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At ppm-ppb base performance for capacitors, it is difficult for the R & D engineer, at the development level, to determine with confidence whether a process or design change improves product quality.

This edition of Tech Topics and the next describe two novel techniques being used by KEMET to help direct process improvement.

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Scintillation Testing of Tantalum Capacitors

by Girdhar Arora

Background

Most electrical failures in tantalum capacitors are attributed to a single failure in the tantalum pentoxide (Ta_2O_5) dielectric film. The dielectric film is typically a few thousand angstroms in thickness (~20 Å/$V_{formation}$) and, in use conditions, is under high voltage stress due to the large electric field across it. Tantalum pentoxide in its amorphous state is insulating (a dielectric) while its crystalline form is, in effect, highly conductive. Catastrophic failure of a tantalum capacitor is normally associated with either local crystallization or field crystallization (bulk) of the dielectric.

Flaws in the dielectric play a major role in the conduction phenomena. The passage of current through these flaws causes changes in them(1). It has also been found that different flaws become active at different voltages. Therefore, it appears that a quantitative measurement of the activation of these flaws at different voltages would serve as a measure of the quality of the dielectric. This measure may in turn be related to the performance of the capacitors in the end-user tests, such as LifeTest and Load Humidity.

Several techniques have been used in the past to measure the quality of the dielectric. Leakage current has been widely used as a measure of dielectric quality. Klein used a redox printing technique to study the distribution of flaws in a flat film(2). Korinek used an activation energy approach by measuring leakage currents at different voltages and at different temperatures to study the dielectric quality(3). Burnham used a breakdown test to study the healing efficiency of the capacitors(4). All of the earlier approaches had serious deficiencies: specialized sample geometry was required (not directly applicable to a tantalum anode), attribute (pass/fail) data resulted, and aggregate quality levels of the dielectric were measured without focusing on the individual flaws. Most studies treated all flaws as equally damaging, which is not the case. Different fault sites in the dielectric have different response to electrical stimuli and only some may be capable of causing a failure. In addition, none of the earlier techniques studies the capacitors as they undergo the total manufacturing process.

As part of our continuous quality improvement efforts at KEMET, we developed a novel technique of studying the dielectric film. Called the Scintillation Test, this technique measures the voltage at which the dielectric film breaks down (scintillates) because of the activation of a single flaw. Since the breakdown occurs at a localized site, and arguably at the electrically weakest area of the film, the test measures the minimum electrical stress (field) required to create a failure. The voltage at which the breakdown occurs is called Scintillation Voltage.

Scintillation Test

A conceptual representation of the test setup is shown in Figure 1. A constant current source provides a fixed current to the device under test, causing the capacitor to charge up. A voltmeter measures the voltage buildup across the capacitor. When the capacitor scintillates, the scintillation causes the capacitor to discharge (at the scintillated site), resulting in a voltage drop measured by the voltmeter.

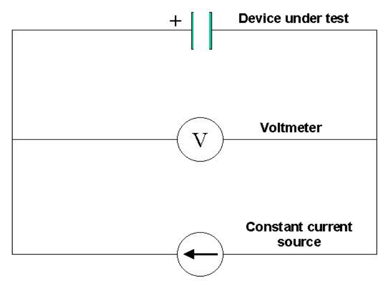

Figure 1. Conceptual Representation of Scintillation Test

A scintillation or dielectric breakdown is considered to be a consequence of very high electric field accompanied by very high instantaneous current densities at the breakdown site. These conditions result in very high thermal gradients in the region raising the local temperature above 500°C. At these temperatures, the solid electrolyte (MnO_2) undergoes a thermal-chemical conversion into lower oxidation state manganese oxides such as MnO, Mn_2O_3 and Mn_3O_4.

These reduced oxides are much less conducting than MnO_2 and act as an insulating plug at the breakdown site, electrically isolating the region from the rest of the capacitor. After the breakdown site and the resulting "short" are isolated, the capacitor recovers and begins to charge up. A typical scintillation test response is shown in Figure 2.

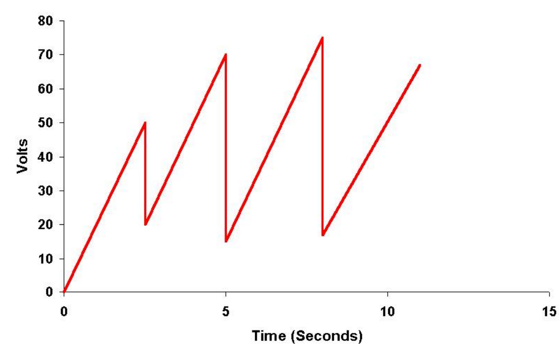

Figure 2. Typical Scintillation Test Data

The scintillation test, in essence, reveals the electrically weakest sites in the dielectric layer. Assuming that a potential failure site will belong to the group of weakest sites in the film, this information becomes very important because most dielectric failures are single-site failures. Comparing the scintillation voltage distribution of two groups of capacitors can then assist us in judging the quality of the dielectric and also in predicting the life of the capacitors in those groups.

