JPL Publication
D - 17920

Getters for Reliable Hermetic Packages

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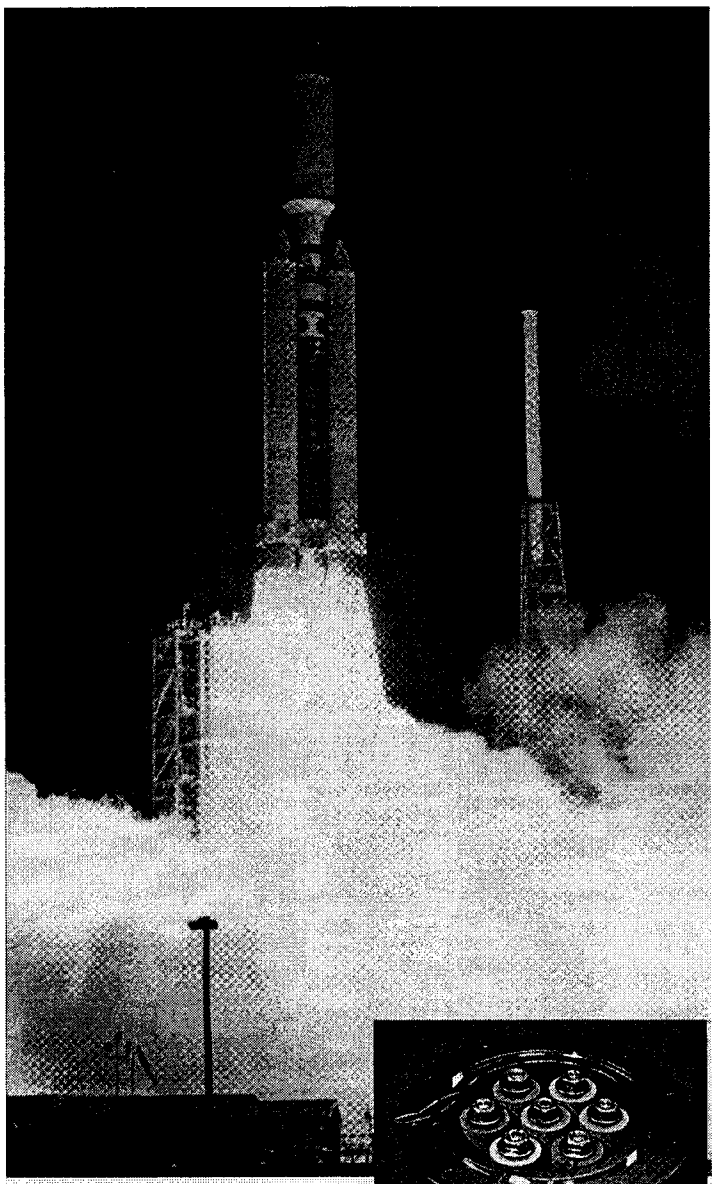
Support: NASA RTOP No. 100336-60.10003 through
Advanced Technology Program Office (JPL)

October 18, 1999



Cassini Launch photo courtesy of NASA

Example of getter rods used on the Huygens Probe

Declaration

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Executive Summary

A variety of sealed-off devices such as cathode ray tubes (CRT's), electron tubes, plasma displays, particle accelerators and colliders, vacuum thermal insulation, ultra-high vacuum (UHV), extreme high vacuum (XHV) systems for semiconductor processing, X-ray tubes, lamps, field-emission displays (FEDs), flat panel displays (FPDs), some microelectromechanical systems (MEMS) and science instruments for space applications, nuclear systems require a vacuum for their successful operation. Maintaining vacuum in extremely small to large volume electronics hermetic packages and vacuum systems depends on the true surface area of the materials exposed to that volume as this is the source of species to be outgassed and it is this outgassing that will finally destroy the vacuum. Destroying the vacuum by outgassing from various materials used may lead to poor performance of the device (e.g., vibratory microgyroscope MEMS device). Getters are routinely used in small and large static systems and similarly getters will be needed if the desired system lifetimes of many years are to be obtained in MEMS and other packages for space applications.

JPL has been working on various types of microelectromechanical systems (MEMS) for space applications. Many high sensitivity microelectromechanical systems such as microgyros and some pressure sensors need to operate in hermetically sealed vacuum electronic packages to realize their full performance characteristics. This vacuum is destroyed by out-gassing of various species such as water vapor, hydrogen, deuterium, tritium, methane, carbon monoxide, nitrogen, oxygen, methane, argon, nitrogen and carbon dioxide from the package surfaces and microleaking or permeation through the package body. The loss of vacuum is particularly serious if organic materials are used in isolated MEMS packaging device. A getter material is

needed to eliminate this problem and to achieve successful MEMS device operation for long duration space applications. The term "getter" refers to materials, which chemically sorb active gases in a vacuum environment. A solution is proposed using a nonevaporable high porosity getter material family such as the type of zirconium-aluminum-iron manufactured by SAES Getters Inc., to solve the hermetic sealing problem associated with the microgyro, other similar MEMS devices and other vacuum systems where hermetic sealing is required. The getter consists of a highly porous and mechanically stable packaging component installed inside the MEMS vacuum packaging chamber and subsequently activated.

The activation of the getter is a key step, which should be performed using a suitable combination of temperature and time. This removes the layer of surface oxides, nitrides, and carbides, by their diffusion into the bulk of the getter and provides a clean metallic surface ready to react with the impinging gaseous molecules in a vacuum environment. Depending on the diffusion constants for the elements (such as H_2, CO, CO_2, H_2O, etc.,) into the getter materials, one can have a more or less effective cleaning of the surface during the activation process. The diffusion rate of various gaseous species present in the package increases upon raising the temperature of the getter material. The activation of the getter must be done when it is exposed for the first time or whenever exposed to air.

The solid-state getters may be either planar or three-dimensional and exhibit good mechanical strength. They must be particle free under the stringent operational conditions in space and on the ground, and they should have a high active surface area that can easily be activated at low temperatures. This minimizes problems such as high ambient temperature that may be detrimental to MEMS devices and other packages during the activation of getter. High porosity combined with a large active surface area of the noevaporable getter will assure

excellent sorption performances at room temperature. There should not be any loss of getter particles before, during, or after activation of the getter in a packaged MEMS device as this may cause failure of the MEMS device. It is critical to maintain the getter's mechanical structure during shocks and vibrations at the time of spacecraft launch and during operation of the MEMS device. The presence of an activated getter material inside the MEMS package will allow achievement of a better vacuum in the hermetically sealed vacuum package. The presence of a getter material inside a MEMS package is needed to avoid a pressure increase above the operational limit of the MEMS device. Sorption of outgassed species by getters permits a greater anticipated lifetime for MEMS devices in hermetically sealed packages.

I have provided an overview on various aspects such as gas sources in the electronics packaging, possible solutions, thermal treatment of packages, package materials, type of getters, applications of getters, preparation, characterization, and activation of getters, leak testing, and advantages and disadvantages of the various types of getters in various applications. This document will guide the researchers to select an appropriate getter for their respective application. The author of this document encourages the user to search for the latest information on this getter technology prior to implementing the concept of a getter to solve the problem in their specific application.

Acknowledgements

The document was put together at the Jet Propulsion Laboratory, California Institute of Technology, Pasadena, CA, under the contract with the National Aeronautics and Space Administration (NASA). It was primarily funded by the NASA Code Q under Research Technology Objectives and Plans (RTOP) and the Center Program Plan, UPN 100336-60.1 through Jet Propulsion Laboratory's Assurance Technology Program Office of Engineering Mission Assurance Division. A review of the document by Mr. Steve Bolin is gratefully acknowledged.

Historical Perspective

The research and development of inorganic or organic getter materials that are able to sorb small quantities of reactive gases such as CO, CO_2, H_2O, O_2 and H_2, began roughly half a century ago to increase the life significantly and to substantially improve the performance of vacuum electron tubes. The first developed getter materials were evaporable getters and they are based on alloys of barium. The term evaporable refers to the method of depositing the alloys as thin films on the inner surface of vacuum tubes. As a final step in a tube's manufacture, it is evacuated and a getter is evaporated by inductive heating to produce a highly reactive film capable of sorbing outgassing components as well as compensating for small leaks in the outer seals. This technology has extended tube life for hundreds to thousands of hours and is still very important today in cathode ray tube's (CRTs) and television picture tubes.

Hermetic Packaging

Electronics packaging is defined as interconnecting, powering, cooling, and protecting integrated circuits. This document aids researchers in protecting the MEMS device performance and other vacuum systems by means of hermetic packaging of MEMS devices and vacuum systems for various ground and space applications.

Condensed moisture on a MEMS device's surface and degassing of gaseous species during the operation may lead to the principle causes of failure in the field. One packaging method designed to prevent performance degradation due to moisture's deleterious effects and outgassing of gaseous species from the packaging materials is the hermetic package. Ceramic or metal is used to form an enclosure to isolate the electronic or microelectromechanical devices from the ambient operating environment. By eliminating condensable moisture and degassed

species from the cavity of the package during the sealing process and preventing the ingress and egress of moisture and other gaseous species at the package perimeter during its operating life. Excellent long-term reliability may be achieved. The word hermetic is defined as completely sealed by fusion, solder, and so on, so as to keep air or gas from getting in or out; in other words, airtight. In hermetic packaging practice, such seals are nonexistent. Small gas molecules will enter the package over time through diffusion and permeation. In light of this permeation and the additional factor of outgassing, there are instances where the use of getters in the electronics packages will be necessary in order to obtain long life.

Introduction

A variety of sealed-off devices such as cathode ray tubes (CRT's), electron tubes, plasma displays, particle accelerators and colliders, vacuum thermal insulation, ultra-high vacuum systems for semiconductor processing, X-ray tubes, lamps, infrared detector dewars, magnetic confinement fusion devices, helium-neon lasers, ring laser gyroscopes, field-emission displays (FEDs) require a vacuum for their successful operation. Maintaining vacuum in extremely small volume packages depends on the surface area of materials exposed to that volume as this is the source for species that outgassed and finally destroy the vacuum. Getters are routinely used in larger static systems and similarly getters will be needed if desired system lifetimes of many years are to be obtained in MEMS and other packages for space applications.

Many high sensitivity microelectromechanical systems (MEMS) need to operate in a hermetically sealed vacuum electronics package to realize their full performance. This vacuum is destroyed by out-gassing of various species (such as water vapor, hydrogen, carbon monoxide, nitrogen, oxygen, and carbon dioxide) from the inner package surfaces and microleaking or

permeation through the package body. The loss of vacuum is particularly serious if organic materials are used in an isolated MEMS packaging device. A getter material is needed to eliminate this problem and achieve successful MEMS device operation for long duration space applications. The term "getter" refers to materials, which chemically sorb active gases in a vacuum environment. A solution is proposed using a SAES nonevaporable, high porosity getter material family (zirconium-aluminum-iron, Zr-Al-Fe) to solve the hermetic sealing problem associated with the microgyro and other similar MEMS devices and vacuum systems where hermetic sealing is required. The getter consists of a highly porous and mechanically stable packaging component that may be installed inside the MEMS vacuum packaging chamber and activated using a suitable approach.

The solid-state getters may be either planar or three-dimensional and exhibit good mechanical strength. They must be particle free under the stringent operational conditions in space and on the ground, and they should have a high active surface area that can easily be activated at low temperatures. This minimizes problems such as high ambient temperature that may be detrimental to MEMS devices during activation. High porosity combined with a large active surface area will assure excellent sorption performances at room temperature. There should not be any loss of getter particles before, during, or after activation of the getter in a MEMS package as this may cause failure of the MEMS device. It is critical to maintain the getter's mechanical structure during shocks and vibrations at the time of spacecraft launch and during operation of the MEMS device. The presence of an activated getter material inside the MEMS package will allow achievement of a better vacuum in the hermetically sealed vacuum package. The presence of a getter material inside a MEMS package is needed to avoid a pressure increase above the operational limit of the MEMS device. Sorption of outgassed species by

getters permits a greater anticipated lifetime for MEMS devices hermetically sealed in packages. For example, the residual gases in travelling wave tube (TWT) have several deleterious effects. The gases interfere with the electron beam and cause the electron beam to scatter, and conversely, the electron beam will ionize the gases and the ions will back stream to the cathode and sputter the cathode surface causing emission current degradation. [1]. Kayali and Ragle have reported an overview of hydrogen effects on GaAs microwave semiconductors in a JPL document. [2]

Watanabe et al., [3,4] have described the alloying effects on absorption and desorption kinetics of deuterium for Zr-Al alloys with mass analyzed thermal desorption spectroscopy using a conventional high vacuum system. They have also found that the temperature dependence of the rate constants, revealed that the activation energies for both the absorption and desorption processes were lowered by increase Al content in the alloys.

