



# **Evaluation of Non-Evaporable Getters for High Vacuum Hermetic Packages**

## **Final Report (JPL D-27440)**

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## 1.0 Executive Summary

Maintaining a controlled ambient (or atmosphere) in an extremely small hermetically sealed device like a packaged microelectromechanical systems (MEMS) depends on being able to pump away or absorb the outgassed gaseous species from the packaging materials that would otherwise destroy the desired ambient. As these devices and package systems depend upon a good vacuum or a controlled atmosphere being present for optimal performance, three hurdles must be passed:

- Proper processing to reduce trapped gasses in the package
- Hermetically sealing the package
- Providing a means to pump away gasses that outgas into the package

This report provides an overview of the state-of-the-art technology on various aspects of packaging a MEMS device in a controlled ambient, focusing on vacuum applications. Proper package processing and getter technology are key to success in this endeavor. This work will help users to understand the key process parameters needed to successfully prepare a package for long service life as well as the basics of gettering technology as used to maintain the desired ambient once the package is sealed.

A technical team (Table 1) was formed to collaborate and leverage activities of high vacuum hermetic sealing process to assess the reliability of various commercial-off-the-shelf (COTS) non-evaporable getters (NEGs), assembled in advanced commercial electronic packages under the NASA Electronic Parts and Packaging (NEPP) Program. One of the primary objectives of the NEPP program is to expedite the infusion of advanced Microelectromechanical Systems (MEMS) technologies, COTS advanced packaging technologies assembled with non-evaporable getters, into present and future NASA missions, to enhance reliability and performance robustness. COTS emerging getter technologies and advanced packaging technologies, which were chosen due to their lower weight, increased functionality, and lower cost will make them excellent candidates for space missions if they are tested, characterized, and qualified to show that they will meet the NASA stringent reliability, life cycle and quality requirements.

**Table 1: Contributors to this report**

Jet Propulsion Laboratory	SST International Inc.	SAES Getters Inc.	Integrated Sensing Systems Inc.
Dr. Rajeshuni Ramesham*	Mr. Paul Barnes*	Mr. Richard Kullberg*	Dr. Douglas Sparks*
Dr. Karl Yee Mr. Jim Okuno Mr. Stephen Bolin			

\*Getters evaluation team members

The team initially planned to evaluate several types of advanced commercial electronic packages and sintered getters and nanogetters for space applications during this task. Because funding was reduced from \$110 k to \$45k by the NEPP management. The level of effort was reduced correspondingly. We have identified an advanced electronic package that has an immediate use to package JPL developed MEMS mesogyro of the Microdevices Laboratory (MDL). Concentrating on this package will produce immediate impact from this NEPP task on this JPL developed technologies. 391 pin grid array (PGA) advanced package, SAES getters and

ISSYS's nanogetters were chosen in this study for evaluation. The prerequisite to evaluate the getter is to hermetically high vacuum seal the package and lid, using suitable sealing process technology and Au/Sn preforms. The key process that has been addressed to package MEMS mesogyro is as follows:

Initially the PGA packages were annealed in a high vacuum for 24 hrs @ 400°C. The lids supplied by the vendors were usually attached with the gold/tin (Au/Sn) preforms. The Au/Sn preform melts at a temperature of ~280°C. Activation of the getter attached to the lid prior to sealing (lid and cavity) is performed in a vacuum at ~400°C, which is apparently not compatible with the melting temperature of the Au/Sn preform attached to the lid. Therefore, the Au/Sn preforms and lids were procured as separate custom package components from the vendor. Initially, a single Au/Sn preform was manually soldered to the sealing ring of the PGA cavity. Several temperature profiles were used to seal the package with the Au/Sn preform and the lid. Several attempts were made to seal the cavity using a single Au/Sn preform. Those attempts resulted in very poor results. We have implemented the use of two preforms together to increase the quantity of the preform and subsequently, spot-welded them to the sealing ring of the cavity instead of manual soldering. A lid was annealed in a high vacuum for 24 hrs @ 400°C without a preform. A cavity with two preforms that was spot-welded to the sealing ring (sealed using High Vacuum (HV-2200) Gated Turbo system at SST International Inc., CA). We have successfully hermetically sealed the 391 PGA assembly without a getter during the several experimental runs performed. This package was leak tested using high pressure He (Helium) bomb leak test that include fine leak and gross leak tests. The leak rates for package # 305 and 306 are  $6.6 \times 10^{-8}$  Atm cc/sec He and  $4.4 \times 10^{-8}$  Atm cc/sec He, respectively. These leak rates are substantially lower than the leak rate fail criteria as per mil-spec 883. These packages were inspected optically and imaged with X-ray non-destructively for any defects/voids in the sealing. We found several voids in the Au/Sn sealing area.

Further work is warranted to clean the surface of the package and other components using suitable plasma cleaning techniques to remove contaminants and assemble the PGAs with the ASIC, resonator and with the getters on the lid. The critical parameter was to optimize the temperature profile to seal the cavity and lid and also activation of the getter being attached to the lid in the vacuum prior to sealing. These two were addressed during the temperature profile development.

The team procured SAES getters from SAES Inc., and deposited nanogetters over the annealed lids that do not have a preform. We have also mounted silicon resonators and the corresponding ASIC in the cavity of the PGA and characterized the resonator in the vacuum of 1 mTorr pressure. We have assembled 6 PGA cavities with the resonators and ASICs. Due to the lack of funds it is necessary to stop work under the NEPP umbrella. This report provides the up-to-date results. Partial support is provided by MDL and Boeing to continue the hermetic sealing process for mesogyro packaging. We have also submitted a proposal to JPL for DRDF (JPL internal) funds to continue this effort to make a reality of packaging the JPL mesogyro for future NASA space applications such as Mars Exploration projects (Mars Science Laboratory, MSL, etc.).

## **2.0 Overview of Vacuum Packaging of MEMS and Related Microsystems (R. Ramesham and R. Kullberg)**

### **2.1 Introduction**

A variety of sealed-off devices such as cathode ray tubes (CRT's), particle accelerators and colliders, X-ray tubes, tube lights, lamps, infrared detector dewars, helium-neon lasers, and others require a controlled ambient of vacuum or gas for their successful operation. Outgassing from surfaces in these systems destroy these controlled ambient over time (days to years). Getters are routinely used in these systems to pump away or absorb this gas and thereby maintain the desired ambient in the system. The same outgassing mechanisms hold in smaller systems, such as packaged MEMS sensors (e.g. motion sensors, gyros, RF MEMS, and infrared sensors). Similarly, getters are needed in order for Sand other microelectronic packages to reach desired system lifetimes of many years. [1-3]

### **2.2 Challenges**

The challenges facing the MEMS industry are three fold:

1. Most MEMS devices are packaged to protect from the external environment
2. MEMS must be able to sense into that external environment
3. MEMS package should be economical if the MEMS device is to be successful in the commercial market. Furthermore, the cost of the MEMS package is very high if the device requires hermetically sealed high vacuum ambient for successful operation

### **2.3 Fundamentals**

Research on inorganic or organic getter materials that are able to sorb small quantities of reactive gases in vacuum devices/packages began late in the 19<sup>th</sup> century. The first use of the term “getter” was by Thomas Edison’s assistant Malignani in 1882. Malignani developed the technique of coating components of incandescent lamps with red phosphorous. Red phosphorous reacts with, or getters, water vapor, thereby breaking the water-tungsten (filament) cycle that limits lamp lifetime. This process is still used today in the lamp industry.

At the beginning of the 20<sup>th</sup> century researchers working to develop electron tubes had great difficulties in obtaining practical tube lifetimes. The lifetime of the tubes was limited by degradation of the internal vacuum due to the outgassing of various species from the inner surfaces. Getters based on alloys or compounds of barium were developed as a successful solution to overcome this problem. These getters are referred to as evaporable getters because they are heated to deposit the barium as a thin film on the inner surface of vacuum tubes. Such films maximize the available gettering capacity of the deposited evaporable getters by creating the maximum available chemically active surface area.

Early forms of evaporable barium getters included pure barium encapsulated in small iron or nickel tubes, barium-thorium alloys and barium-strontium carbonate mixtures. All of these approaches had stability problems. These problems were solved by the development of the BaAl<sub>4</sub> alloy by Paolo della Porta of SAES Getters in the early 1950s. This alloy is stable in air and made practical the high volume use of getters. BaAl<sub>4</sub> getter technology extended vacuum tube life to thousands of hours and is still in use in very important applications like cathode ray tubes (CRTs) for television and computer monitor/display applications.

Gettering technology expanded into non-evaporable getter (NEG) materials technologies during the last half of the 20<sup>th</sup> century. Today non-evaporable getter alloys, using metals such as zirconium and titanium, can be found in applications ranging from Thermos bottles to large physics projects at labs like CERN and Brookhaven National Laboratory (BNL). It is safe to say that many of the vacuum enabled technologies we take for granted today are made practical by the ability of getter materials to provide useful device/system lifetimes. [4]

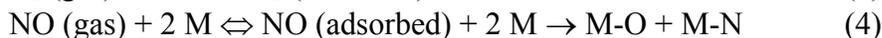
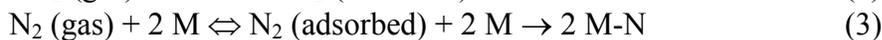
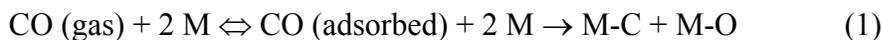
Getters work by the careful implementation of simple mechanisms:

- adsorption of gases in the case of getters such as molecular sieves
- adsorption and chemical reaction in the case of evaporable and non-evaporable getters (NEGs.)

Getters are normally evaluated in terms of their sorption speed and their capacity for various active gases. American Society of Testing Materials (ASTM) standards exist for these evaluations. NEGs are the most inclusive case in terms of the various gettering mechanisms and we will explore how they work in greater detail. 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. In general, getter materials are designed with high surface reactivity and high diffusivity, which provides both a high sorption speed and a large capacity. The bulk diffusivity parameter is particularly important for NEGs whose performance as a bulk getter is enhanced.

