Solid Tantalum Capacitor Dielectric Failure Mechanism and Determination of Failure Rates

by H. W. Holland
DESIGN

To understand the solid tantalum capacitor failure mechanism, some knowledge of the product design and the manufacturing process is necessary. The basic raw material is tantalum. In the first manufacturing step, tantalum is prepared as a fine powder. The powdered tantalum is fed to molds where compression produces a coherent mass, usually in the form of a cylindrical or rectangular shaped pellet. A tantalum lead wire is also embedded in the compressed powder during this operation, the wire extending axially from the end of the pellet.\(^1\) Pressure is controlled so that the pellet density results in a porous body of high internal surface area.

The pellets are sintered at high temperature in vacuum furnaces. Sintering vastly increases cohesion of the tantalum particles and thus assures high electrical conductivity. The pellets also become mechanically rugged structures. Just as important is the purifying effect of the high temperature vacuum bake. Tantalum is a refractory metal, and sintering temperatures range from 1400° to 1900°C. This temperature, in combination with the low furnace pressure (usually less than 10^-4 torr), vaporizes most impurity materials, which are then pumped away in the vacuum system. As will be shown later, failure of capacitors is directly influenced by the residual impurities inevitably left behind.

Sintered pellets are anodized in an acidic bath to form a layer of tantalum pentoxide (Ta₂O₅) on all surfaces reached by the electrolyte. This oxide layer, whose thickness depends upon the applied voltage, is the dielectric of the capacitor. The capacitance value, of course, is inversely proportional to the dielectric thickness. On the other hand, higher voltage ratings require thicker dielectric films. The product of capacitance and anodizing voltage (CV product) is very nearly constant for a pellet of given surface area and is a convenient design parameter.

Anodized pellets are next coated with a semi-conductor “solid electrolyte” that enters and fills the pores to contact as much of the capacitive surface area as possible. The solid electrolyte is manganese dioxide (MnO₂), formed in situ by pyrolysis of a manganese nitrate solution into which the porous pellets are dipped before pyrolysis. Several cycles of dipping in nitrate solution, each followed by thermal decomposition of nitrate to dioxide, are necessary to complete the solid-electrolyte layer.

The remainder of the manufacturing cycle is directed at providing an encapsulation with external lead-wire connections. The positive, or anode, lead is readily obtained by welding to the tantalum wire originally attached to the anode pellet. The cathode connection begins with application of a conductive system over the manganese dioxide. This system is usually composed of succeeding layers of colloidal graphite, silver-loaded paint, and for leaded devices, solder. In the popular hermetically-sealed designs, the solder anchors the pellet inside a metal can to which the negative lead wire is attached. Figure 1 shows this design schematically. In the surface mount tantalum capacitor, the negative termination is a lead frame and is attached by a conductive epoxy.

DEFINITION OF FAILURE

Several criteria could be chosen to define a failure. The nature of the solid tantalum capacitor, however, is such that the short circuit deserves most of the attention. This type of failure is characterized by a sudden rise in leakage current, within a few milliseconds, from nanoampere or microampere magnitude to ampere magnitude in low-impedance circuits. Failure from an open-circuit condition can be produced through faulty manufacture, but these parts are easily detected and scrapped in the plant.

Other definitions of failure would necessarily be centered about changes in leakage current of relatively small degree or changes in the other two important parameters,
capacitance and dissipation factor. Leakage current can be made so small (below 0.01 microampere per rated microcoulomb) that changes of even 100% become insignificant in nearly all applications. Similarly, changes in capacitance and dissipation factor during normal service life are small enough to present no real problem to nearly all applications. No circuit designer, however, can allow for complete short circuit, and this failure mode properly becomes the focus of attention in reliability studies.

MECHANISM OF FAILURE

If the tantalum pentoxide layer, which constitutes the dielectric, were perfectly continuous and of uniform thickness throughout, leakage current would be several orders of magnitude lower than that of the best practical capacitors yet made. The leakage currents normally exhibited must flow, not through the dielectric, but through faults in the dielectric. These faults occur primarily because of impurities in the tantalum metal that have defied efforts at removal. Whenever an impurity is encountered, anodization cannot produce a continuous $\text{Ta}_2\text{O}_5$ layer of uniform thickness. The result is a minute hole or thin spot in the dielectric layer that can be filled with the MnO$_2$ solid electrolyte or with air. Compared with $\text{Ta}_2\text{O}_5$, either of these materials will allow relatively heavy conduction under conditions existing within the capacitor.

