

Leakage Rates through LTCC Substrates for Extreme Environment Applications

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Abstract

A key challenge with many extreme environment applications is the development of substrate technologies that provide the necessary electrical interconnect but also are reliable. Many of these applications also have a requirement for hermetic packaging. As a result, one logical solution is the use of ceramics such as Low Temperature Cofired Ceramics (LTCC) that are very stable and have proven to be reliable under a wide variety of external conditions such as elevated temperatures, and mechanical shock.

A popular approach is to mount components on one side of an LTCC substrate that is then sealed with a lid to create a hermetic environment for those components. Electrical interconnects are generally then feed through the LTCC to the bottom surface. The leak rates of gases or moisture introduced and retained within these structures are difficult to measure accurately using conventional tracer gas technologies. For example a small crack between the via metallization and the ceramic sidewall, created by extreme temperature cycling, could create a pathway into the hermetic volume with a very small leakage rate. However, since the total number of individual vias, or potential leakage points, is very great, the combined leakage rate may not be trivial. Therefore, the ability to quantify these leak rates is critical to high reliability applications.

To resolve this issue the researchers have investigated leak rates through LTCC substrates using krypton-85 tracer gas technology. An important advantage of this technology is that the quantity of gas introduced into a device may be measured directly, thereby allowing detection of gases introduced into and retained in interfacial voids, traces, and other microscopic interstitial passages and connections that may be present in these substrates. This paper will report experimental results using krypton-85 tracer gas technology to identify and quantify very small leakage rates in LTCC substrates both prior to and after exposure to extreme environmental conditions.

1.0 Introduction

A key concern in many electronic systems operating in extreme environmental conditions is excursions or cycling from cold to hot temperatures [1]. There are application when these excursions are either required or the ability to withstand them in a reliable manner facilitates a products design. Examples of the latter include reduced heat sinking or insulation / shielding requirements and or the ability to operate at higher coolant temperatures for liquid cooled systems [2]. In many of these applications, ceramics like LTCC are attractive since they are very stable at temperature extremes and can be easily fabricated into hermetic packages. For example, a common approach is to embed the circuit interconnects in the LTCC; place the components on one side of the substrate and external electrical connections on the other side. A lid can then be used to seal the component side to form a hermetic volume. One potential concern with this approach is that the via structures through the LTCC could potential form a pathway for leakage of gas or moisture into or out of the hermetic volume.

Little published data exists on the leakage rate through these structures, and this is likely due to the very small volume or potential leakage per via. In addition, as fabricated, the vias are filled completely with metal and the bond between the ceramic and the metal is very strong in most cases. However, even a very small leakage rate multiplied by hundreds or in some cases thousands of vias could potential create a reliability problem for sensitive products, and there is little published data on this issue. In addition, thermal cycling caused by extreme environmental conditions could induce defects in the LTCC which could lead to an increase in the combined leakage rate of the via structures.

This paper seeks to address this issue by employing hermetic testing on LTCC samples both before and after accelerated aging created by thermal shock.

2.0 Test Sample Fabrication Process

Test samples have been fabricated using a standard process flow for DuPont 951 LTCC. The use of a standard process and equipment was an important

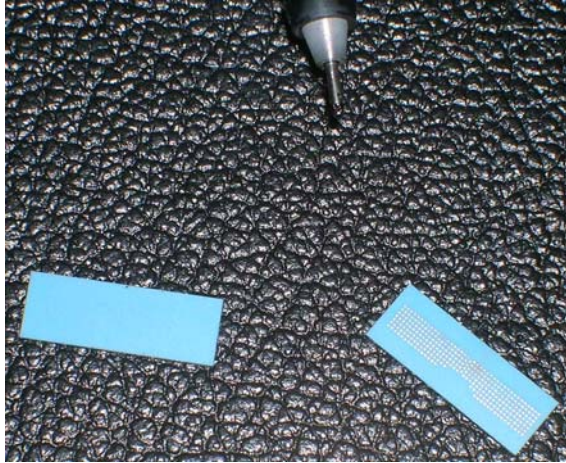


Figure 1: Two of the twelve samples prepared for accelerated aging and hermetic testing. The left sample is from the control set and contains no vias, while the right sample is from the set that contains thru vias.

step, since it is desirable that these test samples be representative of substrates created in a high volume-manufacturing environment. An array of twelve samples was fabricated in a 75 mm (3") square area of a 150 mm (6") LTCC blank. This approach was used to maximize the tape utilization with equipment designed for 150 mm blanks of LTCC. Each blank consisted of four layers of the test sample, since each sheet was cut into four sections that were laminated to form all of the layers used in this four-layer design. While a number of layer thicknesses are available in this material system, the researchers selected roughly the middle of the range with 951P2. This material has a nominal thickness of 165 microns (6.5 mils) and was furnished from the manufacture in precut 150 mm (6") sheets with a punchable backing.

