

SEMI MS10-0912 (Reapproved 0318) TEST METHOD TO MEASURE FLUID PERMEATION THROUGH MEMS PACKAGING MATERIALS

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1 Purpose

1.1 Microelectromechanical systems (MEMS) are miniaturized systems requiring packaging for environmental protection and for interaction with the external environment. While there is a wide range of MEMS devices, a fairly common need is for packaging that allows movement of some of the internal subcomponents of the device during operation. This is in contrast to the typical integrated circuit that requires only that the device be protected chemically and mechanically from the environment and that appropriate electrical interconnections are made. In many cases, hermeticity is critical to MEMS device functionality. In other cases, hermeticity is primarily important to the reliability of MEMS devices, similarly to integrated circuits. To accelerate improvements of packaged MEMS devices, leading to higher levels of marketplace acceptance, a guide to prediction and measurement of permeation of MEMS hermetic packages is required.

1.2 This Document is intended to provide an overview of design considerations and hermetic packaging with particular emphasis on the evaluation of permeation of MEMS hermetic packages having internal cavity volumes that are very small compared to conventional packages.

2 Scope

2.1 This Standard is directed towards defining a common method of measurement of permeation through technical films that are used in sealing hermetic MEMS packages. Areas to be addressed include materials and equipment for producing and evaluating hermetic seals; methods for detection and measurement of leakage; and considerations and recommendations on the evaluation of hermeticity.

2.2 This Standard additionally provides methods for quickly evaluating compliance of a specific hermetic cavity package to application requirements. The Standard may be useful for establishing protocols relating to manufacturing, quality and reliability assurance for all phases of research, development, and production.

2.3 Devices for which this Test Method is expected to be relevant include, but are not limited to, gyroscopes and accelerometers; RF MEMS switches; optical mirrors and switches; pressure sensors; resonators; filters; and microfluidic devices including valves and pumps.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 Users of hermetic cavity packages for MEMS devices require assurance that hermeticity is adequate both initially and over operating life. In turn, designers and manufacturers of MEMS packages must be able to demonstrate that internal ambient conditions of packaged devices meet design requirements:

- manufactured seals have sufficient integrity;
- material permeability to fluids (gases or liquids) present in the operating environment is compatible with maintenance of internal ambient conditions over operating life and;
- outgassing is managed to ensure maintenance of internal ambient conditions over operating life.

3.1.1 The current Document is primarily focused on the evaluation of material permeability coefficients at or near the time of initial manufacture, and in the prediction of the impact of permeation on reliability of hermeticity over operating life.

3.2 The permeation of a hermetic package is related to capillary leaks or permeability properties of the material making the package or the seal. Indeed these characteristics lead to the buildup of air species (including moisture) levels within the package. For purposes of the current Document, it is assumed that the packaging materials and processes have been controlled to a level where outgassing and capillary leaks are considered negligible. This simplifies the calculation of the maximum permeation that can be tolerated according to users' or manufacturers' requirements.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI MS3 — Terminology for MEMS Technology

4.2 JEDEC Standard¹

JEDEC JEP144 — Guideline for Residual Gas Analysis (RGA) for Microelectronic Packages

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *DUT* — device under test

5.1.2 *LPCVD* — low pressure chemical vapor deposition

5.1.3 *MEMS* — micro electro-mechanical systems

5.1.4 *PECVD* — plasma enhanced chemical vapor deposition

5.1.5 *SOI* — silicon-on-insulator

5.1.6 *STP* — standard temperature and pressure

5.1.7 *TEOS* — tetraethyl orthosilicate

5.2 Definitions

5.2.1 *atmosphere* — throughout this Document one atmosphere is defined as being equal to 1000 millibars (= 100 kPa), often referred to as a technical atmosphere.

5.2.2 *desorbability* — the relative capability of a fluid to escape from a solid surface and enter the gas or liquid phase.

5.2.3 *device under test (DUT)* — the device undergoing evaluation.

5.2.4 *dew point* — the saturation point of water, or the temperature to which a given volume of air must be cooled, at constant barometric pressure, for water vapor to condense into liquid water. When the temperature is equal to the dew point the relative humidity is 100%.

5.2.5 *diffusivity* — the rate at which a dissolved fluid will be transmitted through the specimen material. Strictly defined by Fick's first and second laws. Symbol *D* is used.

5.2.6 *equivalent standard leak rate* — the leak rate when the pressure one side of the package is at standard temperature and pressure (~760 mm Hg absolute) while the other side of the package is at vacuum (less than 1 mm Hg absolute), often referred to as the true leak rate, or the leak rate normalized for a unit pressure differential.

