



NBS SPECIAL PUBLICATION 400-9

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Semiconductor Measurement Technology:

ARPA/NBS Workshop II. Hermeticity Testing for Integrated Circuits

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Hermeticity Testing for Integrated Circuits

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PREFACE

The Semiconductor Technology Program serves to focus NBS efforts to enhance the performance, interchangeability, and reliability of discrete semiconductor devices and integrated circuits through improvements in measurement technology for use in specifying materials and devices in national and international commerce and for use by industry in controlling device fabrication processes. Its major thrusts are the development of carefully evaluated and well documented test procedures and associated technology and the dissemination of such information to the electronics community. Application of the output by industry will contribute to higher yields, lower cost, and higher reliability of semiconductor devices. The output provides a common basis for the purchase specifications of government agencies which will lead to greater economy in government procurement. In addition, improved measurement technology will provide a basis for controlled improvements in fabrication processes and in essential device characteristics.

The Program receives direct financial support principally from three major sponsors: the Defense Advanced Research Projects Agency (ARPA),* the Defense Nuclear Agency (DNA),[†] and the National Bureau of Standards (NBS).[×] The ARPA-supported portion of the Program, Advancement of Reliability, Processing, and Automation for Integrated Circuits with the National Bureau of Standards (ARPA/IC/NBS), addresses critical Defense Department problems in the yield, reliability, and availability of integrated circuits. The DNA-supported portion of the Program emphasizes aspects of the work which relate to radiation response of electron devices for use in military systems. There is considerable overlap between the interests of DNA and ARPA. Measurement oriented activity appropriate to the mission of NBS is a critical element in the achievement of the objectives of both other agencies.

Essential assistance to the Program is also received from the semiconductor industry through cooperative experiments and technical exchanges. NBS interacts with industrial users and suppliers of semiconductor devices through participation in standardizing organizations; through direct consultations with device and material suppliers, government agencies, and other users; and through periodically scheduled symposia and workshops. This report describes the results of the second workshop in the ARPA/NBS workshop series. In addition, progress reports are regularly prepared for issuance in the NBS Special Publication 400 sub-series. More detailed reports such as state-of-the-art reviews, literature compilations, and summaries of technical efforts conducted within the Program are issued as these activities are completed. Reports of this type which are published by NBS also appear in the Special Publication 400 sub-series. Announcements of availability of all publications in this sub-series are sent by the Government Printing Office to those who have requested this service. A request form for this purpose may be found at the end of this report.

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Harry A. Schafft

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Semiconductor Measurement Technology:

ARPA/NBS Workshop II.

Hermeticity Testing for Integrated Circuits

by

Harry A. Schafft

Synopses are presented of the six invited talks and two discussion periods of a meeting in which 65 engineers and scientists, representing 36 organizations from private industry and government, participated. Topics ranged from failure analysis and the nature of leaks to evaluations and intercomparisons of bubble, weight, helium, and radioisotope tests. Underlying many of the problems discussed is the lack of a technical basis for specifications on maximum allowable leak rates and contaminant levels; no data are available to relate leak rate to component life. Of concern is the proliferation of test conditions which has complicated testing and test intercomparison efforts, and has resulted, unwittingly, in testing devices to significantly different actual leak rate criteria. Water vapor, sealed-in and that which penetrates the package, is a contaminant of major concern and the difficulties of making sufficiently accurate measurements of water vapor were detailed. The control required in the materials and assembly process to avoid hermeticity failure and false leak indications in ceramic, dual in-line packages was discussed. Finally, the importance of performing some hermeticity tests at elevated temperatures was cited.

Key Words: Bubble test; gas analysis; helium leak test; hermeticity; integrated circuits; measurement methods; microelectronics; radioisotope test; seals; semiconductor devices; weight test.

1. Introduction

The second ARPA/NBS Workshop was held on March 29, 1974, at the National Bureau of Standards (NBS), Gaithersburg, Maryland, and was attended by 65 engineers and scientists, representing 36 organizations. The purpose of the workshop was to define more clearly some of the problems of hermeticity testing, to outline NBS efforts and plans in this area, and to encourage the coordination of efforts in hermeticity test development and standardization.

The workshop was one of a series of meetings intended to address various semiconductor measurement technology problems. These workshops are a part of an effort, Advancement of Reliability, Processing, and Automation for Integrated Circuits with the National Bureau of Standards, sponsored by the Defense Advanced Research Projects Agency (ARPA). This effort is a major element of an NBS program which seeks to develop, and to disseminate to the electronics community, carefully evaluated and well documented test procedures and associated technology to solve measurement

workshop series

ARPA/NBS Program

and standardization problems in connection with the manufacture, procurement, and application of semiconductor devices.

measurement
problems

Severe measurement problems with hermeticity testing had been cited in NBS contacts with a cross section of the electronics community and subsequently in an earlier ARPA/NBS workshop on Measurement Problems in Integrated Circuit Processing and Assembly [1]. The major problems expressed were the lack of measurement correlation for the same test method at different stations and for different test methods at the same station, the discrepancies in the theory on which standards for test procedures are based, the subjective nature of some preferred test procedures, and the scarcity of definitive data linking leak size to device failure.

workshop format

NBS work on hermeticity was begun in response to these measurement needs. The workshop, which represents one part of this work, featured morning and afternoon sessions each with three invited talks and one extended discussion period. The morning session concentrated on failure analysis and hermeticity while the afternoon session focused on the evaluation and inter-comparison of leak test methods and procedures.*

synopses

Following the next, and summary, section are synopses of the invited talks and discussion periods, which constitute the last two sections of the report. The synopses represent the work and opinions of the speakers, who have examined and approved them.

*While detailed descriptions of the leak test method and their variations are available elsewhere [2] [3] [4], a brief description of the generic methods are provided here for the convenience of the reader. Five methods are discussed or mentioned in the text, they are the helium leak, radioisotope, bubble, weight test, and penetrant dye methods. The helium leak and radioisotope methods are similar in that both involve an attempt to force a test gas into the package through a leak. The former uses helium and the latter uses the radioactive isotope krypton-85. The methods differ in that the size of the leak is determined in the former by measuring the amount of test gas, helium, that escapes from the package, and in the latter by measuring the amount of the radioactive gas that is inside the package. Bubble tests involve immersion of the package in a heated fluid and a visual inspection for bubbles as the gas within the package expands and escapes through the leak. Weight test methods involve the measurement of the package weight before and after immersion of the package in a liquid under pressure to force the liquid into the package cavity. A faulty seal is indicated by a weight gain. Finally, penetrant dye methods involve an attempt to force a dye into the package cavity and observing it inside the package, if the package is transparent, or observing where it has leaked out of the package.

2. Summary

A basic problem which implicitly permeated the presentations and discussions during the workshop was the lack of a technical basis for specifications on maximum allowable leak rates and contaminant levels. The reason for this is that no data are presently available which can be used to relate leak rate to component life.

leak rate specs

contaminant
levels

component life

A contaminant of major concern is water vapor, both sealed-in and that which penetrates the package. Compounding this concern are the severe technical difficulties in making sufficiently accurate concentration measurements of water vapor. Specifications on sealed-in water vapor are limited by measurement capabilities rather than device degradation considerations. It was pointed out that because of some confusion about measurement capabilities, the maximum allowed water vapor concentration in devices in one program was set far below present capabilities to measure it. Work is underway to establish packages with controlled concentrations of water vapor for calibrating gas analysis facilities, and to develop a moisture sensor which can be inserted inside the package.

moisture

Of great concern, also was the proliferation of test specifications, in particular, for the helium and radioisotope methods. The numerous testing conditions used lead to needlessly large numbers of tests. Moreover, they grossly complicate intercomparison efforts and, unwittingly, result in significantly large differences in the actual leak test criteria so that, depending on the test method used, large variations in product yield can result. The tests specified in MIL-STD 750 and 883 were cases in point. The causes for these differences are in part due to assumptions about the gas flow regimes involved and in part due to the effects of pressurization times and levels, and on internal package volumes which are not adequately considered.

test specs
proliferation

leak test
criteria
differences

flow regimes

other effects

It is therefore not surprising that the need was repeatedly expressed for standardization of test procedures and for leak rate standards which, with possible correlation factors, could be used to standardize leak rate rejection levels in the industry.

standardization

The nature of leaks in ceramic, dual in-line packages and the control required in the materials and assembly processes to avoid hermeticity failure and false leak indications, were examined in detail. Pertinent not only to these package types is the observation that some packages which can pass hermeticity tests performed at room temperature may fail when tested

leaks in
dual-in-lines

leaks at
elevated
temperatures

at elevated temperatures of from about 55°C to 85°C. Thus, in high reliability applications, hermeticity tests may need to be performed at elevated temperatures.

bubble test
weight test
method

Through extensive use of slides and movies many shortcomings of bubble tests used for gross leak detection were illustrated. A weight test method developed for use on high-reliability production lines was shown to be a sensitive measurement procedure and to be more reliable than the bubble methods. However, because of the relatively limited number of parts that can be tested per hour with current procedures, the method is not presently suited for high-volume products.

NBS work

NBS laboratory work and plans were outlined. In particular, work was described dealing with experimental and numerical examinations of fluid flow mechanisms, with the development of transfer leak standards, and with the evaluation and intercomparison of various test methods now in use. NBS participation in the ASTM round robin experiments for the helium leak test method was mentioned.

meeting ground

radioisotope
round robin

Overall, the workshop served as a meeting ground for workers in the field where problems and plans for the future could be discussed. Of paramount concern were basic deficiencies in measurement methods and technology. An immediate result of the workshop was the recognition, by a number of the attendees, of the need for a round robin intercomparison experiment on hermeticity testing by radioisotope methods, and the decision to organize and participate in such a round robin under the auspices of ASTM Committee F-1 on Electronics.

3. Failure Analysis and Hermeticity

3.1 *IC Packages and Hermetically Sealed-In Contaminants* (Robert W. Thomas*)

topics discussed

This presentation included a brief description of the system developed by the Rome Air Development Center (RADC) to measure the gas ambients inside device packages, a discussion of some of the technical problems involved in making these measurements, a review of what has been found inside packages assembled by different vendors, and discussions of failure modes caused by the presence of water vapor, of the implications of allowing epoxy materials inside packages, and of using epoxies for die attachment.

gas analysis
system

The system used for measuring internal package gas ambients includes a quadrupole mass spectrometer, a data analyzer, and a package

* Code RBRM, Rome Air Development Center, Griffiss Air Force Base, New York 13441.

opener [5]. The last includes an ultra-high vacuum oven for baking out the packages prior to opening. The package-opener chamber is able to accommodate 20 device packages. A block diagram of this system is shown in figure 1. A capacitance manometer is used to measure the total pressure inside the package opener at the time the package is punctured. From the pressure rise, it can be determined if the package has a fine or gross leak. A gross leak is indicated by no increase in pressure. A ballast is used for testing packages with large internal volumes. The gas from the ballast can be bled off gradually and thereby not overload the mass spectrometer.