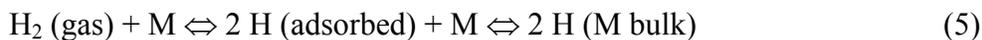
Bulk gettering characteristics are heavily dependent on the amount of active surface area available for reaction with ambient gases. If the getter is operating at room temperature, when, for most gases, very limited bulk diffusion takes place, the surface of the getter eventually becomes saturated, or passivated, and the bulk getter ceases to scavenge gas.

Typical NEG or bulk getter systems are the zirconium based getter alloys. Examples include SAES' St 101 (Zr<sub>0.84</sub>-Al<sub>0.16</sub>) and St 707 (Zr<sub>0.70</sub>-V<sub>0.246</sub>-Fe<sub>0.054</sub>). The zirconium-based system is very reactive with a wide variety of gas molecules such as H<sub>2</sub>, CO, CO<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, and NO<sub>x</sub> 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:



Where M represents the metal constituents of the zirconium based getter alloy.

The exception to this rule is hydrogen and its isotopes. Hydrogen easily diffuses into a getter because it dissociates on the getter surface into atomic hydrogen. The hydrogen atoms easily slip into the atomic lattice of the metal grains as is shown by the following reactions:



Hydrogen in the interior of a non-evaporable getter (NEG) establishes a solid solution that exhibits an equilibrium pressure, which depends on the concentration of hydrogen and the ambient temperature. Sievert's Law describes this relationship:

$$\text{Log}P = A + 2\log q - B/T \quad (7)$$

Where:

$q$  = H<sub>2</sub> concentration in the NEG alloy in torr-liters/g

$P$  = H<sub>2</sub> equilibrium pressure in torr

$T$  = temperature of the getter in Kelvin

$A$  and  $B$  are constants for different NEG alloys

This behavior is very important for ultra high vacuum (UHV) applications, where H<sub>2</sub> is the most important gas being pumped. Unlike other gases, H<sub>2</sub> pumping is reversible, allowing for regeneration cycles that significantly increase getter lifetimes.

If the operating temperature of the NEG getter is high enough, the reaction products (reactions 1-4) will diffuse into the bulk of the alloy, exposing a fresh chemically active surface for renewed adsorption. This "reactivation process" can be carried out continuously at high temperatures or periodically after normal operation at low temperature. For example, at 25°C the pumping speed and capacity for CO in a NEG getter will decrease over time as the CO reacts with the surface. The getter can then be reactivated at high temperature. However, when the operating temperature of the activated NEG getter is above the temperature at which the reaction products begin diffusing (typically > 300 °C,) the pumping speed decays very slowly and the practical capacity of the getter approaches 50% of the stoichiometric capacity of the reaction.

On the other hand, while hydrogen quickly diffuses into the bulk getter, even at room temperature, at higher temperatures (~400 °C for most getter alloys of interest,) the equilibrium reaction (reactions 5-6) is shifted towards the left reducing the hydrogen concentration in the getter bulk.

## 2.4 State of the art

Vacuum continues to be an enabling environment for electronic devices into the 21<sup>st</sup> century. Examples include infrared (IR) sensing systems, inertial navigation systems (gyros etc.), and pressure sensors.

The pressure of the market place to reduce the size of such systems, and thereby reduce their cost, which has spread from the microelectronics industry to all areas of technology. In response to this market pressure researchers and technologists are using semiconductor industry style batch fabrication processes to develop microelectromechanical systems (MEMS) and microoptoelectromechanical systems (MOEMS). MEMS/MOEMS devices need to be connected to, and often be protected from, the outside world. In addition many MEMS devices need to be packaged in a controlled atmosphere or vacuum in order to operate reliably with high performance.

MEMS typically have moving parts that are sensitive to the operating pressure, the partial pressure of water vapor in the package, or both. For example, infra red sensors need to operate in a pressure < 10<sup>-3</sup> Torr in order to be thermally isolated from the outside world and maintain

adequate sensitivity. MEMS gyros increase in their sensitivity or Q as the pressure in a package decreases. Water vapor can also cause stiction, where device components are “glued” together by thin films of water and unable to function. The situation is further complicated by the need for high degrees of hermeticity (leak rates on the order of  $10^{-12}$  atm-cc/sec) and the lack of space to mount getters to control the contaminants in the package.

Consequently, hermetically packaging MEMS devices in a reliable and economical manner is a topic of great interest to the MEMS community. [5] The development of MEMS technology has reached a point where the packaging of the device is proving to be more difficult than the actual device development itself. Many development groups are finding their efforts stymied at this point and interest in MEMS packaging and related topics is at a high level. [1-3] A Specific Example: Controlled Ambient Packaging of Microsensors.

Packaging is a very important aspect to be considered for microsensors. Several microsensor properties are affected by ambient conditions. For example, a microbolometer based infrared night vision system needs to be packaged in a vacuum to thermally isolate it from exterior heat sources that can reduce its sensitivity. In this instance the vacuum serves as an insulator much like it does in a thermos bottle by reducing thermal conduction through removal of gasses present in the system.

Many packaged MEMS have been developed using bonded silicon-glass structures [6]. However, it is difficult 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. [7,8] The other is gas desorption from the surfaces and bulk of materials within the vacuum cavity. These gases must be eliminated in order to create and maintain a high vacuum within a hermetically sealed cavity.

## **2.5 Hermetic Packaging**

Ceramic or metal is typically used to form an enclosure or package to isolate the electronic or microelectromechanical devices from the ambient operating environment. Packaging concepts are extending from the discrete package model to using the MEMS as part of its own package in the wafer level approach. In any case, in order to protect the packaged device the packaging method must be able to be hermetically sealed in order to prevent the incursion of destructive elements from the outside. One definition of the word hermetic, when applied to MEMS packages is complete sealing by fusion, solder, welding or other methods so as to keep air or gas from getting in or out; in other words, airtight.[9] However, in addition to a hermetic seal, the packaging engineer must also concern himself with the condensed moisture on a MEMS device’s surface, and the outgassing of gaseous species during the device operation. Both gas sources may lead to the principle causes of failure in the field. It is important to eliminate condensable moisture, to degas all components prior to sealing, and remove as much gas as possible during the sealing process.

A proper seal will eliminate or reduce the ingress and egress of gaseous species at the package perimeter during its operating life. Examples of gasses that can permeate into the package include water vapor, oxygen, nitrogen, and other components of the external ambient. Unfortunately, perfect hermetic seals are nonexistent. Small gas molecules will enter the package over time through diffusion and permeation destroying the ambient inside the package. In addition, the vacuum can be destroyed by out-gassing of various species (such as water vapor, hydrogen, carbon monoxide, nitrogen, oxygen, and carbon dioxide) from the inner package

surfaces. Also, organic materials are significant sources of outgassing and are to be avoided when ever possible. Any of the above sources of gas can significantly degrade the service life of a packaged MEMS device. Getters are used to pump away these gasses.

Solid-state getters may be either planar or three-dimensional and exhibit good mechanical strength. They must be particle free. Any loss of getter particles before, during, or after activation of the getter in a MEMS package may cause failure of the MEMS device. The getter must be able to survive shocks and vibrations during operation of the MEMS device. To minimize MEMS defects caused by high temperature, the getter should have a high active surface area that can easily be activated at low temperatures. A getter with high porosity combined with a large active surface area will assure excellent sorption performances even at room temperature.

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. For example, an uncooled infrared sensor needs vacuum to thermally isolate it from the environment. In other cases a getter is necessary to control the amount of water vapor present in the package. Hydrogen contamination can also cause deleterious effects. Kayali and Ragle have reported an overview of hydrogen effects on GaAs microwave semiconductors in a JPL document [10].

### 2.5.1 Sources of Gases in a MEMS Package

It is important to know the composition of the residual gas in a vacuum system or 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 also 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 can be expressed in Torr and are shown in Table 2. [11]

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

Table 2: Partial Pressures of Atmospheric Constituents

Almost invariably the majority of gas in a package is water vapor, hydrogen or both. While the source of water vapor is typically water sorbed on the surfaces of the package and device themselves, the source of hydrogen is less obvious. According to Saito et al., [12] the major source of hydrogen gas in a hermetically sealed package is from electroplated nickel in the package housing. For example, Kovar typically electroplated with nickel using sulfamate per QQ-N-290. During the plating process  $H_2$  is evolved on the plated surface through electrolysis. This surface has enough energy to dissociate an appreciable amount of  $H_2$  into the monatomic form, which then diffuses into the nickel itself.

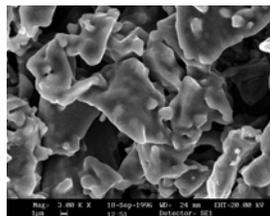
It is known that the temperature must be higher than  $350^\circ C$  to bake-out hydrogen from the iron-base alloys. [12] However, the wire bondability degrades as bake-out temperature of the electronic packaging material increases. The optimum bake-out temperature is based on the amount of hydrogen evolved and subsequently its effect on wire bondability. The optimized hydrogen bake-out temperature was at  $250^\circ C$  for 168 hours for Ni/Au plated Kovar housings based on the residual hydrogen contents measured by Residual Gas Analysis (RGA) and subsequent wire bondability. The evolved hydrogen was reduced from 0.6% to 0.0033% with a post-plating hydrogen bake.

This long bake out proves impractical in many production scenarios. For example, the key concern in a ceramic package that is plated with Ni then Au is to have an adequately pure Au surface for good solder wettability during the sealing of the lid to the package. Empirical work shows that a bake out on the order of 1 hour at  $400^\circ C$  in a vacuum oven at  $1 \times 10^{-5}$  torr results in adequate reduction of the hydrogen concentration without adversely impacting the sealing process.

### 2.5.2 Eliminating Gases In The Package

To eliminate these gases several different methods have been used. In one method, the residual gas in the cavity is evacuated through an opening after anodic bonding. After pump out the opening is plugged by depositing aluminum or silicon monoxide. The aluminum plug can be used as an electrical feed through if desired. In another method, a NEG is placed in the sealed cavity and activated during the sealing process. Both methods may be necessary in order to eliminate both gasses trapped during the sealing process and those outgassing into the cavity over the lifetime of the MEMS.

A typical NEG material used in hermetically packaged MEMS is a  $50 \mu m$  thick microfer substrate coated with a  $250 \mu m$  thick sintered porous Ti and Zr-V-Fe alloy structure. The sintered getter structure is passivated during manufacture to allow handling in atmosphere. In order for the getter to absorb gases it must be activated at around  $400^\circ C$ . Since the anodic bonding temperature is also  $400^\circ C$ , the getter can be activated during the anodic bonding process.



