

## Characterization of Die Attach Adhesives to Identify Contamination Source Present in MEMS Packages

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### Abstract

*Die in hermetic packages are commonly bonded with filled cyanate ester adhesives. These quick-curing, single part adhesives have low-outgassing properties during cure and are approved for high-reliability military products. However, recent investigations at Sandia National Laboratories found that some packages containing MEMS devices, bonded with these adhesives, were rendered useless due to the presence of condensed outgassing products.*

*To characterize the observed contaminants that result from the curing process and adhesives used, Sandia has developed a novel tool for destructively sampling the volatile organic compounds in the packages. A package is sealed in a low-volume, leak tested sampling manifold and is then cracked under static vacuum to release the internal gases. The gas sample is collected, concentrated with a cryo-focuser and introduced into a gas chromatograph interfaced to a mass spectrometer for analysis. In addition to volatile organic compounds, permanent gasses (e.g., nitrogen, oxygen, argon, hydrogen) can be analyzed using a residual gas analyzer. This technique provides useful information for determining hermeticity of the packages by monitoring the pressure of the gas sample. This sample collection technique has been demonstrated on packages ranging in volume from 30 nanoliters to large packages with volumes in the tenths of milliliters.*

*Using this tool, modifications to the cure process (or materials) can be made which can increase the reliability of the MEMS devices. Examples of how this technique has been employed will be discussed including how it has been used to evaluate commercial packaged die (non-MEMS) to comparatively understand cure products left in the hermetic packages after cure and lidding.*

Key words: Mass spectrometry, die adhesives, contaminant characterization, hermeticity testing

### Introduction

Hermetic packages historically have been designed to encapsulate and protect items from the external environment with the goal of extending lifetimes of a wide range of items [1]. Hermetically sealed items include canned foods, medicines, explosives, electronics, and mechanical devices. Package types most commonly are metal, glass or

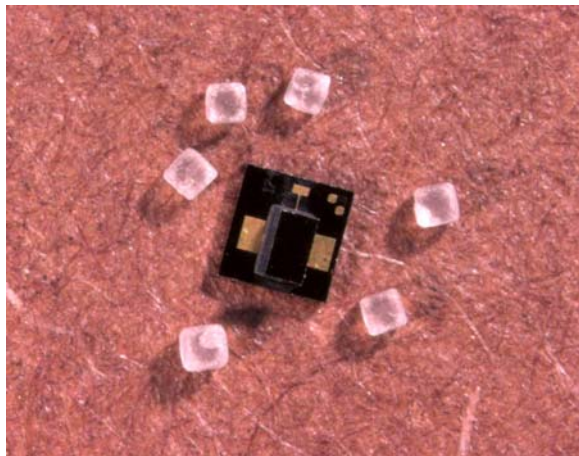
ceramic but can also be made from various polymeric materials [2-3].

The focus of our research has been on hermetically sealed microelectronic devices. Some of these devices contain mechanical parts (MEMS) and some contain hermetically sealed integrated circuits. A series of steps are commonly used to hermetically seal an item. Die are first attached to

the inner surface of the package by using solder or conductive adhesives. The application of adhesive is commonly followed by a cure process which serves two purposes. The first is to harden the epoxy and the second is to desorb any volatile organic compounds from the epoxy. This process is followed by a lid sealing method which can be done under vacuum, controlled gas environments, or air in order to provide a hermetic seal.

In some cases there are limitations to this method depending on the device that is being packaged. The device may not be amenable to soldering due to the excessive heat that is transferred to the device. This is common for MEMS devices that have very small, delicate mechanical parts that are easily overheated, leading to deformation and device failure. Therefore it is often desirable to use conductive adhesives for die bonding. Even though many of these adhesives are certified for high reliability military components and are considered low outgassing compounds, they can be problematic.

This document discusses a novel method for determining if a device is hermetically sealed and how it was used for characterizing die adhesive outgassing products to address contamination issues observed in MEMS and non-MEMS devices. Many packages were tested for hermeticity that ranged from a few tenths of milliliters to several nanoliters. The gas collected can be subjected to residual gas analysis (RGA) to identify permanent gaseous species (oxygen, nitrogen, argon, carbon dioxide, etc.). We have also shown that it can be collected and analyzed for a wide range of organic species using cryofocusing gas chromatography combined with mass spectrometry.