After inserting the devices into the test chamber, the chamber is evacuated using a turbo molecular pump. The packages are kept under vacuum for 24 hours or longer at a temperature of 125°C to remove extraneous contamination on the walls of the chamber and the packages. The ion pump in figure 1 is partially choked off to minimize selective pumping when the package is opened. The device is punctured and the mass spectrometer is set to scan the entire spectrum of mass numbers from 1 to 100, each second. The data analyzer automatically performs digital filtering and background subtraction, computes and applies sensitivity factors, identifies peaks, assigns mass numbers, reads intensities, time integrates the total gas evolved from the package, and finally produces a hard copy of the package ambient analysis. This is all done in a matter of seconds.

system operation

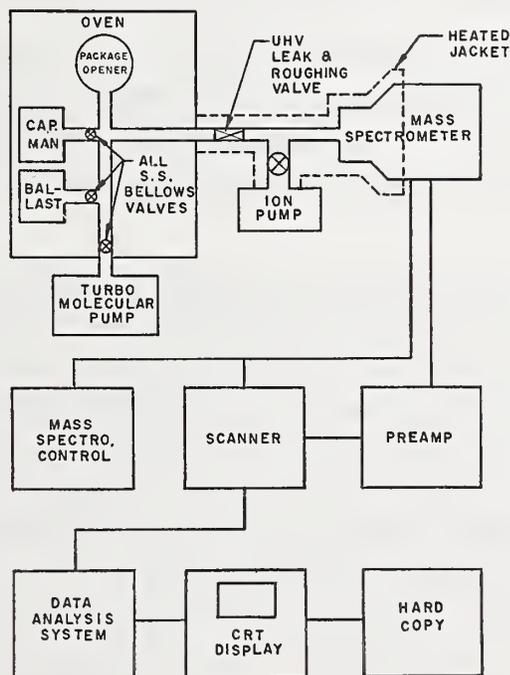


Figure 1. Gas analysis system.

system
sensitivity

With this system an analysis of concentrations as low as 0.1 percent can be guaranteed and trace components down to 1 ppm can be identified.

moisture and
failure

The use of this system, since its development, has revealed that the presence of water vapor appears to be more closely associated with device failure than the presence of any other gas inside device packages. However, the concentration of water vapor is exceedingly difficult to measure accurately.

measurement
problems

Some of the technical problems in measuring the concentration of water vapor accurately relate to establishing a sufficiently low background level of water. In pumping and baking out the package opener chamber, each gas is removed in a time proportional to its sticking coefficient. Water has a particularly large sticking coefficient. To achieve the low background level desired the chamber must be baked out for at least 24 hours.

moisture
background level

high temperature
required

In making the analysis of the internal package ambient it is also important to maintain the walls of the chamber and the tubing to the detector at a high temperature so that the gas will not become attached to the walls and not reach the detector. To make sure that as much of the gas ambient reaches the detector as possible it is important that the entire system be in thermal equilibrium, that the path length between the puncture chamber and the detector be minimized, and because such valves are very effective filters for water vapor, that there be no fine-leak valve between the chamber and the detector.

high conductance
needed

time integration
measurement

Another problem is that gases leave the chamber at different rates so that a time integration of all intensities must be made to insure an accurate measure of all the gas components within the package tested. An example of the relative rate at which different gases evolve is given in figure 2.

moisture
absorbed

An additional complication in obtaining an accurate measure of the concentration of water vapor in the package is the presence of materials that readily absorb water such as epoxies, shellacs, and polyimides. If such materials are present, one tends to obtain not only a measure of the amount of water in the vapor state but also how much has been absorbed by these materials. Actually, an indication of the water in the vapor state at 100°C can be obtained from a time integrated over the first 5 or 10 seconds after the package has been punctured.

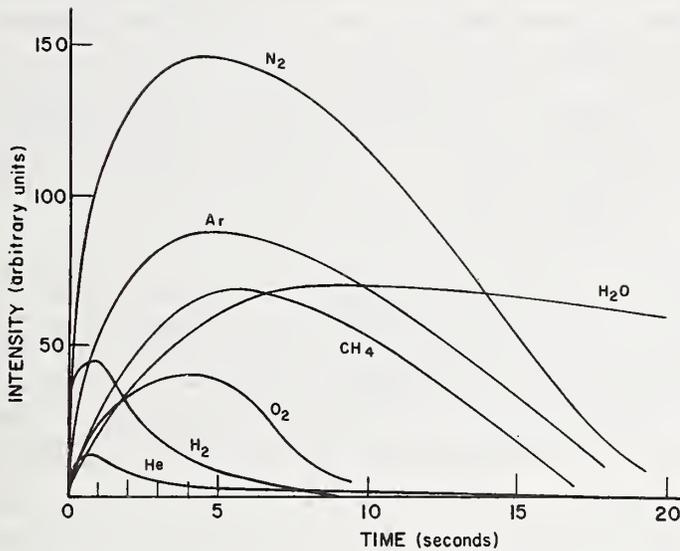


Figure 2. Gas evolution after package opened.

From examinations of many different package types obtained from many different device manufacturers, it has been found that the internal package gas ambient is unique to a given device lot. It has been possible to distinguish packages of the same type assembled by different manufacturers, and to distinguish device lots from a given manufacturer. Gas ambient analysis can provide an indication of the quality control exercised by the manufacturer and can be used to monitor process improvements by examining packages from lots made at different times. From such examinations one can also conclude that there is no consensus in the industry regarding the best gas ambient for package interiors.

package gas
ambients

used to
distinguish lots

When the manufacturer is successful in controlling the gas ambient, the major constituent is nitrogen, the water concentration is typically less than 150 ppm, that of oxygen is less than 1 ppm, and most of the other gas constituents are less than about 500 ppm. When he is not successful, much higher concentrations of water vapor and oxygen are found and significant concentrations of organics are present.

constituent gases

The possibility of inducing temporary leaks in ceramic packages during high-temperature stress tests has been confirmed. This was done by subjecting suspect packages to hot (150°C) and room-temperature neon under pressure (90 psi*). It was found that neon could be injected only under the heated

temporary leaks

* 1 psi = 6804.4 Pa

conditions. The inference is that differences in the thermal coefficient of expansion of the lead frame and glass seal can introduce sufficient stress in marginal seals to allow a leak at elevated temperatures.

burn-in tests

Analyses also revealed that high-temperature burn-in tests alter the gas ambient inside the package. Such tests have resulted in, for example, increases in the hydrogen concentration, decreases in the concentration of water, and increases or decreases in carbon dioxide.

hybrid devices

Experience with large hybrid devices is that manufacturers of such devices appear to feel that less care need be taken with such devices than with monolithic integrated circuits in regard to what can be incorporated inside the package. A case was cited in which a particular group of hybrid devices was failing due to the delamination of chip capacitors caused by excessive moisture in the package. Analyses showed that the gas ambient had an excessive concentration of water. This was despite the precautions the manufacturer had taken in general handling, in baking out the packages, and in back filling the packages with pure nitrogen that was regularly monitored.

materials
inside packages

moisture

However, the manufacturer had used a water soluble flux in sealing the package and had used inside the package such material as tape and shellac which readily absorb water.

specs on
moisture

This is just one example of the damage that water can cause if present in sufficiently large concentrations. It is not yet possible to define objectively a concentration at which degradation will occur but it is felt that it will be possible to do so soon. The Air Force Space and Missile Systems Organization (SAMSO) has recently incorporated an RADC recommended upper limit of 500 ppm of water vapor inside packages for devices purchased on a sensitive high-rel space program. Such a limitation will lead to greater reliability and 500 ppm is a reasonable level, one to which four or five laboratories in the country can measure. However, a lower limit may be needed for devices that are very sensitive to the presence of water vapor, such as MOS devices and devices with poorly passivated, thin film resistors.

well-sealed
packages

It is possible to seal packages well enough to maintain a low level of undesirable trace gases, such as water vapor, over long storage periods. This was demonstrated in tests of Apollo logic circuit devices, enclosed in ceramic, glass-sealed packages in 1965 and 1966 [5]. Unusually high background levels of hydrogen ($\sim 1\%$) and helium ($\sim 0.5\%$) were found in these devices, however. The hydrogen is believed to be the result of outdiffusion from metal parts during the period that the components had been baked, prior to final sealing. The helium is believed to have diffused through the glass bottom of these packages.

Some concern was expressed about the uncertain effects of hydrogen on wire bonds. Hydrogen may significantly degrade wire bonds at elevated temperatures but more work needs to be done to determine if and under what conditions such degradation can occur. The Apollo devices were not subjected to high temperatures where the wire bond might have been affected by the presence of hydrogen.

effect of
hydrogen

The results from a contract [6] involving the evaluation of a variety of epoxies for packages and for die attachment indicate that the epoxies have a large water content. Analysis of the internal gas ambient of devices with such epoxies showed from 1.9 to 2.3 percent water vapor. The existence of such high levels of water casts doubt on the appropriateness of their use in high reliability devices where the presence of water vapor can cause degradation.

epoxies in
packages

The importance was underscored of establishing maximum allowable levels of water vapor in the internal package ambients based on the sensitivity of the different device classes to the presence of water vapor. This work has been hampered by the difficulty in making precise water vapor measurements. Accordingly, work has begun at RADC to establish packages with controlled concentrations of water vapor to be used as standards which could be used at the different gas analysis facilities to calibrate their equipment. Such a standard is needed because of the poor agreement between the different facilities in the measurement of water.

specs on
moisture

water vapor
concentration
standards

Also, there is a need for a cheaper method for determining the control of the gas ambients within packages than the system now in use to do that. The test of a single device now costs about \$85. A contract [7] was recently let to develop a method to insert a sensor in packages and, on a lot sampling basis, establish that the gas sealed in the packages contains less than a certain quantity of water.

moisture sensors

3.2 *Failure Mechanisms Related to Hermeticity; The Nature of Leaks* (M. Douglas Keene*)

The causes for and the nature of the leaks that can occur in ceramic, solder-glass dual-in-line packages were reviewed. The reason for limiting the scope of the talk to this package type was the contention that this type is presently used for over 95 percent of all hermetic integrated circuits sold in the world and that it will continue to be the primary hermetic package in the marketplace for the foreseeable future. An estimated 30 million such packages are assembled each month in the United States.

leaks in ceramic
packages

* Fairchild Semiconductor, IC Division, 464 Ellis Street, Mountain View, California 94040

sealing
processes

In preparation for discussing the causes for seal failure, the processes involved in sealing this type of package were outlined. The glass used to seal together the metal lead frame and the ceramic base and cap is introduced as a slurry mixture of vitreous glass beads suspended in an organic binder. In general, the sealing process involves the heating of this mixture in a way which allows the organic binder to evaporate and the glass to transform from its initial vitreous or liquid state to a devitrified or crystalline state. In particular, the devitrification process begins with an initial heating in a belt furnace of the ceramic base with the lead frame resting on a slurry layer of glass beads and organic binder. The glass melts, the binder begins to burn off, and the frame sinks into the glass and becomes partially bonded to the base. Devitrification of the glass, which begins during this process, continues during the subsequent semiconductor die attachment process which involves a glass to metal eutectic bond made at temperatures as high as 425°C. After wire bonding and visual inspection, the ceramic cap is put in place and final sealing is performed at a temperature in the range from 450 to 525°C.