**Figure 1:** SEM photograph of a typical sintered porous NEG microstructure  
(Courtesy of R. Kullberg, SAES Inc.)

Care must be taken that a getter appropriate to the process be used. For example, a getter that is exposed to high concentrations of gas and high temperature must have properties that make it “frittable” or able to be activated in a vacuum after such exposure.

### 2.5.3 Mitigation of Hydrogen Degradation

A common gas that degrades the ambient of hermetically sealed MEMS packages is hydrogen. This hydrogen is inherently trapped in the various metallization layers used to manufacture these devices and their packages. An example of hydrogen entrapment is the capture of hydrogen released during gold plating processes. This hydrogen goes into solution in the gold film and later outgasses into the cavity, destroying the desired ambient.

Saito et al. [12] have recommended a three-pronged approach to mitigate hydrogen degradation. Practically speaking there are two key steps to reduce hydrogen outgassing to acceptable levels. Step one is to utilize the Sievert’s Law mechanism by baking out gold plated components in a vacuum prior to assembly and sealing of the device. Step two is to utilize a getter in order to capture any remaining hydrogen that outgasses into the device.

### 2.6 Applications of Getters

Getters are used as an enabling technology in a broad array of technologies and industries. To meet the needs of such a broad array of applications many different types of getters are manufactured. The following table 3 gives examples of the widely divergent technologies where getters are used.

Markets for Getters	Key Applications
Display devices	Color & monochrome cathode ray tubes.
Light sources & Lamps:	High intensity discharge Fluorescent, cold cathode
Vacuum Insulation	insulated pipes, solar collectors, vacuum bottles, and vacuum insulation
Electronic devices & Flat Panel Displays	Infrared detectors, night vision tubes, x-ray image intensifiers, pressure transducers, vacuum fluorescent displays, x-ray tubes, photomultipliers, vacuum interrupters, field emission displays, plasma displays.
Vacuum Insulation Panels (VIPs)	VIP’s for refrigeration industry
Vacuum systems	System processing pumps, physics projects, super conductors, <i>in situ</i> pumping
Gas purification technologies	Semiconductor industry/small, large, area and <i>in situ</i> purifiers, analytical instruments, analytical and customer service.
Microelectromechanical Systems (MEMS)	Microgyros, Pressure sensors, Accelerometers, DMDs, RF MEMS, microbolometers, etc.

**Table 3:** Getters markets and their applications

## **2.6.1 Microelectromechanical Systems (MEMS)**

Gettering a MEMS device/package has severe constraints that must be met. The getter must have a large active surface area in order to deal with the large outgassing load expected in relation to the working volume of the package. In addition the getter must not damage the device during the activation process. Consequently it must be activated at relatively low temperature (300 - 500°C.) The getter must also exhibit high sorption performance at room temperature, be free of particles and possess good mechanical strength.

### **2.6.1.1 Types of Getters used in MEMS**

As previously mentioned, getters are classified as being either evaporable or non-evaporable getters. For hermetically sealed MEMS packages evaporable getters are non-applicable as large internal surfaces areas are required on which to deposit them. To maintain the desired ambient in MEMS packages two types of getters are typically used. These are non-evaporable getters and moisture getters or 'dryers.' In addition, as the packages are very small, often well under 1 cm<sup>3</sup> in volume, the getter must be small as well.

### **2.6.1.2 Non-evaporable getters (NEG)**

Various techniques are used to impart to NEG materials special mechanical characteristics (low free particles) and particularly high porosity and surface areas to maximize their capacity when working at room temperature. Various manufacturing techniques are used to obtain getters of preformed shapes and good mechanical stability [13].

One type of getter that meets these requirements uses a screen printing technique that allows the manufacture of porous getters coatings meeting the requirements for a MEMS package.[14,15] The finished getter is called a High Porosity Thick Film (HPTF) getter. HPTF getters have a very high porosity in order to maximize both the chemically active surface area of the getter alloy and the conductance of gas through the getter microstructure. High porosity combined with a large specific, or active, surface area assures good sorption performance at room temperature. The porosity of HPTF getters is about 60 – 65%. The specific surface area, expressed in terms of ratio of active surface area over geometrical area, is on the order of 20 – 30 cm<sup>2</sup>/cm<sup>2</sup> for typical coatings. The density of the coating is 2.0±0.3 g/cm<sup>3</sup>. The sintered porous matrix 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 they are used in.

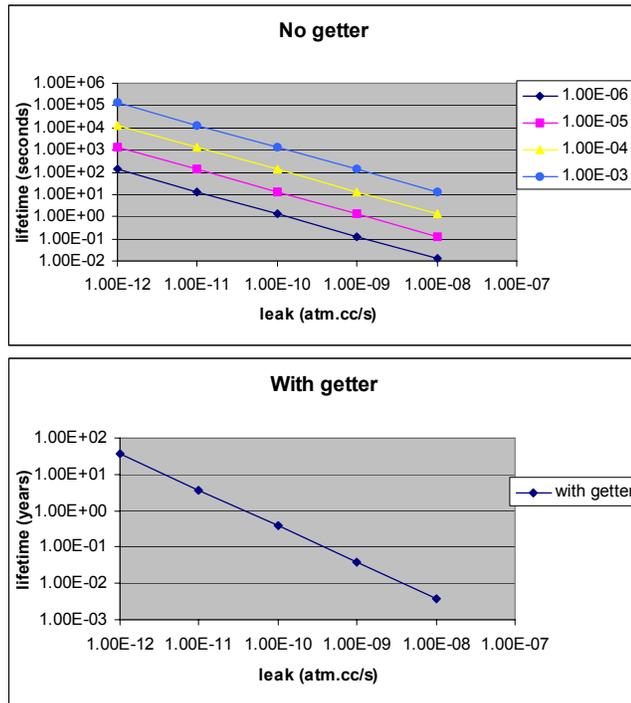
NEG technology is moving beyond sintered structures into new methods of manufacturing. New methods are needed because the envelope within a wafer bonded MEMS doesn't have the necessary volume to accommodate even a HPTF getter, yet the gas loads to be gettered are similar. Getters using advanced coating techniques based on PVD processes have been reported that give similar performance to HPTF, yet are two orders of magnitude thinner. [16]

### **2.6.1.3 Moisture Getters**

There has been continuing concern regarding the presence of water vapor in hermetically sealed semiconductor devices. Hermetically sealed microelectronic devices used in military, space, medical and other applications requiring high reliability may not exceed a limit of 5,000

ppm by volume of water vapor content at the time of fabrication (MIL STD 883, Method 1014.) [17]

Hermetic packages must also demonstrate that they pass a He leak rate test to  $10^{-8}$  atm-cc/sec. Even this rate is too high for real world applications, though it represented a real world limit of detection when it was chosen many years ago. The following curves illustrate the real world tradeoffs of various leak rates. As you can see, even with a getter leak rates appreciably better than the mil-spec are required if life times in years as opposed to seconds are required.



**Figure 2:** Lifetime with and without getters (Courtesy of Ro. Kullberg, SAES Inc.)

In spite of these precautions, 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 ways by which water vapor finds its way to the inside of the package. These include leakage through the various seals, in particular, organic seals which are very permeable to moisture. In addition, the materials used in construction of the package itself can be water sources. An example is epoxies used for such purposes as die attach, which outgas water vapor. Even the sealing atmosphere itself may be contaminated with moisture if not prepared with extreme care.

Tominetti and Renzo [18] examined devices sealed with different types of epoxy. They were able to detect various gas and moisture concentrations to an accuracy of 1 to 10 ppm in the electronic package. The pressure inside these packages was 1 atmosphere of nitrogen. With time they were able to measure the intrusion of water into the package through the epoxy seal. Tests were made with and without a chemical dryer. Without the SAES CaO chemical dryer, moisture inside the package reached 10 ppm in less than one day. With the SAES CaO chemical dryer, the moisture level inside the package was  $<1$  ppm after 21 days. The high surface activity CaO dryer developed by SAES was able to cope with the moisture intrusion through the epoxy

seals. Interestingly, the CaO also reacted with the CO<sub>2</sub> within the device keeping the CO<sub>2</sub> level to <1 ppm for 14 days.

### Using Getters

In order to use a getter to successfully maintain the ambient in a hermetically sealed device several steps need to be followed:

1. Identify the source and type of gas to be gettered.
2. Determine the rate at which the gasses desorb into the package and the total amount of gas to be pumped over the designed service life of the device.
3. Choose the type of getter to be used, insuring its compatibility with the device and processes used to manufacture and package the device.
4. Size the getter in order to have adequate capacity, plus a safety margin, in order to pump the expected gas load.
5. Integrate the getter into the package design inclusive of getter activation methods.

## 2.7 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 [15]

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:

$$q = q_0 t^{-\nu} \quad (8)$$

Where the time factor ( $\nu$ ) 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 desorbs by diffusion from the bulk of a material.

The quantity of gas released can be obtained by integrating equation 8 over the desired time period, t:

$$q = \int q_0 t^{-\nu} dt = q_0 \int t^{-\nu} dt \quad (9)$$

$$= q_0 \frac{t^{1-\nu}}{1-\nu} \quad \text{for } \nu \neq 1 \quad (10)$$

$$= q_0 \ln t \quad \text{for } \nu = 1 \quad (11)$$

$t_0$  is assumed to be 1 hour for these calculations as the instantaneous outgassing rate at  $t=0$  is impractical to measure.

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.

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. 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. This only worsens the situation. An additional parameter to keep in mind in doing these estimates is that part of the getter capacity can be used up during bake out. The getter quantity has to be suitably dimensioned in order to maintain a sufficient gettering capacity to cope with the outgassing load during lifetime.