**Figure 1. Example of a 30 nanoliter package (surrounded by table salt) which was analyzed to determine if it was hermetically sealed.**

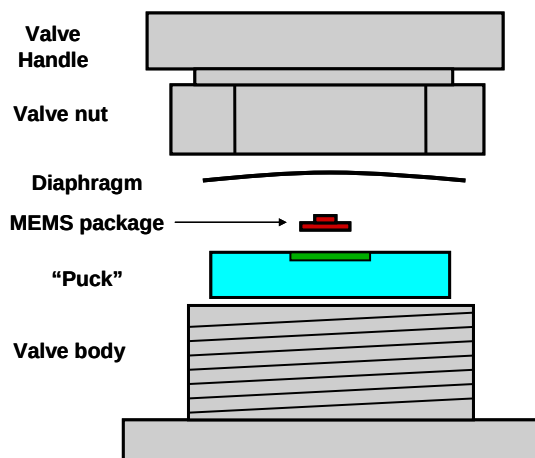
### Common Methods for Leak Testing MEMS devices

Often it is necessary to confirm that packages have been properly sealed and will remain hermetic over their functional lifetimes. Two techniques are commonly used for the detection of gross and fine leaks. Gross leak testing is conducted by submerging the component of interest into highly volatile fluorocarbon liquids. Once submerged, the devices are left to soak and uptake the solution. When this is complete the component is removed from the first solution and placed into a second solution. The part is then monitored for the release of bubbles indicating a leak.

Fine leak tests are most commonly conducted using helium. The component is exposed to helium for a specific time and pressure is monitored over time for a change that would indicate the incorporation of helium into the package. The parts can then be monitored for helium uptake using a helium leak detector. The above methods have many limitations associated with their sensitivity and therefore detection limits and the size of package they can accommodate. We have implemented a novel solution to these problems and have successfully demonstrated the effectiveness of this method for monitoring hermeticity, characterizing internal gas composition, and identifying possible contaminants. This has been applied to a very diverse set of devices and has proven very effective.

### Crush Valve Design and Description

A crush valve was designed and tested to enable the destructive testing of hermetically sealed packages to determine the extent of hermeticity and collect and analyze internal gases present to characterize the internal gaseous environment and identify possible contaminants that may alter performance and lifetimes of high reliability components. Commercially available valves were modified by machining out the valve body and replacing it with custom stainless steel “pucks” which are designed to accommodate a specific package [4]. This ensures that the volume is minimized enabling extremely small packages to be tested. Once the puck has been inserted into the valve the package being tested is placed into the puck and the valve nut is tightened down to create a seal inside the valve assembly. Figure 2 shows the critical components associated with the crush valve design.



**Figure 2. Overview of the major components of the crush valve design which is used for hermetic MEMS device testing.**

Packages are placed into the crush valve and sealed. The valve is leak tested to ensure a leak tight seal is obtained by evacuating the valve manifold and monitoring the pressure rise. The valve is connected to a high vacuum system which evacuates the valve and corresponding manifold. Once fully evacuated and confirmed to be leak tight the valve is isolated under static vacuum and the valve handle is turned resulting in the package being crushed. The pressure is monitored and can be used to determine the amount of gas present in the package as well as if the package is hermetically sealed. If the package is not properly sealed, the internal gas in the package will be evacuated during the pump down step and no pressure rise will be measured after crushing. Several valve ports are also present on the crush valve manifold which serve and ports to attach nitrogen gas which serves as a backfill and a sweep gas. An additional port also exists for the collection of released gases for analysis by cryo-GC/MS for full organic component characterization.

In addition to monitoring the pressure and ability to conduct cryo-GC/MS measurements, a residual gas analyzer is incorporated into the system to allow for real-time monitoring of gaseous species that have evolved. A pico-liter pulse valve developed by Siemens for use on the Quantra mass spectrometer is used to inject picoliters of gas into the RGA. This has proven very effective for continuously monitoring the internal gas from packages that contain only nanoliters of gas.

## Materials and Methods

Packages were placed into the puck which was inserted into the valve body. The valve nut was tightened down onto the body and sealed. The valve

was opened to high vacuum (approximately  $2 \times 10^{-9}$  Torr) and allowed to pump down for approximately 20 minutes. The pump down removes any residual gas present in the valve manifold and also will evacuate the internal volume of the device being tested if it contains a leak. Once the manifold was fully evacuated the crush valve was isolated under static vacuum and the package was crushed. The pressure after the package was breached was measured.