causes for
failure

process control

sealing
temperature
too high

The causes for seal failure derive from inadequate control over these processes and from improper handling. Inadequate control of the heating process and of the quality and the mixture ratio of the glass and binder can result in a defective seal. If the sealing process exposes the glass to too high a temperature, the organic binders will not burn off before the glass devitrifies. This can result in gases frozen into the seal in small bubble-like cavities. Structurally weak seals, which may be porous, can also result. Such excessive temperatures could occur, for example, if a furnace belt stopped due to a power failure, if a base were left too long on a heater block in preparation for die attachment, or if the die attachment process were too slow.

frame-attach
temperature too
low

Cavities and porous seals can also occur if the temperature at which the frame is attached to the base is too low. In this case the glass under the frame does not outgas sufficiently. Blow holes can then result during the final sealing operation due to the build-up of gas pressure. Such insufficiently high temperatures could be caused, for example, by increasing the furnace belt speed in order to shorten the processing time.

seal materials

Care must also be exercised in controlling the seal materials. Too high a concentration of the binder can result in its not being burned off during the sealing process. The glass mixture must also be evaluated to make sure that it does not exhibit any abnormal devitrification behavior. For example, some lots of glasses have exhibited a partial devitrification at an abnormally low temperature which has prevented the complete removal of

the binder during the sealing process. The use of differential temperature analysis [8] on incoming glasses now helps to eliminate this problem. glass additives

Another potential problem with the incoming glass is that additives designed to adjust the thermal expansion coefficient may not be in the proper balance. Packages made with such glasses may fail later in temperature cycling tests. Such glasses can be eliminated in incoming inspection by making thermal expansion tests.

Inadequate control over the mechanical handling of packages can result in the degradation of well-made package seals. Excessive physical stress on the package leads can be transmitted to the brittle glass seals. A number of examples of handling procedures where excessive stress could be applied were cited: lead clipping; lead flexing, to facilitate or perform socket insertion; and package dropping, such as might be experienced in automatic handling and in bulk loading. handling

Some electro-tin plating processes used to bright-finish the external lead frames of the sealed packages can also cause leaks. Acids used in this process can deplete the glass, particularly adjacent to the frame. Such leaks are most prevalent in parts that have been exposed to plating solutions for extended periods of time and those that have been replated, as in attempts to strip and replate tin-plated leads that oxidized during burn-in tests. plating

Finally, the problem of false leaks was discussed. Test methods presently used rely on the detection of a test gas or liquid that has penetrated the package cavity through an opening in the package seal either while it is still inside the package (radioisotope and weight gain methods) or as it escapes from the package (helium and bubble test methods). Surface porosity of the seal material, as produced by inadequate control over the seal materials and sealing procedures, can trap the test gas or liquid and can erroneously indicate a hermeticity failure. false leaks

"False cavities" with volumes nearly as large as 10^{-2} cm³ can sometimes be found in the seal material near the ends of the package, separate from the package cavity. The amount of seal material used may be selected only on the basis of providing a desired seal thickness in the area of the lead frame. If too little material is used, it may happen that the surface tension of the glass will pull the seal material towards the frame to leave a "false cavity" at either end of the package during the sealing process. The glass in this area may be weaker than that near the frame and package cavity and, as a result, cracks or a porous seal may develop to provide a leak to the outside of the package. Such a "false cavity" would give an indication of a leak porosity

indistinguishable from one to the device cavity by the helium or radioisotope methods. To determine if a false leak exists, a dye penetrant test is used.*

surface
contamination

False leaks, usually only seen in helium leak testing, can be caused by fingerprints, pencil marks, and dirt on the surface of the device. A single fingerprint was shown experimentally to increase an initial leak rate reading of 10^{-7} atm cm³/s[†] to an erroneously high level of 5×10^{-6} atm cm³/s. A solvent rinse followed by a high pressure air blast after helium pressurization reduces this problem significantly. Some users have found that a 10-min bake at 100°C, following the helium pressurization, improves the repeatability of the test and improves the correlation with radioisotope methods by eliminating surface adhering gases.

3.3 *Engineering Relationship Between Leak Rate and Component Life* (George R. Neff^x)

testing
experience

A summary was given of the results of an independent testing organization's experience with hermeticity testing of not only semiconductor devices but also a great variety of other components such as lamp displays, relays, thermostiches, glow plugs, thermal probes, and circuit boards [9]. The talk was prefaced with the statement that data to substantiate all of the remarks could not be released at the workshop because this information was the property of the customers who had paid for the work that had been done by the organization.

customer test
requirements

Customer test requests for the semiconductor devices that have been received and tested over the last few years are summarized in table 1. Here the ranges of internal volumes are listed as are the leak-rate ranges and limits that customers have specified. The primary hermeticity test method employed is the radioisotope method; secondary methods are the helium and bubble tests.

confusion over
tests

Generally, customers are confused about what kinds of hermeticity tests should be used and what minimum leak rate levels should be required. One case cited involved a customer who had brought in a number of hybrid circuits, each costing several hundred dollars, with no instructions other than that they should be tested for hermeticity.

* See footnote on page 32

† See footnote on page 14

^x Iso Vac Engineering, Inc., 6220 San Fernando Road, Glendale, California 91201.

Table 1. Summary of Customer Requests

| Type | Internal Volume | Leak rate ranges and limits of tests requested by customers | | | Percent of type received tested for hermeticity | Test Specification |
|-----------------------|--------------------------------------|---|------------------------|--------------------------------------|---|------------------------------------|
| | | Gross | Fine | Wet | | |
| | cm ³ | atm cm ³ /s | atm cm ³ /s | atm cm ³ /s | | |
| IC's ¹ | 10 ⁻³ to 10 ⁻¹ | 10 ⁻³ to 10 ⁻⁶ | 10 ⁻⁸ | 10 ⁻¹ to 10 ⁻⁵ | 40 | 750 ² /883 ³ |
| Diodes | 10 ⁻⁵ to 10 ⁻¹ | 10 ⁻⁴ to 10 ⁻⁶ | 10 ⁻⁹ | 10 ⁻¹ to 10 ⁻⁵ | 35 | 750/TX ⁴ |
| Transistors | 10 ⁻³ to 10 ⁻¹ | 10 ⁻⁴ to 10 ⁻⁶ | 10 ⁻⁷ | 10 ⁻¹ to 10 ⁻⁶ | 75 | 750/883 |
| Hybrids | 10 ⁻¹ to 10 | 10 ⁻³ to 10 ⁻⁶ | 10 ⁻⁸ | 10 ⁻¹ to 10 ⁻⁶ | 75 | 750/IV ⁵ |
| Plastics ⁶ | 10 ⁻³ to 1 | 10 ⁻⁴ to 10 ⁻⁶ | 10 ⁻⁷ | 10 ⁻¹ to 10 ⁻⁵ | 35 | IV/750 |

¹Integrated Circuits
²MIL-STD 750 [2]
³MIL-STD 883 [3]
⁴MIL-STD 19500/286B
⁵ISO Vac generated specification which is usually some modification of the specifications in MIL-STD 750 and 883.
⁶Cavity-type

In addition to the hermeticity test procedures given in MIL-STD 750 [2] and MIL-STD 883 [3], or combinations of the two, it has been very useful to perform the tests at elevated temperatures of from 55 to 85°C, representing the operating temperature range of many devices. It has been found that some devices which pass fine leak tests performed at room temperature, fail when tested at higher temperatures. Such leaks are apparently the result of weak seals which fail under the stress resulting from differences in the thermal coefficients of expansion of the constituent parts of the seal. This behavior is not restricted to one package type but rather has been found in a wide variety of package types.

tests at elevated temperatures

Of greatest concern to customers is the presence of moisture in the package; of somewhat less concern is the presence of oxygen. At the same time, however, there are no data to indicate at what concentrations moisture has an adverse effect on a device. A number of experiments have been performed by workers in the field in attempts to determine such critical concentrations but they were not successful.

moisture reject level

A consensus is believed to exist among investigators in the field that there is a relationship between the relative concentrations of oxygen and water that will penetrate a package, and that the ratio of oxygen to water is about 7:1. [See Section 3.4 for a further discussion of the validity of such a ratio.]

moisture/oxygen relationship

For a given internal package volume, a set of curves was shown of percent oxygen concentration in the package versus time after sealing for different leak rates. These curves were calculated assuming molecular flow, pure nitrogen as the initial internal gas ambient, and a normal atmospheric condition external to the package. It has been possible to substantiate these curves by the general procedure of first establishing a leak rate for the package using the radioisotope method and then performing a gas analysis at a known time after the package had been sealed. With such curves and

using oxygen to measure moisture level

assuming the 7:1 ratio of oxygen to water one would be able to determine the time after sealing that a given concentration of water would be obtained for a given leak rate. Assuming further that one can agree on a maximum level of moisture in the package that would not cause device degradation one could establish the maximum tolerable leak rate for a desired lifetime or level of reliability for the device.

flow regimes vs.
leak rate

It was asserted that molecular flow equations can generally be applied to the kinds of leaks that are encountered. The distribution of measured leak rates* for all components tested by a radioisotope method [10] at Iso Vac over the last few years is indicated in figure 3 as are the relative number of components whose seal leaks can be described by viscous and by molecular flow. The flow regimes were identified with a radioisotope method by measuring the concentration of Krypton 85 (Kr^{85}) after successive exposures to Kr^{85} under pressure. If the increase in the concentration of Kr^{85} remaining in the package is linear, a molecular regime is indicated; while if an exponential increase is observed, a viscous regime is indicated. Except for leak rates greater than about 10^{-4} atm cm^3/s , most of the leaks found can be described using molecular flow equations. Because of these results, a molecular flow regime is assumed when using the radioisotope method. It should be pointed out that the devices tested and represented in figure 3 are a biased sample in that they were generally considered to be suspect by the customer who provided them. Thus, the shape of the distribution would not be representative of all devices.