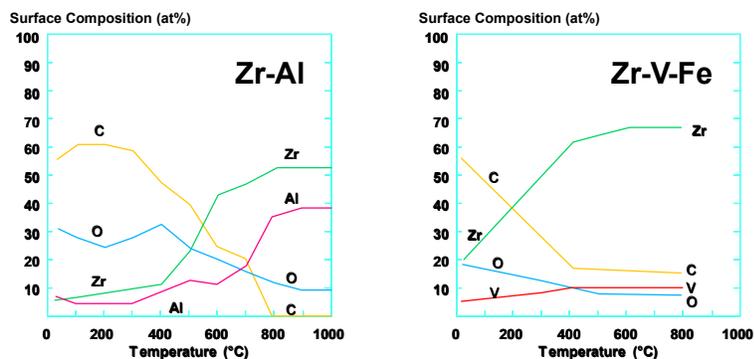
## 2.8 Sorption Performance

The capacity of a getter is defined by its sorption performance, i.e. how much and how fast can it pump various gases. Typically this data is presented in the form of a sorption curve with total mass pumped on the x-axis and pumping speed on the y-axis. These curves usually show performance for two gas species, CO and H<sub>2</sub>. These gases were chosen because they reflect the differing pumping mechanisms present. In addition CO reflects the average kinetics of the reaction of a gas on a getter surface. Most gas mixtures pump in the same speed range as CO, while pure oxygen will be much faster and pure nitrogen much slower. Tests to determine the sorption performance of a getter are typically performed per ASTM standard procedures. The standards are F 798 – 82 for NEG's and F 111 – 72 for barium getters.

## 2.9 Activation of NEG's

As delivered from the manufacturer a non-evaporable getter's (NEG) surface is passivated so that it may be safely handled in atmosphere on the factory floor. This passivation layer can consist of oxides, carbides, and nitrides. In order to make the getter ready to pump away gases in a vacuum space it must be activated. The activation process consists of heating the getter to an adequate temperature to cause the passivation layer to diffuse into the bulk of the grains of the getter alloy. This mechanism is of intense interest to getter researchers and the users as the necessary activation temperature and required time present process consequences and opportunities when engineering a vacuum dependent system.

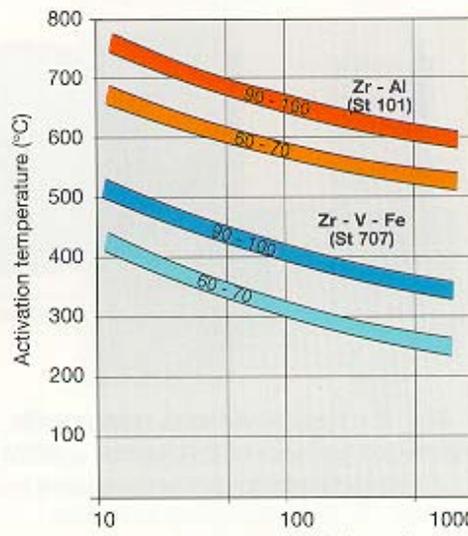
The activation mechanism has been discussed in the literature on the basis of theoretical considerations combined with the results of numerous sorption test results and mass-spectrometric analyses. The application of surface analysis techniques provides a clear and definitive interpretation of the mechanism as it appears from Figure 6. [19,20] The following curves prepared by Watanabe et al [21,22] demonstrate the compositional changes on NEG surfaces when subjected to heat:



**Figure 3:** Change in surface composition of Zr alloy getter materials when heated under a vacuum. (Courtesy of R. Kullberg, SAES)

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 of Zr and Al increases whereas O<sub>2</sub> and C concentration dramatically decreases. [19] These compositional changes clearly illustrate the diffusion of the passivation products into the getter bulk and the diffusion of fresh chemically active zirconium to the getter surface, thereby creating chemically active sorption sites on that surface.

The activation process is not only related to activation temperature but also to time of activation. However, it is typically more strongly dependent on temperature for a diffusion mechanism. [7,8,20,23] Many 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 4 shows the various degrees of activation obtained with different temperature/time combinations for two typical getters (Zr-V-Fe and Zr-Al).



**Figure 4:** Activation efficiency for different temperature/time combinations (in percent of full activation) [24,25]

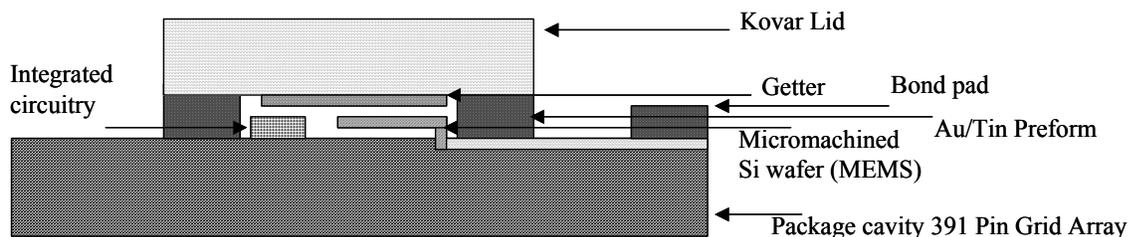
From an engineering perspective the time-temperature dependency of the activation process presents opportunities for process optimization. For example, a lower activation temperature can be used over a longer time period to protect temperature sensitive components. On the other hand, a getter with a higher activation temperature can be used when a process requires a relatively high temperature bake out. This allows the bake out to proceed without sacrificing the getter as an *in-situ* pump during the bake out.

### 3. High Vacuum Hermetic Package Assembly Processes and Evaluation of Non-Evaporable Getters (NEGs) (R. Ramesham)

#### 3.1 Background

The vacuum level in the electronics package assembly can directly affect MEMS device performance. The level of vacuum in an hermetically sealed advanced electronic package of  $10^{-4}$  to  $10^{-5}$  Torr pressure will be of significant use for the MEMS device performance and their functionality. Activation of the getter is a part of the package assembly process, which controls the ambient and the pressure inside the package to improve the quality, reliability, and functionality of the MEMS device for long-term space mission applications. [1-3,26-30] The three main functions of MEMS packages/assemblies are mechanical support, protection from the environment, and electrical connection to the other system components. A survey has been made for a variety of commercial-off-the-shelf (COTS) electronic packages during the initial stages of the research work and constantly there after. Several types of advanced packages were procured for the assembly and evaluation of non-evaporable getters (NEGs) for various JPL/NASA projects. This project was de-scoped due to the funding reduction from 110k to 45k by the NEPP program and project management. Therefore, this task has been scoped to evaluate only one type of advanced electronic package that is of primary significance to packaging of JPL's high profile technically visible micro and mesogyro project for NASA's navigational applications.

Conventional anodic bonding process produces internal cavity pressures in the range of 100 – 400 Torr range [31] while glass frit and solder sealing process produces cavity pressures of 1-2 Torr [32,33]. A package cavity pressure of 1.4 Torr was obtained with glass frit sealing due to squeeze-film damping. This pressure increases as the aging of the package increases. One technically possible solution to this serious problem is the incorporation of a getter to absorb the trapped and desorbed gas components left in the package micro cavity. Esahi and others [31, 34] first applied getters to MEMS devices during the 1990's. Non-evaporable getters (NEGs) were used in the package cavity of a ceramic package to maintain the internal vacuum. The NEGs were thermally activated through annealing the complete package with the getter or by Joule heating. The high temperature activation process step in a high vacuum or hydrogen-containing reducing ambient is strictly required to remove the inherent surface oxide layer that was formed during the high temperature sintering process. Particles of 2 to 3 microns may be generated during cutting of getters. These particles may cause electrical shorts, impede motion and shift resonant frequencies of the MEMS microstructure components inside the cavity.[31,34,35] To overcome the drawbacks to NEGs a new technical approach to MEMS gettering was developed by Spark et al. [35] Figure 5 shows the schematic cross-sectional illustration of the 391-pin grid array package that has been chosen in this study to evaluate the non-evaporable and nanogetters for the future JPL/NASA application.



**Figure 5:** Cross-sectional illustration of the vacuum packaging with a getter (not to scale)

Commercially available 391 ceramic pin grid array cavities with the kovar lids were procured. Usually there will be a Au/Tin preform attached to the lid. A Au/Tin preform will melt around  $\sim 280^{\circ}\text{C}$ . The activation of the getter attached to the lid is performed at  $400^{\circ}\text{C}$  for 7 minutes. Apparently, there is a process incompatibility during the assembly process of lid, preform, and the cavity. Therefore, we have procured the package cavities and the lids without preforms. We have procured the freestanding preforms and tac/spot/laser/ welded the preform to the package cavity at the seal ring region. The lids were procured without Au/Sn preforms. The lid side, oriented towards the cavity, was patterned with deposited nanogetter at ISSYS Inc. The NanoGetter is comprised of a proprietary; patent pending, multilayer structure and the film layers are in the range of  $50 - 5000\text{\AA}$  thickness range. We have also procured micromachined resonators and the associated electronics to monitor the resonance frequency of the resonator as a function of vacuum or to monitor the vacuum level in a sealed electronic package. A prerequisite to evaluating the NEG getters is to hermetically high vacuum seal the electronic package. The resonator and the corresponding electronics were mounted in the cavity using vacuum compatible high vacuum epoxy. We used the vacuum compatible epoxy instead of Au/Tin alloy solder attachment due to the lack of funds and the lack of time to complete the task and its objectives. Au/Tin alloy will be tried in the future when the funds are available. The surface morphology of the NanoGetter is shown in figure 2 as per ISSYS Inc. The thin film deposition method enhances the ability to easily integrate the NanoGetter into a typical MEMS process flow. [35]

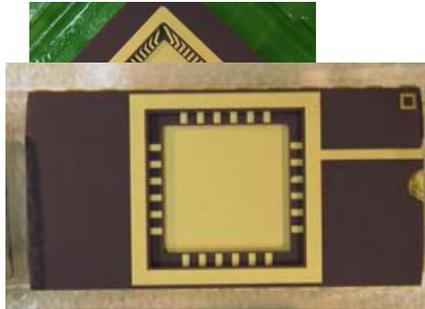
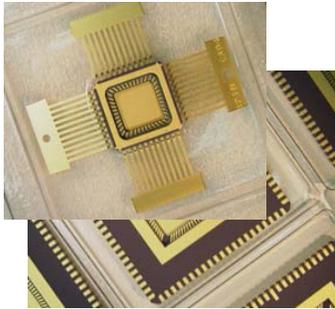
Optimizing the assembly and manufacturing of MEMS packages has placed an important focus on quality and reliability of MEMS performance. For a package to survive and perform flawlessly in a hostile environment, materials and processes will require better controls. Optical switching is becoming more popular where light is routed directly using microoptoelectromechanical systems (MOEMS). Packaging of these devices is becoming more complex because of the need to protect the devices from environments, etc. When designing soldering into an assembly, four important factors must be taken into consideration; materials, physical, process compatibility, and cost-effectiveness. Soldering has good thermal and electrical conductivity and requires low energy for application and the resulting joints are impermeable to gases and liquids.[36,37] The primary requirement for soldering is that the metal surfaces to be joined are in a solderable condition. If the surface is not clean and solderable, the molten solder will not flow properly resulting in non-wetting, dewetting, and voiding.[36,37]