Two sets of sample designs have been included on this panel of parts; the first design included no internal features and no vias, while the second design contained 268 150 μm diameter vias. The first design was used as a control since no cuts or penetrations were made in the ceramic, no leaks should be observed. The second set of samples was used to determine the combined leakage rate of these hundreds of thru vias both before and after accelerated aging.

The vias were created with a standard Ushio CNC punching system using a 150 μm punch and die set. Since all of the layers for all of the parts were created from only one 150 mm LTCC blank, a single punching step was required to form all of the vias within these samples.

Once the layer had been punched, the layer receives via fill in the form of Dupont 6141 silver via fill ink. This step was performed with a standard stencil printing process, and an AMI MSP-485 screen printer

operating in contact mode. The stencil used was provided by Sefar Printing Solutions and was composed of 150 $\mu\text{m} \pm 25 \mu\text{m}$ (5.5-6.5 mil) openings in a 25 μm (1 mil) stainless steel foil that was mounted to a 300 mm (12") aluminum frame. A plastic squeeze and a relatively fast printing speed were used to create a slight overfill condition. No capture pads were used in these parts; therefore, no metallization was printed on either surface of the tape.

Once the via had been filed the 150 mm blank was cut into four sections, manually stacked in a fixture, and laminated with a uniaxial press and the tape manufacturers recommended conditions. Punched structures in each of the 75 mm tape sections were used to align each to pins in the alignment fixture and therefore to each other.

Once the lamination process was complete, the entire 150 mm panel was fired, using the tape manufacturer's recommended profile, and then diced to size with a dicing saw. A resin-bonded diamond-dicing blade was used in conjunction with a Disco DAD320 dicing saw. While coarse grits blades can be utilized, the authors have found that diamond particle sizes larger than $\sim 30\text{-}40 \mu\text{m}$ produce rough edges and chipping of the LTCC material. Finer particle sizes, approximately 15 – 20 μm , coupled with low spindle speeds ($\sim 20,000$ rpm) and slow feed rates (0.25 in /sec) produce high quality cuts with minimal chipping even with a single pass through



Figure 2: ESPEC Thermal Shock chamber used for accelerated aging of the samples.

thick substrates.

While it is well known that machining parts to size in the green state is possible, dicing after firing provides a higher degree of dimensional accuracy. The key difference is that post machining the parts after firing eliminates any variation in this critical dimension caused by shrinkage variations. However, care was taken to ensure a smooth edge quality of the samples through the use of a fine saw blade. A coarse saw blade or inappropriate cutting conditions may have let to microscopic cracks on the edge surfaces that could have appeared as virtual leaks.

3.0 Accelerated Aging

All of the twelve-diced samples then underwent an initial Hermetic test as described below. After the initial testing, all of the samples were randomly placed inside of a ESPEC thermal shock chamber, shown in figure 2. This system can rapidly transfer the samples from a hot zone to a cold zone in a repetitive fashion to simulate the thermal shock created by the environmental conditions to which the component or system will be exposed.

While there are, a wide range of standard shock profiles that can be utilized most of these standards are limited to maximum temperatures of $\sim 155^{\circ}\text{C}$. Since the researchers are interested in more extreme environments, a modification of a common automotive standard shock profile was selected. While the standard normally specifies -40°C to $+155^{\circ}\text{C}$, the researchers expanded the range from -40°C to $+200^{\circ}\text{C}$. Ideally, a higher temperature would have been selected for the hot zone; however, 200°C is the highest temperature that the majority of air-to-air thermal shock systems can maintain without sacrificing temperature uniformity. The selected profile, as measured via a set of thermocouples and an external computer, is illustrated in Figure 3. All of the samples were exposed to 500 cycles of this profile in a continuous manner. Data from five different thermocouples that were located throughout the chamber were used to compile figure 3, which is just a sample of the 500 cycles, and ensure the uniformity of the temperatures within the chamber.