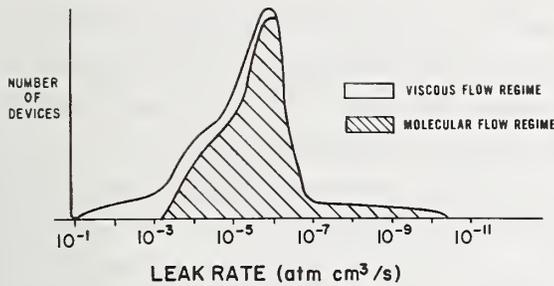
flow through
channel

If one were to theoretically examine the flow through a single hole, the molecular flow regime would apply only for leak rates less than 5×10^{-7} atm cm^3/s , in obvious conflict with the results shown in figure 3. This only serves to point out that one is generally not dealing with leaks in packages that can be described in terms of a single, well-defined hole.

molecular flow
regime

If a package leak be in the molecular flow regime then the pressure decay within the package, while the package is held under vacuum,

* Leak rate is generally considered to be the quantity of gas (in pressure-volume units) flowing through a leak per unit time, where the pressure on the low-pressure side of the leak has a negligible effect on the flow rate. It is implicitly assumed that the gas is at room temperature, is air, and is at one atmosphere pressure on the high-pressure side of the leak. Leak rate is commonly given in units of atm cm^3/s (10 Pa m^2/s) which is equivalent to a mass flow rate of about 4×10^{-5} g mol/s.



| Leak Rate Range (atm cm ³ /s) | Percent of Total Number of Leaking Parts Tested |
|---|--|
| 10 ⁻³ to 10 ⁻⁴ | ~ 10% |
| 10 ⁻⁴ to 10 ⁻⁵ | ~ 30% |
| 10 ⁻⁵ to 10 ⁻⁶ | ~ 50% |
| 10 ⁻⁶ to 10 ⁻⁷ | ~ 6% |
| >10 ⁻³ , <10 ⁻⁷ | ~ 4% |

Figure 3. Distribution of Measured Leak Rates of All Devices Tested

obeys very closely, the relation

$$P_t = P_0 \exp(-kt) \quad (1)$$

where P_t = internal pressure at time t (atm),
 P_0 = initial pressure at time $t=0$ (atm),
 k = conductance/volume (s^{-1}) and
 t = time (s).

Large deviations are found if the flow regime through the seal leak is a mixture of molecular and viscous flows.

It has been possible to get good correlation between the leak rates obtained from helium and radioisotope methods, particularly for the larger volume devices. The procedure used is to establish a leak rate with a radioisotope method [10] using Kr⁸⁵, assuming molecular flow, and then correct the reading to a helium equivalent. The package is checked with a helium mass spectrometer for any residual helium. If none is found, the package is pressurized with helium for a time to allow a 10 percent concentration of helium within the package. This time is calculated as indicated in MIL-STD 883, using the helium equivalent leak rate obtained from the radioisotope method. Correlation is established if the leak rate measured by the helium leak test is as predicted. If there is particular concern about the flow regime an experiment may be performed to see if the pressure decays as predicted by eq (1).

correlation -
helium and
radioisotope
tests

It was pointed out that eq (1) applies only for non-sticking gases and so does not apply to water vapor. The presence of appreciable concentrations of moisture also makes the measurement of leak rates more difficult. Kr⁸⁵ does not penetrate or flow out of a package with a leak in a predictable way if appreciable moisture is present. In fact, moisture can plug leaks. And, for this reason, a dry radioisotope test must be performed before a wet one.

moisture and
leak rate
measurements

moisture
absorption

A problem also occurs in the leak detection and internal gas analysis of devices where the package material absorbs appreciable moisture before encapsulation. In this case, the moisture that evolves from the material can block the entrance of the Kr⁸⁵ leading to a misleadingly low leak rate unless unusually long pressurization times are used. Also, the moisture absorbed by the package material requires some time to evolve into the package cavity. Thus, if a gas analysis is performed soon after encapsulation, which is usually the case, a misleadingly low concentration of moisture is measured.

3.4 Discussion Period

gas analysis
problems

In response to a question about concentrating gases for analysis, Thomas pointed out that gas analysis must be done in a dynamic system. Gases from inside the package cannot be stored or concentrated because they cannot be differentiated from the gases evolving from the package and the wall of the analysis system. Evidence of the complex dynamic nature of such measurements is that an initial decrease in the concentration of water vapor may be observed upon opening the package. This results from a temporary decrease in the background level of water vapor caused by an increase in system pressure with the opening of the package which in turn decreases the outgasing of water from the stainless steel walls of the test chamber.

need for baking
gas-analysis
system

Thomas was questioned about the need to bake out his system and make measurements with the analysis system at a high temperature. He replied that such high temperatures are necessary in order to reduce the background level of water vapor in his system in a reasonable time so that the required measurement sensitivity can be achieved. He added that he usually must bakeout the system for 24 to 48 h to reduce the water vapor background sufficiently. The system is baked at 125°C. The temperature is reduced to 100°C during measurements.

bake-out
economics

The long bake times mentioned by Thomas elicited some cautioning remarks about the need for practical test methods keyed to the requirements of large scale production. For example, if a test requires bake times of 24 to 48 h this requirement would be incompatible with the need to produce parts costing only pennies a part. Thomas replied that if used as a sample test method, such a test might be economically feasible.

moisture sources

In response to a question about the sources for the moisture found in packages, Thomas pointed out that not only are the walls of ceramic packages a sink for moisture which later serve as a source after encapsulation but that there are other less obvious sources present.

While packages may be encapsulated in a dry box with a moisture content of a few parts per million, the moisture concentration in the encapsulated devices may be as large as a few thousand parts per million. This can happen if, for example, the tubing through which the sealing gas passes gives off moisture. If left stagnant for 4 to 8 h, sufficient water may penetrate nylon and plastic tubing or outgas from stainless steel tubing to contaminate the package ambient. To minimize moisture content one needs to have dynamic flow conditions. Thomas added that some dry boxes are supplied with neoprene gloves which allow the moisture from the operator's hands to pass easily into the dry chamber; gloves made of butyl rubber do not allow water penetration.

Besides degradation to thin film resistors, a questioner asked if there were any other effects that water would have on a device. Thomas mentioned a paper by Nagasime et al. [11] which showed that for glass passivation with a concentration of phosphorus in excess of 8 percent, the presence of moisture at 120°C for 4 h had the effect of removing the phosphorus from the glass leaving behind a matrix in the glass that would absorb moisture and releasing sodium atoms that the phosphorus glass was intended to getter.

moisture-induced
degradation

In regard to performing low-temperature electrical tests it is important to be aware that the rapidity of cooling can affect test results. Thomas mentioned that if the package is cooled too rapidly, any moisture present in the package may condense and freeze on the walls of the package. Thus, much less degradation of the electrical parameters of the device may be seen than if the cooling is performed more slowly to allow more moisture to condense on the semiconductor die.

low-temperature
tests and
moisture

A member of the workshop mentioned that all his experience with the various analytical laboratories in the country has led him to the conclusion that all analyses of the concentration of water vapor in packages by these laboratories are "not worth a plugged nickel." It is for this reason that he felt the 7:1 ratio of oxygen to water vapor, that Neff mentioned, was potentially so important and should be documented.

analysis for
moisture

Thomas disagreed with the contention that the measured concentration of oxygen can be used as a measure of the amount of moisture in a device package. He seldom sees small quantities of oxygen with his gas analysis system. A reason for this is that hydrogen is the major residual gas present in a thoroughly baked out system. Hydrogen reduces the metal components so that if a package with a small quantity of oxygen is opened, the oxygen will strike the high temperature walls, form an oxide, and never reach the detector. Unless there exists a concentration of at least a few percent of

relation between
moisture and
oxygen

oxygen in the package, no oxygen will be measured. He added that even for packages sealed in open air, they never find more than about 5 percent oxygen with their gas analysis system.

more on moisture
and oxygen

Neff responded by saying that the 7:1 ratio was actually based on theoretically and empirically derived equations that predict such a ratio. He mentioned that people have attempted to experimentally verify this ratio but, in his opinion, have thus far been unsuccessful. He added that they could have chosen another gas for which another ratio would have been obtained. Oxygen was chosen because some feel it is detrimental to devices.

leak valves

Thomas has found that in his mass spectrometer analysis system, he is not able to predict the calibration of leak valves for different gases based on any parameters that can affect passage through the leak. He finds that he must resort to empirical means.

moisture sensors

Considering the many serious problems of making accurate analyses of gas ambients in packages, Thomas suggested an alternative approach. A suitable sensor could be placed inside the package to measure the moisture sealed in and determine the level of moisture penetration for packages with different leak rates. Using this approach, one could obtain an indication of what leak rates to test for to be assured that no significant amount of moisture can enter the package in a given time. RADC is, therefore, looking for some sensor that can reproducibly and quantitatively measure the amount of water vapor in the package.

measurement
standards needed

Neff reiterated his frustration at the lack of any standardized leak rate specifications. A member of the audience added that he had found no rationale for the leak rates specified in MIL-STD 883. It appeared, he said, that the levels were essentially arbitrarily chosen rather than selected on the basis of fundamental considerations regarding reliability, component life, etc.

present reject
levels arbitrary

life tests and
leak rates

Keene mentioned some reliability studies that he performed involving long term life tests (10 kh) at elevated temperatures of devices with known leak rates of from 10^{-4} to 10^{-8} atm cm^3/s . He found significant device degradation for the devices with leak rates in the 10^{-4} to 10^{-5} atm cm^3/s range.

diffusion of
water vapor and
other gases

Ruthberg mentioned the results of some preliminary calculations of the time it would take for water vapor to diffuse through tubes of

various radii and for different ambient temperatures. The results indicated a strong temperature dependence for the diffusion coefficient and that it might take years for moisture to enter a package through a leak channel while it would take only seconds for a test gas such as helium or Kr⁸⁵ to do the same. Thus, for some leak sizes for which packages would be rejected, the time for water vapor to diffuse into the package would be many times the expected life of the device.

A basic question that needs answering, concluded Ruthberg near the end of the discussion period, is what can leak rate measurements such as are now being performed really indicate about the reliability of the devices being tested. Also, should one expend efforts to determine true leak rates or should one attempt to relate the behavior of the device with the results that are obtained for one of the methods now used.

leak rates and
reliability

4. Evaluation and Intercomparison of Leak Rate Measurement Procedures

4.1 *Weight Test Method for Hermeticity Testing* (Aaron Der Marderosian* and Paul Nelson*)

By way of documenting the need for a better test method for gross leaks that led to the development of the weight test method, early efforts with a variety of bubble test methods were described. These tests were to detect gross leaks in devices to be used by Raytheon in the Apollo space craft and the Lunar Excursion Module [12]. Using calibrated capillary leaks and packages with various sized holes, the problems of subjectivity and the selection of leak criteria for bubble tests were shown with extensive use of slides and movie film. The primary faults of these tests are the inability, at least on a production line, to detect packages with gross leak rates (greater than about 10⁻² atm cm³/s) and the tendency to reject too many non-leaking packages.

gross leak tests

bubble tests

The need for a more reliable method for detecting gross leaks led to the development of the weight test method. The method, in brief, consists of weighing the device before and after being pressurized in a test liquid; the leak criterion is a package weight gain of more than 1 mg after removal from the test liquid.

weight test

The method has been found to be satisfactory for testing devices with leak rates in the range of 5 x 10⁻⁶ to 10 atm cm³/s. Metal packages with intentionally made holes to cover a range of leak rates were used to test the effectiveness of the method. A group of 25 such packages and 10 nonleaking packages has been introduced into device manufacturer lots to audit the

use of weight
test

* Raytheon Co., 528 Boston Post Road, Sudbury, Massachusetts 01776

standard production line leak testing procedures. For a sequence of 42 lots including a total of about 150,000 parts, only one leaker was accepted but that was due to an operator calculation error. Since then the test system has been automated to avoid such operator errors.

test procedure

The weight test procedure was described as follows:

1. Clean devices by dipping them in clean filtered Freon TF* for at least 2 min.
2. Bake devices for 30 min in an oven preheated to 125°C (to remove any Freon that may have gotten inside the package).
3. Weigh each device on a balance that automatically categorizes in 0.5 mg increments. (This procedure can be operated at a rate of about 10 parts per min.)
4. Place devices in a combination vacuum-pressure vessel and reduce pressure to less than about 1 Torr (1.3×10^2 Pa) for 1/2 h.
5. Introduce, at the reduced pressure, FC-77 fluorocarbon liquid to submerge devices. Then slowly increase pressure to 100 psig ($\sim 6.9 \times 10^5$ Pa) and maintain it for 2 h.
6. Reduce pressure to room pressure in vessel, remove devices, and store devices under FC-77 fluorocarbon at room pressure. Transfer time shall be less than 1 min.
7. Remove ten devices of the same weight category from the FC-77 reservoir and allow to dry for 2 min.
8. Weigh each device and determine weight category. All ten devices must be weighed within 4 min after removal from the reservoir. (This time limitation is to detect package with gross leaks and those with crack-like openings through which the test fluid can quickly flow out of the package by capillary action.)
9. Separate all devices that deviate more than two weight categories. Reject any such device that shows a gain in weight. Recycle any such device which shows a weight loss.