Homes and Corrigan [5] have described the interaction of H_2O and O_2 with evaporated titanium gettered surfaces under ultra high vacuum (UHV) conditions. The use of getters to reduce the concentration of oxidizing species in the residual gases in electron tubes is an established technology of well proven effectiveness. Use of getters is proposed to maintain the high initial reflectivity of bare evaporated aluminium reflecting coatings in a proposed satellite spectrometer. The wavelength range over which the instrument is to operate is 90 – 120 nm, where even a slight oxidation causes a serious loss of reflectivity. The major source of oxidizing species in such a spectrometer would be outgassing from the apparatus itself. [6]

Sources of Gases

It is important to know the composition of the residual gas in the vacuum system or a hermetically sealed electronics package. Outgassing from the walls of the vacuum chamber, the interaction of these gases with the hot filaments of gauges or MEMS devices, leaks, the permeability of the materials of construction and the type of pumping mechanism used will all produce a residual atmosphere bearing no relation to the composition of the normal atmosphere. The constituents of the normal atmosphere are mainly nitrogen and oxygen in the ratio of 80 - 20% together with small traces of rare gases, carbon dioxide, argon, neon and helium, and a variable quantity of water vapor (depending on ambient temperature and humidity conditions). The abundance of each gas is conveniently expressed as the pressure that each constituent contributes to the total. These partial pressures are expressed in torr and are shown in Table 1. [7]

Table 1: Partial Pressures of Atmospheric Constituents

Nitrogen	596 Torr
Oxygen	159 Torr
Argon	7.1 Torr
Carbon dioxide	0.23 Torr
Krypton	7.6×10^{-4} Torr
Xenon	6.8×10^{-5} Torr
Neon	1.4×10^{-2} Torr
Helium	3.8×10^{-3} Torr
Hydrogen	3.8×10^{-4} Torr
Water vapor	7 Torr, but depends on relative humidity

Hydrogen Evolution from Packaging Materials

According to Saito et al., [8] the major source of hydrogen gas in a hermetically sealed package is from electroplated nickel in the package housing. KOVAR is typically electroplated

with nickel using sulfamate per QQ-N-290; A40 was electroless plated with nickel-phosphorus per MIL-C-26074Ni/Au. Then the plated test specimens were placed in aluminum housing. It was baked out in a nitrogen furnace and subsequently laser welded to conduct a storage life test. In both unbaked and baked (150°C) A40 experiments, hydrogen reached a constant level in the hermetically sealed housing after 300 hours indicating all the hydrogen in electroplated nickel being baked out. In contrast, electroplated KOVAR continued to evolve hydrogen, indicating that the hydrogen in plated nickel diffuses into KOVAR at a much higher rate than into aluminum in A40. The bake-out of hydrogen from the electronics packaging materials is considerably more beneficial for A40 and other aluminum-based alloys than KOVAR and iron-base alloys.

It is known that the temperature must be higher than 350°C to bake-out hydrogen from the iron-base alloys.[8] However, the wire bondability degrades as bake-out temperature of the electronic packaging material increases. The optimum bake-out temperature is based on the amount of hydrogen evolved and subsequently its effect on wire bondability. The optimized hydrogen bake-out temperature was at 250°C for 168 hours for Ni/Au plated Kovar (Fe/Ni/Co) and A40 (Al/Si) housings based on the residual hydrogen contents measured by Residual Gas Analysis (RGA) and subsequent wire bondability. For KOVAR housings, the evolved hydrogen was reduced from 0.6% to 0.0033% with a post-plating hydrogen bake. For A40 housings, the hydrogen content (0.002% after the hydrogen bake) is significantly lower than that of Kovar.

Why use Getters?

Getters offer very high pumping speeds for reactive gases such as H_2, H_2O, CO, CO_2, N_2, and O_2, thereby complementing the capabilities of conventional dynamic vacuum pumping

technologies. Getters will be particularly applicable when a vacuum chamber or electronic package housing is in a static condition. For example, one of the Thomas Edison's assistants used a coating of red phosphorus inside a light bulb to remove water vapor in order to increase significantly the lifetime of light bulbs.

Mechanism of Gettering [9]

Nonevaporable getters are alloys of metals from the IV-A Group (Ti, Zr, Th) of the periodic table of the elements. The metals in a getter are capable of dissolving their own chemically formed oxides, nitrides, and carbides in the solid-state form at elevated temperatures, thus, creating a clean, renewable, and highly reactive metal surface. The activation temperature of the metals (Ti, Zr, Th) is relatively low (in some cases, below 500°C), making materials useful in vacuum applications where the environment is not sealed. By employing a nonevaporable getter as an *in situ* pump in a vacuum chamber, greater pumping speeds are attained (along with lower base pressures) than could be achieved without the getter. Getters eliminate active gases from vacuum environments by chemically binding the gaseous molecules to their surfaces. The sorption process starts with a clean, metallic, getter surface. During heating, the getter is activated by the diffusion of surface-adsorbed gas molecules into the bulk of the getter; thus exposing a fresh metal surface for renewed chemical reaction. Then the reaction process continues until the surface saturates with the reaction products.

Gettering involves the removal of atoms and molecules from the gas phase by chemical reactions on an active surface. Getters are characterized by their chemical affinity for different gases and by the diffusivity of each chemisorbed species into the bulk of the getter material.

Getters are normally evaluated in terms of their sorption speed and their capacity for various active gases.

These gettering characteristics are heavily dependent on the amount of active surface available for reaction with gases. If the getter is operating at room temperature, when, for most gases, very limited bulk diffusion takes place, the surface of the getter can become saturated with reaction byproducts relatively quickly.

An exception to this approach is hydrogen and its isotopes. Hydrogen can easily diffuse in to a getter because it moves about essentially as a proton. Hydrogen in the interior of a nonevaporable getter (NEG) establishes a solid solution that exhibits an equilibrium pressure that depends on the concentration of hydrogen and the ambient temperature. This behavior is very important for ultra high vacuum applications, where H_2 is the most important gas being pumped. Unlike with other gases, H_2 pumping is completely reversible, allowing for regeneration cycles that increase getter lifetimes indefinitely. In general, getter materials are designed to achieve high diffusivity, which provides both a high sorption speed and a large capacity. This parameter is particularly important for NEGs, which operate as bulk getters.

The zirconium-based system is very reactive with a wide variety of gas molecules such as H_2, CO, CO_2, O_2, N_2, and NO_x to form essentially nonreactive oxides, carbides, and nitrides. Generally, the reactions proceed by dissociative chemisorption followed by a reaction to form the resulting oxide, carbide, or nitride:

$$CO\ (gas) + 2\ M \Leftrightarrow CO\ (adsorbed) + 2\ M \rightarrow M\text{-}C + M\text{-}O$$

$$CO_2\ (gas) + 3\ M \Leftrightarrow CO_2\ (adsorbed) + 3\ M \rightarrow M\text{-}C + 2\ M\text{-}O$$

$$N_2\ (gas) + 2\ M \Leftrightarrow N_2\ (adsorbed) + 2\ M \rightarrow 2\ M\text{-}N$$

$$NO\ (gas) + 2\ M \Leftrightarrow NO\ (adsorbed) + 2\ M \rightarrow M\text{-}O + M\text{-}N$$

Where M represents constituents of the zirconium based getter alloy. Hydrogen compounds seem to be an exception, with the hydrogen being dissociatively chemisorbed then dissolved into the metal bulk:

$$H_2 \text{ (gas)} + M \Leftrightarrow 2\ H \text{ (adsorbed)} + M \Leftrightarrow 2\ H \text{ (M bulk)}$$

$$H_2O \text{ (gas)} + 3\ M \Leftrightarrow 2\ H \text{ (adsorbed)} + M\text{-}O + 2\ M \Leftrightarrow M\text{-}O + 2\ H \text{ (M bulk)}$$

Depending on the temperature, the reaction products that will diffuse into the bulk of the alloy, and finally, providing a fresh clean surface for renewed adsorption and a subsequent chemical reaction. The reactivation process can be carried out by sorption at high temperature either continuously or periodically after normal operation at low temperature. At 25°C the pumping speed for CO quickly falls due to surface saturation, but at 400°C it extends far beyond this level due to diffusion of reaction product compounds into the bulk. On the other hand, hydrogen shows reduced capacity at high temperatures (~1000°C) due to equilibrium desorption and solubility effects. Hydrogen quickly diffuses into the bulk even at room temperature. Its total capacity is significantly increased due to its higher solubility at the lower temperature.

Mitigation to hydrogen degradation

Saito et al., [8] have recommended in their research work a three pronged approach to mitigate hydrogen degradation. Eliminate the hydrogen source and bake-out hydrogen prior to seal. The elimination of the hydrogen source can be implemented by reducing hydrogen entrapment during electroplating. Another mitigation is to use a hydrogen getter. There are many types of commercially available hydrogen, which can be implemented to reduce the hydrogen contents in a hermetic package.

Vacuum Packaging of Microsensors [10]

Packaging is a very important aspect to be considered for microsensors. Many packaged pressure sensors and accelerometers have been developed using bonded silicon-glass structure [11]. The squeeze film damping effect should be reduced in capacitive accelerometers to improve their dynamic response. In infrared sensors, thermal conductance between a sensing element and its package should be minimized for high sensitivity. The high quality factor (Q) required in resonant sensors for high resolution and wide dynamic range that can be obtained by reducing air dampening. [12] The sensitivity of thin diaphragm pressure sensors is very high if the reference cavity pressure is kept extremely low.[13] For these purposes, vacuum-sealed cavities are widely required.

It was not possible to make a high vacuum cavity by using anodic bonding of glass-silicon in vacuum. Two residual gas sources that pose a problem for vacuum sealing have to be considered. One is gas generation during the anodic bonding process.[14] The other is gas desorption from the inner surface of the sealed cavity and the getter materials. These gases must be eliminated in order to create a high vacuum cavity.

High vacuum sealing methods are classified into two categories. In one type, the residual gas in the cavity is evacuated through an opening after anodic bonding and then the opening is plugged by depositing aluminium or silicon monoxide (method A). The aluminum plug can be used as an electrical feedthrough if desired. In the other, a small vacuum pump is placed in the sealed cavity. A nonevaporable getter is used as a small vacuum pump (method B). Method C is the combination of methods A and B.

The NEG material used is a Ni/Cr ribbon of 0.005 mm thick, 2 mm wide and 6 mm long covered with a mixture of porous Ti and Zr-V-Fe alloy. Before, activation, the NEG does not

absorb gases because oxides and carbides cover its surface. To absorb gases, the NEG must be activated at around 400°C. Since the anodic bonding temperature is also 400°C, the NEG is activated during the anodic bonding process. The active metallic surface is formed by the activation because surface oxides and carbides are diffused into the bulk. Then the NEG can absorb the residual gases.

In method A, evaporation of Al and silicon monoxide (SiO) on the opening was carried out under a pressure of 10^{-6} Torr after the glass-silicon anodic bonding process. Before evaporation, the gas inside the cavity was evacuated through a channel for 2 hours. The opening was plugged with Al thicker than 8 μm. The pressure inside the cavity was approximately 200 Torr. In the case of SiO, evacuation was carried out with substrate heating at 170°C. The opening was plugged with 2 μm with SiO. The estimated pressure inside the cavity was about 60 Torr.

In method B a NEG is placed in the sealed cavity. At first no special pre-heat-treatment was performed to the NEG getters to desorb gases before the anodic bonding process. The anodic bonding process was carried out under a pressure of 10^{-6} Torr. After the anodic bonding process in vacuum. The NEG was heated up to 400°C and reactivated so as to absorb the residual gases in the cavity. Before reactivation, the pressure inside the cavity was around 400 Torr so that the diaphragm was inflated in the vacuum chamber. As the reactivation time was increased, the diaphragm became flat. Even after long NEG reactivation, the pressure inside the cavity was not lower than 5 Torr. It is assumed that the gettering function of the NEG was saturated.

In order to desorb gas from the NEG, a heat treatment was carried out at 300°C for 1 hour before the anodic bonding process. The anodic bonding process was successively carried out in

vacuum. This heat treatment was also applied for the above process (method B) with the NEG. The pressure in the cavity of the device with the NEG was 4×10^{-5} Torr. The cavity pressure was decreased from 400 Torr to 4×10^{-5} Torr by preheating at 300°C. This shows the preheating is quite effective in desorbing the gases from the NEG. The NEG was reactivated during the anodic bonding process and an additional reactivation was not carried out. With additional reactivation of 1 hour the pressure was lower than 1×10^{-4} Torr.

In method C desorption of gases from the NEG, silicon and glass, anodic bonding, evacuation of residual gas in the cavity through a channel, plugging the opening, and reactivation of the NEG. All these processes, except the last one have to be performed continuously in vacuum. The NEG was reactivated at 400°C for 2 hours. The pressure inside the cavity was lower than 1×10^{-5} Torr. No change was observed over 2 months, which indicates that leakage through the plugged opening was very small.

To avoid dust from the NEG coming into the cavity, it was formed on the backside of the wafer and was connected to the reference cavity through a small hole.

How a Nonevaporable Getter Pump Works

The gas sorption or pumping mechanism has four distinct stages; activation, adsorption, diffusion, and reactivation. As received, the getter is protected from the atmosphere by a thin surface film. This is dissolved into the bulk of the getter during activation in vacuum at an elevated temperature, typically 350 – 800°C. Adsorption and diffusion occur during pumping, with some active gases forming reaction products that are concentrated near the surface. Eventually, these reaction products start to reduce pumping speed. During activation, again at

higher temperature, reaction products are distributed more evenly throughout the getter material to leave a clean, active surface. [15]

Theory

Knize and Cecchi [16,17] have presented a theory of bulk getter operation that includes both adsorption, desorption kinetics, and bulk diffusion. The theory is applicable to diatomic gas, which adsorbs dissociatively on a getter material, which also exhibits time independent sticking co-efficient. Exact numerical solutions have been presented for a wide variety of parameters characterizing pumping and desorption scenarios. The experimentally observed behavior of the Zr-Al bulk getter is described accurately by the proposed theory. The characteristics of both pumping and desorption for a wide range of conditions are predicted closely by the proposed model. This consistent agreement for the entire range of getter operation comprises substantial evidence for the validity of the theory.

Getter Model

Knize and Cecchi [16,17] have summarized the theoretical and experimental aspects of phenomenology of bulk gettering. The theory includes both surface adsorption/desorption kinetics and bulk diffusion as well as external parameters such as the pressure and temperature. For the case of pumping at a constant pressure, the theory predicts that the pumping speed is pressure independent when the surface-limited adsorbed flux is less than the surface-limited flux. For higher pressures, the pumping speed becomes limited by the diffusivity and falls as one over the square root of pressure. The initial desorption is also shown to be diffusion limited. Subsequently, the desorption becomes surface kinetics limited and then the concentration

decreases as reciprocal of time (1/time). The experimentally observed behavior of ZrAl and ZrVFe bulk getters is accurately described by the theory.