Surface contamination or trapped gas can be the cause for some defects, which will reduce the reliability of the package and the device.[36,37] Voids generated during the bonding process will increase the device operating temperatures and weaken the bond area, directly reducing the reliability of the package. Co-efficient of thermal expansion (CTE) of the materials must all be compatible to prevent cracking during heating and cooling by minimizing the effect of the thermal stress.[36,37] Metallurgical incompatibility of materials and processing conditions may manifest through poor wetting, excessive erosion, and formation of undesirable phases.[38]

The first step in the assembly process is to mount MEMS components and the associated electronics to the cavity using a eutectic solder (Gold/Tin), which reflows at  $280^{\circ}\text{C}$ . As the solder is heated to its melting point the liquefied solder then penetrates both bonding surfaces. An intermetallic bond develops, which is also known as wetting. Tests were performed in which the components were processed in a vacuum. There are two copper electrodes extending into the chamber that provide a platform for fixturing.[36,37]

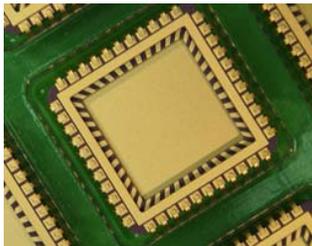
The fixturing is mostly comprised of a graphite material, which is configured as a holding/locating device as well as the heat source. The fixture is placed on the electrodes and an electrical current is passed from the electrode to the graphite via a transformer and phase fired controlled power pack. A thermocouple was fixed to the graphite interfacing with the controller to provide accurate ramp rates, dwell times, and cool downs. The graphite resistive element is heated; precise control of the temperature profile is accomplished. Graphite was chosen because it is inexpensive, easy to machine, a good thermal conductor and is not wetted by the majority of molten alloys. Graphite also has the merit of “mopping up” oxygen in an oxidizing atmosphere to form CO and CO<sub>2</sub> [36-38]. The graphite selected was of a semiconductor grade where the properties are superior to conventionally used graphite materials. The material is isotropic, densely packed with small uniform grains and has a coefficient of thermal expansion closely matching the majority of materials chosen to manufacture the MEMS assembly.

**3.2. Potential advanced electronic packages:** We have procured the following packages (Figure 6) for the study to assess the package reliability with NEG's that are hermetically sealed.



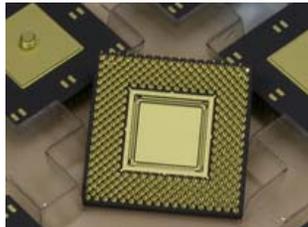
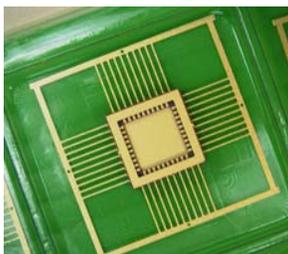
**Cerquad base 44 lead package    64 Pin Ceramic Pin Grid Array (PGA)**

**84 Pin Lead Less Chip Carrier (LCC)    Side braze 24 lead package**



**44 J-leaded chip carrier**

**Transistor Outline (TO) – 10 Lead package**

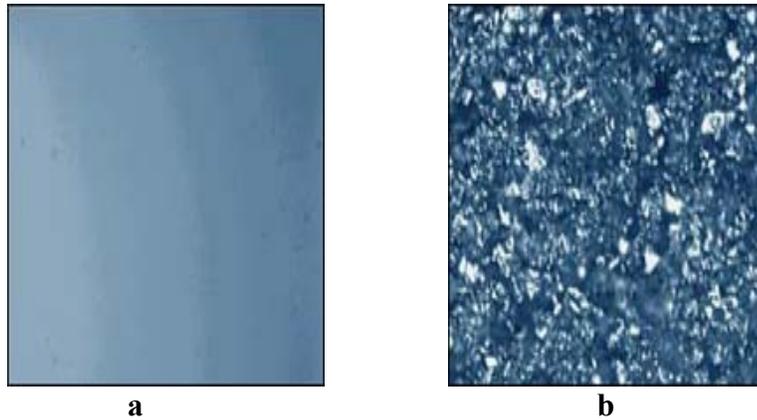


**Flat pack 40 leaded chip carrier    391 Ceramic Pin Grid Array**

**Figure 6:** Optical photographs of the advanced packages chosen to evaluate the getters for future NASA applications.

### 3.3. Types of non-evaporable getters

There are two types of non-evaporable getters that were chosen in this study. One is nanogetters (figure 7) that are procured from Integrated Sensing Systems Inc., and the other is sintered non-evaporable getters procured from the SAES Getters Inc. (Figure 1)



**Figure 7: Optical photomicrographs of an NEG surface and a thin film NanoGetter surface.** (Courtesy of D. Sparks, ISSYS)

### 3.4 Preforms

Clean, dry, Gold/Tin (Au/Sn) solder preforms (Figure 8) are attached to the necessary components of the assembly using either tack welding, spot welding, laser welding, or direct soldering operation.



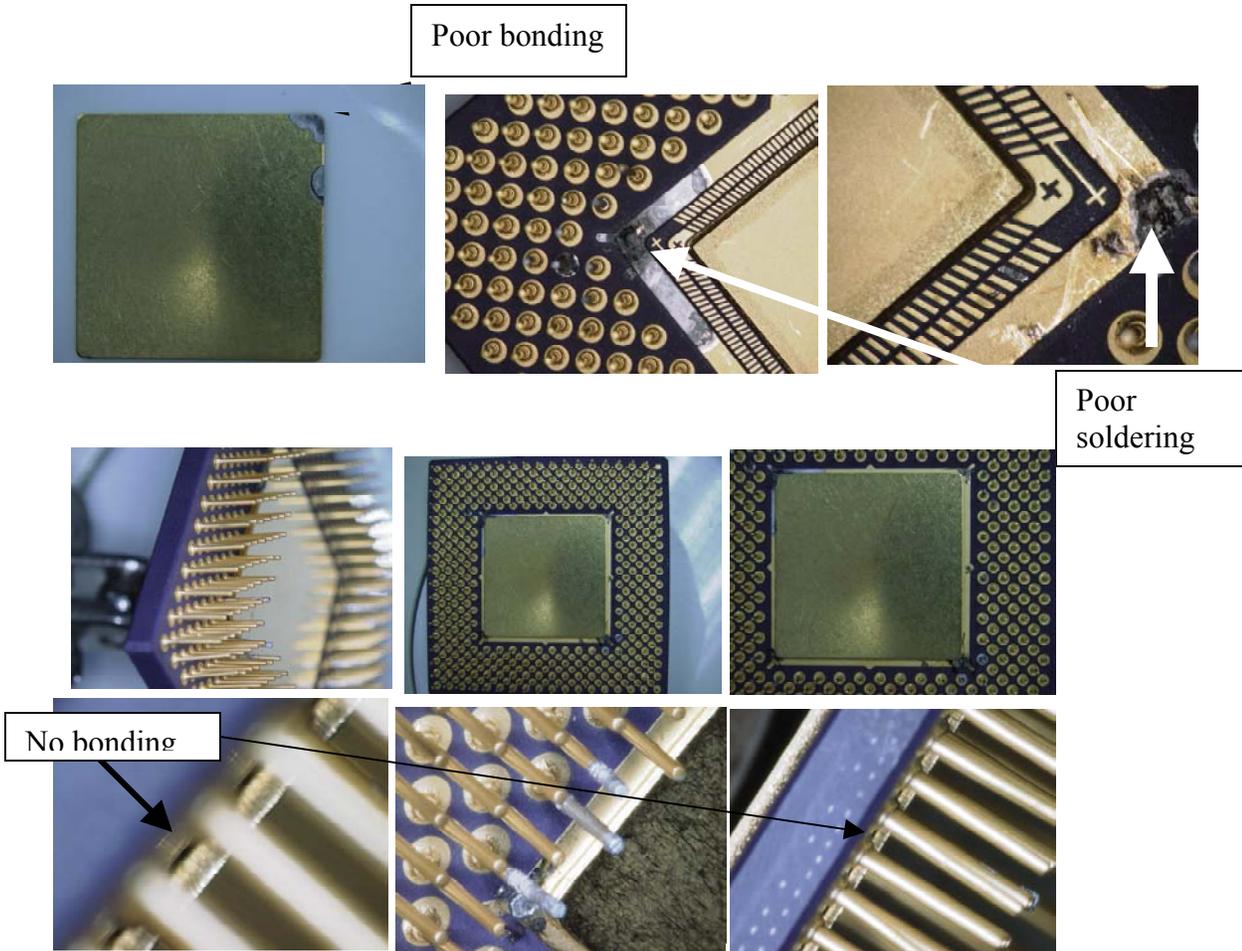
**Figure 8: Free standing Au/Sn preform to use with 391 pin grid array (PGA) packages.**

## 3.5 Experimental Results and Discussion

### 3.5.1 Manual soldering

The Au/Sn preform was attached manually with a soldering iron, having the smallest soldering tip available in the lab at SST international Inc. This resulted in poor bonding. Figure 9 show the optical photographs of the soldered area of the preform over the package seal ring after bonding in the vacuum. We were able to take the lid off of the package using only a forceps. The team has tried over 20 experimental runs (varying various parameters) to bond the cavity to the lid since this is a critical step to package a getter and a gyro in a commercial package. The team has tried to evaluate the Au/Sn attachment options and employed laser

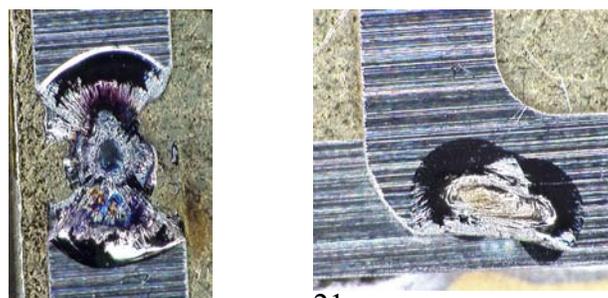
welding to attach Au/Sn preforms to the seal ring of the package cavity. No x-ray imaging was performed since the poor bonding was seen using optical microscope. No solder was observed along the edge of the lid. Solder was completely melted and crept over the electrical array leads of the advanced electronic package due to gravity, surface tension, wetting angle, mounting fixture (facing down in the vacuum chamber) and also the temperature that is beyond the melting point of the preform.