* Certain commercial materials are identified here in order to adequately specify the experimental procedure. In no case does identification imply recommendation or endorsement by the National Bureau of Standards nor does it imply that the material identified is necessarily the best available for the purpose.

Particulate contamination of the test fluids was mentioned as a problem. To reduce this problem the fluid is recycled through a 1- μ m particle filter. A dryer, consisting of activated alumina, was also added to remove any water present in the test fluid. To eliminate another problem, a plastic disc is floated on the test fluid in the vacuum-pressure vessel of step 4. This is done to avoid the absorption into the test fluid of the nitrogen gas used in achieving the pressure in step 5. Absorbed nitrogen causes the test fluid to effervesce during depressurization and push out some of the fluid that may have entered the package.

problems with
test fluids

A method, developed by workers at Westinghouse, was mentioned briefly, and described as being as sensitive as the weight test method. This method consists of exposing the package to alcohol under pressure and then measuring the surface conductance between internal leads. Concern was expressed, however, about using in such a test a fluid which could conceivably degrade devices which may have passed the leak test yet have had a trace of the fluid enter the package.

another method

4.2 Evaluation of Helium and Radioisotope Methods (Ralph E. McCullough*)

This report emphasized the variability of results obtained when various semiconductor device packages are tested for hermeticity with the helium and radioisotope methods under the different test conditions specified in MIL-STD 750 [2] and MIL-STD 883 [3]. The differences in the test conditions specified in the two military standards, presented in table 2, were reviewed

variability of
reject levels

Table 2. Test Conditions of MIL-STD 750 and MIL-STD 883 for (A) the Helium Method and (B) the Radioisotope Method

| A. Helium Method | | | | | |
|------------------|-----------------------|----------------|-------------------|------------------------|------------------------|
| Standard | Package Volume | Pressurization | | Leak Rate Reject Level | |
| | | Time | Pressure | Indicated | Calculated |
| | cm ³ | h | psig [†] | atm cm ³ /s | atm cm ³ /s |
| MIL-STD 750 | all | 4 | 60 \pm 5 | 5 x 10 ⁻⁸ | NA |
| MIL-STD 883 | \leq 0.1 | 1 | 60 | 5 x 10 ⁻⁸ | 5 x 10 ⁻⁷ |
| MIL-STD 883 | \geq 0.1; \leq 10 | 1 | 60 | 5 x 10 ⁻⁷ | 5 x 10 ⁻⁶ |

| B. Radioisotope Method | | | | | |
|------------------------|----------------|----------------|-------------------|------------------------|--|
| Standard | Package Volume | Pressurization | | Leak Rate Reject Level | |
| | | Time | Pressure | | |
| | | h | psia [†] | atm cm ³ /s | |
| MIL-STD 750 | all | 0.1 | 73.5 | 1 x 10 ⁻⁸ | |
| MIL-STD 883 | all | 0.2 | 73.5 | 5 x 10 ⁻⁸ | |

* Texas Instruments Incorporated, P. O. Box 5012, Dallas, Texas 75222

[†] 1 psi = 6894.4 Pa; 60 psi = 4.14 x 10⁵ Pa; 73.5 psi = 5.07 x 10⁵ Pa

and it was shown how these differences lead to marked differences in the calculated leak rate rejection levels and, therefore, the number of parts that would be rejected [13].

leak rate tests

Data developed from testing devices having a wide range of leak rates were presented. Three different packages were examined each having different internal volumes. Tested were a ceramic dual-in-line package with a volume of 0.015 cm^3 , and TO-5 and TO-3 metal packages having volumes of 0.086 and 1.07 cm^3 , respectively. The packages were vacuum baked after each test and measured prior to retesting to assure that there was no residual tracer gas.

test data - ceramic packages

Leak rate histograms for the ceramic package obtained for the different tests are reproduced in figure 4. The distribution of measured leak rates as determined from the helium method test conditions of MIL-STD 750 and shown in figure 4A differ considerably from that of the distribution of calculated leak rates determined from the test conditions of MIL-STD 883 and shown in figure 4B. Many parts that would be rejected if the test were performed according to MIL-STD 750 would be accepted if tested according to MIL-STD 883. The discrepancy between the two methods is seen by using the equation in the MIL-STD 883 method to calculate an actual leak rate from the machine reading.

discrepancies

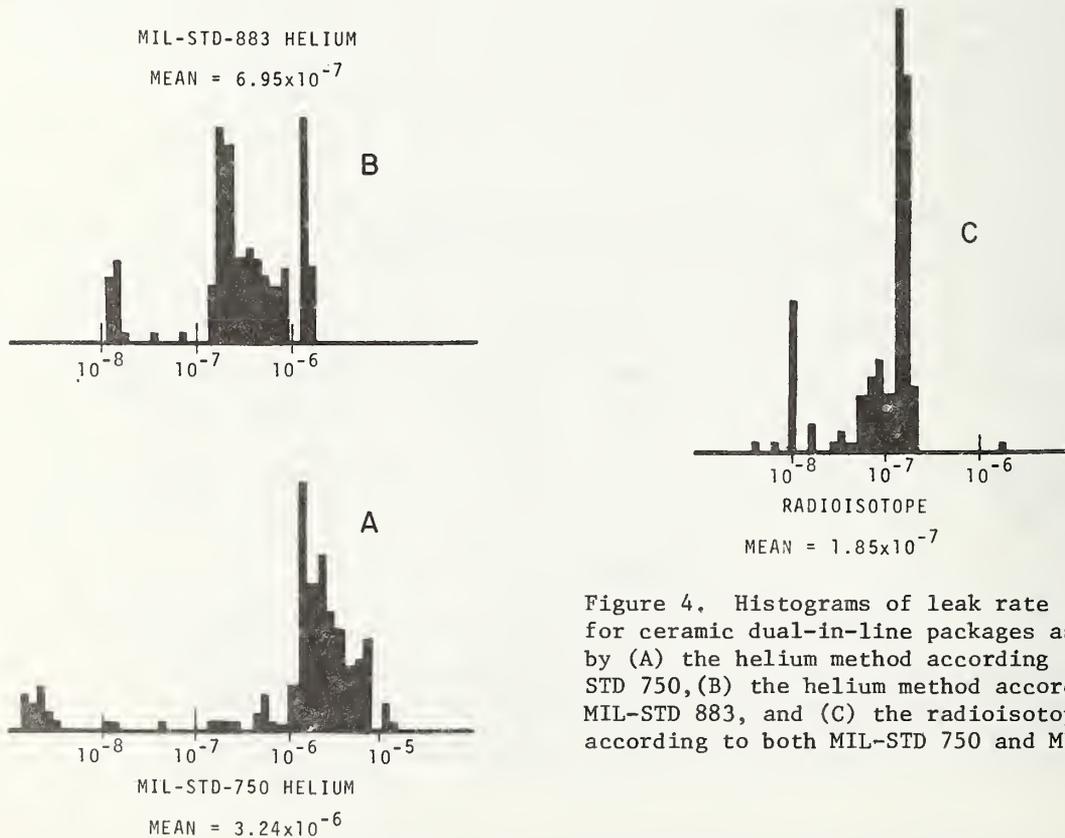


Figure 4. Histograms of leak rate ($\text{atm cm}^3/\text{s}$) for ceramic dual-in-line packages as tested by (A) the helium method according to MIL-STD 750, (B) the helium method according to MIL-STD 883, and (C) the radioisotope method according to both MIL-STD 750 and MIL-STD 883.

The 5×10^{-8} atm cm³/s leak rate reject level for MIL-STD 750 transforms to a calculated actual leak rate of 4.5×10^{-8} atm cm³/s. This rejection level is over an order of magnitude more severe than the 5×10^{-7} atm cm³/s for MIL-STD 883. The distribution of leak rates as determined with the radioisotope test is essentially unchanged for the two pressurization times in MIL-STD 750 and 883 and so only one distribution need be shown in figure 4C.

The degree of correlation between the radioisotope and helium methods as performed by MIL-STD 750 is shown in figure 5A while that as performed by MIL-STD 883 is shown in figure 5B. In the latter, it was felt that there was a fairly good correlation between the two methods up to a leak rate of about 10^{-7} atm cm³/s. For larger leak rates there is a large, unexplained deviation which has been seen for many package types. In the leak rate range where the reject criteria lie, the correlation between the radioisotope and helium is as good as it is because the pressurization times and levels have been considered.