Simple probability indicates that the larger the capacitive area, the larger the number of impurity sites to be encountered. If impurities were randomly distributed over the underlying area of tantalum, the number of fault sites would be directly proportional to the size of the anode pellet. However, nearly all the surface area is reached via pores in the pellet. Also, it will be recalled that final purification is accomplished during sintering. If the pellet’s size is increased (especially by increasing cross-section) the mean free path for impurity removal becomes much longer. Consequently, capacitors with large anodes present a difficulty in manufacture beyond that indicated by a simple size relationship.

There is, in addition, a depthwise distribution of impurities in any given particle of tantalum within the pellet. This distribution is also derived from the purification process, those impurities near the original surface being more easily removed during sintering. Figure 2 represents this situation when anodizing takes place. The layer of tantalum near the original surface has been reacted electrochemically to form, $\text{Ta}_2\text{O}_5$. To form a thicker $\text{Ta}_2\text{O}_5$ film will obviously require reaction that penetrates deeper and encounters higher impurity densities. Thicker dielectric films are necessary for higher voltage ratings; these ratings consequently become more difficult to produce.

It has been stated that each impurity site in the dielectric produces leakage current when a conductive material is present. Leakage currents would be many, many times larger than they are if it were not for a change in MnO$_2$ structure that takes place opposite the fault sites. As D.C. voltage is applied to a newly manufactured capacitor, high current densities are produced in the faults. Since the faults are small (in the Angstrom-unit range), large total currents are not necessary to produce extremely high current densities. The high current produces Joule heating in the MnO$_2$ opposite the fault. At elevated temperature, MnO$_2$ undergoes spontaneous reduction to lower oxides. The lower oxides coincidentally display much higher electrical resistivities, effectively reducing the leakage current that originally produced the heating. The magnitude of this effect can be deduced by noting that MnO$_2$ has a resistivity of approximately 10 ohm-cm, while Mn$_2$O$_3$ has a resistivity of approximately $10^4$ ohm-cm.

If a relatively small fault site is encountered, the mechanism just described may

2. In this discussion it is assumed that manufacturing variables such as powder particle size, pellet density, and sintering time and temperature remain fixed. These are degrees of freedom in design that can be used to alter the magnitude of the size effect.

3. Figure 2 does not purport to show the exact appearance of Ta$_2$O$_5$ at fault sites, but illustrates that defects in the dielectric follow from impurities in the metal substrate.
permanently reduce the leakage current associated with that site to a very low value. If a large fault site is encountered, the amount of heat produced may be sufficient to destroy a yet larger area of dielectric through crystallization of the normally amorphous oxide. An avalanche condition is then produced, leading inexorably to a short-circuited capacitor. Between these extremes are intermediate conditions producing capacitors with various leakage currents. Some of these are on the fringe of stability. For these, a high external circuit impedance limits the fault current and assists the MnO₂ “healing”, whereas a low impedance circuit produces catastrophic failure. It is thus important to keep life-test circuit impedance low to produce worst-case conditions in reliability studies. Scintillation (or flicker) is a phenomenon favored by high-impedance circuits. This term describes a momentary pulse of current that is shut off by “healing” action. Low-impedance testing usually converts scintillation to catastrophic failure.

STATISTICAL STUDIES OF FAILURE

The postulated mechanism of failure may have a somewhat different beginning to the same end if it is agreed that impurities migrate during service, subsequently causing a dielectric fault. This possibility should cause no alarm if the chosen mathematical model can allow for such heterogeneity of cause. Indeed, it should be recognized that production lots can probably never be made such that every capacitor is precisely identical to every other capacitor; neither can one lot be made identical to every other lot. These difficulties should be overcome if entire lots are tested and the survival data fitted to a distribution for each lot.

Behavior of solid tantalum capacitors in service follows a pattern that is entirely consistent with the failure mechanism described above. Two service conditions (assuming constant circuit impedance) are important: temperature and applied D.C. voltage. At any given combination of temperature and voltage, the average leakage current will trend downward during life test. The “healing” effect apparently continues to form better and better barriers at the fault sites. Occasionally, there will be a catastrophic failure when leakage current rises in avalanche fashion. In these instances, a localized failure in the dielectric, resulting from perturbation of the healing equilibrium, has apparently taken place. The rate at which failures occur in any given group of capacitors can be determined by periodic counting of the number failed. Such counts form the basis for prediction of the probability of failure during anticipated service life.

Failure rate (usually expressed as percent failed per thousand hours of operation) of any device can increase, decrease, or remain constant with respect to time. With some devices (such as the human being) all three situations are found: a high infant mortality, decreasing to a constant rate caused by random events, and finally a wear-out period when the rate increases. Only the first of these periods has been found applicable to solid tantalum capacitors: that is, the failure rate decreases and continues to decrease for times at least in excess of 70,000 hours. Obviously, application of a very large over-stress can cause immediate failure of all parts, so it is possible to select conditions where failure rate does not decrease with time. This aspect will be treated more thoroughly in a discussion of “acceleration.”