**Figure 9:** Optical photographs of the area of soldering using preform and bonding.

### 3.5.2 Laser welding

The melting point of the preform (Au/Sn) is substantially lower than that of gold (Au), which is present over the seal ring of the cavity. It was not very difficult to laser weld the preform to the seal ring. Laser welding has resulted in an outcome that could lead to a potential fault in the Au/Sn reflow. A laser welded spot may be the potential site for leaking in the assembled package after sealing. Figure 10 show the optical photographs of the laser welding of the preform to the seal ring of the cavity.





- Load the packages into the graphite fixture
- Load the graphite fixture into the vacuum chamber
- Insert the thermocouple to control the temperature profile and close the lid
- Program the temperature profile (after optimizing)
- Pump down or evacuate the chamber using turbo pump and mechanical pumps
- Backfill and purge the vacuum chamber with inert gas
- Evacuate the vacuum chamber and turn on the temperature/heat profile to pre-bake the fixture with package components below the eutectic point of the preform (Au/Sn)
- Turn off the vacuum and spike the chamber with inert gas, still on the vacuum side of atmosphere, to enhance the thermal conductivity.
- Ramp to final reflow temperature and held for desired time.
- Backfill with final gas pressure to 60 psig while solder is molten to eliminate voids.
- Turn off heat during the cool down process.

### 3.6 Processing aspects

Consideration of the overall process and practicality of the several issues determine the best methods for successfully assembling the MEMS resonator package. Figures 13 and 14 are the scanned copies of the experimental run and temperature profile of the successful bonding experiment.

HR	MIN	SEC	TEMP	EVENT	STATUS		
PROFILE NUMBER:	4						
GAIN1:	30						
GAIN2:	50						
HR	0	MIN	0	SEC	1	LIFT	UP
HR	0	MIN	0	SEC	5	VAC	ON
HR	0	MIN	2	SEC	0	VAC	OFF
HR	0	MIN	2	SEC	5	GAS1	ON
HR	0	MIN	2	SEC	20	EXH	ON
HR	0	MIN	3	SEC	20	GAS1	OFF
HR	0	MIN	3	SEC	25	EXH	OFF
HR	0	MIN	3	SEC	30	VAC	ON
HR	0	MIN	13	SEC	0	HEAT	ON
HR	0	MIN	15	SEC	0	260	
HR	0	MIN	45	SEC	0	260	
HR	1	MIN	15	SEC	0	260	
HR	1	MIN	45	SEC	0	260	
HR	2	MIN	12	SEC	0	LIFT	DOWN
HR	2	MIN	15	SEC	0	260	
HR	2	MIN	16	SEC	0	370	
HR	2	MIN	23	SEC	0	370	
HR	2	MIN	23	SEC	1	HEAT	OFF
HR	2	MIN	33	SEC	0	VAC	OFF
HR	2	MIN	33	SEC	10	GAS1	ON
HR	2	MIN	33	SEC	20	EXH	ON
HR	2	MIN	45	SEC	0	GAS1	OFF
HR	2	MIN	45	SEC	5	EXH	OFF

Figure 13: Scanned copy of the experimental run of the successful bonding process.

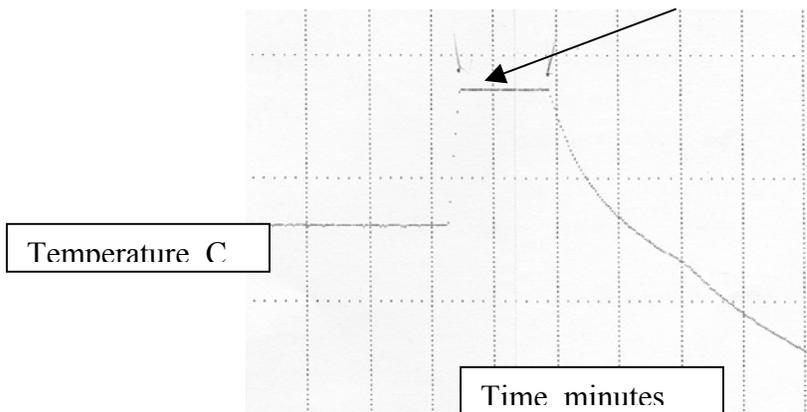
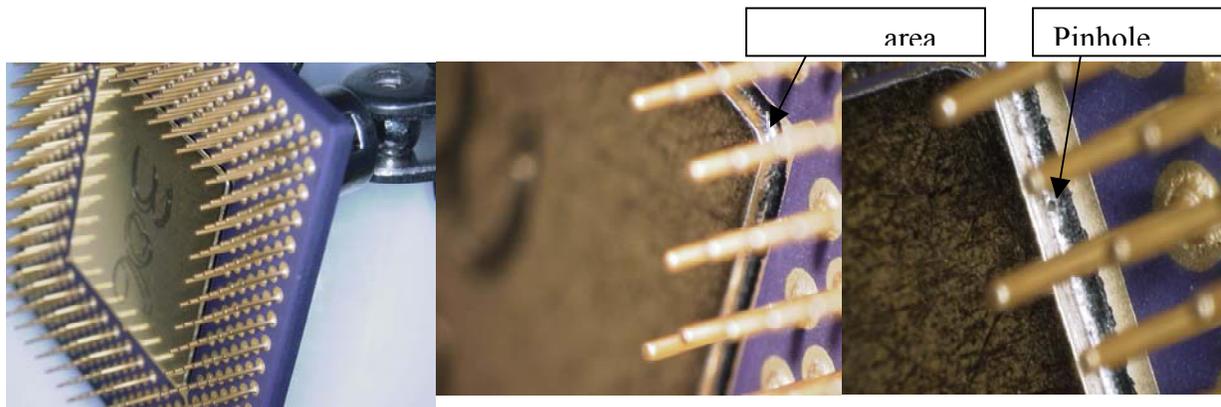


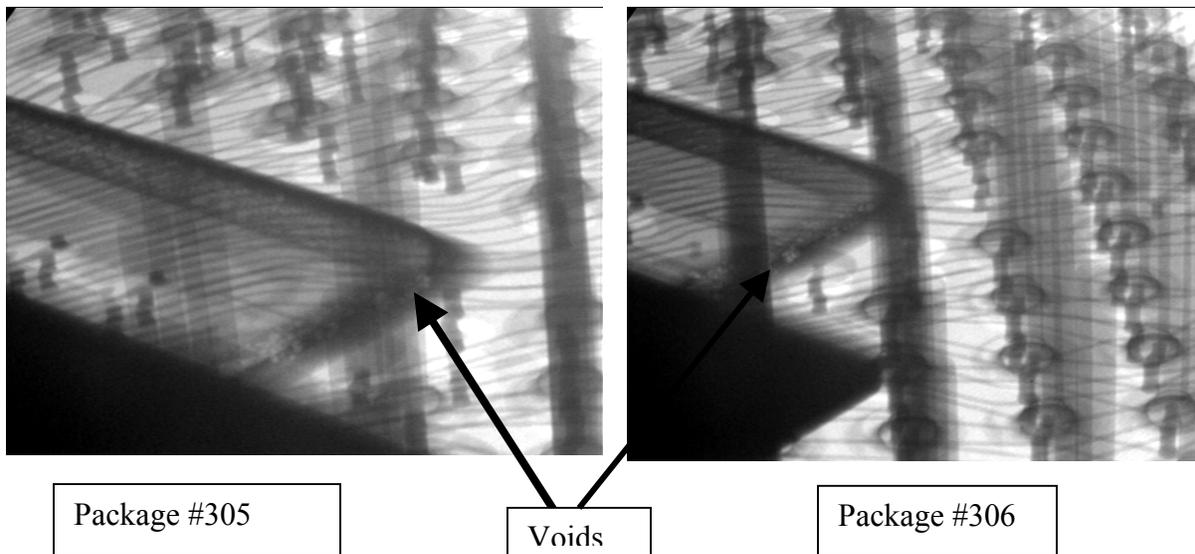
Figure 14: Temperature profile

### 3.7 Non-destructive evaluation (NDE) of sealed packages

Figure 15 shows the optical photographs of the sealed 391 PGA package without a resonator and associated electronics. There were six packages with resonators and associated electronics assembled inside the cavity. Optimization of the sealing process in terms of temperature profile is a very important step to achieve the reflow conditions inside the vacuum of  $10^{-6}$  Torr pressure to result in a high vacuum hermetic sealing. This temperature profile also includes an activation of the nonevaporable getter to maintain the vacuum after the package is vacuum-sealed. Several test runs (over 24) were made with packages without NEG getter, resonator, and associated electronics to save funds and the effort. In the last test run, the sealing was good and Helium leak bomb tests showed they passed the fine leak and gross leak tests. We have observed some pinholes in the bonded area. One can minimize the pinholes by cleaning the package with selective gas plasma to reduce the instantaneous surface oxides formed in the sealing bond area. We have inspected the packages with x-ray imaging equipment at JPL. Figure 16 show the x-ray images of sealed packages of #305 and #306. There were several voids in the sealing/bonding area that were revealed as a result of x-ray imaging. These voids could be stress concentrators in a long run and eventually decrease the reliability sealed packages. Plasma treatments could improve the sealing process by reducing the outgassing and oxides on the seal ring area and subsequently reduce the voids and increase reliability.