4.0 Microscopic Analysis

Samples were prepared and examined before and after testing with either a high magnification light microscope and / or a Scanning Electron Microscope to identify any cracks that may have formed during the aging process. An representative example of these images is illustrated in figure 4, after accelerated

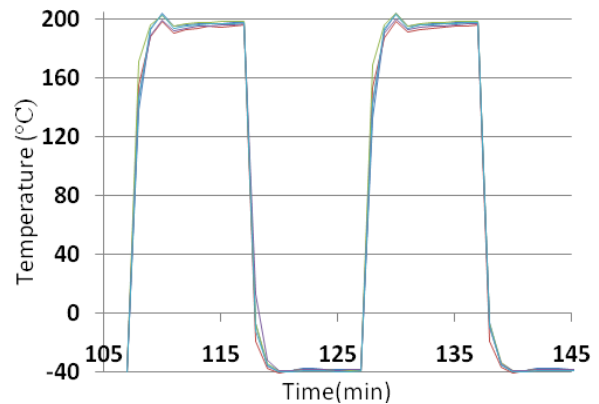


Figure 3: Temperature of samples recorded during thermal shock. Little deviation exists between the five-thermocouple measurements.

aging. No visible cracks were observed between the vias and the ceramic itself in any of the samples.

5.0 Hermetic Testing

A radioisotope technique utilizing krypton-85 is used for hermetic testing in this research. Since details concerning the fundamental principles of radioisotope leak detection and its application to the hermetic testing of devices ranging from microelectronic components to energetic devices are thoroughly described in the literature [3-6], only a cursory description of the method is presented in this paper. At the same time however, literature pertaining to the application of the radioisotope method in order to verify the hermetic integrity of LTCC substrates is sparse. For that reason, it is important to mention the most prominent advantages to the application of the krypton-85 leak detection technique to the LTCC materials of this study.

A. The Radioisotope Leak Detection Method

In this research, a Radiflo® Mark V leak detection apparatus [7] is used to expose the LTCC components to a gas mixture composed of 0.01% (by volume) ^{85}Kr in air at elevated pressures for specified periods of time. In the Radiflo® process, upon completion of the bombing cycle, the ^{85}Kr /air gas mixture surrounding the device in test is automatically recovered and returned to storage within the apparatus. Test devices are subsequently removed from the bombing chamber and delivered to a scintillation crystal detection apparatus for radiation measurement. Both gamma rays and beta particles are produced in the decay of ^{85}Kr [4]. The gamma rays are generally able to penetrate the walls of the test article with relatively little attenuation, and the

resulting signal produced by the scintillation crystal counting apparatus is used to calculate the amount of ^{85}Kr gas retained within the test article. Beta particle emission from the test device is measured using a Geiger-Mueller tube. Since beta particles are easily shielded, indication of beta particle emission may be attributed to the liberation of ^{85}Kr from the surfaces of the device, direct flow of gas from interior cavities or crevices, or a combination of these effects.

The period of time between completion of the pressure bombing cycle and the scintillation crystal measurement is commonly termed the “dwell” time. For small cavity devices exhibiting large (gross) leak rates, the dwell time becomes an important process parameter whose ramifications must be understood. The principal concern is that, given a gross leak from a small cavity device, the tracer gas contained within the component will escape before the scintillation measurement can be completed. This situation can be addressed to a limited extent through minimization of the dwell time, but the possibility of inadvertent acceptance of a grossly leaking small cavity device is always a possibility using conventional tracer gas leak detection methods – either ^{85}Kr radioisotope or helium mass spectrometry (HMS) methods [5, 6].

An important advantage to the radioisotope leak detection is that its range of applicability can be greatly extended to identify gross leaks from small cavity devices using two innovative techniques. One involves placing a small quantity of a ^{85}Kr “getter” material within a device [4] greatly extend the time of retention of ^{85}Kr within the device. Since the LTCC specimens subject of this research do not contain internal cavities and inclusion of getter material in the substrate is not desirable, this option is not feasible. Instead, a Radiflo® “wet” test method [6,8] is used in an attempt to ascertain the possible presence of porosity, cracks, surface contamination, or other imperfections that may be present in the LTCC substrates. With the inclusion of the wet test method, the radioisotope technique is ideally suited to this research.