Types of Getters

Getters are classified as being either evaporable or non-evaporable getters. An evaporable getter is a material that forms an active surface layer upon being deposited by evaporation. Since it exists as a thin film, an evaporable getter functions primarily at its surface. Evaporable getters make up for having relatively little bulk capacity by having a relatively large surface area. [18]

Hydrogen Getter Requirements

The following criteria may be used in selecting materials for gettering formulations:

1. Both reactants and products should be as chemically inert to their environment as far as possible except for hydrogen reactivity.
2. Reactants and products should have very low vapor pressures to ensure long term stability.
3. The gettering reaction should be irreversible or lead to extremely low hydrogen equilibrium concentrations.
4. The forward reaction rate should be sufficient to keep the dynamic partial pressure of hydrogen low during corrosion or outgassing in practical systems.
5. The reacting system should be mechanically convenient to incorporate into various types of systems.
6. Getters should be passive devices requiring no external energy sources for initiation

A gettering formulation fulfilling these requirements would ensure the irreversible removal of hydrogen without detrimental side reactions.

Evaporable Getters

An evaporable getter is a solid material that forms an active surface layers upon being deposited by evaporation in a vacuum chamber. An example of such a getter is $BaAl_4$. When $BaAl_4$ is heated to above 1000°C, it decomposes and barium is released to form a gettering film on the wall of the package in a vacuum.

Figure 1 shows early barium getters in receiving tubes. A "U" shaped channel filled with BaF_4 is mixed with nickel powder to assure an exothermic reaction and a reproducible evaporation of barium when the ring was heated with a radio-frequency coil placed coaxially to the getter, outside the vacuum device.

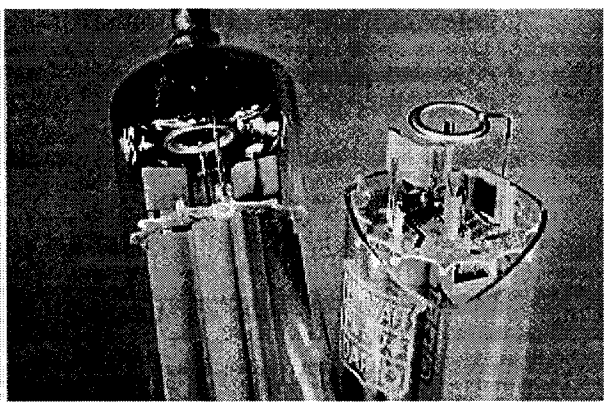


Figure 1: Early Barium Getters in receiving tubes [18]

A getter based on a barium pill embedded in a nickel flag and protected with a capsule made with aluminum foil and a perforated nickel cover. The first large shipment of this product by SAES Getters Inc., was a total disaster since most of the getters exploded and the barium deteriorated to carbonate. [18]

The accelerating voltage was reduced to low values where the energy of the electrons was not high enough to make the electrons penetrate the barium deposited on the screen. To solve this problem, the nitrogen doping method was invented whereby nitrogen is released by a N_2 containing compound (Fe_4N) added to the getter; N_2 is released during the heating process, just before beginning of the evaporation. The barium atoms, evaporated during the initial stages of the exothermic reaction, are scattered by N_2 and deposited mostly onto the cone-area without depositing an excessive quantity on the screen; this gives the additional advantage of an increased porosity of the resulting film and ensures a much higher speed of adsorption, particularly for the less active gases. The life of the first rectangular cathode ray tube (CRT) was only of 100 hours. The invention of a new design ring getter made possible on increase of life to 10,000 hours.

In the field of evaporable getters, it has been possible to ensure meeting the continuous and more and more stringent requirements of tube technology evolution by innovative concepts such as: ring getter, exothermic getters, nitrogen and delayed nitrogen doping, total yield getter, the soft flash getter, the high yield getter, the frittable getters, the low argon getter, and the more recent low particle getter.

In transmitting tubes as well as in power tubes, the high operating temperature and the high voltages were making the use of evaporated barium thin films impossible. Also in other applications the barium film is not ideal. The possibility of short-circuits due to the evaporated

film can also be a serious problem. In many cases there is no appropriate way to heat the barium getter by radio frequency (RF) because of the metallic envelope of the vacuum device. For various considerations, the need of non-evaporable getters (NEG) with high speed and capacity for all active gases was then well understood.

Thorium-cerium alloy was used, as a getter until later it was discovered that it was not desirable candidate since thorium is slightly radioactive. [19,20]

Nonevaporable Getters (NEG)

A nonevaporable getter is usually activated through the application of heat, but the temperature needed to drive-off sorbed gases is well below that required for the material to change phase. Of the two types of getters (evaporable and nonevaporable), NEGs have a wider range of applications.

The main field of application of NEGs is in sealed-off vacuum devices such as microwave tubes and X-ray tubes. NEG alloys are also being used as auxiliary vacuum pumps in a variety of ultra high vacuum (UHV) machines such as storage rings, synchrotrons, particle accelerators, and nuclear fusion devices. The research into new types of getters at SAES Getters Inc., (Italy and USA) is resulting in materials that are suitable for *in situ* use inside electronic devices, such as plasma displays and microelectromechanical systems (MEMS). Packaging is an important consideration with MEMS to maintain the stability and reduce the size and cost of these devices. The most difficult task in the MEMS packaging technology is to maintain a suitable vacuum level inside the package. Although, a micromachine is bonded in high vacuum, a good vacuum may not be maintained, due to outgassing produced during the bonding process.

A nonevaporable getter placed inside the package and activated during the bonding process can sorb the gases produced during the bonding as well as the lifetime degassing processes.

First generation evacuated micromachines require a vacuum in the order of 10^{-3} torr, thus requiring a getter to maintain that level of vacuum. SAES Getters Inc. is working towards solutions for situation requiring vacuum cavities created inside the chip itself and also compatible with the semiconductor fabrication technology. These gettering materials must function adequately while being extremely small and free of loose particles and thin enough to meet the ever-shrinking packaging requirement of MEMS. [21]

The sorption speed for all the active gases is greatest for a zirconium-aluminum (84-16%) alloy [22]. Figure 2 shows the sorption efficiency for different compositions of Zr-Al getter alloy. The most popular shapes of the finished getters are pellets and rings. A technique to coat metal ribbons with a relatively thin layer (50 – 100 μm) of the alloy without binders has drastically increased the geometrical surface area and the gettering speed. Figure 3 shows a metallic strip coated with a nonevaporable getter (St 101). This alloy operates ideally at 350 – 450°C with a very low H_2 equilibrium pressure. At the beginning, the getter has to be activated at 700 – 900°C for some time to obtain a clean reactive surface. The getter can also be reactivated many times. For hydrogen, sorption is reversible, and it is released during activation but it is immediately reabsorbed when the getter cools down. Later it was realized that there was also a need for a getter alloy, which could be activated at lower temperature (400 – 450°C). Another major objective was a non-evaporable getter with good performance over a wide temperature range and also at room temperature. [23]] An alloy of zirconium, vanadium and iron (St 707) was discovered to have the required performance. [24,25] The small pill of this

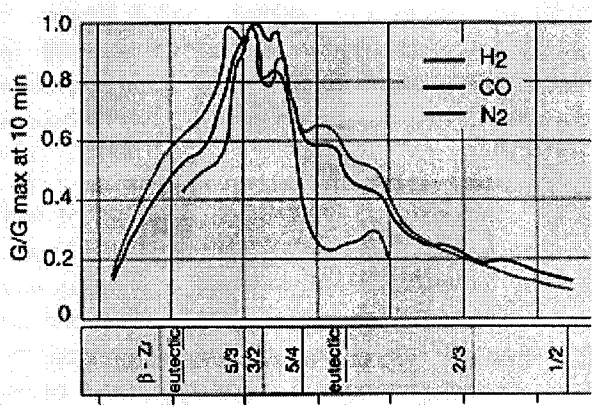


Figure 2: Sorption efficiency for different compositions of Zr-Al getter alloy [18]

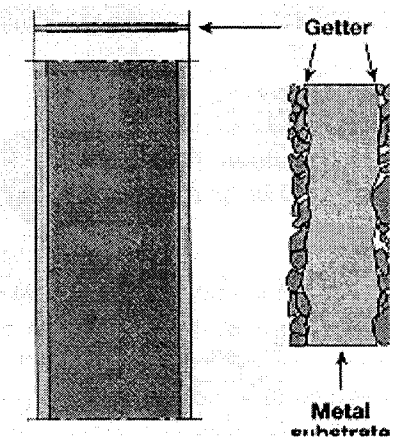


Figure 3: Nonevaporable getter strip [18]

alloy was able to assure an excellent vacuum for the life of the device (Stainless Steel Vacuum bottles such as thermos) taking care of outgassing and of the microleaks. A zirconium-iron (St 198) alloy used for lamp applications is able to sorb all other active gases except nitrogen. [26] A zirconium-nickel alloy (St 199) is able to sorb water with still negligible hydrogen pressure.[27] A number of titanium and titanium-zirconium alloys have been developed for special applications.[28] Techniques are aimed at imparting to NEG materials with special

mechanical characteristics (low particles) and particularly high porosity and surface areas to maximize their capacity when working at room temperature (porous sintered getters). Figure 4 shows the typical getter with internal heater. In the circle, a scanning electron micrograph of the microstructure. [29,30] The getter materials can be pure metals such as titanium or zirconium, but often are mixtures of these metals with other getter alloys, sometimes with the addition of antisintering components. Special techniques such as plasma spraying and, particularly, electrophoresis (high porosity thick film – HPTF – getters), have been used to obtain getters of preformed shapes and good mechanical stability. [31]

Getters are characterized by various parameters such as their chemical affinity for different gases and the diffusivity (diffusion into the bulk) of the chemisorbed species other parameters such as safety, workability, and toxicity must also be considered. The overall characteristics of getters are normally evaluated in terms of sorption speed and capacity for different gases, under various working conditions, values are obtained by dynamic or static measurement methods. [22] They are strictly related to the physicochemical parameters of affinity and diffusivity and also to the actual surface available for the reaction with the gas. If

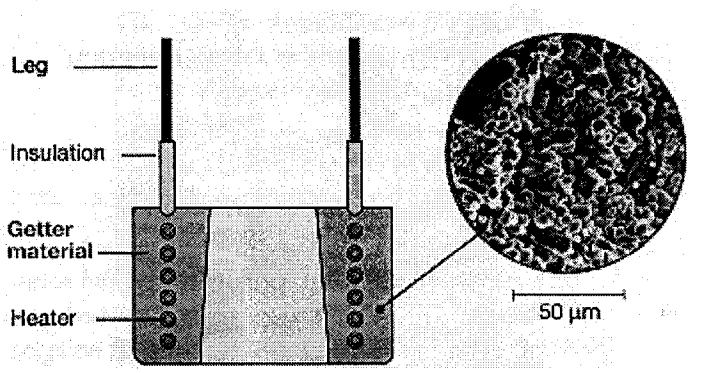


Figure 4: Typical porous getter with internal heater. In the circle, a scanning electron micrograph (SEM) of the microstructure of the getter [18]

the getter is working at room temperature, when, apart from H_2, practically no (or very limited) bulk diffusion takes place, the surface of the getter has to be maximized since the getter capacity corresponds to the surface saturation.

The capacity is greatly enhanced at high temperatures, since the diffusion of dissociatively chemisorbed species is promoted; the resulting practical capacity (corresponding to the amount of gas sorbed to a point where the sorption speed has decreased of a certain percentage of the initial value (usually 90 – 95%)) is, however, less than the stoichiometric capacity (when the getter materials has completely reacted with the gas). Getter materials are studied with the aim of getting a high diffusivity to increase both the sorption speed and the capacity. This parameter is particularly important for nonevaporable getters, which generally operate as bulk getters. H_2 equilibrium pressure for ultra-high-vacuum (UHV) applications, the real mechanism of the reactions taking place at the getter surface, the effect of radiations on sorbed species, the actual surface composition, mathematical models for the optimization of the getter configurations is a very complex process. [17]

Knowledge of gettering and the performance of getters could benefit an extensive use of various analytical techniques including X-ray photoelectron spectroscopy, Auger surface analyses, scanning electron microscopy, x-ray diffractometry, vacuum microgravimetry, surface area measurement and mass spectrometry.

A fundamental area of investigation concerns the activation process of getters. This is intended to lead to the achievement of an unsaturated getter surface ready to react with the active gases such as H_2, CO, N_2, H_2O, CO_2, and O_2 usually present in vacuum devices.

In the case of evaporable getters activation is performed by evaporating the getter material thus creating a free, unsaturated, normally large metallic film ready to chemisorb gases. [32,33,34]

The saturation process is more complex when dealing with the nonevaporable getters. Activation is carried out by properly heating the getter material and promoting the bulk diffusion of oxygen, carbon and nitrogen from a passivating surface layer until the surface is sufficiently clean to start sorbing the impinging gases. Figure 5 shows the schematic representation of the activation process of nonevaporable getter materials.

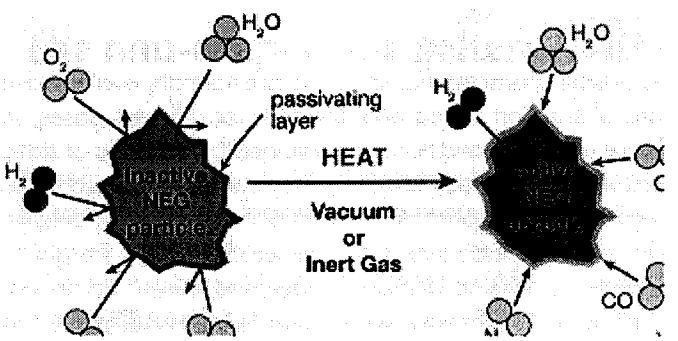


Figure 5: Schematic representation of the activation process for NEG [18]

The mechanism was described on the basis of theoretical consideration combined with the results of numerous sorption test results and mass-spectrometric analyses. The application of surface analysis techniques provides a clear and definitive interpretation of the mechanism as it appears from Figure 6. [35,36] The surface composition of a Zr-Al getter vs. temperature is analyzed by means of X-ray photoelectron spectroscopy (XPS) during activation. The surface concentration is of Zr and Al increases whereas O_2 and C concentration dramatically decreases. The activation temperature of this alloy is in the range of 700 – 900°C. Zr-V-Fe low temperature activable getter (St 707) the surface turns out to be essentially metallic at about 400 – 500°C according to sorption test measurements.[35]

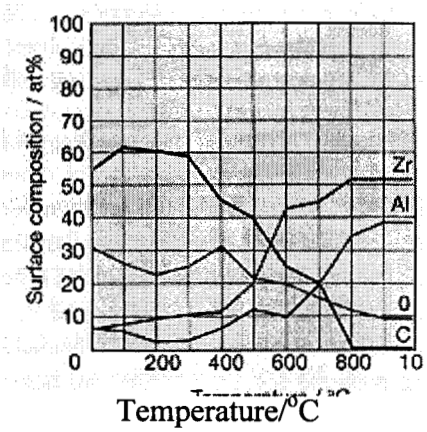


Figure 6: Change of surface composition of a Zr -(16%) Al getter alloy, as a function of temperature. XPS analysis [18, 34]

The activation process is not only related to activation temperature but also to time of activation. However, it is expected to be more strongly dependent on temperature for a diffusion mechanism. [34,36, 37] Lots of tests and studies have been made on getter materials to determine quantitatively the effects of the temperature and time parameters on the degree of activation. Full activation corresponds to the maximum sorption speed obtainable or to the nearly complete removal of the passivating layer. Partial activation can often be perfectly acceptable. Figure 7 shows the various activation degrees obtained with different temperature/time combinations for two typical getters (Zr-V-Fe and Zr-Al).