5.2.7 *fluid transmission rate* — the quantity of fluid passing through a unit area of the specimen per unit time under the test conditions. The fluid transmission rate depends on the barrier properties of the material and the partial pressure difference between the two sides of the specimen. Expressed in units of cm³ (at STP)/ cm²-sec., or moles/cm²-sec. Acronym FTR may be used.

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5.2.8 *frost point* — the temperature to which a given volume of air must be cooled, at constant barometric pressure, for water vapor to condense into ice. Since ice has a stronger bonding between neighboring molecules, it is more difficult for water molecules to escape a frozen surface.

5.2.9 *hermetic package* — completely sealed with minimal communication of either gases or liquids between the interior and the exterior of the package over operating life.

5.2.10 *hermetic vacuum package* — hermetic package sealed with vacuum in the interior of the package.

5.2.11 *material permeability* — the tendency of fluids to directly migrate through the walls of the package. Bulk permeability depends on chemical composition and diffusion, as well as solubility and desorbability. For example, diffusion of fluids may be accelerated along grain boundaries. Stainless steel in half-hard condition will have small grain sizes relative to package wall thickness, while in fully annealed condition the grain size may be comparable to package wall thickness. In the latter case material permeability is increased although the chemical composition is unchanged.

5.2.12 *measured leak rate* — the leak rate of a given package as measured using a specific set of operationally defined conditions and test media, often referred to as the apparent leak rate.

5.2.13 *micro electro-mechanical system (MEMS)* — a term used to describe micron-scale structures, sensors, actuators, and technologies used for their manufacture (such as, silicon process technologies), or combinations thereof.

5.2.14 *near hermetic package* — at least a portion of the package materials is formed of materials having permeability as high as 10^{-8} to 10^{-10} atm-cc/sec. An example of such a material is liquid crystal polymer (LCP).

5.2.15 *outgassing* — release of gases absorbed on interior package surfaces and devices or evolution of gases due to chemical change of enclosed materials.

5.2.15.1 For example, epoxies, silicones, and Teflon all tend to outgas water vapor. As the pressure is reduced below about 100 millitorrs, much of the load in a vacuum hermetic package may be from outgassing. In addition, outgassing may be due to virtual leaks, or the slow release of gases that have been inadvertently trapped within the enclosure.

5.2.16 *partition coefficient or equilibrium constant* — the ratio of gas concentration at the solid-gas interface at the package exterior to the partial pressure of the gas in the surrounding medium. Symbol K is used.

5.2.17 *permeability coefficient* — a measure of the capability of a specimen to permit the flow of fluids through its surrounding walls. The product of permeance and the wall thickness. Defined as the volume of fluid passing through a material of unit thickness, per unit area and unit time, under unit partial-pressure difference between the two sides of the specimen. Determined by both the geometrical and material properties of the specimen. Symbol P is used, or $P'O_2$ to express the permeability coefficient specifically of oxygen gas, for example.

5.2.18 *permeance* — a measure of the ability of a solid specimen material to conduct fluid. Also defined as the volume of fluid which, under steady conditions, crosses unit area of the sample in unit time under unit pressure difference and at constant temperature. Permeance depends only on the specimen itself. Symbol Q is used.

5.2.19 *permeation* — the penetration of a permeate (fluid = gas or liquid) through a solid.

5.2.20 *quasi-hermetic package* — hermetic over a limited amount of time. Conventional package materials such as metal, glass or ceramic are used but an organic adhesive material forms the seal.

5.2.21 *relative humidity* — the ratio of the actual vapor pressure to the vapor pressure in the state of equilibrium with the liquid.

5.2.22 *seal integrity* — a measure of the quality of the package seal. Evaluation of the seal material for any paths that could allow environmental conditions to ingress the cavity or egress from the cavity and the bond quality between the package and seal material or the seal material and the lid is necessary to determine the seal integrity. The width or thickness of the seal material may also be important to the integrity, based on the permeability of the material and/or the structural strength required for the application.

5.2.23 *solubility* — the relative capability of a fluid being dissolved into a material.

5.2.24 *time zero* — the beginning point of the operating life of a packaged device, generally the time at which the hermetic seal is actually formed. (Note that when surface mount reflow follows formation of the hermetic seal, this

definition holds only assuming that the high reflow temperatures do not irreversibly alter the hermetic seal. Otherwise, time zero would begin at completion of surface mount reflow.)

6 Permeation

6.1 Permeation of fluids through package materials causes a change in the internal package pressure. As such, package materials must be chosen carefully.

6.2 MEMS building block materials must be qualified for permeability. Materials of commercial interest having specifically defined deposition conditions are addressed. See Appendix 3.