In the metal TO-5 package the shapes of the leak rate distributions for the different methods and procedures were similar, as seen in the

correlation data -
ceramic packages

test data -
TO-5 packages

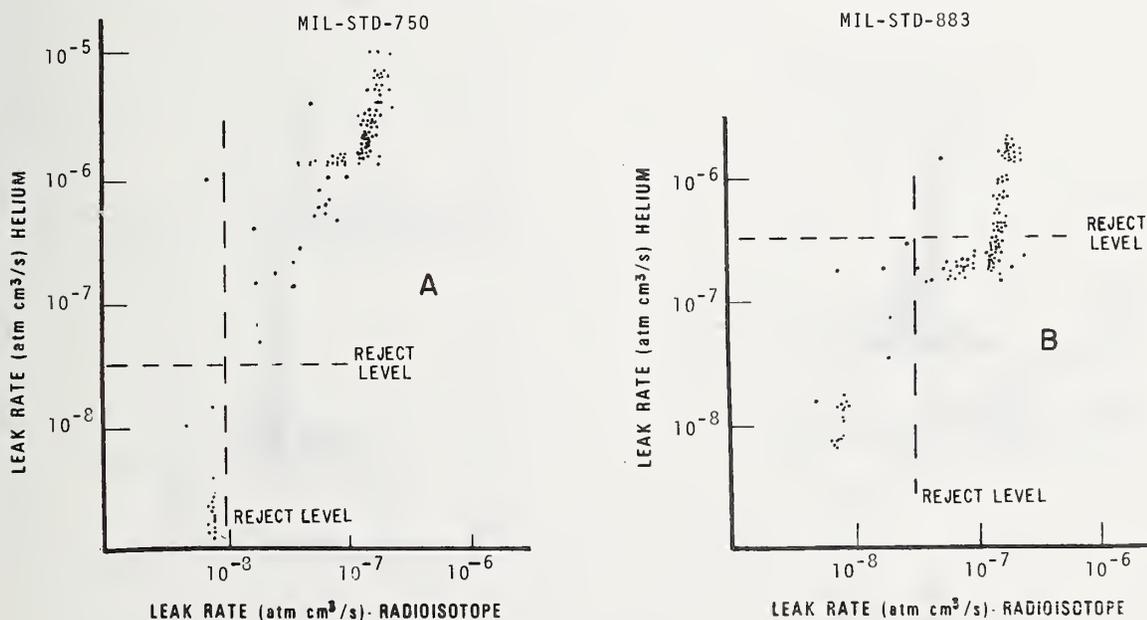


Figure 5. For ceramic dual-in-line packages, leak rates measured by the helium leak method versus those measured by the radioisotope method for test conditions specified in (A) MIL-STD 750 and (B) MIL-STD 883.

histograms in figure 6. This is characteristic of results from metal-can type packages. The lack of similarity noted in the ceramic parts is possibly due to the porosity of the ceramic material used in those packages.

correlation data -
T0-5 packages

For this metal package type, the correlation between the radioisotope and helium test methods appears to be better than for the ceramic packages, especially for the MIL-STD 750 test. The correlation data are shown in figure 7.

test data -
T0-3 packages

Leak rate distributions for the T0-3 metal packages show a similarity in shape although the spread in the distribution for the MIL-STD 883 helium test is smaller than that of the others. Histogram data are shown in figure 8. Correlation data presented are shown in figure 9.

problem with
MIL-STD 750

Two problems with the helium test methods for leak rates in the large internal volume package were pointed out. For the MIL-STD 750 procedure, the actual or calculated reject level is 3.6×10^{-8} atm cm^3/s for the T0-3 package whereas for the smaller T0-5 package it is 4.5×10^{-8} atm cm^3/s , as calculated from the equation given in MIL-STD 883 to relate instrument leak rate reading to a calculated leak rate. Thus, there is a more restricted test criterion for large volume packages than for small volume packages.

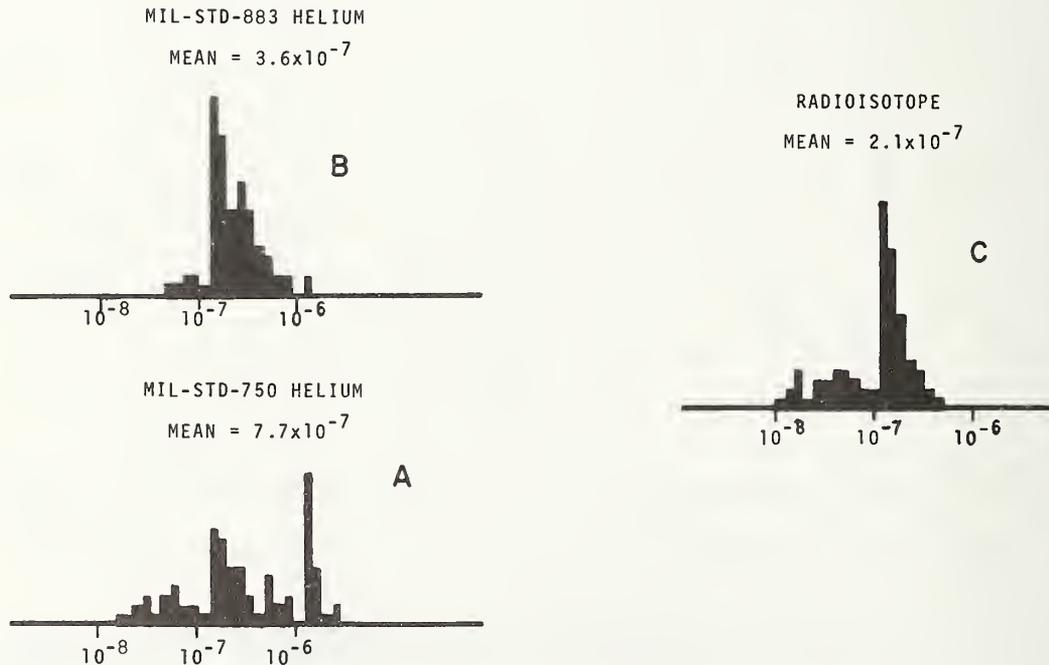


Figure 6. Histograms of leak rate (atm cm^3/s) for T0-5 packages with internal volume of 0.086 cm^3 as tested by (A) the helium method according to MIL-STD 750, (B) the helium method according to MIL-STD 883, and (C) the radioisotope method according to both MIL-STD 750 and MIL-STD 883.

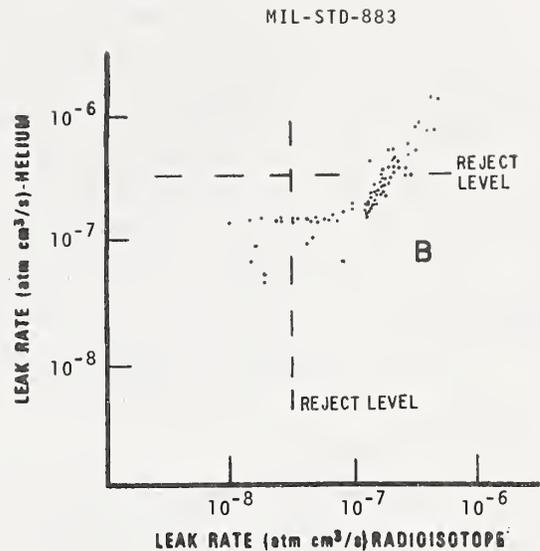
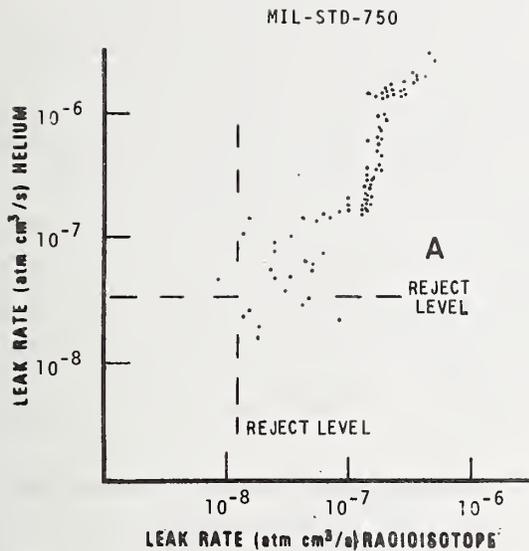


Figure 7. For TO-5 packages with internal volume of 0.086 cm³, leak rates measured by the helium leak method versus those measured by the radioisotope method for test conditions specified in (A) MIL-STD 750 and (B) MIL-STD 883.

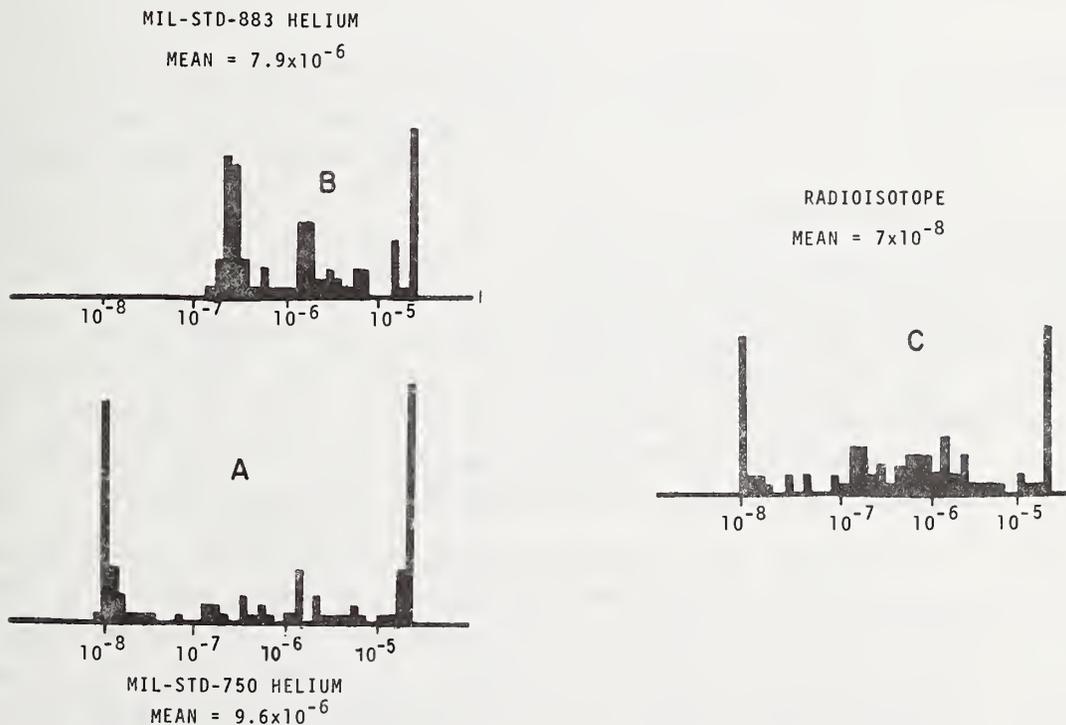


Figure 8. Histograms of leak rate (atm cm³/s) for TO-3 packages with internal volume of 1.0 cm³ as tested by (A) the helium method according to MIL-STD 750, (B) the helium method according to MIL-STD 883, and (C) the radioisotope method according to both MIL-STD 750 and MIL-STD 883.