Another area of important investigations has been related to the interaction of getter materials with H_2 and its isotopes. Both are of interest for UHV applications concerning plasma physics and experimental nuclear fusion where tritium (T_2) is involved. [38-41] Different getter materials exhibit different equilibrium pressures that usually follow Sieverts' law for relatively low concentrations. The sorption of H_2 isotopes, in contrast to other active gases, is reversible and corresponds to the formation of a solid solution. This allows the adoption of sorption and

regeneration cycles by suitably adjusting the operation temperatures as a function of the hydrogen concentration in the getter and the desired working pressure. This can be of great interest in plasma physics experiments. Figure 8 shows examples of equilibrium pressure Sieverts' plots for common getter materials. [42]

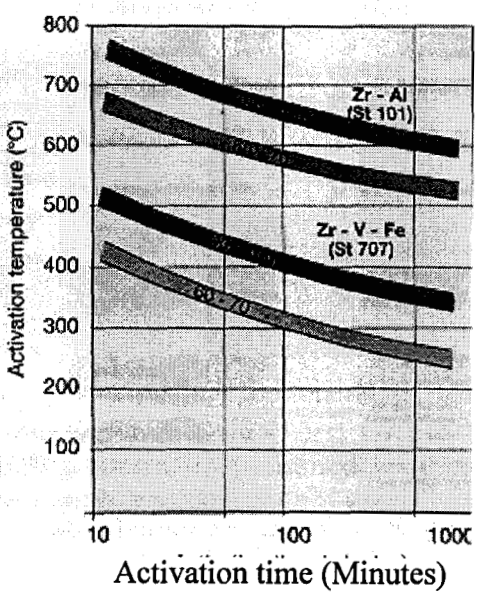


Figure 7: Activation efficiency for different temperature/time combinations (in percent of full activation) [18]

Magnesium Rich Rare Earth Alloy Getters

Xi et al., [43] have studied the application of magnesium rich rare-earth alloys such as La_2Mg_{17} and $La_2Mg_{16}Ni$ for hydrogen absorption. The results show that these alloys have high gettering rate and sorption capacity for hydrogen in a vacuum. Pure magnesium, evaporated as a thin film, had a very small hydrogen sorption capacity. The evaporation of Mg increases the surface area for adsorbing hydrogen so that the hydrogen gettering rate of the evaporated alloy is greater than that of the bulk material.

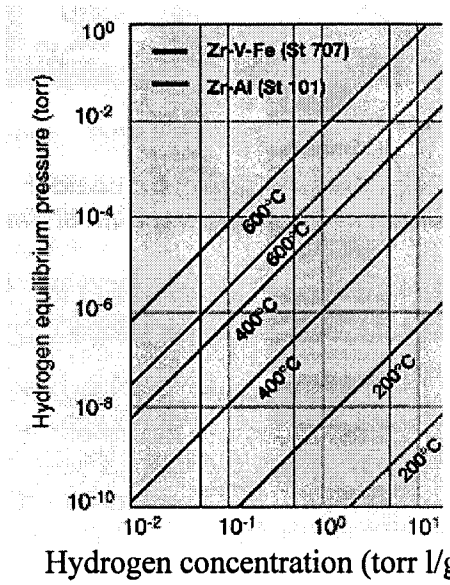


Figure 8: Hydrogen equilibrium pressures of common getter materials (Sievert's plots at different temperatures) [18]

Zirconium-Nickel Selective Hydrogen Getters

Kuus and Martens [44] have shown a substantial gettering rate difference between pure zirconium and a sintered mixture of zirconium and nickel. It was concluded that this is due to the immediate absorption by nickel. Cracks are formed as a result of the hydrogen absorption. The catalytic activity of nickel metal is combined with the effect of crack formation and with a surface enlargement caused by the cracks. Zr-Ni based getters inside high-pressure discharge lamps have major advantage over pure zirconium or zirconium-aluminum alloy getters because Zr-Ni getter can be used at a much lower temperature while at high temperature there is not a big difference.

Metal Hydrides

The term metal hydrides covers a very wide variety of materials, but it may be defined as compounds containing a metal to hydrogen bond. Metal hydrides may be divided into three

groups, depending upon the nature of the metal-hydrogen bond, ionic, covalent, or metallic. Metallic hydrides may be used as getters to remove all traces of hydrogen gas from special vacuum tubes. [45] In thermonuclear fusion technology, including weapon systems, it is important to remove and collect tritium gas from some systems for safety reasons because of the radioactivity of the isotope. A hydrogen getter material should be able to react rapidly with the gas and also have high thermal stability (i.e., low dissociation pressure). Most getter materials that are presently in use are alloys of zirconium (e.g., ZrNi, ZrV_2, $ZrAl_2$) with dissociation pressures ranging from 10^{-5} to less than 10^{-7} MPa at room temperature. Heat transfer is the major factor affecting rates of reaction, poisoning of the hydride by impurities in the hydrogen gas.

Metal hydrides acting as reversible getters have been proposed for interim storage and transport of tritium. The most extensively investigated and widely used getter is uranium. This nuclear material is, however, subject to restrictions and, in addition, its high chemical reactivity makes precautionary safety measures mandatory. Therefore, alternative getter metals and alloys, such as titanium and zirconium/cobalt, are used because they are chemically more stable towards air and water. [46,47]

Organic Hydrogen Getters

The concentration of hydrogen, if allowed to build-up in closed systems, may exceed the normally accepted safety limit of 4% by volume. It may also cause malfunction of electrical components, as well as hydrogen embrittlement of metals. The gettering of hydrogen from closed systems in the past has involved the use of noble metal catalysts in combination with molecular sieves composed primarily of platinum or palladium. A platinum or palladium molecular sieve can, in the presence of oxygen, effectively getter the hydrogen by combining the

hydrogen with oxygen to convert the molecular hydrogen into water. However, these types of getters are not effective for use in closed systems void of oxygen. Both platinum and palladium form stable hydrides in the absence of oxygen and upon subsequent exposure to the atmosphere are extremely pyrophoric. To overcome this difficulty, Courtney and Harrah [48] have developed a solid, unsaturated organic-catalyst combination that can irreversibly getter hydrogen from closed systems via chemical reaction. The unsaturated organic is 1,6-diphenoxy-2,4-hexadiyene or dimerized phenyl propargyle ether (DPPE), and the catalyst is 5%-$CaCO_3$. The compounded getter has been shown to be non-explosive and even when completely hydrogen saturated, non-pyrophoric. Kinetics studies have shown that the getter reacts with sufficient speed to prevent hydrogen build-up in closed systems containing reactive metals and water.

Trujillo and Courtney [49] have determined the reaction rates for the hydrogenation of twenty-seven acetylenic materials, (both compounds and polymers) on calcium carbonate-supported palladium catalysts. Certain of these organic-catalyst formulations are capable of rapidly and quantitatively removing hydrogen from an environment at low hydrogen pressure (100 mm Hg) and ambient temperature. Inductive, steric and catalyst poisoning factors that involved in determining the rate of hydrogen sorption for any given alkyne.

Fabrication

Preparation

All those getter alloys are produced by standard vacuum induction melting and casting methods. The as-cast ingots are reduced to powder which is then used to obtain various gettering

structures such as compressed pills, coated strips, high efficiency getter pumps, and gas purification devices.

Della Porta et al., [50] have identified the alloy of greatest activity, later produced under the trademark of St101. There were several unresolved questions due to the difficulties of preparing samples of the single phases of the Zr-Al system of sufficiently high purity and homogeneity with conventional melting techniques. Barosi [51] has developed a cold crucible melting technique (40 kW), under vacuum (10^{-6} Torr), with high frequency induction heating. After the first melting, the ingots were broken up and remelted twice more to obtain good homogeneity. Using this melting technique over 30 alloys of Zr-Al were produced where Al composition varied from 6 – 37% with a high purity and homogeneity. These alloys were used to examine the Zr-Al system activity towards gases such as CO, N_2 and H_2.

In vacuum devices there are often problems for getter material accommodation due to limited available space. Restricted space may not allow the use of evaporable getters [52], which require large surfaces on which they can be deposited as a film. Nonevaporable getters are usually prepared in limited configurations (by pressing or mechanically coating powders of getter materials), and are not always compatible with small and specific geometries. High porosity thick getters (50 – 250 microns) can be deposited on any metallic substrate including device parts. The porous structure of this getter family ensures a large available surface for the sorption process. As a result of this high porosity, these materials have very good gettering performance even at room temperature. High porosity getters have been successfully applied to substrates such as Ni, nickel-chrome, stainless steel (SS), Ti, and Zr. On each of these substrates the getter coating exhibits strong adhesion due to the production of a diffusion layer at the interface during high-temperature sintering. [53]

In many applications the getters have to work at relatively low temperatures to avoid damaging internal components and their excessive degassing. At these low temperatures the bulk diffusion of the sorbed gases is normally very small (except for H_2) and therefore the active surface must be maximized. This means that large specific surfaces and porous structures have to be used. Existing porous getters normally require activation temperatures of 700 – 900°C but in several cases this is not easy or even possible to achieve. A new generation of porous getters which, maintains a relatively large surface area and porosity are obtained via a sintering process between zirconium and Zr-V-Fe alloy powders. They combine the large specific surface and porous structure necessary for surface sorption of gases at low temperature with the characteristics of high diffusivity, which makes possible efficient sorption possible after activation at relatively low temperature (400 – 500°C).

The complex layered structures used in integrated circuit metallization contain hydrogen. The hydrogen is introduced both as a contaminant during deposition, and as a component of the forming gas (90% N_2 + 10% H_2) which is commonly used as an ambient during annealing. Gettering of hydrogen by titanium layers has been proposed as the explanation for the observation that the annealing of electron-beam-induced radiation defects in MOS devices is retarded by the presence of a Ti layer close to the devices. [54]

St172 porous getter is composed of St707 and Zr powder in about the same proportion. The mixture is sintered in a high vacuum high temperature furnace. The getter mass can be prepared in different shapes with or without an embedded heater. An embedded heater can be used to activate the getter or maintain it at its operating temperature. Porosity is obtained by adding Zr-V-Fe, to the Zr powder along with, particles of materials such as graphite that are inert for the gases to be sorbed.

Getter Configurations

Several different getter configurations are available from SAES Getters Inc., to optimize the performance of getters, for specific application. Typical getter configurations available are compressed pills, coated strips, cartridges, and modules.

Outgassing and Getter Dimensioning

Outgassing data (rate versus time) is necessary to assess the amount of getter material needed to capture the gas released during device operation. Such data can be obtained through a specific outgassing test [55, 56]

To calculate the total quantity of gas outgassed over a time period (t) measured in seconds, it is customary to assume a time dependence of the outgassing rate (q) of the type:

$q = q_o t^{-\nu}$

where the time factor (ν) is nominally estimated to be equivalent to 1 for gases such as carbon monoxide (CO) or nitrogen which are desorbed from the surface of a material. The time factor is estimated to be equivalent to 0.5 for gases such as hydrogen, which desorb by diffusion from the bulk of a material.

By integrating the measured outgassing rates from typical packages it can be seen that the vacuum service life will be quite short if the outgassed species are not trapped in some manner.

The total gas load $(Q) = q_o (t^{1-\nu}-1)/1-\nu$

The reality for vacuum service life in the package is very different than the optimistic perspective that something so small would hold a vacuum for the needed lifetime of many years. In vacuum maintenance terms, the ratio of surface areas outgassing into the volume is very large

in comparison to the volume when compared to traditional large static systems such as IR Dewars and vacuum bottles.

An additional parameter is that part of the getter capacity, which is used up during baking. The getter quantity has to be suitably dimensioned in order to maintain a sufficient gettering capacity to cope with the outgassing load during lifetime.

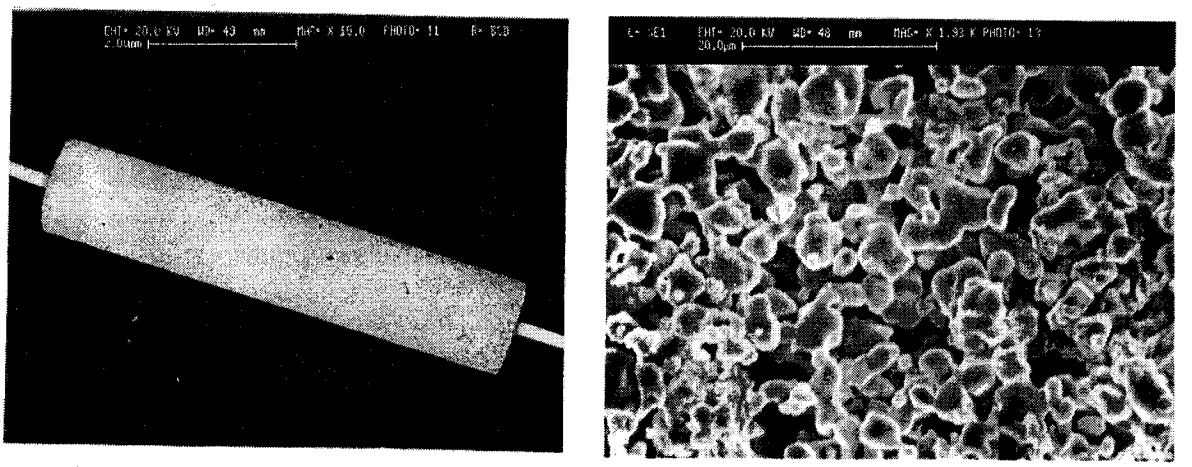
Characterization

An i*n situ* getter has a limited life since they require periodic reactivation because of surface saturation the gettering materials. Secondary ion mass spectroscopy (SIMS) was used to characterize the interaction of active gases with the getter material during reactivation. The data show the migration of active elements such as carbon and oxygen into the bulk of the getter materials as temperature increases.