6.3 Depending upon the material and deposition method, the film structure may be porous and/or metastable even at room temperature. Sputtered films may contain enough (several atomic %) of the sputtering gas species (usually Argon) to cause significant outgassing later. Plating solutions may include organics or other chemicals that will be included in the plated layers and later evolve into outgassing species. Air species (including moisture) absorbed during fabrication may be desorbed later.

7 Detection and Measurement of Hermetic Package Permeability

7.1 Permeation involves three steps. First, a fluid surrounding the material being permeated absorbs into the solid at the solubility limit. Second, the fluid diffuses away from the surface, driven by the concentration gradient towards the interior of the package. Third, the fluid that has diffused to the package interior desorbs. The first two steps are generally rate controlling, with desorption occurring very rapidly by comparison. In equation form:

$$P = K \times D \quad (1)$$

where:

D = the diffusivity in cm^2/s .

K = the solubility in cm^3 (at STP) / $\text{cm}^3 \times \text{Pa}$.

P = the permeability coefficient in units of cm^3 (at STP) $\times \text{cm} / \text{cm}^2 \times \text{s} \times \text{Pa}$.

The above equation implicitly assumes that 100% of the fluid diffusing through the package walls desorbs. For MEMS packages where the interior surface to volume ratio is very high, it might become important to separately consider desorption. Data is not currently available to support such consideration.

7.2 Diffusivity (D) is defined by Fick's first and second laws:

$$J = -D \frac{\partial \phi}{\partial x}; \quad \frac{\partial \phi}{\partial t} = -D \frac{\partial^2 \phi}{\partial x^2} \quad (2)$$

where ϕ is the concentration in g/cm^3 .

7.3 The partition coefficient K , also called equilibrium coefficient, is the ratio of fluid concentration at the solid-fluid interface to the partial pressure of the fluid in the surrounding medium, $K = \frac{c}{p}$. Solubility follows Henry's or Sievert's Law, depending on the fluid species.

7.3.1 Henry's Law states that the amount of a given fluid dissolved in a given type and volume of solid (liquid) is directly proportional to the partial pressure of that fluid in equilibrium with that solid (liquid):

$$p = k_H \times c = K \times c \quad (3)$$

where:

p = the fluid partial pressure in Pa.

k_H = the Henry's constant of the material in Pa/mol.

c = the concentration of the dissolved fluid at the interface in cm^3/m^3 , or in mol/m^3 .

7.4 Diffusivity, solubility and desorbability follow Arrhenius relationship with temperature:

$$D = D_0 e^{\frac{-E_D}{kT}} \quad (4)$$

where:

D_0 = a constant in cm^2/sec .

E_D = an activation energy in eV.

k = Boltzmann's constant = 8.62×10^{-5} eV/Kelvin.

T = the temperature in Kelvin.

Note that E_D is always a positive value in that equation, that is, D always increases with T . A phase transition of the permeated material (such as a glass transition temperature T_g) or the permeating species (such as a fluid critical point) may affect the values of D_0 and/or E_D .

7.5 The temperature dependence of the solubility coefficient is determined as:

$$K = K_0 e^{\frac{-E_S}{kT}} \quad (5)$$

where:

K_0 = a constant in cm^3 at STP/ $\text{cm}^3 \times \text{Pa}$.

E_S = an activation energy in eV.

Note that E_S is usually a negative value in that equation, that is, K usually decreases with T . A phase transition of the permeated material (such as a glass transition temperature T_g) or the permeating species (such as a gas critical point) may affect the values of K_0 and/or E_S .

7.6 Clearly, since $P = K \times D$, the temperature dependence of the permeability coefficient can be expressed as:

$$P = P_0 e^{\frac{-E_P}{kT}} \quad (6)$$

where:

P_0 = a constant in cm^3 at STP $\times \text{cm} / \text{cm}^2 \times \text{sec} \times \text{Pa}$.

$E_P = E_D + E_S$ is an activation energy in eV.

Note that E_P may be a positive or negative value in that equation, that is, P may increase or decrease with T , depending upon the signs and magnitudes of E_D , E_S and E_P ; however, in practice E_P is usually a positive value, that is, P usually increases with T . A phase transition of the permeated material (such as a glass transition temperature T_g) or the permeating species (such as a gas critical point) may affect the values of P_0 and/or E_P . Clearly, these phase transitions should be taken into account when performing hermeticity accelerated stresses.

7.7 Solubility can also be expressed in terms of molecules/ cm^3 by applying the ideal gas law:

$$K = \frac{c}{p} = \frac{\rho}{nR_0T/V} = \frac{M}{R_0T} \quad (7)$$

where:

p = the pressure differential in Pa.