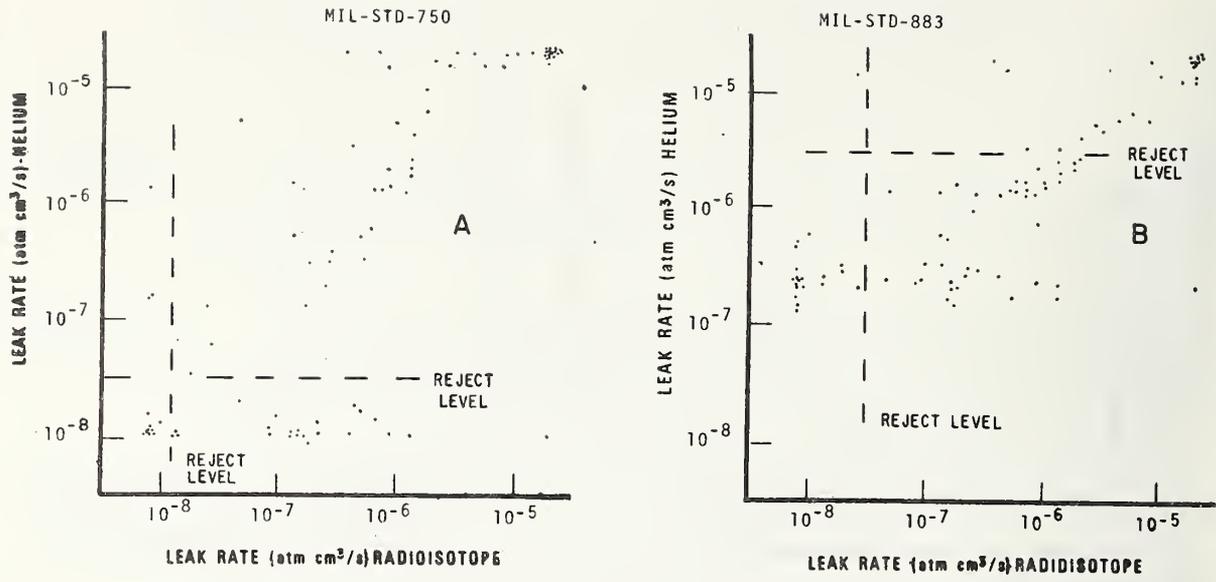


Figure 9. For TO-3 packages with internal volume of 1.0 cm³, leak rates measured by the helium leak method versus those measured by the radioisotope method for test conditions specified in (A) MIL-STD 750 and (B) MIL-STD 883.

However, if one assumes that the density of an impurity gas inside the package such as water vapor is an important consideration for device reliability, then the more restricted test criteria should be placed on smaller volume packages.

problem with MIL-STD 883

For the procedure specified in MIL-STD 883, the use of the 5×10^{-6} atm cm³/s reject point corresponds to an instrument reading of about 10^{-4} atm cm³/s which is normally beyond the range of most machines. If one uses the 5×10^{-7} atm cm³/s instrument reading reject point one really is using a calculated reject point of 2×10^{-7} atm cm³/s. Thus, there is a choice between two reject criteria that are over an order of magnitude different, when compared as calculated or as indicated readings.

test method correlation

In summary, it is possible to obtain good correlation between the helium and radioisotope test methods in the range of interest. However, to do this it is necessary to take into consideration that the leak rate indications on helium leak test instruments are dependent on internal package volumes and on pressurization levels and times. Furthermore, there is a desperate need for the development of standard leak packages of known volume and leak rate with which to calibrate systems not only with regard to the instruments' indicated leak rates but with regard to the pressurization levels and times. Another need is to decide what leak rate reject levels are relevant and appropriate for reliability and life requirements of devices.

standard leak needed

relevant reject levels needed

4.3 An Overview of the NBS Hermeticity Effort (Stanley Ruthberg*)

This talk began with a listing of five elements which are or will be included in the NBS effort. They are (1) an examination of fluid flow mechanisms by both numerical and experimental analyses for particular geometries and pertinent fluids, (2) the development of transfer leak standards, (3) an evaluation and intercomparison of measurement methods, (4) the application of guidelines developed from the first three elements for theoretical models to real packages with real leak channels, and (5) the development of new measurement methods. The primary efforts at NBS currently involve the first three of these elements and they were discussed in the talk.

NBS work

In the discussion of fluid flow mechanisms it was shown that without reference to gas flow models no correlation can be made between the measured and true leak rates. It appears that part of the reason for poor correlation between the helium and the radioisotope methods is the use of equations that are not all derived for the same gas flow regimes. To illustrate the effect of assuming different gas flow models for the helium leak test, plots of true versus measured leak rates for the helium leak test derived by two groups and for both laminar and molecular gas flow regimes are reproduced in figure 10.[†]

flow regimes and test methods

molecular and laminar flow

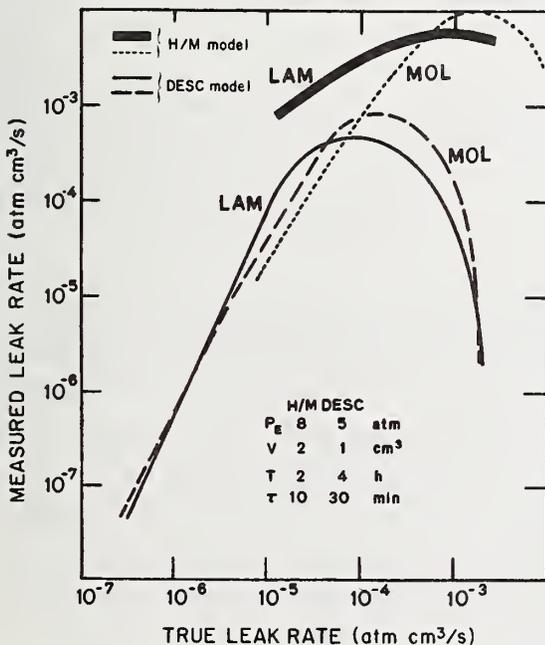


Figure 10. Measured leak rate versus true leak rate for laminar (LAM) and molecular (MOL) flow regimes as determined by the models of Howl and Mann (H/M) and of the Defense Electronics Supply Center for the test conditions indicated where P_E is the tracer gas pressurization, V is the internal package volume, T is the pressurization time, and τ is the time after pressurization that the leak test is performed.

* National Bureau of Standards, Washington, D.C. 20234

[†] The indicated or measured leak rate of a package depends on, for example, the level of pressurization and time after depressurization as the helium gas escapes from the package which in turn affects the gas flow regime involved. The curves in figure 10 relate the measured to the calibrated or true leak rate which is determined from a standard leak under standard test conditions.

Two sets of curves for molecular and laminar flow are shown. The upper set is from Howl and Mann [14] while the lower set is from the Defense Electronics Supply Center [4]. Even if the test conditions shown in the figure were the same, there could be significant differences between the sets because of the different assumptions made by the two groups in the derivation of these relationships.

measured and
true leak rate

To complicate the matter of relating measured leak rate to true leak rate, the actual flow regime is likely to be some mix of laminar and molecular flow regimes. To pursue this further and to suggest how sensitive the degree of mixing may be to the nature of the leak, the results of some preliminary calculations were shown for expected flow regimes and for different sized leak channels using the equation of Knudsen [15]. This equation which was originally formulated on a semi-empirical basis, gives correct behavior for transition flow at subatmospheric pressures. In this equation the total flow rate, Q_T (atm cm³/s), is given by

total flow rate

$$Q_T = Q_L + ZQ_M$$

where Q_L is the laminar flow (atm cm³/s),

Q_M is the molecular flow (atm cm³/s), and

Z is a function of the ratio of the average mean path, λ , to the radius of the leak channel, R .

flow regimes for
channel leak

Flow regimes for channel leaks of length 0.1 cm but with different radii and for two pressure differentials were considered and the results are summarized in table 3. For one atmosphere of pressure on the upstream side of the channel, P_2 , and zero pressure on the downstream side of the channel, P_1 , the flow rates vary from about 10^{-2} to 10^{-9} atm cm³/s for channel radii from 10 to 0.1 μ m. The flow regime varies from 95 percent laminar flow for the 10- μ m radius channel to complete molecular flow for the 0.1- μ m radius channel. Increasing the pressure to 5 and 1 atm for P_2 and P_1 , respectively, shifts the flow regime toward laminar flow. This change in pressures has, for example, changed the regime from completely molecular flow to 50 percent laminar flow for the 0.1- μ m radius channel. The implication is that in the process of testing the hermeticity of a package with such a leak where pressures P_1 and P_2 would be changing, the flow regimes could also be changing significantly.

factors affecting
flow

Additional factors affect flow. There exists a transition length, L_E , or distance that the gas must flow in the channel before uniform, laminar flow is achieved. There is also a gas flow limit, Q_{comp} , at which compressibility effects tend to reduce the flow rate. There are other limits, Q_{turb}

and Q_{crit} , which are the flow rates for turbulent and sonically choked flow, respectively, which also tend to reduce the flow rate. The values for L_E , Q_{comp} , Q_{turb} , and Q_{crit} for leak channels 1 mm in length for different radii and pressures P_2 and P_1 are shown in table 4. For $P_2 = 5$ atm and a 10- μ m radius channel, the transition length is greater than the length of the channel so that laminar flow is not achieved anywhere in the channel. Further, the flow, Q_T , has surpassed the compressible flow limit, Q_{comp} , by an order of magnitude. Even for the 5- μ m radius channel, $Q_{comp} \approx Q_T$. At a more typical pressurization of 10 atm and for a 5- μ m radius, L_E is a significant fraction of the channel length and the compressible flow limit is exceeded by almost an order of magnitude.

Table 3. Types of flow and flow rate through cylindrical leak channel of 1 mm long for different channel radii, R, and different upstream, P_2 , and downstream, P_1 , pressures.

| P_2 | P_1 | R | Q_T | Q_T/Q_M | Q_L/Q_T | $R/\bar{\lambda}$ |
|-------|-------|---------|------------------------|-----------|------------|-------------------|
| atm | atm | μ m | atm cm ³ /s | | | |
| 1 | 0 | 10 | 1.2×10^{-2} | 15.5 | $\sim .95$ | 100 |
| 1 | 0 | 5 | 9.4×10^{-4} | 8.2 | $\sim .90$ | 50 |
| 1 | 0 | 1 | 2.1×10^{-6} | 2.3 | $\sim .50$ | 10 |
| 1 | 0 | 0.1 | 1.1×10^{-9} | 1.0 | 0 | 1 |
| 5 | 1 | 1 | 3.9×10^{-5} | 9.6 | $\sim .90$ | 60 |
| 5 | 1 | 0.1 | 6.3×10^{-9} | 1.7 | $\sim .50$ | 6 |

Table 4. Gas flow rate, transition length, and gas flow limits for a cylindrical leak channel 1 mm long for different radii, R, and different upstream, P_2 , and downstream, P_1 , pressures.

| P_2 | P_1 | R | Q_T | L_E | Q_{comp} | Q_{turb} | Q_{crit} |
|-------|-------|---------|------------------------|----------------------|------------------------|------------------------|------------------------|
| atm | atm | μ m | atm cm ³ /s | mm | atm cm ³ /s | atm cm ³ /s | atm cm ³ /s |
| 5 | 1 | 10 | 3.3×10^{-1} | 1.3 | 4×10^{-2} | 4.9×10^{-1} | 6.5×10^{-1} |
| 5 | 1 | 5 | 2.1×10^{-2} | 9.7×10^{-3} | 1×10^{-2} | 2.4×10^{-1} | 1.6×10^{-1} |
| 5 | 1 | 1 | 3.9×10^{-5} | 2.4×10^{-4} | 4×10^{-4} | 4.9×10^{-2} | 6.5×10^{-2} |
| 10 | 1 | 10 | 1.4 | 6.1 | 4×10^{-2} | 4.9×10^{-1} | 1.3 |
| 10 | 1 | 5 | 7.5×10^{-2} | 0.34 | 1×10^{-2} | 2.4×10^{-1} | 3.3×10^{-1} |
| 10 | 1 | 1 | 1.4×10^{-4} | 6.5×10^{-4} | 4×10^{-4} | 4.9×10^{-2} | 1.3×10^{-2} |

flow dependence
on gas

All these data were calculated for air. Other gases will exhibit different leak rates and compressible flow limits. Values relative to air are shown in tables 5 and 6. From these data it can be seen that, for example, helium can be used at much higher pressures than air before exceeding the compressible flow limit. Also, the leak rates can differ significantly from that of air for different gases and that these differences may vary significantly with channel radius. For helium gas, the leak rate relative to air changes from about unity for a 10- μm radius channel to 2.7 for a 0.1- μm radius channel. Pure krypton 85 shows a much smaller variation while argon shows very little variation at all.

other
complications

There are still more complications and some relate to the fact that so far the data presented are for one-directional flow, into the interior of the device. The flow rate out is dependent on the internal volume, leak size, and pressurization time. Actually, the limiting effects of gas flow for gross leaks and the possible mix of flow regimes for very small leaks are academic for the helium and radioisotope methods. It is the intermediate leak-rate range that is of significance and should be studied carefully because it is in this range where the reject level is located and factors of 2 or 3 in the measured leak rate values can very significantly affect production yield.

