Variable Leak Rate Phenomena in Glass to Metal Seals

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ABSTRACT:

The term "one-way leaker" (1) is sometimes used to describe a hybrid which passes all Mil-Std leak requirements yet still provides a Residual Gas Analysis (RGA) spectrum containing large amounts of air. The controversial assumption generally made is that the part was hermetic under one set of conditions and 'leaky' under a different set of conditions. The controversy stems from the fact that the most common method of forming a glass to metal seal in Kovar packages is by means of oxide bonding. Such seals, once broken, should not be expected to reseal themselves. This paper details the results of a failure analysis of an RGA failure with a power hybrid case where the bow in the base of the package was found to have resulted in sufficient stress during burn-in to create a 'one-way' leaker. The leak location was subsequently confirmed by dye penetrant testing. A conclusive experimental verification of how the leak rate of such a glass to metal seal can vary with pressure and/or temperature is presented.

Keywords: RGA, Moisture, Variable Leaker, One-way Leaker

Introduction

Over the years Teledyne has conducted several investigations into the cause of RGA failures. The reasons for the failures have varied but generally have fallen into three broad categories relating to package seal, temperature exposure or internal material properties. For the most part the latter two types are well understood by hybrid manufacturers and the appropriate hierarchy of preseal and post seal temperature exposures are used for a given combination of organic materials in the package that allow the achievement of <5000ppm moisture (2-3).

The more difficult issues that continue to appear from time to time are related to the quality of the package seal, particularly with glass to metal seals. The most recent example of such an investigation occurred with a space program in which a power hybrid was found to contain greater than 5000ppm of moisture. After extensive investigation it was concluded that the package had been stressed in burn-in by over-clamping the parts in such a way as to 'crack' the glass seal of one or more leads. This conclusion was reached on the basis of the high oxygen/argon and low helium content which accompanied the high moisture and the subsequent discovery of the leak path by means of die penetrant testing. The most difficult issue with the analysis was how to explain the fact that the part was capable of passing the Mil-Std-883 fine leak requirement with measured leak rates well into Be 10^{-9} Atm. cc/sec range.

This paper will present the details of the investigation into that failure as well as the results of experiments conducted by Raytheon for TET on similar failures that conclusively show the variable leak rates that apparently 'good' glass to metal seals can exhibit as a function of pressure and temperature.

Failure Investigation

The hybrid in question was constructed in a molybdenum based power package with 12 copper leads, butt welded to Kovar feedthroughs (see Figure 1). The package was felt to be of robust design and had never failed RGA previously. The RGA results were obtained as part of a DPA by the customer and are shown in Table 1 along with the data from a subsequent confirmation DPA test as well as data from 5 samples rejected at TET as x-ray failures.

Figure 1
Side View of Power Package RGA Failure

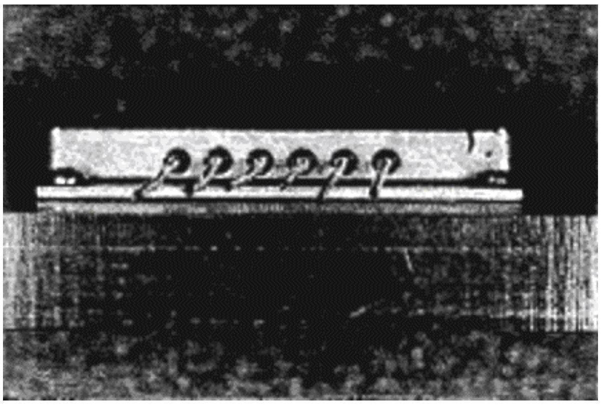


Figure 2
Inside Stereomicro View
Pin 6 Black Light 12X 32 Seconds

Additional data taken after 1000 hour life test of units from the same lot are shown in Table 2. Again 3 of 5 units exhibit abnormal levels of argon and moisture. This time the oxygen levels are very low, however. The probable reason for this is discussed in a latter section.

A fault tree analysis was performed but the potential causes of failure were quickly narrowed to two basic areas; mechanical overstress of he package or defects in the glass to metal seals. As seen in Table 1, five of seven units had significant oxygen levels and accompanying increases in argon (normal ratio of 0_2:Ar in air is 20:1). Since there are no known sources of argon internal to the hybrid and the usual background level of argon in the sealing chamber at TET is 125 to 200ppm, anything over that level in an RGA test is considered evidence of a leak at some point in the history of the unit. The oxygen is monitored during the sealing operation and sealing halted if oxygen exceeds 100ppm.