Watanabe et al., [3] have characterized the activation process of nonevaporable getters and absorption/desorption of hydrogen isotopes by x-ray photoelectron spectroscopy (XPS), secondary ion mass spectroscopy (SIMS) and thermal desorption spectroscopy (TDS). Alloying of nickel to zirconium gave rise to considerable modification of the getter properties including activation temperature, activation energy for hydrogen absorption, heat of absorption, the selective pumping property and the kinetic isotope effect. The activation temperature of Zr-alloy getters varies with the alloying element and the composition. This process is controlled by the balance of the diffusivity of the surface contaminants and the stability of the oxides and carbides in the bulk. The mechanisms of the adsorption and desorption process, however, do not change with alloying. It is expected that a suitable choice of the alloying element and composition will lead to improvement in the activation temperature, absorption rate, equilibrium pressure,

selective pumping property, resistivity against impurity gases and extent of the isotope effects. The technique used for measuring the thermal outgassing was a known dynamic conductance technique using a quadrupole mass spectrometer gas analyzer.[57]

Scanning electron microscopy (Figure 9) may be used to investigate the porosity of the porous getter.[58] Accessibility to the gas to be sorbed is also proven by the relatively high surface area measured by the usual BET technique using Kr as the test gas. The values obtained



(A) (B)

Figure 9: Scanning electron micrographs of the St172 getter obtained from SAES Getters Inc.[58]

are around 1000 cm^2/g, which corresponds to about 1.7×10^{17} adsorbed Kr atoms per getter (0.36 gr). This large surface area should allow good sorption characteristics at room temperature, while the presence of the Zr-V-Fe alloy appears to be responsible for the good gettering efficiency after activation at relatively low temperatures.[59].

Mechanical coherence and stability of the present porous getters have also been verified by ultrasonic treatment in Freon, which shows no instability problems.

Gong-Le and Xian-de [60] have studied the mechanism of absorption and desorption of hydrogen for the Zr/Al getter alloy based on kinetic measurements of thermal desorption by means of the flash desorption technique.

Composition of some of the getters [61]

- St101: 84% Zr + 16% Al
- St707: 70% Zr + 24.6% V + 5.4% Fe
- St121: 7 parts St101 and 3 parts St707
- St122: 3 parts St101 and 7 parts St707
- St122: Zr – V – Fe – Ti
- St198: 76.5% Zr + 23.5% Fe

Sorption Performance

The room temperature sorption characteristics of the porous coatings have been determined for H_2 and CO. The pumping speed was measured as a function of the sorbed quantity by means of the dynamic method.[10]. Getter activation was carried out in the temperature range of 300 – 500°C. Sorption tests were performed at room temperature at a constant pressure of 4×10^{-6} mbar.

Tests were conducted to evaluate the sorption performance of the coatings and to determine the influence of the coating thickness on sorption characteristics. The measurements have shown that the getter capacity increases proportionally with the thickness. The behavior of the sorption curves confirms that the getter structures are highly accessible to gases. In the case of CO sorption it is evident that surface saturation is achieved at a sorbed quantity of about 0.7 cc mbar/cm^2 and about 1.4 cc mbar/cm^2 for the 100 μm and 200 μm thick coatings, respectively. The observed increase of capacity proportional with the thickness is in good agreement with the measurements of porosity and apparent density. In fact, controlling the porosity of the film

independenty of the total film thickness allows complete exploitation of the increased quantity of getter material. The presence of the Zr-V-Fe alloys as a component of St122 mixture also improves gettering efficiencies after activation at relatively low temperatures. Tests performed on St122 coatings have demonstrated that activation of the getter at 500°C for 10 minutes is effective in giving excellent sorption performances. It is possible to reach full activation for the getter under these conditions, as heating at higher temperatures does not significantly improve the getter's activity. It is possible to obtain comparable sorption characteristics of the coatings after heating at temperatures lower than 500°C for several hours. Heating the getter at 400°C for 2 hours allows the achievement of very active surfaces and sorption performance very close to those of the full-activated getter.

During the production process, MEMS devices are subjected to low bake-out temperatures for many hours. It is possible to exploit this bake-out process to obtain a significant activation of the getter even in low temperatures range (300 – 350°C). Getter activation performed at 350°C for 6 hours or at 300°C for 18 hours assures good sorption activity. After heating, coatings show pumping speed and CO capacities similar to those of a full activated getter (about 80 – 90%). The initial pumping speeds of the getters after activation are quite high. This is very useful in maintaining the desired base pressures in the packages, as the initial outgassing rates are quite high as well. Due to processing system conductance limitations, it is often the case that the achievable base pressure in the packages prior to sealing is higher than desired. In such an instance, the high initial getter pumping speeds can be achieved to serve as an *in situ* pumping system to finish removing gas from the package. [62] The presence of an internal active getter during the bake-out process can allow reducing the baking time to achieve a better vacuum at the seal-off of the package.

Nagasaka and Yamashina [63] have reported the gettering rates of hydrogen and deuterium by titanium and zirconium in a temperature range of 500 – 700°C at pressures of 10^{-4} – 10^{-2} Torr. The gettering rates remained constant in the initial period of reaction and later the rate began to decrease according to the increment of hydrogen concentration in the solid, finally becoming zero when the concentration attained equilibrium value. It was also found in their studies that the gettering rate of hydrogen is about two times faster than that of deuterium under the same conditions in both Ti and Zr.

Temperature measurement

The getters may be activated at the chosen temperature by passing current through the embedded heater. The temperature of the getters during activation may be monitored by a thermocouple welded to the central zone of the lateral surface. After activation, the getters may be allowed to cool down to the sorption temperature before starting use as a getter. Activation of the test getters may be performed by induction heating and depending on the temperature monitoring the temperature with an optical pyrometer or a thermocouple.

Activation

St171 nonevaporable getter has been proposed for use in a Defense Satellite Communications System (DSCS) travelling wave tube (TWT) by Lampert et al., [1]. The activation of the getter was accomplished by a solid-state chemical reaction that produces a near-metallic Zr surface. Residual gases given off during the getter activation process are principally hydrogen, H_2O, CO, and CO_2. Getter activity proceeds by forming stable chemical compounds such as ZrO_2, ZH_2, ZrC, etc., by reaction of gases with the clean Zr metal. These changes in the

Zr surface may be monitored by using x-ray photoelectron spectroscopy (XPS) and line of sight residual gas analysis (LOSRGA).

Activation of the nonevaporable getter (St 171) may be accomplished in several ways.

1. the getter may be slowly heated in steps of heater current to attain 900°C.
2. the getter may be flashed to 900°C, i.e., the heater current may be immediately set up to the 900°C equivalent.
3. Another approach is to closely simulate the treatment the St171 nonevaporable getter would receive if installed in a TWT during the preprocessing and processing steps.

Activation is performed using a suitable combination of temperature and time to remove the layer of surface oxides, nitrides, and carbides, by diffusion into the bulk, thus providing a clean metallic surface ready to react with the impinging gaseous molecules. Depending on the diffusion constants of the getter materials, for species such as H_2, CO, CO_2, H_2O, etc., one can have a more or less effective cleaning of the surface during the activation process. The diffusion rate increases upon raising the temperature. The activation of the getter must be done when it is exposed for the first time or whenever exposed to air.

Many high sensitivity microelectromechanical systems (MEMS) need to operate in a hermetically sealed vacuum electronic package to realize their full performance. This vacuum is destroyed by out-gassing of various species such as water vapor, hydrogen, carbon monoxide, nitrogen, oxygen, and carbon dioxide from the package surfaces and microleaking or permeation through the package body. The loss of vacuum is particularly serious if organic materials are used in MEMS packaging. A getter material is needed to eliminate this problem and achieve successful MEMS device operation for long duration space applications.

Solid-state getters may be either planar or three-dimensional and exhibit good mechanical strength. For use in MEMS applications they must be particle free under the stringent operational conditions in space and on the ground, and they should have a high active surface area that can easily be activated at low temperatures. This minimizes problems such as high ambient temperature that may be detrimental to MEMS devices during activation. High porosity combined with a large active surface area will assure excellent sorption performance at room temperature. There should not be any loss of getter particles before, during, or after activation of the getter in a packaged MEMS device as this may cause failure of the MEMS device. It is critical to maintain the getter's mechanical structure during shocks and vibrations at the time of spacecraft launch and during operation of the MEMS device. The presence of an activated getter material inside the MEMS package will allow achievement of a better vacuum in the hermetically sealed vacuum package. The presence of a getter material inside a MEMS package is needed to avoid a pressure increase above the operational limit of the MEMS device. Sorption of outgassed species by getters permits a greater anticipated lifetime for MEMS devices in hermetically sealed packages. A procedure to activate the getter inside a MEMS package for possible space applications is proposed.

Activation of the nonevaporable getter may be achieved using the following procedure that may be appropriately modified for a particular MEMS device:

- The activation parameters are temperature, pressure, time, and method of heating.
- Getters should be handled only with clean tools, rubber or plastic gloves and never with bare hands.
- Getters may be ultrasonically agitated in high purity isopropyl alcohol (IPA) for a few seconds and dried in an oven.

- For long-term storage, a clean, dry ambient is desirable. Getter may be stored in a phosphorus pentoxide or a silica gel air desiccator, a vacuum desiccator, or under a dry nitrogen atmosphere.
- Weld the getter appropriately in the MEMS packaging container.
- Pump until the pressure in the MEMS packaging chamber is less than 1×10^{-6} Torr or 1×10^{-4} Pascals.
- Activate the getter by heating to the activation temperature of the getter material. The activation temperature and time are functions of the getter material and the activation technique.
- Activation of the getters may be accomplished by resistance heating.
- The heating rate of the getter should be controlled to avoid excessively high system pressure due to outgassing. The time required to attain activation temperature should not be included in activation time at the activation temperature.
- Monitor the maximum pressure during activation using a pressure gauge; this will give the relative value of the gas content of the getter.
- Allow the getter to cool to its test temperature while pumping down. Pinch off will be performed carefully at the final stage.

For bulk getters, the rate-determing step in the sorption process is diffusion from the surface to the bulk. Bulk diffusion of the active gases occurs relatively slowly at room temperature, so the gases are mainly adsorbed onto the surface of the getter. In the case of H_2, however, the diffusion rate is already high at room temperature, and H_2 continuously diffuses into the bulk of the getter. The initial measured pumping speeds for CO, N_2, H_2O, and H_2 are about 800, 450, 1100, and 2100 l/sec, respectively. As the exposed surface of the getter becomes saturated, the

pumping speed of the active gases falls off, while H_2 continues to pump at the same rate. Pumping performance must be periodically restored by a reactivation step (heating under vacuum), diffusing the adsorbed gases into the bulk of the getter material.

Effect of Radiation

Recently, NEGs have come into use in atomic fusion experiments and in particle accelerators.[64] Pumping speed of the NEG strip is high in comparison with its size and some NEG strips are not influenced by magnetic fields. The NEG is capable of pumping all active gases such as CO, O_2 and N_2. It forms a solid solution with H_2 and its isotopes. H_2 and its isotopes can be pumped reversibly according to Sieverts law.[65] However, the NEG is almost incapable of pumping non-active gases such as methane.

Temperature and pressure changes have occurred under direct synchrotron radiation (SR) exposure of the NEG strip. As soon as the NEG is exposed to SR the pressure rises more than ten times, but it immediately begins to decrease. The initial pressure rise is assumed to be due to the desorption of contamination on the first layer of getter material. The residual gas spectrum during exposure of the NEG strip to synchrotron radiation has a large hydrogen peak, which is attributed to the release of sorbed hydrogen due to direct exposure. When the NEG is allowed to be charged up positively by proton incidence, there is possibility of enhanced desorption by electron stimulated desorption (ESD).[66]

Applications

The attributes of NEG getter pumps make them useful in a variety of UHV applications, which include semiconductor-processing equipment, both current and future. Their high

hydrogen pumping speed can be key in achieving lower base pressure in UHV processing equipment such as molecular beam epitaxy (MBE) and ion implantation systems, and in future x-ray synchrotron radation sources for x-ray lithography. The absence of vibration and magnetic fields makes them especially useful in applications involving electron columns, such as e-beam lithography processing equipment and electron microscope based analytical equipment. Low vibration levels also reduce particle contamination. The pumping selectivity of NEG pumps, especially their zero effective pumping speed for argon, can be advantageous in sputtering systems and low pressure chemical vapor deposition (LPCVD) equipment. It can also be used for *in situ* purification of process gases, which can result in purer films.[67]

The use of getters has expanded in applications ranging from small and simple, to big and complex devices, from industrial mass-produced devices, to special experimental machines with different demands in terms of vacuum degree and geometrical constraints.

A recent notable application is in the getter pump developed for the CERN high-energy particle accelerator in Geneva.[68] The accelerator, a 27 km (kilometer) long aluminum, vacuum chamber, contains a 20 km strip of Zr-Al getter material which in conjunction with several small sputter ion pumps, has produced vacuum levels below 10^{-12} Torr. The getter pump is regenerable by heating and is expected to require regeneration between ten and twenty times over a twenty-year period.

