging Application

Species	Exemplar Material Outgassed 1 hr at 250°C in Glass Ampule	Tool Headspace after 100 Hr 250°C Bake
Blanket	Pure N ₂	Natural air
N ₂	94.10	76.10
O ₂	0.08	0.11
Ar	0.04	0.89
CO ₂	0.03	11.50
H ₂ O	2.04	10.70
H ₂	2.40	0.11
CH ₄	1.20	<0.01
C ₆ H ₆	0.15	<0.01
Organics	<0.01	0.56

AVOIDING HARSH INTERNAL ENVIRONMENTS

Materials; Component Manufacturers

Select package parts with attention to composition, surface chemistry, cleanliness, and outgassing behavior.

Select attachment and protective materials with volatility properties suited to expected field service conditions.

Deploy getters engineered to capture and bind volatiles discussed in this paper. Appropriate getter products are readily available.

Processes; Component Manufacturers

Assemble in clean environments with humidity control.

Cure all organic materials completely per manufacturer recommendations; do not shortcut cure schedules nor overload curing ovens.

Engineer post-cure pre-seal bakes to maximize loss of volatile materials before seal without altering material physical or chemical properties.

Pre-seal bake assembled components in-process. Bake in vacuum below 10mTorr if possible.

Execute robust processes faithfully. Do not overload ovens or short-cut cycle times.

Do not re-expose pre-seal baked components to ambient air.

Seal in moisture-controlled equipment under pure N₂ or He.

AVOIDING HARSH EXTERNAL ENVIRONMENTS THAT MAY CAUSE OR EXACERBATE HARSH INTERNAL ENVIRONMENTS

Processes; In-House or Contract Assemblers

Fully inform component suppliers of assembly and handling process conditions: soldering and baking temperature profiles, plus extremes of thermal cycling, mechanical stress, thermal or mechanical shock, and vibration.

Fully inform component suppliers of product test conditions and requirements with respect to above-mentioned factors.

Field Service; Customers and Users

Fully inform component suppliers and assembly providers of product application expectations, especially with respect to temperature and physical usage factors.

Procure products designed, tested, and fully characterized for conditions that the service requires.

Do not install units in field service where temperatures could exceed cure or pre-seal bake process temperatures.

CONCLUSION

Several electronic component failure modes are triggered by harsh chemical environments of certain gas compositions within sealed enclosures. Moisture is the principal concern, but oxygen, hydrogen, ammonia, and hydrocarbons each pose unique threats. Harsh internal environments occur mainly due to materials outgassing, but also due to blanket seal gas impurities and/or loss of package seal integrity. Harsh environments are readily induced, and can be

aggravated, by harsh external temperatures and/or mechanical shock or vibration. Careful materials selection, robust process engineering and control, and avoiding harsh field service conditions that exceed pre-seal process treatments preclude harsh environments inside hermetically sealed enclosures.

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