Table 1
RGA Test Data on Various Power Hybrids
of Lot in Question

S/N / RGA Gas	DPA	DPA Retest	X-Ray Rejects				
	439	434	413	409	415	431	403
Nitrogen	93.9%	91.1%	90.6%	93.2%	91.7%	93.0%	91.5%
Oxygen	2.4%	2.0%	4.3%	0.6%	0.3%	ND	ND
Argon	1030 ppm	1338 ppm	2389 ppm	816 ppm	271 ppm	137 ppm	188 ppm
CO_2	718 ppm	830 ppm	651 ppm	654 ppm	374 ppm	100 ppm	81 ppm
H_2O	0.53%	1.0%	1.2%	0.72%	0.13%	787 ppm	767 ppm
Hydrogen	47 ppm	ND	ND	279 ppm	1833 ppm	669 ppm	2124 ppm
Helium	3.0%	5.7%	3.5%	5.3%	7.7%	6.8%	8.2%
Fluorocarbon	88 ppm	ND	ND	ND	ND	ND	ND
CH4	24 ppm	ND	ND	ND	150	ND	ND
NH3	ND	ND	ND	26 ppm	ND	ND	ND
Package Bow (mil)	2.3	3.0	1.6	2.5	1.6	2.1	2.2

Table 2
RGA Test Data for Units
After 1000 Hours at 125°C

S/N / RGA Gas	Life Test Units				
	405	420	425	428	432
Nitrogen	92.2%	71.2%	94.6%	99.3%	92.1%
Oxygen	ND	ND	ND	ND	3355 ppm
Argon	174 ppm	2861 ppm	5343 ppm	4318 ppm	450 ppm
CO_2	112 ppm	1300 ppm	401 ppm	107 ppm	256 ppm
H_2O	0.1%	1.5%	0.77%	0.28%	0.78%
Hydrogen	2384	2637	ND	ND	ND
Helium	7.4%	17.7%	3.9%	ND	6.7%
Fluorocarbon	ND	ND	774 ppm	ND	360 ppm
CH4	ND	ND	ND	ND	ND
NH3	ND	ND	ND	ND	ND
Total HC	78 ppm	8.8%	125 ppm	27 ppm	170 ppm
Package Bow (mil)	2.5	2.5	3.8	2.7	2.8

Because the units were passing fine leak test in every case at less than 10^{-8} Atm. cc/sec., the focus of attention was placed on how air could have gotten into the unit. Both DPA samples were sent for dye penetrant testing at a local lab. The first unit (S/N 439) gave an indicator at the glass to case interface of one feedthrough and the second (S/N 434) at the pin to glass interface (see Figure 2). Both required at least 30psi over internal pressure before the leak was evident. In the cue of S/N 434, before an indicator was observed, the unit had to be clamped to a thick aluminum plate in the same fashion as employed during it's bun-in. Admittedly no way exists to test that the unit might still pass fine leak test after the dye penetrant test. It is assumed therefore that the glass seal was under sufficient compression to exclude dye initially, but a path opened when the package was placed under external pressure.

Both DPA samples were sent to the package manufacturer for cross-sectional analysis. No flaws could be found, even in the area of known dye penetration. They did indicate however that the oxide at the interface was "thinner than usual". They also confirmed the presence of a natural convexity in the base of the package caused by their manufacturing process. Since the unit was designed to be clamped at the four corners, the result of a dome in the middle of the bottom of the package would be the creation of forces on the walls of the package during clamping that would depend on the size of the dome. The height of the dome for each of the units is provided along with the RGA data in Tables 1 and 2. While the dependence is not absolute, there appears to be higher levels of argon and moisture as the height of the dome increases. The lack of a clear trend suggests that variations in the glass to metal seal integrity may also be a contributing factor.

Calculation of the rate of moisture or oxygen ingress into packages has been carried out by many people (2),(4-5). Employing equation 1 below and using the internal volume of the device in question and assuming a leak period of 160 hours, SN 439 with 2.4% oxygen would have had to have had a leak rate for air of approx. $2x10^{-6}$ Atm. cc/sec.

$$L = V/t \ln [Co/Co\text{-}C] \quad \ldots\ldots\ldots\ldots \quad 1$$

Where L = Leak rate of O_2
V = 11.2 cc
Co = 21%
C = 2.4% for S/N 439
t = $5.8x10^{+5}$ sec

For Helium this corresponds to a leak rate of approx. $5.5x10^{-6}$ Atm. cc/sec, about three orders of magnitude greater than the observed leak rate.

As the time between seal and RGA test was significantly longer than 160 hours, the argument could also be made for a somewhat lower leak rate over a longer period. This does not coincide, however, with the repeated measurement of extremely low leak rates both during fabrication and during the investigation. The possibility of a much higher leak rate for a short duration was also ruled out on the basis of seal records during that period. As TET uses approx. 10% He in the sealing atmosphere, the unit can be tested for leak rate within a short period after seal. No unusual failure rates or anomalies were noted.