In situ pumping with high efficiency gettering panels is utilized in the vacuum vessel of plasma machines for experiments on nuclear fusion. Getter pumps are utilized for the recovery and handling of hydrogen isotopes and exploiting the reversible character of the sorption of these gases. They are used in particle accelerators, where the vacuum requirements are very stringent (pressure of 10^{-7} Pa or even better).

The getter materials and configuration developed are generally the result of a specific study of the application considered, also into account the nature of the gas and the gas load expected to be sorbed, often based on extensive outgassing and permeation rate measurements. For many devices, gas analyses of prototypes are often performed to finalize the getter type and use conditions.

High and Ultra-High Vacuum

A new generation of pumps has been developed using steel ribbons coated with the St 101 (Zr – Al) alloy. Figure 10 shows a cartridge type getter pump with control unit.[18] The getter material is heated for activation by an internal heater. In getter modules, the ratio between spacing and depth of the individual fins was optimized to give the external surface of the device a high sticking probability. These modules can be assembled together in panels with thousands of liters/sec of sorption speed. [69,70] Figure 11 shows a getter panel.[18] The activation and operation temperature in this case is obtained by direct passage of current through the getter strip. Due to the extremely low equilibrium pressure of hydrogen, these modules represent an ideal combination with an ion-pump for which the pumping speed for hydrogen rapidly decreases below 10^{-7} Torr. [71,72] A simple ribbon coated with the St101 alloy has been in the large electron positron collider operating in Geneva since 1989, where 80% of its 27km length is pumped by non-evaporable getters. Sputter-ion pumps are just pumping the residual argon or the possible methane. In special vacuum experiments in a suitable chamber with St 707 getter, the CERN's scientists have obtained pressure in the low 10^{-13} mbar range.

Getter modules or getter pumps are now used in most large accelerators, particularly in the synchrotron light machines where a very high speed and capacity is needed to take care of

the large outgassing due to the impinging Synchrotron radiation. [73] Big gettering panels have also been tested inside the Tokamak chambers in neutral particle injectors. [74]

Figure 10: Cartridge type getter pump with control unit
(SORB-AC Cartridge pump) [18]

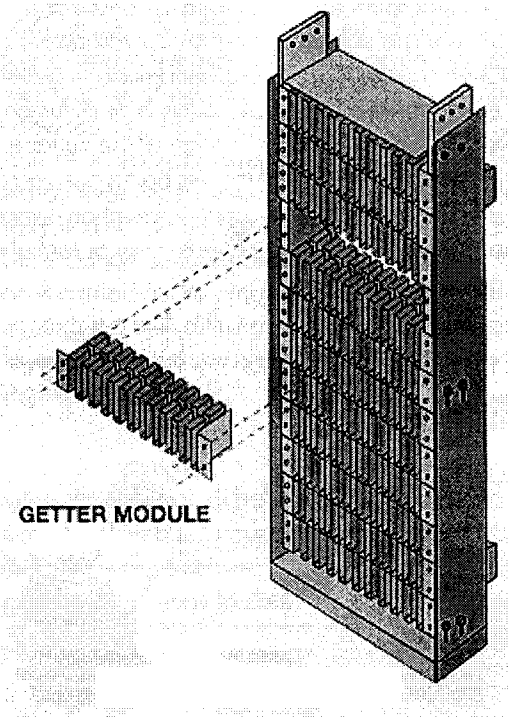


Figure 11: Getter panel [18]

Medium and Low Vacuum

Getters in strip and pill shape have been used and studied for industrial solar collectors, dewars, vacuum thermally insulated panels, etc. where the purpose of vacuum is just for thermal insulation. The nonevaporable getters pills were used in a stainless steel thermos to keep good vacuum. The use of getters in lamps is now also well established. [75,76] Both barium getters and nonevaporable getters are used depending on the lamp types. Barium getters are used in high-pressure sodium lamps. The barium should assure a high vacuum within the jacket, which surrounds the arc tube for getting an efficient thermal insulation. Traces of moisture are always present within the jacket at the tip off and some H_2 is released from the glass during lamp operation. Residual hydrogen within the discharge affects negatively the ignition of the lamp and considerably shortens the life. The combination of barium and nonevaporable getters has been adopted to solve this problem.

Gas Purification

Getter materials do not sorb rare gases and therefore they have been used to purify helium for analytical purposes and decrease the level of impurities in this gas down to the partial pressure range of 10 mbar (1 ppm) or less. [77] Figure 12 shows a getter based gas purifier.[18] Argon and helium (rare gases), nitrogen and hydrogen are now purified with getter-based materials.

Gettering has proven to have a significant impact on vacuum technology. Getters cover the role of maintaining and improving vacuum, allowing reaching pressures in the pico-torr region, with an ever-increasing importance. They have also found new applications, still linked to vacuum technology concepts, such as gas purification.

Business Area	Key Market/Applications
Display devices	Color & monochrome cathode ray tubes.
Light sources & Vacuum Insulation	Lamps: High intensity discharge Fluorescent, cold cathode insulated pipes, solar collectors, vacuum bottles, and vacuum insulation
Electronic devices & Flat Displays	Infrared detectors, night vision tubes, x-ray image intensifiers, pressure transducers, vacuum fluorescent displays, x-ray tubes, photomultipliers micromachines, vacuum interrupters, field emission displays, plasma displays.
Vacuum Insulation Panels (VIP) for Vacuum systems	VIP's for refrigeration industry System processing pumps, physics projects, super conductors, *in situ* pumping
Pure gas Technologies	Semiconductor industry/small, large, area and *in situ* purifiers, analytical instruments, analytical and customer service.
Microelectro-Mechanical Systems (MEMS)	Microgyro, Pressure sensors, etc.

Moisture Getter

Modern packages used for electronic devices, such as integrated microcircuits, hybrid microcircuits and surface mounted devices that function as transistors, diodes, resistors, capacitors, transducers, and such, are designed to protect the circuits and sensitive components mechanically and environmentally, and to provide a functional interface to macroscopic interface, such as a printed circuit board. Such packages are usually constructed from metal or ceramic and hermetically sealed in a moisture free atmosphere to minimize the possibility of

corrosion during usage. Hermetic seals may be formed by soldering, welding, brazing, and sealing through glass.

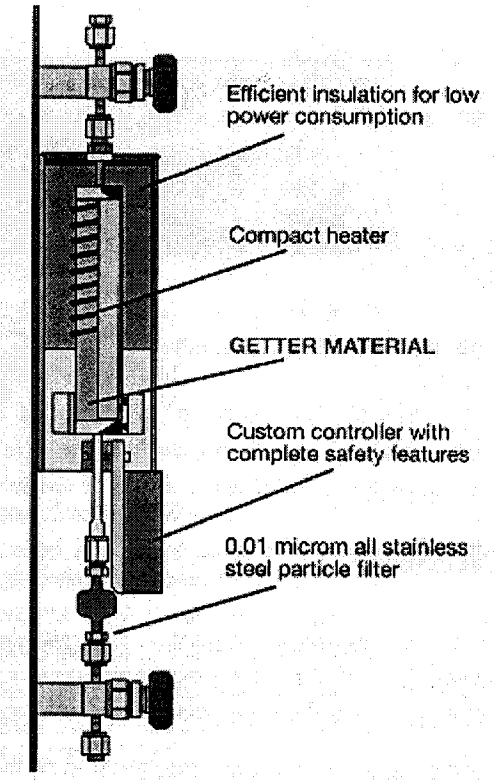


Figure12: Getter based gas purifier [18]

There has been continuing concern regarding the presence of water vapor in hermetically sealed semiconductor devices. Hermetic microelectronic devices used in military, space, medical and other applications requiring high reliability have an upper requirement limit of 5,000 ppm by volume of water vapor content at the time of fabrication. Package leak rate is limited to 10^{-8} atm-cc/sec maximum to prevent leakage of a significant amount of moist ambient air into the packaging during the device's useful life. In spite of extreme precaution, it is difficult to manufacture hermetic packaging for microelectronic devices with low water vapor content and to maintain it during its useful life. There are various channels water vapor finds its way to the inside of the enclosure:

1. Leakage through the various seals, which are not perfect and can be easily damaged.
2. Epoxies outgassing inside the packaging
3. The packaging material itself outgasses a certain amount of moisture.
4. The sealing atmosphere may be contaminated with moisture if not prepared with extreme care.

Andrew Shores [78] has reported experimental data on the enormous moisture gettering capacity of the coating (Staydry 1000 of Staystick Inc.,) at various temperatures at equilibrium with a wide range of water vapor concentrations. Results have shown that the atmosphere in the cavity stays dry 2 to 3 orders of magnitude longer than without the getter. This allows leak rates to be as high as $10^{-5} - 10^{-6}$ atom.cm^3/sec while the water vapor concentration stays below 5000 ppm for 20 years or longer. Data also suggests the feasibility of eliminating packaging bakeout, sealing with ambient gases and the extended use of plastic sealers.

Getters for Tritium

Kherani and Shmayda [79] have shown that uranium and titanium storage beds have good operating characteristics for application as temporary and permanent tritium storage media. Uranium, however, has certain drawbacks such as it is pyrophoric material and chemical toxicity.

Space Science Exploration

Getters have shown their utility in space exploration. The particular experiments in which they seem most suited concern the analysis of planetary atmosphere by means of mass spectrometry. The Pioneer Venus space mission of NASA [80] launched on May 20th 1978 consisted of two spacecrafts, the orbiter and a multiprobe. The multiprobe carried a mass

spectrometer to investigate the atmospheric composition and the isotope ratio of rare gases, from the altitude of 76 km down to the planet's surface.

The Galileo mass spectrometer also makes use of nonevaporable getters and a sputter ion pump, since they can be simply adapted to space flight.[81] There are no moving parts and they have very low power requirements. It seems important at this point to emphasize how the sputter ion pump and NEG compliment each other. One undertakes the function that the other cannot fulfill.

SAES Getters were part of the Cassini Ion and Neutral Mass Spectrometer (INMS) and Huygens probe gas chromatograph mass spectrometer (GCMS). Cassini was launched in October 1997 and is scheduled to arrive in July 2004 to explore the atmosphere of Saturn and its rings and moons. The Huygens probe is deployed to the surface of Titan, the largest moon to study the atmospheric chemistry and surface topography. Nonevaporable getters were also used in the Brilliant Eyes Ten-Kelvin Sorption Cryocooler Experiment (BETSCE) conducted by NASA.

Microelectromechanical Systems (MEMS)

Microelectromechanical systems (MEMS) combine electronic and mechanical components. MEMS often contain with moving parts such as tiny membranes, valves, cantilever beams, and bridges, which can be mass-produced, on silicon wafers. They can detect minor temperature changes, measure acceleration, or open and close a valve that's is smaller than a pinpoint. Microsensors and microactuators interface the external non-electronic environment with a microelectronics integrated circuit.

The most developed commercial applications of MEMS are related to the automotve market. An example is the acceleration sensors used in automotive airbag triggering systems. Further commercialization in this market will include applications such as side-impact airbag activation, low-G accelerometers for anti-lock braking systems, position sensing for inertial navigation, electromechanical switches, resonance devices, motion detectors, temperature sensors, dynamic control systems, and advanced infrared devices. Packaging of MEMS is important to maintain stability and reduce the size and cost of fabrication. Often, shrinking the sensor size can create a device 10 times less expensive or having improved performance. Research relating to new applications demonstrates the need for a vacuum or controlled environment. Examples of such applications include capacitive accelerometers, resonators, microgyroscopes, thermal radiation sensors, and microbolometers. Even if the micromachine is bonded in vacuum, good vacuum may not be maintained due to outgassing produced during bonding process. A nonevaporable getter placed inside the cavity and activated during the bonding process sorbs the gases produced and solve this problem.

The getter must have high a large active surface in order to deal with the large outgassing load expected in relation to the working volume of the package.

The getter must become active at relatively low temperature (300 - 500°C) and exhibit high sorption performance at room temperature and good mechanical strength.

Several techniques were studied for the fabrication of such a planar getter. The use of screen printing technique permits the attainment of all the requirements for a MEMs package getter.

A special screen printing process has been developed for the preparation of porous getter coatings. A suitable getter paste (Zr 70% – V 24.6% – Fe 5.4%, St 122), wherein the St122

getter mixture is combined with a special binder, is deposited on metallic substrates (nichrome and titanium) by means of printing techniques. Such techniques allow a precise control of the thickness of getter coatings, which typically ranges from 50 - 250 μm. The printing process is typically performed through metal masks on chem-milled substrates. This makes it possible to prepare getter layers in almost any desired pattern and in many preformed shapes to accommodate the requirements of different device and package geometries. Geometries can vary from simple squares, rectangles, or circles to complicated shapes with coated and uncoated areas. After drying, getter coatings are sintered at high temperatures, exceeding 800°C, in a high vacuum furnace. The thermal treatment consolidates the coating and the adhesion of the getter powder to the substrate is obtained due to the formation of a diffusion layer at the interface.

The screen-printed HPTF (High Porosity Thick Film) getters have maximized surface to volume ratio and have very high porosity to favor the accessibility of the gases to be pumped to the internal getter areas. High porosity combined with a large specific, or active, surface area should assure good sorption performance at room temperature. The realized planar getters present a porosity of about 60 – 65% and specific surface area, expressed in terms of ratio of active surface area over geometrical area, of about 20 – 30 cm^2/cm^2 for typical 100 μm thick coatings. The specific load of the coating is of 2.0±0.3 g/cm^3. These getters exhibit porosity and a specific load independent of the thickness of the coatings.

The sintered porous matrix, based on an active metal such as titanium, incorporating the Zr-V-Fe getter alloy and applied onto a metal substrate, gives the getter structure attractive mechanical characteristics to withstand shocks and vibrations without any loss of particles and to meet the special working conditions of the devices.

Leak Testing [82]

The practice of leak testing of hermetic packages has been described in refs.83,84. The spectrum of leak rates of interest is broadly divided into two regimes. Leaks in the range 10^{-1} to 10^{-4} are termed gross leaks, where as those in the range 10^{-5} to 10^{-12} are termed fine leaks.