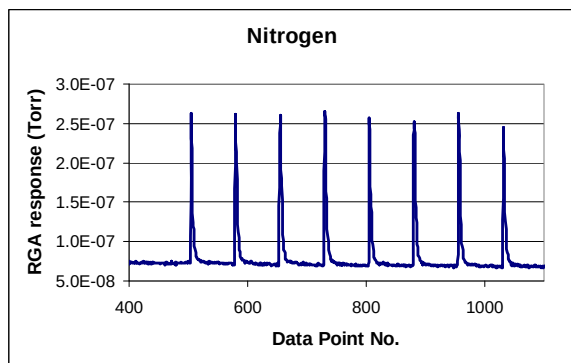
Permanent gases are analyzed in real-time using an Extrel RGA and a picoliter pulse valve produced by Siemens applied automation. This enables the gas composition to be determined. For example, if the package was sealed under nitrogen, there should not be other gases present if the packages are still hermetically sealed; however, the presence of air components such as argon and oxygen indicates a probable leak.

Further gas analysis was also conducted using an Entech 7100 preconcentrator, which is interfaced with a Jeol GC Mate II mass analyzer. Grab samples of the gas were collected and backfilled with house nitrogen to a pressure of 760 Torr. Ten cubic centimeters of the gas was injected into the cryo-GC/MS and analyzed for the presence of trace organics. Compounds were positively identified by library matching the mass spectra.

## Experimental Results

### Gas Composition

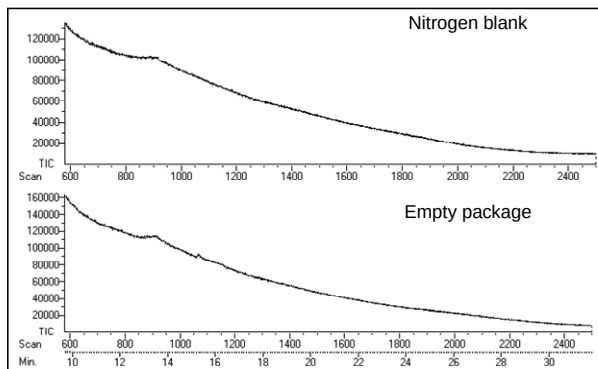
Several different packages were analyzed to determine if the packages were hermetically sealed. The packages were sealed in a nitrogen environment. These packages showed that they contained nitrogen after being pumped down to base pressure of  $10^{-9}$  Torr. The presence of nitrogen and absence of other air components suggested that packages were hermetically sealed. The RGA trace is shown in Figure 3 with each peak representing a pulse of gas that was introduced into the RGA through the picoliter pulse valve. These results proved to be highly reproducible which enables statistical analysis. If the packages were not hermetically sealed other gases such as argon and oxygen would be observed and the package would not contain any gas pressure after the pump down was completed.



**Figure 3.** Example of an RGA trace showing the presence of nitrogen in a package that was 30 nanoliters in volume.

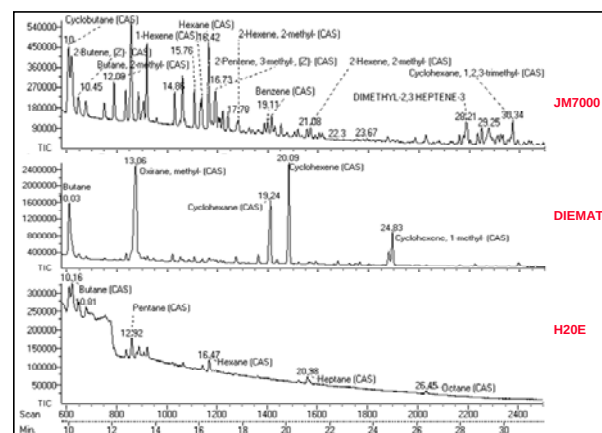
### Identification and Characterization of Contaminants

To address and characterize the cause of MEMS device failure due to contamination, the crush valve manifold was modified to enable grab samples to be collected after monitoring the pressure and permanent gas composition of crushed packages. A test matrix was designed to identify the source of contamination and mitigate contaminant removal. Experiments were conducted on empty packages that did not contain die, packages with die that had not been bonded, and packages that had undergone die bonding with several different adhesives. The goal of these experiments was to identify the contaminants present, isolate the source of the contaminants, and modify processing steps or materials to prevent device failure. Figure 4 shows the cryo-GC/MS results for a nitrogen blank (top) and an empty package (bottom) that did not contain a die or any adhesive. These experiments demonstrated that the empty package was not contributing to contaminant formation.