**Figure 15: Optical photographs of the sealed 391 PGA package without a resonator and associated electronics**



## Figure 16: X-ray images of hermetically vacuum-sealed 391 PGA packages (#305 and #306)

### 3.8 Leak testing (MIL-STD-883E, Method 1014.9)

The purpose of this test is to determine the effectiveness (hermeticity) of the seal of the advanced pin grid array package for eventual getter evaluation and MEMS gyro device evaluation and packaging assessment. Fine and gross leak tests were conducted in accordance with the requirements and procedures described in the mil-spec at JPL in the failure analysis (FA) laboratory. Testing order was fine leak followed by gross leak tests as per the mil standard. The packages were placed in a sealed chamber, which is then pressurized with a tracer gas of Helium for the required time and pressure. The Helium, FC-72 vessel Pressure was 60 PSIG. The packages were left for a minimum time in pressurized vessel of 2 hours. The pressure was then relieved and each package was transferred to another chamber, which were connected to the evacuating system and a mass spectrometer type leak detector. The chamber was evacuated; any tracer gas, which was previously forced into the assembled package specimen is leaking will thus be drawn out and will be indicated by the leak detector as a measured leak rate. The approximate calculated volume of the cavity of the package is about 1.3 cc. The rejection limit for this volume of 1.3 cc is about  $2 \times 10^{-7}$  atm cc/sec He. (determined using variable method as per my discussion with Jim Okuno)

The packages were placed in a vacuum/pressure chamber and the pressure reduced to 5 Torr and maintained for 30 minutes minimum. A sufficient amount of type I detector fluid was admitted to cover the devices. The fluid was admitted after minimum 30 minute period but before breaking the vacuum and the packages were then pressurized. When the pressurization period was complete the pressure was released and the packages were removed from the chamber without being removed from a bath of detector fluid for more than 20 seconds. When the packages were removed from the bath they were dried for  $2 \pm 1$  minutes in air prior to immersion in type II indicator fluid, which was maintained at  $125^{\circ}\text{C} \pm 5^{\circ}\text{C}$ . The packages were immersed with the uppermost portion at a minimum depth of 2 inches below the surface of the indicator fluid one package at a time. The package was observed against a dull, nonreflective black background through the magnifier, while illuminated by the lighting source, from the instant of immersion until expiration of a 30-second minimum observation period.

Figure 17 show the equipment used in evaluating the high vacuum hermetically sealed packages for fine leak and gross leak testing, and also the mass spectrometer facility.

#### 3.8.1 Tracer gas helium (He) fine leak test

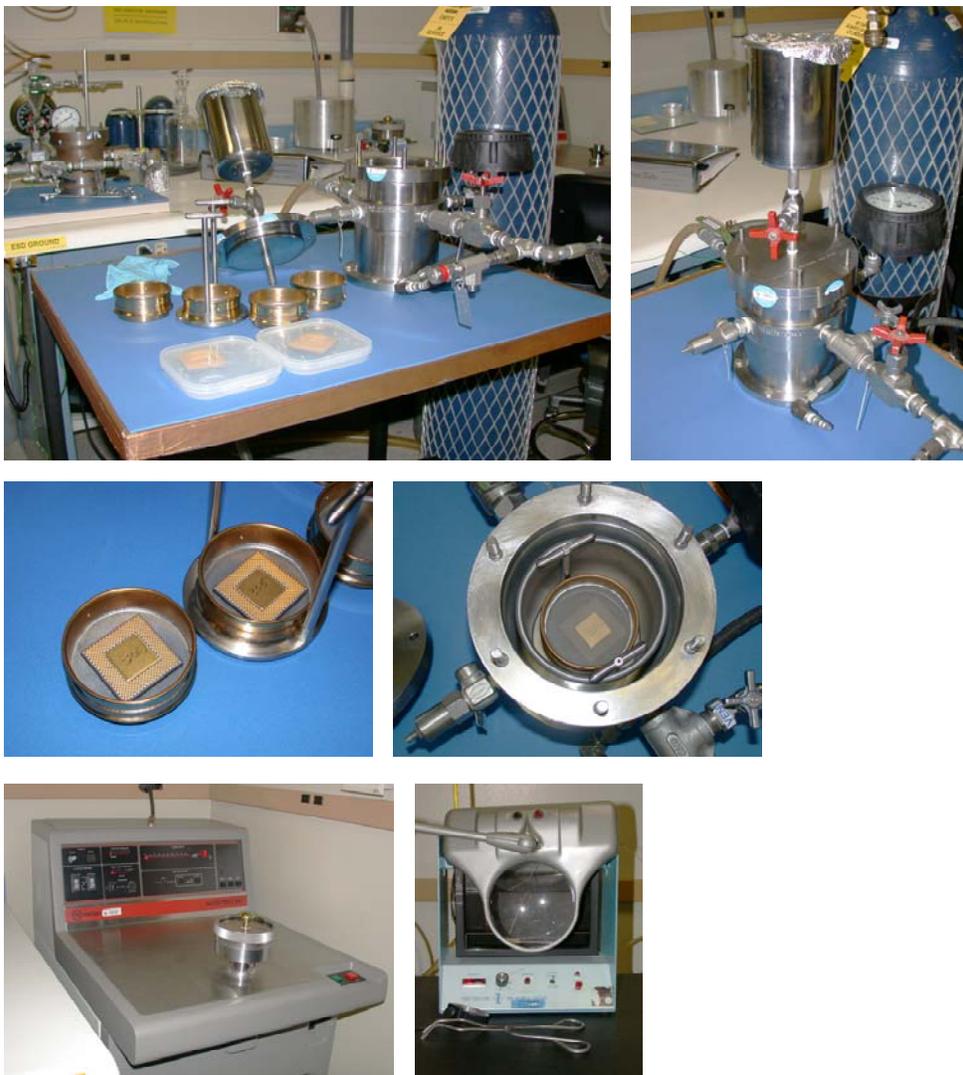
The apparatus consists of suitable pressure and vacuum chamber and mass spectrometer-type leak detector present and properly calibrated for a helium leak rate sensitivity sufficient to read measured helium leak rates of  $10^{-9}$  atm cc/s and greater.

- ❖ The observed leak rate for the package (#305) was  $6.6 \times 10^{-8}$  atm cc/sec He tested as soon as the package was taken out of the pressure vessel and monitored the leak rate with a mass spectrometer leak detector. When the same package was measured after a few minutes the leak rate was  $4 \times 10^{-8}$  atm cc/s He. As per the first and second measurements the package passed the fine leak test.

- ❖ The observed leak rate for package (#306) was  $4.4 \times 10^{-8}$  atm cc/sec He as soon as the package was taken out of the pressure vessel. As per the first measurement the package passed the fine leak test.

### 3.8.2 Perfluorocarbon (FC-40) gross leak

A vacuum chamber was used for the evaluation and subsequent bombing the package up to 105 psi. A suitable observation container provided provisions to maintain the indicator fluid at a temperature of 125°C under illuminated conditions. A magnifier with a magnification in the range of 1.5X to 30X allowed for observation of bubbles emanating from packages when immersed in the indicator fluid. Detector fluids were used as per MIL-STD-883 (perfluorocarbon containing no chlorine and hydrogen). As per gross leak experimental testing results packages #305 and #306 have passed the gross leak testing.

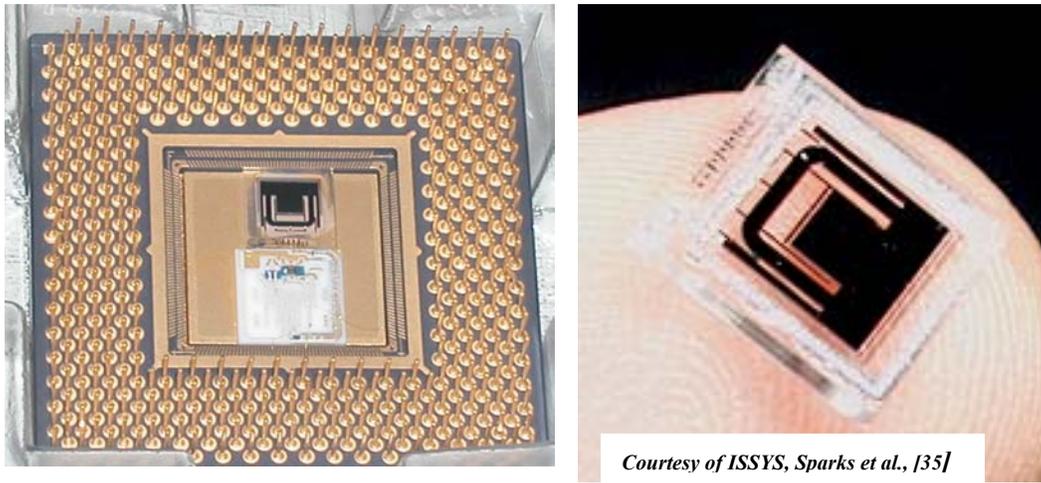


**Figure 17:** Optical photographs of the equipment used in evaluating the high vacuum hermetically sealed packages for fine leak and gross leak

testing, and also the mass spectrometer facility.

### 3.9 Packaging of resonator with its electronics to monitor vacuum

Figure 18 show the 391 PGA assembled with a resonator and associated electronics to monitor the resonator characteristics in the vacuum of the hermetically vacuum-sealed package. Resonator characteristics were monitored in a vacuum chamber at a 1 mTorr pressure. The results are shown in Table 4. Due to the lack of resources, we have not continued the work to seal the resonators with electronics to determine the functionality of the resonators and the functionality of the getters after sealing. We have not performed any thermal cycling on the sealing reliability for long-term reliability assessment. These will be addressed when the funds are available in the future either under NEPP or another funding umbrella.



**Figure 18:** Optical photographs of the PGA assembled with a resonator and electronics.

Device#	1	2	3	4	5	6
Resonant Frequency, Hz	9438	8820	8018	6810	8036	9385
Gain, db	-15	6.7	-1.4	6.16	-1.4	-5
Bias, V	5	2	5	5	5	5
Q	3500	3835	2970	6810	4230	3910

**Table 4:** Experimental test data (Q factor etc.) of the resonators in a vacuum

## 4.0 Conclusions and Future Activities

### 4.1. Conclusions

- The team has successfully assessed a process to high vacuum hermetic seal large 391 advanced pin grid array packages for the first time.