Gross-leak testing:

Gross leaks are tested by using a fluorocarbon liquid. The package to be tested is first subjected to a vacuum-evacuation step ($\geq$ 5 Torr, 1-h dwell) to facilitate easy entry of the test liquid if a leak exists. Following the evacuation soak, the package is immersed in 3M fluorocarbon liquid (FC-84) without breaking the vacuum. It is left to soak in it for 30 min. The filled package is then immersed in another fluorocarbon liquid (FC-40) having a higher boiling point of FC-84. Any FC-84 that had leaked into the package during fill phase will now expand in volume and appear as bubbles observable through a magnifying window in the FC-40 liquid container.

Negative-ion detection method

This technique uses a gas analyzer specially tuned to detect FC-84 molecules from packages filled with the FC-84. The filled packages are heated in a metal chamber maintained at 125°C and attached to the gas analyzer.

Fine-leak testing

The common method of testing for fine leaks uses helium gas as the testing medium. First, the package is subjected to high-pressure helium gas in a pressure chamber or bomb.

Typically the pressure used is 60 psig and the dwell time is 2 h. After the pressurization step, the packages are introduced, either singly or in small batches, into a vacuum chamber attached to a helium mass spectrometer. If any helium evolution is detected during the evacuation step, the whole batch is rejected.

Radioactive tracer gas

This technique may be used for fine leak tests and also serves to test for gross leaks. The package is subjected to pressurization in dry nitrogen gas into, which is mixed about 1% Kr-85 radioactive tracer gas. After the pressurization, the package enters a scintillation counter, which detects the gamma emission that accompanies the beta decay of Kr-85. Because the gamma rays can easily penetrate the package walls, the scintillation count directly yields a measure of the Kr-85 concentration within the package.

Helium leak rates measured in this test could be orders of magnitude larger than helium leak rates that would occur under use conditions – 1 atm pressure in air [95]. This difference is due to the predominant contribution of viscous flow of the gas during bombing and subsequent testing in vacuum and to the absence of viscous flow under use conditions. Further, the penetration of moisture into the package could be significantly slower than for He because of the tendency of the former to absorb on the walls of the leak path.

Advantages and Disadvantages

Non-evaporable getter pumps have several features, which distinguish them from other UHV compatible pumps such as cryopumps and turbomolecular pumps. These include low power requirements, small size, lightweight, ease of use and freedom from pump oil, vibration,

or noise. Since they work on the principle of chemical sorption and cleaning of the getter surface by thermal diffusion into the bulk, they have no moving parts. This results in high reliability and low maintenance, the only replaceable components being the getter cartridge and in some cases the heater.

Gettering materials provide passive, chemically based pumping. Evaporable getters have only limited application (primarily in vacuum tubes) because they function only in sealed environments at high activation temperatures of around 1200°C.

Nonevaporable or bulk getters are based on sintered zirconium alloy. They provide gettering at temperatures of 550°C, which is lower compatible with semiconductor fabrication processes and materials. The chemical pump can not only provide lower vacuum pressures than traditional pumps used alone, it can also speed throughput by reducing downtime.

Getters are extensively used in most of the sealed-off vacuum devices, such as electron tubes, where they act as the only pump. They have also been adopted in the more advanced application of plasma physics and particle accelerators as *in situ* main pumps in unsealed vacuum systems. [86] Non-evaporable getters present better flexibility, fewer constraints in use and, in some configurations, higher sorption performance than evaporable getters. [87] The new ternary alloy presents the remarkable advantage of becoming active at lower temperatures (300 – 500°C) than those usually required by the traditional NEG (700 – 900°C). The disadvantage of the NEGs is reactivation of the getter is required whenever it has been exposed to air.

NEG pumps using commercially available getters such as St101 (konstantan strip onto which a bonded mixture of Zr 84% - Al 16% manufactured by SAES Getters Inc.) have already been used successfully in the UHV system of devices having abnormally high gas loads with very little space for pumping. However, in reliably applying the high electrical power required

for activation of the getter modules, technical difficulties have been encountered which make the operation possible, but delicate. [88]

Strict choice of materials, heat treatments, cleaning procedure and an elaborate bakeout system have reliably reduced the overall outgassing rates to the 10^{-13} mbar l sec^{-1} cm^{-2} range with negligible leak occurrence. For electron coolers and gas jet targets, which, in operation, provide large gas loads and virtually no space for pumping, the task of providing very low pressures becomes extremely difficult. [89] High gas throughputs can be obtained with getters such as sublimated titanium or nonevaporable getters at the expense of continuous operation at elevated temperature using large amounts of electrical power. The heat generated may be difficult to remove in the space available and it is usually detrimental for the required low base pressure.

After reaching a certain concentration limits, Getter materials, sorbing H_2, exhibit embrittlement phenomena. The St121 and St122 getter materials have a very good resistance to high H_2 loads and to thermal fatigue. The embrittlement limit for St121 coating has been found to be as high as 90 l.torr/g.

Tests were performed to determine whether the presence of the pump in the chamber could affect particle contamination or various thin film characteristics (including sheet resistance, sheet resistance uniformity, stress, and reflectivity). No performance variations were found for any of those metrics when compared to films deposited without the nonevaporable getter. [90]

The deposited films were analyzed using total reflection X-ray fluorescence (XRTF) and secondary ion mass spectroscopy (SIMS) techniques to determine whether a chemical pump such a nonevaporable getter affects the purity of deposited films. Aluminum films deposited at the beginning and end of the getter life (before reactivation) were analyzed for atomic composition.

For both films, the atomic concentrations of the elements making up the getter materials were found to be at the level of equipment detection, indicating that the getter is not a source of contamination.[90]

Applied Materials Inc. has realized reduced downtime, which effectively improves the productivity. For a typical 0.5 μm, four-level logic processes, a 28% reduction in maintenance downtime was achieved, translating to a greater than 11% increase in wafer starts /week and more than a 7% reduction in baseline cost/wafer.

NEG pumps are compact and light in weight. A getter wafer module type pump with 1250 l/s H_2 pumping speed weighs only about a kilogram; a cryopump with the equivalent H_2 pumping speed would weigh about 22 kg, a turbomolecular pump about 45 kg, and ion pump about 450 kg. NEG pumps can be mounted at any angle. Though cartridge pumps have optional water cooling jackets, cooling is not generally required. The pumps can also be supplied in nonmagnetic versions. NEG cartridge pumps are configured with standard UHV-compatible flange mounts or as modules for *in situ* pumping applications.

One disadvantage of NEG pumps is that since they work on the principle of chemical sorption they have finite capacities. When their cartridges are saturated they must be replaced. In typical UHV setups, however, getter lifetimes are in excess of one year.

NEGs can be used in vacuum devices not only to take care of outgassing but also to act as an *in situ* pump during processing. This can influence the process characteristics such as decreasing outgassing rates during lifetime and reducing process time.

The Zr-V-Fe alloy is an efficient getter, which can sorb all active gases usually after activation at relatively low temperatures (300 – 500°C). In many applications such a temperature is usually reached during the baking process and therefore two aims can be reached at the same

time: degassing of the tube, thus reaching low outgassing rates after processing and activating the getter as an internal mini-pump or *in situ* pump both for first improving the processing efficiency and then to cope with the outgassing load during the life of the sealed device.

The getters used must have maximized surface to volume ratios and high porosity to favor accessibility of the gas to be pumped to the internal areas of the getter. In some applications it is advisable not to perform the activation of the getter materials at high temperature (e.g., 700 – 900°C), as occurs with usual getters, such as Ti and Zr-graphite types known as St171 getters. To avoid permanent damage of some internal vacuum device components or simply to save power, the tolerable activation temperatures may be in the range of 400 – 500°C. In order to meet both requirements, a porous getter has been developed, which can be activated at relatively low temperatures, while still maintaining a good sorption behavior.

Nakamura and Hoshi [91] have demonstrated reversible getter pumps using ZrNi deuteride (hydride) as a getter material. This reversible getter pump system has the advantage of conventional methods, e.g., it can completely recover hydrogen isotopes, it can work at low temperatures (room temperature to 200°C), it is fast, and it consumes little energy. The disadvantage of this method is that ZrNi dueteride (hydride) disintegrates into fine powder and care must be taken not to lose the powder into the vacuum. It is possible to contain getter particles by enclosing the getter materials within a porous container.

Turbomolecular pumps are widely used in high vacuum and ultra high vacuum applications. However in UHV conditions, where hydrogen is usually the dominant species, the performance of turbomolecular pumps tends to decay due to their poor pumping ability or compression ratio for this gas. Pozzo et al., [92] have shown that the addition of a NEG pump in a system equipped with a turbomolecular pump is synergistically effective in reducing the

ultimate pressure by one or two orders of magnitude. The improvement is mainly due to the increase of the pumping speed for hydrogen. They have shown that the final pressure can be reduced by two orders of magnitude, from 10^{-9} to 10^{-11} mbar in the test chamber, compared with the turbomolecular pump alone when an NEG pump is used in conjunction with the turbomolecular pump.

Pressures in the range of 10^{-13} and low 10^{-14} Torr range have been reached by means of nonevaporable getter pumping. The lowest measured pressures are probably due to measuring limitations rather than to the pumps or materials employed.[93] The sintered getter bodies increase surface area and capacity, requiring less frequent reactivation and facilitating greater overall life of the pump.

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Glossary

Alloy:	A solid-state solution or compound formation of two or more metals. Alternatively, a combination of metals resulting in a phase containing some of each constituent.
Assembly:	A hybrid circuit, which includes discrete or integrated components that have been, attached to the next level of package, usually a card.
Avogadro's law:	Equal volumes of all ideal gases measured at the same temperature and pressure contain the same number of molecules.
Avogadro's number:	The number of molecules in a gram molecule of gas or any substance is a universal constant and is 6.023×10^{23}.
Binder:	Materials (organic or inorganic) added to thick-film compositions and to unfired substrate materials to give sufficient strength temporarily for prefire handling.
Bondability:	Those surface characteristics and conditions of cleanliness of a bonding area which must exist in order to provide a capability for successfully bonding an interconnection material by one of several methods, such as ultrasonic or thermocompression wire bonding.
Braze:	A joint formed between two different materials by formation of liquid at the interface.

Brazing:	Joining of metals by melting a non-ferrous, filler brazing metal, such as eutectic gold-tin alloy, having a melting point lower than that of the base metals. Also known as hard soldering.
Ceramic:	Inorganic, nonmetallic material, such as alumina, beryllia, or glass-ceramic, whose final characteristics are produced by subjection to high temperatures. Often used in forming ceramic-substrates for packaging semiconductors.
Chip:	The uncased and normally leadless form of an electronic component part, passive or active, discrete or integrated. Also referred to as a die.
Chip Carrier:	Special types of enclosure or package to house a semiconductor device. It has electrical terminations around its perimeter, or solderpads on its underside, rather than an extended lead frame or plug-in pins.
Compression seal:	A seal between an electronic package and its leads. The seal is formed as the heated metal, when cooled, shrinks around the glass insulator, thereby forming a tight compression joint.
Conduction:	Thermal transmission of heat energy from a hotter region to a cooler region in a conducting medium.
Conductive adhesive:	An adhesive material, usually epoxy, that has metal powder added to increase electrical conductivity. Usual conductor added is silver.

Conductive epoxy: An epoxy material (polymer resin) that has been made conductive by the addition of a metal powder, usually gold or silver. Best common conductors are silver, copper, and gold.

Convection: Transmission of thermal energy from a hotter to a cooler region through a moving medium, such as air or water.

Creep: Nonrecoverable deformation proceeding at relatively low strain rates, less than about 10^{-6}/sec, usually associated with sufficiently high temperature to allow significant rates of diffusion.

Crystallization: Formation of crystalline phase out of amorphous materials during high-temperature processing. Undesirable or uncontrollable crystallization is called divitrification.

Custom design: A form of design in which the choice and arrangement of components and wiring on a package may vary arbitrarily within tolerances from a regular array.

Dalton's law of partial pressures: The total pressure excerted by a mixture of gases is equal to the sum of the partial pressures excerted by the individual components.

Die: Integrated circuit chips as cut (diced) from finished wafer.

Die Bond: Mechanical attachment of silicon to substrate usually by solder, epoxy, or gold-silicon eutectic, including interface metallurgies on chip and substrate. The die bond is made to the back (inactive) side of the chip with the circuit side (face) up.

Dielectric:	Materials that does not conduct electricity. Generally used for making capacitors, insulating conductors (as in crossover and multilayered circuits), and for encapsulating circuits.
Dielectric Constant:	The term used to describe a material's ability to store charge when used as a capacitor dielectric. It is the ratio of the charge that would be stored with free space to that stored with the materials in question as the dielectrics.
Direct Chip Attach (DCA):	A name applied to any of the chip-to-substrate connections used to eliminate the first level of packaging.
Discrete Component:	Individual components or elements, such as resistors, capacitors, transistors, diodes, inductors, and others, as self-contained entities.
Dry Processing:	Pressing and compacting together of dry powdered materials with additives in rigid die molds under heat and pressure to form a solid mass, usually followed by sintering to form shapes.
Electroless Plating:	Metal deposition, usually in an aqueous medium, which proceeds by an exchange reaction between metal complexes in the solution and the particular metal to coated, the reaction does not require externally applied electrical current or potential.
Electroplating:	Deposition of an adherent metallic coating onto a conductive object placed into an electrolytic bath composed of a solution of the salt of the metal to be plated. Using the terminal as the anode (possibly of the same metal as the one used for plating), a DC current is passed

through the solution affecting transfer of metals ions onto the cathodic surface.

Encapsulation: Sealing up or covering an element or circuit for mechanical and environmental protection.

Eutectic: A term applied to the mixture of two or more substances with the lowest melting point possible between those components.

Evacuation rate: The pressure in a vacuum system falls as gas is removed, and the rate of gas removal is the throughput of the vacuum pumps. Assuming the pumping speed to remain constant, the throughput falls as the pressure falls, and is in fact exponential, given by:

$P_2 = P_1 \exp -(St/V)$

Where P_2 = pressure (torr) at time t (seconds), P_1 = pressure (torr) at time t = 0, V = system volume (liters) and S = pumping speed (l/s).