**Figure 4.** Total ion current for a blank and empty package

Further experiments were conducted on packages that contained die that were bonded with various adhesives using the manufacturer specified cure times and temperatures. These results are summarized in Figure 5. The top chromatogram shows the total ion current for JM7000 die attach adhesive. This material contained many organic species that could accumulate inside of the package. The middle trace is from the analysis of DIEMAT adhesive. This material did not generate the same signature as JM7000 but did contain many of the same organic products. The bottom chromatogram is from H20E adhesive. This material contained the least amount of contaminants and a different organic signature than the other two materials. These experiments demonstrated that the source of contamination was the die bonding adhesives. In all cases many organic contaminants were observed and identified, and each of the die adhesives has a very distinct signature that can be easily distinguished with this technique.

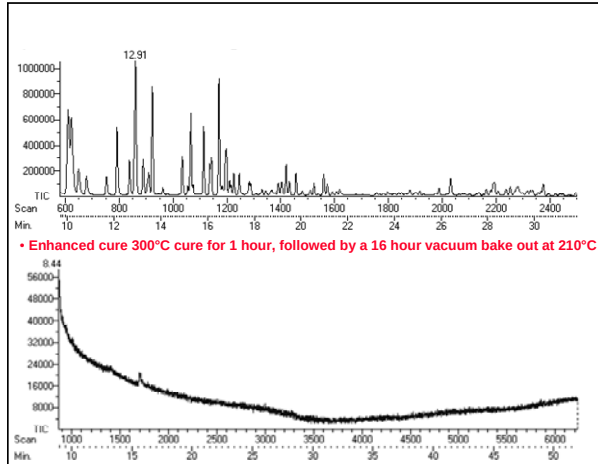


**Figure 5.** Total ion current for three packages, each containing a different conductive adhesives commonly used for die bonding

### Mitigation of Contamination Issues

Once the source of contamination was identified and the contaminants characterized, the next step was to identify changes that could be made to alleviate the source of contamination. It was concluded that a series of tests must be conducted to modify the cure process by changing temperatures and cure times to minimize the contamination source without altering device functionality. Therefore, an enhanced cure method was designed to remove volatile adhesive offgassing products before lid seal. The enhanced cure was conducted at 300°C for one hour which was followed by a vacuum bake at 210°C. This proved to be the most effective method

for removing the contamination while still using the same adhesives. Figure 6 summarizes the results for the enhanced cure method. The top chromatogram is for JM7000 following the manufacturer suggested cure of 300°C for 15 minutes. The enhanced cure shown on the bottom trace confirmed that the contaminants had been reduced to levels that were undetectable using cyro-GC/MS.



**Figure 6. Total ion current for JM7000 following manufacturer specified cure times and temperatures (top). The bottom chromatogram shows the results for the modified cure which incorporated the use of elevated temperatures and vacuum bake.**

### Conclusions

This method and experimental design has proved to be an effective method for studying high reliability hermetic devices. We have demonstrated that it is useful for determining what permanent gases are present (nitrogen, oxygen, argon, carbon dioxide, etc.) inside of sealed packages and the pressure. By incorporating a picoliter pulse valve and minimizing volumes extremely small packages (nanoliter internal volumes) were analyzed and the internal gas composition characterized.

Modifications to the crush valve manifold enabled grab samples to be collected and analyzed

using cryo-GC/MS to identify the contamination source and fully characterize organic products produced from the offgassing of die attach adhesives. These experiments revealed that three commonly used adhesives that are considered low outgassing contained significant volatile products that can potentially impact device function. All three adhesives tested contained highly reproducible and distinguishable trace organic gas signatures.

Altering cure times and temperatures from manufacturer specifications resulted in the removal of the contaminants and have restored device functionality over specified lifetimes. We have also developed different processing methods to address variability in the amounts of adhesive used, different package volumes and configurations.

This technique has proven to be a versatile method that is effective for packages of all sizes and configurations. Essentially any package can be studied using this method. In addition, relatively inexpensive modifications can be made to valves or other hardware to accommodate any size of package. We concluded that the contamination present in the packages tested was due to insufficient cure times.

### References

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