The conclusion was reached that the probable leak period was consistent with the time spent in burn-in under a clamped condition. An explanation was needed, however, for the apparent transient nature of the leak rate. The next section details the results of experiments performed on another package type, but with a similar RGA condition (see for example data in Table 3) that conclusively demonstrates how pressure and/or temperature effects can cause just such a three orders of magnitude variation in leak rate.

Table 3
RGA Test Data for a Digital Hybrid

RGA Gas	Serial Number	
	20	56
Nitrogen	82.2%	74.7%
Oxygen	2.58%	ND
Argon	1713 ppm	184 ppm
CO_2	1.06%	2.01%
H_2O	0.81%	0.34%
Hydrogen	ND	ND
Helium	12.2%	21.8%
Fluorocarbon	8773 ppm	ND
NH3	0.13%	1.16%
HC	ND	171 ppm

Test Approach

The apparatus exists at Raytheon to pressurize a hermetic device, either internally or externally, while monitoring the opposite side for He. A tube is soldered to a hole drilled in the lid of the package under test. By applying pressurized helium to the outside and connecting the tube to a mass spectrometer or vice versa, the actual leak rate can be measured as a function of both pressure and/or temperature. The package used for these experiments was a 34 pin plug-in Kovar package of a one piece bathtub construction whose only similarity to the units discussed above was the unexplained oxygen/argon spectra. The RGA failure, S/N 20, can be seen in Table 3 to have over 2.5% oxygen and a correspondingly high level of argon and moisture. S/N 56 was sent as a control since it had passed the RGA test and had only the normal background level of argon.

Both units were run through a sequence involving 3 externally pressurized runs in a row at 22°C followed by 3 internally pressurized runs. A single run for each unit was then made at 125°C while pressurized externally, followed by an internal pressure run. The later sequence was repeated at -11°C, the lowest temperature that could be achieved.

Figure 3 - Teledyne Hybrid S/N 22
Helium Leak Tests at 22°C

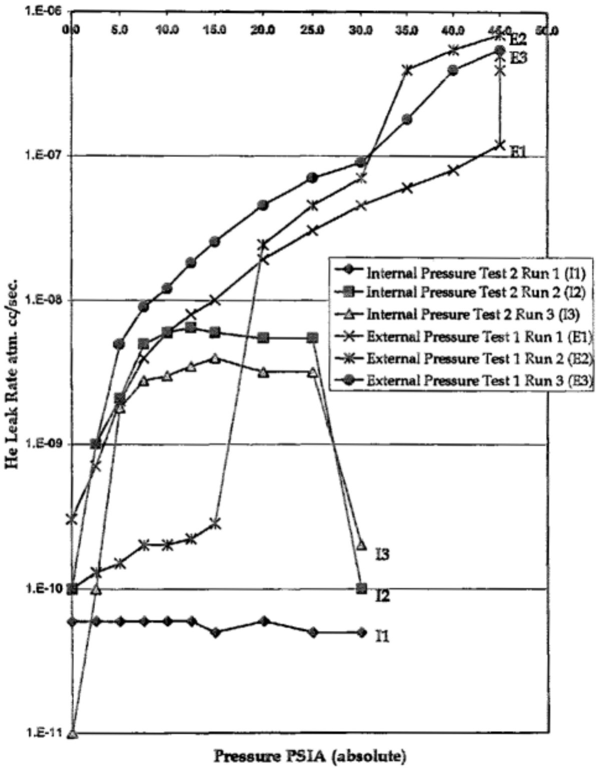
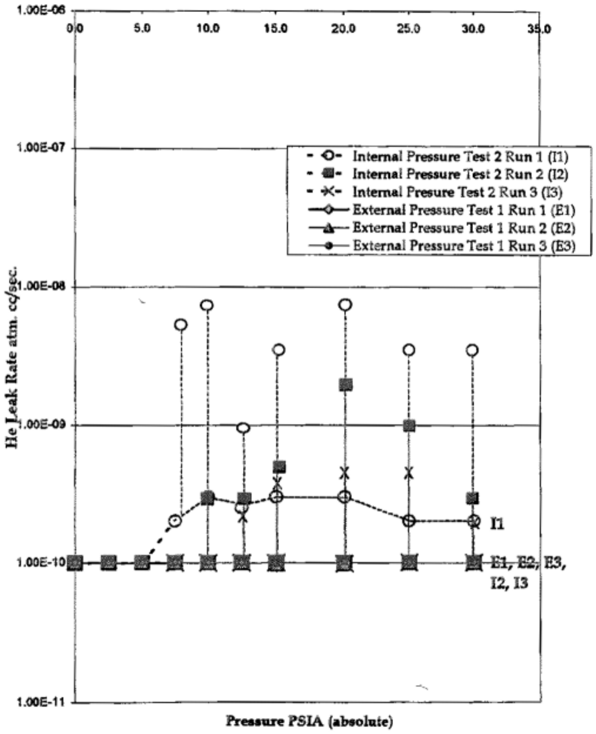