ρ = the fluid density in g/cm^3 .

V = the volume in cm^3 .

N = the number of moles.

R_0 = the gas constant, equal to $8.314 \times 10^5 \text{ m}^3\text{Pa}/\text{K}\cdot\text{mol}$.

T = the temperature in Kelvin.

M = the fluid molar mass in kg/mole.

7.8 Permeation of a package can be considered as a mass flow from exterior to interior. This can be considered as analogous to electric current, or charge flow, according to Ohms law:

$$I \left(\frac{\text{coulombs}}{\text{sec}} \right) = \frac{V(\text{volts})}{R(\text{ohms})} \quad (8)$$

7.9 The electrical resistance of a conductor and current can be further defined by the material and geometry properties:

$$R = \rho \left(\frac{L}{A} \right) = \frac{1}{\sigma} \left(\frac{L}{A} \right); \quad I = \sigma V \left(\frac{A}{L} \right) \quad (9)$$

where:

ρ = the resistivity, a material property with units of ohm-cm.

L = the length of the conductor in cm.

A = the cross-sectional area in cm².

σ = the conductivity, the inverse of resistivity.

7.10 Now consider a package having a seal of thickness, t , width, W , and perimeter length, l , (see Figure 1). The cross-sectional area $A_C = t \times l$. The mass flow can be defined simply as:

$$I_{gas} = Pp \left(\frac{A_C}{W} \right) \quad (10)$$

7.11 Clearly the permeability coefficient P is analogous to electrical conductivity, and partial pressure p , the driving force for permeation, is analogous to voltage in an electrical circuit.

7.12 Further, the permeation of a package can be easily estimated by considering the various elements of the package as resistive components. Similarly to adding electrical resistors in parallel, the total resistance is determined by adding reciprocals, then taking the inverse.

7.13 Permeation is one component considered in modeling the lifetime of a hermetic package. As the cavity pressure of a hermetic package changes over lifetime, the difference in the partial pressure between the exterior and interior of the package decreases. This can be addressed mathematically. However, for significant reductions in the pressure differential, the package is already compromised. Therefore, for all practical purposes the simpler equations are useful and provide sufficient accuracy in predicting the package lifetime.

7.14 There is a lag time between initial application of differential pressure across the package walls and the time that the diffusing fluid appears in the interior. It is useful (see § 13.7; Moisture Ingress into Non-Hermetic Enclosures and Packages – a Quasi-Steady State Model for Diffusion and Attenuation of Ambient Humidity Variations) to define a time constant τ such that:

$$\tau = \frac{VL}{AP} + \frac{L^2}{2D} + \tau_G - \tau_O \quad (11)$$

where:

V = cavity internal volume.

L = cavity wall thickness.

A = cavity wall area (perpendicular to the concentration gradient).

P = permeability coefficient for the cavity wall material.

D = diffusion rate through the cavity wall.

τ_G = time constant associated with internal gettering (Note: Considered negligible for this method).

τ_O = time constant associated with internal outgassing (Note: Considered negligible for this method).

For thin or non-absorbing walls the first term dominates. In the case of thick or absorbing walls, the second term of the equation dominates. For typical MEMS packages where the volume, V , is small, the second term will generally dominate. In that case, the time constant is proportional to the square of the wall thickness. It is therefore very advantageous to have thick and/or absorbing walls for a MEMS cavity, since in that case the internal volume or wall area has little effect. See § 13.7 (Measuring Mass Flows in hermetically sealed MEMS & MOEMS to ensure device reliability) regarding the proper measurement of moisture in small packages.

7.14.1 The effect of gettering (outgassing) is to decrease (increase) the rise of the partial pressure of the relevant fluid in the package interior. Gettering and outgassing are very important considerations in designing hermetic packaging; however, both are beyond the scope of the present document and are not considered for this method.

8 Examples

8.1 Two examples of geometry and material choices are included below. The procedure for calculating whether a package will meet requirements is to:

- 8.1.1 Determine the materials and geometries to be evaluated.
- 8.1.2 Calculate the flux of each specific gas of interest at STP conditions.
- 8.1.3 Calculate the dew point of water at the lowest operating temperature.
- 8.1.4 Estimate the flux of each specific gas at worst case temperature.
- 8.1.5 Compare flux and dew points to requirements.
- 8.1.6 Adjust materials or geometries if requirements are not met and repeat the analysis.

8.2 *Example 1* — Consider a specific package with a lifetime requirement of 5 years. Assume permeation of oxygen at 25°C through a square polymer seal material. For convenience, assume an oxygen partial pressure differential of 2×10^4 Pa (~0.2 atm).