The pressure falls to half its original value in 0.69 V/S seconds to quarter that value in twice as long, to 1/8 that value in twice as long again an so on. At low pressures the evacuation rate falls off as a result of leakage and outgassing from system walls.

Failure: The temporary or permanent impairment of device function caused by physical, chemical, mechanical, electrical, or electromagnetic disturbance or damage.

Failure Rate: The rate at which devices from a given population can be expected (or were found) to fail as a function of time (e.g., %1000 hr. of operation)

Fatigue:	Used to describe the failure of any structure caused by repeated application of stress over a period of time.
Filler:	A substance, usually ceramic or metal powder, used to modify the properties of fluids or polymers.
Flux:	In soldering, a material that chemically attacks surface oxides so that molten solder can wet the surface to be soldered, or an inert liquid which excludes oxygen during the soldering process.
Getter:	The term getter is usually referred to materials, which chemically sorb active gases in a vacuum environment.
Gram molecule or mole:	Quantity of gas (or any substance) having a mass equal to its molecular weights in grams.
Gram molecular volume:	Volume occupied by a gram molecule of gas is a universal constant found experimentally to be 22.414 liters at 760 torr and $0^{o}C$ (molecular weight in pounds occupies 359 ft^{3})
Hermetic:	Sealed so that the object is gastight. The test for hermeticity is to fill the object with a test gas – often helium- and observe leak rates when placed in a vacuum. A plastic encapsulation cannot be hermetic as it allows gases to permeate.
Hybrid module:	A special carrier of hybrid microcircuits and other components interconnected as a unit, or as a component of an electronic subsystem. The module may be of single construction or made up of submodules, each usually with a compartment to house hermetically packaged hybrids and discrete passive component

parts, such as transformers, axle-lead resistors, etc. Nonhermetic hybrid modules generally are parylene coated.

Inert Atmosphere: A gas atmosphere such as helium or nitrogen that is nonoxidizing or nonreducing to metals.

Insulators: A class of materials with high resistivity. Materials that do not conduct electricity. Materials with resistivity values of over $10^6\Omega$ cm are generally classified as insulators.

Integrated circuit: A microcircuit (monolithic) consisting of interconnected elements inseparably associated and formed in situ on or within a single substrate (usually silicon) to perform an electronic circuit function.

Interconnection: The conductive path required achieving connection from a circuit element to the rest of the circuit.

Interface: The boundary between dissimilar materials, such as between a film and substrate, or between two films.

Leak rates: A leak rate is the throughput of a small hole from atmospheric pressure to the vacuum space, and is conveniently measured in torr ls^{-1}.

INVAR: A trademark of International Nickel Co., for a very low thermal expansion alloy of nickel and iron.

KOVAR: An alloy of iron (53%), cobalt (17%), and nickel (29%) with thermal expansion matching alumina substrate and certain sealing glasses. Most common lead frame and pin material.

Laser Soldering: The technique in which heat to reflow a solder interconnection is provided by a laser, usually a longer wavelength YAG or CO_2 laser. The joints are heated sequentially, and cooled rapidly.

Metal Migration: An undesirable phenomenon whereby metal ions, notably silver, are transmitted through another metal across an insulated surface, in the presence of moisture and an electrical potential.

Metallization: A film pattern (single or multilayer) of conductive material deposited on a substrate to interconnect electronic components.

Multichip Package: An electronic package that carries a number of chips and interconnects them through several layers of conductive patterns. Each one is separated by insulative layer and interconnected via holes.

Out gassing rates: An outgassing rate of a system is the rate at which gas is desorbed from system walls to be pumped away by the vacuum pump, and is expressed as throughput. It is dependent on the surface area of the system from which desorption is taking place. The outgassing rate of a surface is therefore expressed as throughput per unit area, or torr l $s^{-1}cm^{-2}$. Typically a clean unbaked stainless steel surface has an outgassing rate, after one or two hrs pumping, of 10^{-8} to 10^{-9} torr l s^{-1} cm^{-2}. Outgassing rate are particularly influenced by surface history such as exposure to moist atmosphere, baking etc.

Overcoat: A thin film of insulating material, either plastic or inorganic (e.g., glass or silicon nitride), applied over integrated circuit elements for

the purposes of mechanical protection and prevention of contamination.

Passivation: The formation of an insulating layer directly over a circuit or circuit element to protect the surface from contaminants, moisture, or particles.

Passive Components (Elements): Elements or components such as resistors, capacitors, and inductors, which do not change their basic character when an electrical signal is applied. In contrast transistors, diodes, and electron tubes are active components.

Partial pressures: The partial pressure excerted by any one component of a mixture of gases is the pressure excerted by that component if it occupied that volume alone.

Paste: Synonymous with composition or ink when relating to screenable thick-film materials, usually consisting of metal or ceramic powders dispersed in organic vehicles.

Peel Strength (Peel Test): A measure of adhesion between a conductor and the substrate. The test is performed by pulling or peeling the conductor off the substrate and observing the force required.

Permeability: The property of a solid plastic material that allows penetration by a liquid or gas.

Pemeability: Constructional materials, especially glasses and elastomers, are permeable to the light constituents of the atmosphere. Permeability rates are expressed in the same terms as outgassing rates.

Phase Diagram:	State of a metal alloy or ceramic over a wide temperature range. The phase diagram is used to identify phases as a function of composition and temperature.
Plating:	A condensation of the word Electroplating or Electroless Plating that describes the coating of a metal on plastic or other surfaces with metal that is electrolytically or chemically deposited from a bath.
Pumping speed:	The speed of a pump is defined as the volumetric rate flow into the pump (liter/second) where the volume is measured at the pressure at the pump inlet (torr).
Radiation:	The combined process of emission, transmission, and absorption of thermal energy between bodies separated by empty space.
Relative Humidity (rh):	The ratio of partial pressure of water in any gas at a particular temperature to the saturated vapor pressure of water in the same gas at the same temperature, usually expressed in percent.
Reliability:	The probability of survival of a component, or assembly, for the expected period of use. Expressed mathematically, R = 1- Probability of failure during the expected life.
Reliability:	1. The duration or probability of failure-free performance under stated conditions. 2. The probability that an item can perform its intended function for a specified interval under stated conditions.
Reliability, mission:	The ability of an item to perform its required functions for the duration of a specified mission profile.

Resin:	A term used for an organic polymer that when mixed with a curing agent crosslinks to form a thermosetting plastic.
Sealing:	Joining the package case header (or chip carrier base or substrate) with its cover or lid into a sealed unit. For hybrids, sealing connotes an important finishing operation in fabricating a hybrid microcircuit, signaling the stage when the assembly, in the form of a populated package, becomes a bona fide hermetic (nonhermetic) device.
Sintering:	Heating a metal or ceramic powder, thereby causing the particles to bond together to form monolithic body.
Solder:	A low melting-point alloy used in numerous joining applications in microelectronics. The most common solders are lead-tin alloys.
Solder Glasses:	Glasses used to package sealing that have a low melting point and tend to wet metal and ceramic surfaces.
Solderability:	The ability of a conductor to be wetted by solder and to form a strong bond with the solder.
Soldering:	The process of joining metals by fusion and solidification of an adherent alloy having a melting point below about 300°C.
Sputtering:	The removal of atoms from a source by energetic ion bombardment. The ions are supplied by plasma. The sputtering process is used to deposit films for various thin-film applications.
Storage life:	The length of time can be stored under specified conditions and still meet specified requirements.

Stress Corrosion: Refers to the degradation of mechanical properties of brittle materials by crack propagation due to the acceleration of applied stress in the presence of corroding atmospheres such as water.

System: A composite of equipment and skills, and techniques capable of performing or supporting an operational role, or both. A complete system includes all equipment, related facilities, material, software, services, and personnel required for its operation and support to the degree that it could be considered self-sufficient in its intended operational environment.

System effectiveness: A measure of the degree to which an item or system can be expected to achieve a set of specific mission requirements, and which may be expressed as a function of availability, dependability, and capability.

Temperature cycling: An environmental test where the film circuit is subjected to several temperature changes from a low temperature to a high temperature over a period of time.

Thermal Coefficient of Expansion (TCE): The ratio of the change in dimensions to the change in temperature-per-unit starting length usually expressed in cm/cm/^{o}C. The acronyms TCE and CTE are synomous.

Thermal Conductivity: The rate with which a material is capable of transferring a given amount of heat through itself.

Thermal Cycling: A method to impose a cyclic stress on an assembly of microelectronic components by alternately heating and cooling in an oven. It is used for accelerated reliability testing of assemblies.

Thermal Fatigue: Failure of a structural element from repeated temperature excursions, wherein the load develops from thermal expansion mismatch of dissimilar metals.

Thermal Gradient: The plot of temperature variances across the bulk thickness of materials being heated.

Thermal Mismatch: Difference in thermal coefficients of expansion of materials, which are bonded together.

Thermocompression Bonding: A process involving the use of pressure and temperature to join two materials by interdiffusion across the boundary.

Thermosonic Bonding: A bonding process, which uses a combination of thermocompression, bonding ultrasonic bonding. It is done on what amounts to a gold-wire thermocompression bonder with ultrasonic power applied to the capillary.

Thick Film: A film deposited by screen printing processes and fired at high temperature to fuse into its final form. The basic processes of thick-film technology are screen-printing and firing.

Thin Film: Thin film referes to a coating layer of thickness in the range of from a few atomic layers to a few microns. The important feature distinguishing thin films from thick films, though, is not so much the difference in thickness as the method of deposition, which takes

place by a variety of techniques such as chemical vapor deposition, evaporation, or sputtering.

Thin-Film Packaging: An electronic package in which the conductors and/or insulators are fabricated using deposition and patterning techniques similar to those used for integrated circuit chips.

Through Hole: A hole connecting the two surfaces of a printed-circuit structure.

Throughput: Throughput is the mass flow rate of gas past any point in a vacuum system and at equilibrium is constant for all points in the vacuum system. Mass flow rate is measured gram.second or the number of molecules per second. Since pressure (torr) is a measure of the molecular concentration, i.e., number of molecules per liter, and the volumetric flow rate is given as litre/second, then the product torr.litre/second is proportional to the number of molecules passing any point in a vacuum system per second and hence the throughput.

Time to form a monolayer: The number of molecular impacts per second is a difficult quantity to comprehend but it can be expressed in the more useful way as the time required for the surface to become covered by a single layer of molecules. At a pressure of 760 torr and 20°C the time to form a monolayer of nitrogen molecules is 3×10^{-9} seconds. At a pressure of 10^{-9} torr and 20°C the time to form a monolayer of nitrogen molecules is about 2000 seconds.

Ultrasonic Bonding:	A process involving the use of ultrasonic energy and pressures to join two materials.
Useful life:	The number of life units from manufacture to when an item has a repairable failure or unacceptable failure rate.
Vacuum Deposition:	Deposition of a metal film onto a substrate in vacuum by metal evaporation techniques.
Via:	An opening in the dielectric layer though which a riser passes, or else whose walls are made conductive.
Wafer:	Commonly, a slice of a semiconductor crystalline ingot used for substrate material when modified by the addition, as applicable, of impurity diffusion (doping), ion implantation, epitaxy, etc., and whose active surface has been processes into arrays of discrete devices or Ics by metallization and passivation.
Wave Soldering:	The technique for solder application and reflow in which a jet of liquid solder is directed at the two metallic points to be interconnected. The technique usually involves processing steps to apply flux and remove excess solder.
Welding:	Joining two metals by applying heat to melt and fuse them with or without filler metal.
Wetting:	The spreading of molten solder or glass on a metallic or nonmetallic surface, with proper application of heat and flux.
Wireability:	The capability of a package to permit the interconnection of subpackages mounted on it and terminals attached to it measured as

the probability of wiring success. It is near one when sufficient wiring capacity, via availability and terminal access, are present.

Wirebond: A completed sire connection whose constituents provide electrical continuity between the semiconductor die (pad) and terminal. These constituents are the fine wire; metal bonding surfaces like die pad and package land; and metallurgical interfaces between wire, and metals on both the chip and substrate.

Wirebonding: The method used to attach very fine wire to semiconductor components in order to interconnect these components with each other or with package leads.

Biography of Author

Dr. Rajeshuni Ramesham is working as a Senior Member of Engineering Staff at the Jet Propulsion Laboratory, California Institute of Technology, Pasadena, CA, and USA. His present research work focus on the reliability of packaging and interconnects associated with the microelectromechanical systems (MEMS) applications and advanced adhesion and bonding issues in microelectronics, and photonics packaging. He also works on the application of polycrystalline synthetic diamond for MEMS, electrochemical, electroanalytical, and corrosion resistant coating applications. He worked for Auburn University, Auburn, AL for little over nine years. His research work at Auburn University addressed the fundamental issues involved in diamond processing techniques, heat dissipation techniques, and electrochemical applications of diamond. He has extensive experience in diamond film growth, photolithography, thin films, semiconductor processing, characterization, microstructure fabrication, electrochemical evaluation of diamond coatings, etc. He has received an outstanding research performance award from the Electrical Engineering Department of Auburn University. Dr. Ramesham has worked for the NASA's JPL, Caltech, Pasadena, CA, as a NRC RRA from Oct 1985 to Oct 1988 in Thin Film Laboratory (today it is MDL). He has received 7 awards from NASA for the research work he performed. He has published over 93 refereed journal and proceedings articles and has made 60 national and international conference presentations. He has given invited presentations at the national and international conferences. He received the best research paper award from the IEEE Alabama Section. He is a co-chair for Microelectromechanical activities at the SPIE's Micromachining and Microfabrication Conference and International Conference on Mechanics in Medicine and Biology. He has been selected by Who is Who in the World. He has two patents and two patents are pending and five NASA technical disclosures. He has

offered a short course on "Fabrication of Thin Film Diamond Microstructures" at the First international Conference on the Applications of Diamond Thin Films and Related Materials, August 17-22, 1991, Auburn, AL. Dr. Ramesham received his Ph.D from the Indian Institute of Science, Bangalore, India. He received a fellowship from the DoE, India during graduate studies. He has obtained 2nd rank at the M.S. and received a county first rank at the B.S. level. He received a National Merit Scholarship during his M.S. studies.