Figure 4 - Teledyne Hybrid S/N 56
Helium Leak Tests at 22°C



Both units were then internally pressurized to 15psig and placed in FC-77 to observe any bubbles. When none were observed they were clamped in a fixture to minimize flexure of the walls and internally pressurized to 60psig and again placed in the FC-77 tank. As a final step the units were soldered shut and tested with the traditional bombed He leak test method for comparison of the existing leak rates with previously obtained values.

Test Results

The results of the variable pressure tests at 22°C are plotted in Figures 3-4. The graph in Figure 3 shows that S/N 20 was extremely pressure sensitive, although somewhat erratic. The leak rate variations produced with external pressure were not reproduced on the initial internal pressure run. On the next two runs however a 15 fold increase in leak rate was observed at pressures up to 25psia that disappeared as the pressure exceeded 25psia.

The data in Figure 4 shows that S/N 56 had no leak rate dependence on external pressure. An unexplained transient leak rate was observed, however, under internal pressure conditions. This "burst" leakage occurred for short periods at me $5x10^{-9}$ Atm. cc/sec level before settling at a steady $4x10^{-10}$ Atm. cc/sec level on the first run. The burst behavior only was observed on the other runs.

The effects of pressure at high temperature were similar although not as severe. At cold temperatures the leak rates were typically lower under both internal and external pressure conditions. under external pressure at 125°C, the leak rate of S/N 20 never exceeded $1x10^{-8}$ Atm. cc/sec at up to 45 psia. while at –11°C the rate actually dropped to $<10^{-11}$ Atm. cc/sec. Under internal pressure, S/N 20 reached a rate of only $6x10^{-6}$ Atm. cc/sec at 125°C and $6x10^{-11}$ Atm. cc/sec. at –11°C.

For S/N 56, the temperature-pressure tests indicated no change in the leak status. No bubbles were observed from either unit under the pressurized fluorocarbon test. The subsequent He leak tests showed leak rates again in the low 10^{-9} Atm. cc/sec. range.

Discussion

S/N 20 is clearly a pressure sensitive leaker. The exact leak rates appear to vary depending on the history of the unit. This is hardly unexpected given the peculiar nature of the leak path. The values of the leak rates shown are qualitatively consistent with a leak created during burn-in for both types of package. A period of at least a week would have been required for the latter unit, with its volume of 7.2 cc, to achieve the observed O_2/Ar levels.

In both cases the 'external' pressure would have to have been applied mechanically. Clamping units with a domed bottom to a heatsink result in forces applied to the package that are in the same direction as those achieved with external air pressure.

It should also be noted that forces of similar value and magnitude can be achieved in both centrifuge testing at 5000g and fine and gross leak bombs at 30 psig or more (values permitted in both cases by Mil-PRF-38534). During the acceleration test, the units are in vacuum and see the pressure for short periods only (i.e. 1 min.). With the bombed leak test conditioning, the pressure can be applied for up to 24 hours and can be repeated several tines in the life of a unit but here again, as with acceleration testing, the atmosphere at the time would not contain sufficient O_2/Ar/H_2O.

With a view of the leak as transient, the RGA spectra for the life test units can be understood. The phenomena of hydrogen and oxygen reacting in the presence of a catalyst such as Ni to form H_20 at as low as room temperature is documented (7-8). In the absence of additional sources, the residual oxygen indicated by the presence of argon would have been used up over the 1000 hours of unclamped burn-in (of the three different burn-in conditions specified for the unit in question only one required clamping). Kovar is a well known source of hydrogen (9). The levels of hydrogen in units with low argon can be seen in Tables 1 and 2 to be much higher than in the apparent leakers.

Conclusions

- Oxide bonded glass to metal seals can exhibit variable leak rates under certain pressure conditions.
- This type of variable seal cannot generally be detected by the current Mil-Std bombed helium leak test methods, even if the allowable leak rate were lowered by an order of magnitude.
- Moisture levels are increased initially by leakage and subsequently by reaction of residual oxygen with hydrogen outgassed from the package base metal.
- The term "one-way leaker" is a misnomer as at the time the leak is present the usual laws of gaseous diffusion apply. The descriptive "onetime leaker" might be more appropriate.

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