Table 5. Transition length and gas flow limits of He and Kr⁸⁵ relative to air.

| Gas | Q_C | Q_T | L_E |
|------------------|-------|-------|-------|
| air | 1 | 1 | 1 |
| He | 2.8 | 8.1 | 0.11 |
| Kr ⁸⁵ | 0.62 | 0.65 | 1.5 |

Table 6. Leak rates for different gases relative to air of 1.0-mm long cylindrical channels of different radii.

| Gas | Channel Radius | | |
|------------------|------------------|-----------------|-------------------|
| | 10 μm | 1 μm | 0.1 μm |
| air | 1 | 1 | 1 |
| Ar | 0.83 | 0.84 | 0.85 |
| He | 1.09 | 1.77 | 2.7 |
| H ₂ | 2.22 | 2.90 | 3.62 |
| Kr ⁸⁵ | 0.74 | - | 0.59 |

These data demonstrate that further assessment of gas flows is needed, both numerical and experimental. Experimental work requires the use of stable and precise leaks for the study of both flow behavior and, when sealed to known volumes, leak rate characteristics of real packages. Measurements of flow rates as a function of gas species and pressure conditions by mass spectrometers and manometers are being planned with instruments that are available or in assembly in NBS's laboratories. The Howl and Mann theory [13] is now being tested for large-volume devices in a round robin conducted by the ASTM Committee F-1 on Electronics.

more work needed

In regard to transfer standards and methods for evaluating these standards, the development of suitable test bodies is important to the solution of many measurement problems. Not only would such test bodies, or transfer standards, be helpful in experimentally discriminating between theoretical models, but they could allow routine and repeated checks of leak rate measurement performance as well as intercomparison of different measurement stations and methods.

transfer standards

A suitable method for measuring flow rates is necessary for the development of such test bodies. Leak rates greater than 10^{-5} atm cm³/s can be measured by a rate-of-rise technique in measured volumes with absolute reference manometers [16]. The conductance of such leaks can then be calculated. A combination of leaks for which the conductances have been so measured and calculated can be assembled to form a flow divider which in turn can be used to measure leak rates in the range from 10^{-5} to 10^{-10} atm cm³/s. In conjunction with an ultra high vacuum system and mass spectrometer, NBS will have the capability of measuring quickly and directly any leak for any well-behaved test gas.

flow rate measurements

An important capability of a transfer standard is the incorporation of a calibrated leak into an envelope designed so that the resultant test body can be used repeatedly without degradation. One possible design for such a test body is a refinement of the type presently being used in the round robin experiment mentioned above. The refinement involves the use of a smaller test volume and a construction with a glass less permeable to helium. Another possible design is shown in figure 11 where a stainless steel tube is sealed at one end with a demountable aluminum-foil, high-vacuum seal [17] and at the opposite end could be attached to a porous plug or crimped as shown in the figure. Stable test bodies with known leak rates measured for the tracer gas used: would allow direct calibrations with improved precision of the particular leak test techniques used, would allow a direct comparison of the capabilities of the different methods in use, and could serve on the production line to test for measurement integrity.

calibrated leak

use of such leaks

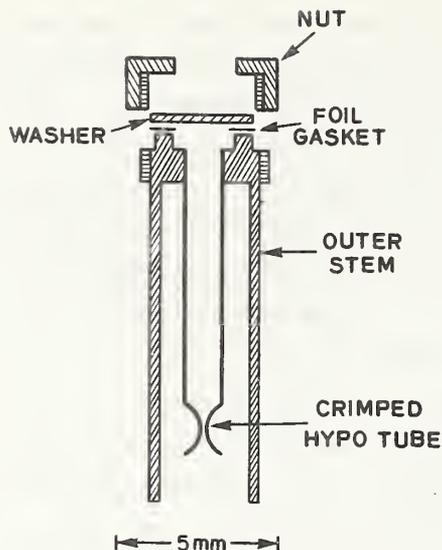


Figure 11. Design for transfer standard

4.4 Discussion Period

data scatter

Neff was concerned that the audience did not realize that the data shown by McCullough, which indicated relatively small scatter of data points in the plots of the helium versus the radioisotope test results, were obtained by engineers using the test equipment under well-controlled conditions. Were this done in a production-line environment, he noted that the scatter in data points would be far greater. McCullough readily agreed that this was so but added that within the relatively narrow leak-rate range about the reject level for a given package type, the difficulty in controlling conditions even on a production line was not so great.

reject levels
for Helium test

In reference to a question from the audience, McCullough agreed that there should be more reject levels to more finely cover the range of internal package volumes in use for the helium leak test.

package damage

It was pointed out that with some of the larger volume package types, the high pressures specified can result in package damage. However, if the pressure level is reduced the sensitivity of the test method will be severely reduced unless the pressurization times are increased to inconveniently long periods.

nature of leaks

Keene showed slides of a number of packages prepared with a dye* to define the area and extent of typical leaks found in ceramic packages.

* Parts were pressurized at 100 psi (6.9×10^5 Pa) for 1h in a fluorescein dye diluted 10:1 with trichloroethelene and then baked after pressurization to dry the dye.

Keene's purpose was to show that none of the leaks exhibited any well-defined tubular holes. Rather, they all indicated that the leak consisted of a system of defects or cracks in the glass or ceramic material.

Keene was asked if he has experienced many problems with the absorption of Kr⁸⁵ on the surface of ceramic packages. He replied that one can detect surface absorption of Kr⁸⁵ by using a beta detector. However, in actual applications where he has radioisotope and helium test instruments next to each other, he does not have surface absorption or problems with fingerprints, pencil marks, etc. with the radioisotope test but does with the helium leak tester. Neff suggested that the reason for this is the shorter pressurization times required for the radioisotope method. If these times were as long as those in the helium leak test, surface absorption would also be a problem with the radioisotope method.

surface
absorption

Thomas reviewed some of the anticipated changes in MIL-STD 883 relative to hermeticity tests. These changes are listed in Appendix A. One of the projected changes is to prevent gross leak testing before making a fine leak test to avoid the possibility of plugging a fine leak. This generated considerable discussion in that other procedures and tests, such as a thermal shock test, could also serve to plug a fine leak. One participant summed up this part of the discussion by saying that there would be "no peace in the family" until what is done to the part before performing the hermeticity test is adequately specified.

MIL-STD 883
changes

plugging leaks

Thomas also indicated that perhaps too much emphasis was being placed on gross and fine leaks at the expense of learning what is inside the package to start with. This became part of an extended discussion on the role and the generation of moisture inside the package and the need to correlate leak rates for moisture to the leak rates of helium and Kr⁸⁵. One pointed out that they had spent much time at the workshop talking about one problem (moisture inside the package) and measuring another (the penetration and escape of helium and Kr⁸⁵).

moisture in
packages

These discussions led to a reiteration of the need for work to determine what levels of contaminants, such as moisture, could be tolerated and from that establish some generally agreed upon leak rate reject level to which all could test to.

setting reject
levels

At the end of the discussion period and the workshop, after the discussion had turned to essentially manufacturing and procurement problems, McCullough reminded the participants that NBS cannot solve such problems. Rather, he said, NBS can and should provide the industry with the measurement tools so that we will know that what we think we are measuring is actually what we are measuring!

NBS's role

Acknowledgement

The active participation of the speakers and the attendees, which made the workshop a success, are acknowledged here with pleasure. Thanks go to the speakers who provided me with copies of the visual materials used in the talks and with written materials which assisted greatly in the preparation of this report. Special thanks go to Stanley Ruthberg, leader of the NBS hermeticity project, who served as the technical chairman of the workshop and who provided me with technical insight that was very valuable in the preparation of the report. I also wish to express my gratitude to W. Murray Bullis for his editorial assistance, to Edgar C. Watts for his work with the figures, to Mary Jane Ronas for her skilled help in proofreading the final draft, and to Ellen Y. Trager for her diligence in typing the final and camera-copy draft.

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APPENDIX A

Anticipated Changes in Method 1014 of MIL-STD 883

I. TEST CONDITION A - *Helium Fine Leak Test*

Three methods will be allowed:

1. Fixed Method: Same as present method using fixed preconditioning parameters.
2. Flexible Method: Use formula to determine preconditioning parameters for specific package cavity volume. As an alternative, preconditioning parameters for a series of package cavity volumes may be selected from a table.
3. Packaged in Helium Method: Use to test package sealing process.

II. TEST CONDITION B - *Radioisotope Fine Leak Test*

Minor changes

III. TEST CONDITION C - *Fluorocarbon Gross Leak Test*

Of the two present methods, only the one requiring pressurization will be required with some changes in the preconditioning parameters.

IV. TEST CONDITION D - *Penetrant Dye Gross Leak Test*

Minor changes

V. TEST CONDITION E - *Weight Measurement Leak Gross Test*

New Test. Similar to weight test described in Section 4.1

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| 15. SUPPLEMENTARY NOTES | | | |
| 16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.) Synopses are presented of the six invited talks and two discussion periods of a meeting in which 65 engineers and scientists, representing 36 organizations from private industry and government, participated. Topics ranged from failure analysis and the nature of leaks to evaluations and intercomparisons of bubble, weight, helium, and radioisotope tests. Underlying many of the problems discussed is the lack of a technical basis for specifications on maximum allowable leak rates and contaminant levels; no data are available to relate leak rate to component life. Of concern is the proliferation of test conditions which has complicated testing and test intercomparison efforts, and has resulted, unwittingly, in testing devices to significantly different actual leak rate criteria. Water vapor, sealed-in and that which penetrates the package, is a contaminant of major concern and the difficulties of making sufficiently accurate measurements of water vapor were detailed. The control required in the materials and assembly process to avoid hermeticity failure and false leak indications in ceramic, dual in-line packages was discussed. Finally, the importance of performing some hermeticity tests at elevated temperatures was cited. | | | |
| 17. KEY WORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons) Bubble test; gas analysis; helium leak test; hermeticity; integrated circuits; measurement methods; microelectronics; radioisotope test; seals; semiconductor devices; weight test. | | | |
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