- Identified a potential advanced package to package a JPL developed gyro to infuse this technology for future JPL and NASA navigation applications.
- Identified a problem area and rectified the process by changing the protocol of the sealing processes. This protocol is compatible with the getter activation process.
- Assessed and developed a temperature profile to seal large commercial packages.
- Optical inspection revealed good sealing resulted during the test runs.
- X-ray imaging showed there are several pinholes in the sealed area.
- The team used Au/Sn preforms to seal the package robustly.
- Attached successfully the Au/Sn preforms over the seal ring of the package cavity rather than lid due to the process incompatibility.
- Incorporated a step to activate the getter during the temperature profile development in a high vacuum environment.
- Identified nanogetters and non-evaporable getters to package JPL developed gyro.
- Helium leak bomb test results showed the sealing was very good. Both the packages have passed fine and gross leak tests as per military specifications.
- Assembled resonators and electronics in to the 391 PGA cavity and monitored/measured the resonator characteristics in vacuum.
- Leveraged funds with other NASA projects at JPL.

#### 4.2 Future Activities

- Develop temperature profiles for a variety of advanced packages of various thermal mass and size to achieve high vacuum hermetic seal.
- Seal the 391 PGA containing the resonator and electronics and measure the resonator characteristics in vacuum after hermetically sealed. Assess the leak rate using the He Leak bomb test and correlate with resonator data.
- Seal the 391 PGA containing the JPL gyro and measure the resonator characteristics. Assess the leak rate using He Leak bomb test and correlate with resonator data.
- Perform fine and gross leak tests after the resonators are packaged.
- Develop the process to attach the resonator using inorganic solid-state solder instead of high vacuum compatible organic materials.
- Life testing of sealing and resonator using thermal cycling and NDE analysis.
- Improve the process by incorporating hydrogen and argon plasma treatment to decrease the voids in the sealing area.
- Assess the activation of a non-getters and non-evaporable getters since we have achieved a reasonably good sealing process.
- Employ optical methods to non-destructively evaluate robustness of the sealed packages.
- The thrust of technology into micro and nano systems continues unabated. So does the push to do things ever cheaper. A key to success for these systems is good packaging, yet such packaging costs money. Future research in the area of economical packaging and getters to maintain the ambient in the packages will be key to the commercial success of micro and nanosystems that require controlled ambient.

#### 5.0 References

1. R. Ramesham, JPL D 17920, “Getters for Reliable Hermetic Packages’ 1999

2. R. Ramesham R. Ghaffarian, and N.P. Kim, "Reliability issues of COTS MEMS for Aerospace Applications", Proceedings of the International Society for Optical Engineering (SPIE), Editors: R.A. Lawton, W.M. Miller, G. Lin, and R. Ramesham, Vol 3880, pp. 83-88, 1999.
3. R. Ramesham and R. Ghaffarian "Challenges in Interconnection and Packaging of Microelectromechanical Systems (MEMS)", **Proceedings of 50th Electronic Components and Technology Conference** Proceedings (IEEE, ECTC-2000).
4. B. Ferrario, Foundations of Vacuum Science and Technology, edited by J.M. Lafferty, J. Wiley and Sons, 1998, Chapter 5.
5. C. O'Neal, A. Malshe, S. Singh, and W.D. Brown, "Challenges in the packaging of MEMS", Proc. of the International Symposium on Advanced Materials, pp. 41-47 (1999).
6. M. Esashi, N. Ura, and Y. Matsumoto, Anodic bonding for integrated capacitive sensors, Proc. MEMS '92, Travenmunde, Germany, Feb. 4-7, 1992, pp. 43-48
7. K. Ichimura, et al., J. Vac. Sci. Technol., A5 (2) (1987) 220.
8. K. Ichimura, M. Matsuyama, and K. Watanabe, "Alloying effect on the activation processes of Zr-alloy getters," J. Vac. Sci. Technol. A5, pp. 220-225 (1987).
9. Hal Greenhouse, "Hermeticity of Electronic Packages," William Andrew Publishing Norwich, NY, USA/Noyes Publishing (np), Park Ridge, NJ, USA. 1999
10. S. Kayali and D. Ragle, "Hydrogen Effects on GaAs Microwave Semiconductors," JPL D -14765, June 18, 1997, 35 Pages.
11. N.T.M. Dennis and T.A. Heppell, High Vacuum Series, L. Holland, Vacuum System Design, Edited by N.T.M. Dennis and T.A. Heppell, Chapman and Hall, London, 1968.
12. Y. Saito, R. Griese, J. Kessler, R. Kono, and J. Fang, IEEE 1995 Microwave and Millimeter-Wave Monolithic Circuits Symposium, pp. 119-122.
13. J. Verhoeven and H. Vandoveren "Interactions Of Residual Gases With A Barium Getter Film As Measured By AES And XPS" Journal Of Vacuum Science & Technology 20 (1): 64-74 1982
14. R.C. Kullberg, "Processes and materials for creating and maintaining reliable vacuum and other controlled atmospheres in hermetically sealed MEMS packages," SPIE Proceedings Vol. 3880, 1999 pp.75-82.
15. Corrazza and R.C. Kullberg, "Vacuum maintenance in hermetically sealed MEMS packages," Micromachined Devices and Components IV, P.J. French and K. Chau, Vol 3514, pp. 82086, SPIE, Bellingham 1998.
16. M. Moraja, M. Amiotti, and R.C. Kullberg, "New getter configuration at wafer level for assuring long term stability of MEMS" SPIE Proceedings Vol. in print, 2003 pp.260-267.
17. U.S. Department of Defense, [http://www.dsccl.dla.mil/Downloads/MilSpec/Docs/MIL-STD-883/std883\\_1000.pdf](http://www.dsccl.dla.mil/Downloads/MilSpec/Docs/MIL-STD-883/std883_1000.pdf)
18. S. Tominetti and A. Renzo, Determination of Impurities in Organic Light Emitting Diode Displays Using A Dedicated Mass Spectroscopic Technique, SID 01 Digest, p 660. 2001
19. M. Sancrotti , G. Trezzi, and P. Manini, "An X-Ray Photoemission Spectroscopy Investigation Of Thermal-Activation Induced Changes In Surface-Composition And Chemical-Bonds Of 2 Gettering Alloys - Zr<sub>2</sub>Fe Versus Zr<sub>0.57</sub>V<sub>0.36</sub>Fe<sub>0.7</sub>" Journal Of Vacuum Science & Technology A-Vacuum Surfaces And Films 9 (2): 182-189 Mar-Apr 1991 and G. Sancrotti, G. Trezzi, P. Manini, J. Vac. Sci. Technol., A9 (2) (1991) 187.
20. Barosi, Proc. IV Int. Symp on Residual Gases in Electron Tubes Florence, 1971 – Academic Press (1972) 143.
21. K. Watanabe and K. Ichimura, K. Ashida, Fusion Technology, Vol. 14, Sept. 1988, pp. 729.

22. M. Watanabe, C. Takeda, S. Tada, H. Anada, S. Ikenu, K. Ashida, and K. Watanabe, *Fusion Technology*, 21 (1992) pp. 763-768.
23. J.L. Cecchi, *J. Nucl. Mat.*, 111-112 (1982) 305.
24. R.J. Knize and J.L. Cecchi, *Fusion Technology*, Vol. 14, Sept. 1988, pp. 503.
25. R.J. Knize and J.L. Cecchi, *J. Appl. Phys.*, 54(6), June 1983, pp. 3183.
26. R.M. Caloi and C. Caretti, "Getters and gettering in plasma display panles," *J. Vac. Sci. and Tech.* A16, pp. 1991-1997, 1998.
27. F. Sciuccati et al., "*In-situ* pumping with NEG during vacuum processing", *Vacuum*, 38, pp. 765-769, 1988.
28. S. J. Kwon et al., "Influence of getter activation and aging in a frit-sealed emission display panel", *Journal of vacuum Sci. and Tech.*, B 18, pp. 1227-1231, 2000.
29. C. Benvenuti and P. Chiggiato, "Pumping characteristics of the St707 NEG", *J. Vac. Sci. Tech*, A14, pp3278-3282, 1996.
30. B. Ferrario, "Use of getters in vacuum systems", *Vacuum* 37, pp. 375-377, 1987.
31. H. Henmi, S. Shoji, Y. Shoji, K. Yoshimi, and M. Esahi, "Vacuum packaging for microsensors by glass-silicon anodic bonding," *Sensors and materials A*, 43, 1994, 243.
32. D. Sparks et al., "Chip-sclae packaging of a gyroscope using wafer bonding," *Sensors and Materials*, Vol. 11, 1999, 197.
33. D. Sparks et al., "Wafer-wafer bonding of nonplanarized MEMS surfaces using solder," *J. Micromech. & Microengr.* Vol. 11, No. 6, 2001, 630.
34. M. Esahi, et al., "Vacuum-sealed silicon micromachined pressure sensors," *Proc. IEEE* Vol. 86, No.8, 1998, 1627.
35. D. Sparks, R. Smith, M. Straayer, J. Cripe, R. Schneider, S. Massoud-Ansari, N. Najafi, *Transducers'03*, 12<sup>th</sup> International conference on solid state sensors, actuators and Microsystems, Boston, June 8-12, 2003.
36. P.W. Barnes, "Flux-free and void-free fiber optic packaging assembly process" Presented at the Optoelectronic Packaging Workshop, IMAPS, October 2002.
37. G. Pangelina, "MEMS production and yield improvement using a flux-free/void free assembly process", in "Reliability, Testing, and Characterization of MEMS/MOEMS II", edited by R. Ramesham and D.M. Tanner, pp. 213-219, 2003.
38. G. Humpston and D. Jacobson, "Principles of soldering and brazing" ASM International First Printing, Materials Park, Chapter 4, (a) p. 114, (b) p. 21, 1993.

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**Dr. Rajeshuni Ramesham** is a Member Engineering Staff at the Jet Propulsion Laboratory, California Institute of Technology, Pasadena, California. 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. Before joining JPL, Dr. Rameshan spent 9 years at Auburn University, Auburn, Alabama. His research work at Auburn University addressed the fundamental issues involved in diamond processing techniques, heat dissipation techniques, and electrochemical applications of diamond.

Dr. Rameshan has received many awards for his work. These include an outstanding research performance award from the Electrical Engineering Department of Auburn University and 7 awards from NASA for the research work he performed. He has published over 100 refereed journal and proceedings articles and has made 80 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 chair for Microelectromechanical activities at the SPIE's Micromachining and Microfabrication Conference and International Conference on Mechanics in Medicine and Biology. Dr. Ramesham received his Ph.D from the Indian Institute of Science, Bangalore, India.

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