Application to KEMET Chip Manufacturing Process

A brief flow chart of the tantalum capacitor manufacturing process is shown in figure 3. Scintillation tests were done on a sample of capacitors from a lot of 10µF/35 volt anodes after formation of the dielectric. The negative connection was made by immersing the anode in a liquid electrolyte of 0.1M nitric acid at room temperature in a container with a stainless steel cathode. During the scintillation test, each capacitor was charged with a 200μA current up to 100 volts. The first scintillation voltage (weakest site) of each capacitor was recorded. All the scintillation voltages from the sample were plotted as a histogram to show their distribution. The process was repeated for a sample of capacitors drawn after each of the listed processes in Figure 3 from the same lot of anodes.

Formation of Ta_2O_5 Dielectric

↓

Solid Electrolyte (MnO_2) Impregnation

↓

Reformation

↓

Solid Electrolyte (MnO_2) Impregnation

↓

Reformation

↓

etc.

Figure 3. Electrochemical Process -Tantalum Capacitor Manufacturing

The quality of the dielectric after formation is very good, as would be expected. After the dielectric formation step to a maximum of 150 volts, none of the capacitors scintillated up to maximum test voltage of 100 volts. While the dielectric film may still have flaws, in a strict technical sense (because of impurity atoms, for instance) these flaws are not electrically active below 100 volts. When formed anodes are processed through the impregnation process (multiple cycles of in-situ thermal conversion of manganese nitrate into manganese dioxide solid electrolyte, within the pores of the anodized tantalum anode), the quality of the dielectric degrades drastically. Scintillation voltages as low as 45 volts are observed in some capacitors after impregnation is completed.

The distribution of scintillation voltages after impregnation is broad and continues to broaden after each successive cycle of manganese nitrate thermal conversion. The reduction of scintillation voltages of tantalum anodes and broadening of their distribution clearly shows the extent of damage done to the devices as they proceed through manufacturing. It is important to point out that this damage is not related to lapses in quality control or to the processes of any one tantalum capacitor manufacturer. This dielectric damage is a direct consequence of chemical and physical changes that thermo-chemical conversion of manganese nitrate causes to the very thin amorphous tantalum pentoxide. Details of these changes can be found in the literature [5].

Reformation is a traditional process to repair the damaged dielectric film. While it is well known that reformation is necessary because of the benefits seen at end-of-the-line testing or in life tests, a direct measure of the extent of repair is obviously a very valuable process improvement tool. Scintillation testing was used to measure the quality of the dielectric after two different reformation processes to study their effectiveness in improving the quality of the tantalum pentoxide dielectric. Figure 4 shows scintillation voltage distribution of anodes before reformation and after those two reformation processes. The first scintillation voltage of each anode in the test samples is plotted against the percentile ranking of that anode in the sample distribution. For instance, if an anode scintillated at 60 volts and 50% of the anodes in its group scintillated below 60 volts, then the anode will be plotted on the graph with an abscissa of 60 volts and an ordinate of 0.5.

In Figure 4, both process A and B improved the scintillation voltage distribution of anodes remarkably from the state before the processes. However, process B is more effective than process A because it results in higher scintillation voltages and tighter distribution. Another insight available from this data is that while reform has improved the quality significantly, there is still room for further improvement of the process. The quality of the dielectric is not as good as it is right after the formation process. This realization has been a strong motivation to further improve our processes.

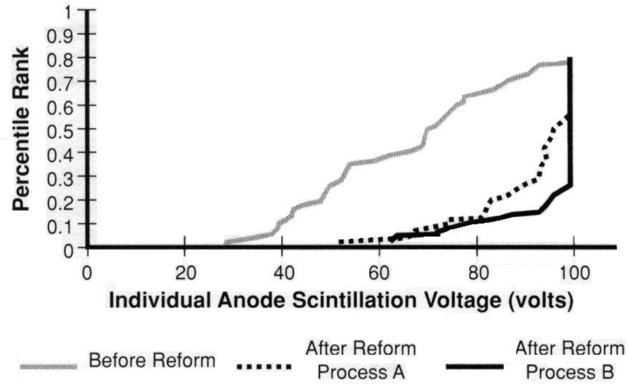

Figure 4. Comparison of Reformation Processes

While degradation of the dielectric during the impregnation process and subsequent improvement in reformation are widely known in the industry, as these processes are used by all tantalum capacitor manufacturers, this test is perhaps the only method of directly quantifying the extent of changes occurring in the dielectric during the manufacturing process. The test has several key merits. It provides a direct measure of the quality of the underlying dielectric. It focuses only on the electrically weakest sites in a capacitor and ignores those that are not affected by electrical stimuli, thereby reducing noise from the data. It is a stress-to-fail type test by which inferences about the capacitor's long-term performance may be drawn.

KEMET has used the scintillation test at several other points in our process to isolate the processes and the conditions that damage the quality of the tantalum capacitors. Using this information, we are leading the way in improving tantalum capacitor technology to produce even more reliable parts for our customers.

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