B. Radioisotope Test Procedures

The specific radioisotope test procedures used in this study are based on those suggested for incorporation in the most recent version of MIL-STD-750E, Test Method 1071.9 [8]. As discussed above, the necessity for inclusion of the wet test is due to the recognized difficulty in performing reliable gross leak tests using any of conventional leak testing procedures in the Military Standards for those components with extremely small internal cavities (generally less than 0.001 cm^3). In radioisotope “wet”

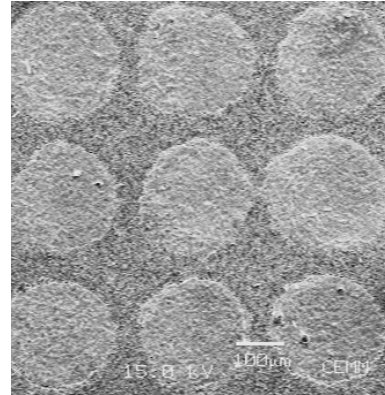


Figure 4: SEM image of vias through an LTCC sample after accelerated aging.

gross test, devices are exposed to a red dye penetrant under vacuum followed by pressure conditions, then solvent washed by acetone, followed by leak testing using the Radiflo® process. In this role, the red dye penetrant acts as both an indicator of surface imperfection sites and a ^{85}Kr getter for leak detection. Magnaflux Spotcheck® solvent removable red dye penetrant (SKL-SP1) is used for this research.

A “well” scintillation crystal with a cylindrically-shaped cavity measuring 1.5-inches in diameter by approximately 2.5 inches deep is used to detect gamma emission. Four ^{85}Kr standards with nuclide content traceable to the National Bureau of Standards are used to calibrate the crystal. Individual standards are placed at the bottom of the well crystal and the resulting scintillation rates are recorded. Through knowledge of the original ^{85}Kr content of the standard and correcting for radioactive decay since its production, the crystal counting efficiency (expressed as cpm/ μCi) is determined. The counting efficiencies well crystal is found to be 14,000 cpm/ μCi (+/- 10%). Crystal counting efficiencies are generally extremely stable and are rarely found to change. Nevertheless, the counting efficiency of the crystal is checked for accuracy before each test series using the calibration standards

Specifically, tests are applied in the following order:

- Radioisotope Dry Gas Gross Leak Test
A bombing pressure of 90 psia for 36 seconds is used. (This follows specific revisions recently suggested for adoption into MIL-STD-750E, Test Method 1071.9 test condition G₂ [8])
- Radioisotope Fine Leak Test
A bombing pressure of 90 psia for 6 minutes is used. (This follows MIL-

STD-750E, Test Method 1071.9 test condition G₃ [8])

- Radioisotope Fine Leak Test
A bombing pressure of 90 psia for 24 hours is used. (This is a custom test introduced to determine if any ⁸⁵Kr diffusion and solubility into the LTCC substrate is indicated.)
- Radioisotope Wet Gross Leak Test
Devices are exposed to red dye penetrant under vacuum and pressure conditions (10 minutes each cycle), Acetone washed, and Radiflo® tested at a bombing pressure of 90 psia for 6 minutes. (This follows MIL-STD-750E, Test Method 1071.9 test condition G₁ [8])
- Radioisotope Wet Gross Leak Test
Devices are exposed to red dye penetrant under vacuum and pressure conditions (3 hours each cycle), Acetone washed, and Radiflo® tested at a bombing pressure of 90 psia for 24 hours. (This is a custom test developed for this study. The solubility of tracer gas in the LTCC substrates is expected to be insignificant and the volume attributed to cracks - if they occur - will likely be extremely small. Therefore, anticipated scintillation rates as calculated considering substrate free volume and porosity estimates, in conjunction with crystal counting efficiency and nuclide content are also relatively low. Therefore, unusually long pressure bombing periods are introduced in an attempt to maximize test sensitivity.)

6.0 Results & Conclusions

All of the samples created in this study were observed to have no cracks or defects either before thermal shock or after thermal shock that could be identified with optical or electronic microscopy techniques.

The extensive Radiflo® testing was unable to identify any leakage from these parts or scintillation attributable to krypton-85 retained within the substrates themselves, either before or after accelerated aging. There was also no statically significant difference between measurements made on the control samples and the vias filled samples, since all of the radiation counts observed have been found to be within the background level. This

indicates that any leakage from the substrates or retention of krypton-85 within the substrates must be very small and below the threshold of detection.

7.0 Future Work

These results demonstrate the robust nature of the LTCC technology and demonstrate that no significant leaks are present in samples of this configuration even after harsh environmental exposure. However, the testing of more complicated substrates, with internal volumes, is also of interest and may be used to establish a minimal leakage rate for larger complex parts with thousands of vias.

8.0 References

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