The characteristic decreasing failure rate makes statistical treatment more difficult than the frequently-assumed constant failure rate. There is, however, a distribution function, published by Waloddi Weibull, that can conveniently treat variable failure rates. It is stated in its useful two-parameter form as:

\[ F(x) = 1 - \exp \left( - \frac{x^\beta}{\alpha} \right) \]

where \( x = \) time
\( \beta = \) the "shape parameter"
\( \alpha = \) the "scale parameter"

\( F(x) \) gives the cumulative fraction of the group that has failed to time \( x \). The prob-
ability of failure at any particular time $x$ is given by the first derivative of the cumulative function:

$$F'(x) = \frac{\beta}{\alpha} x^{\beta-1} \exp\left(-\frac{x^\beta}{\alpha}\right)$$

The fraction of the group surviving at time $x$ is one minus the cumulative fraction failed:

$$1 - F(x) = \exp\left(-\frac{x^\beta}{\alpha}\right)$$

The instantaneous failure rate is the probability of failure at time $x$ divided by the fraction of the original group surviving at time $x$:

$$Z(x) = \frac{F'(x)}{1 - F(x)} = \frac{\beta}{\alpha} x^{\beta-1}$$

To make the easy use of the Weibull function, a special graph paper has been constructed, based on the following transformation:

$$1 - F(x) = \exp\left(-\frac{x^\beta}{\alpha}\right)$$

$$\frac{1}{1 - F(x)} = \exp\left(-\frac{x^\beta}{\alpha}\right)$$

$$1n \frac{1}{1 - F(x)} = \frac{x^\beta}{\alpha}$$

$$1n 1n \frac{1}{1 - F(x)} = \beta 1n x - 1n \alpha$$

The latter equation is the form of a straight line with slope $= \beta$ and y-intercept $= -1n \alpha$. The grid of Figure 3 has $1n x$ as the principal abscissae and $1n 1n \frac{1}{1 - F(x)}$ as the principal ordinate. Auxiliary scales permit direct plotting of $F(x)$ (cumulative fraction failed) on the ordinate versus $x$ (time of failure) on the abscissa.

A straight line is obtained when solid tantalum capacitors are put on life test and the cumulative failures plotted versus time on the Weibull grid. The significance of $\beta$ should now be noted. When $\beta = 1$, failure rate is constant; when $\beta > 1$, failure rate is increasing; and when $\beta < 1$, failure rate is decreasing. Observed slopes of solid tantalum capacitors are uniformly less than unity unless stressed well above their temperature-voltage ratings or previously damaged in some way (e.g., by reverse polarization at high voltage). Once the line has been established, $\alpha$ and $\beta$ are readily obtained, and the instantaneous failure rate at any time can be calculated. Extrapolation will determine the failure rate at some time in the future, or conversely, the time required before the failure rate falls below some given value.

**ACCELERATION**

Solid tantalum capacitors carry rated voltages that are intended to apply at $85^\circ C$ or below. Supplemental ratings above $85^\circ C$ are made to $125^\circ C$, but permissible voltage is decreased accordingly. This rating scheme fits the concept described above: temperature and voltage are the two important and inter-related conditions determining anticipated life in service. It is well recognized that, at any given temperature below the maximum $125^\circ C$, voltages lower than rated will lead to improved reliability (i.e., lower failure rates). Similarly, higher voltages will lead to higher failure rates.

Since failure rate is increased by increased voltage, the failures that would ordinarily be observed over a long period of time are made to occur within a shorter time span by application of voltage above rated. Any aging effects are thus "accelerated". For example, application of 1.1 times the rated voltage might mean that every hour on test under this condition and at $85^\circ C$ is equivalent to four hours under the conditions of rated voltage and $85^\circ C$. If very high voltages are applied, the failure mechanism might change. There is thus a limit to the degree of acceleration beyond which consistent relationship to applied voltage is no longer obtained.

**APPLICATIONS TO DETERMINE FAILURE RATE**

Use of the Weibull distribution as a model was shown above, and acceleration factors were described. Combining the two provides
Knowing $\alpha$ and $\beta$, it is possible to calculate the instantaneous failure rate, $Z(x)$, at any time. It is convenient to determine $Z(x)$ at one hour. The earlier equation,

$$Z(x) = \frac{\beta}{\alpha} x^{\beta-1}$$

is now modified by a factor of 105, to convert to the conventional units of percent failed per thousand hours, and by the acceleration factor, $A$, to convert to real time:

$$Z(x) = \frac{\beta}{\alpha} x^{\beta-1} \times \frac{10^5}{A}$$

when $x = 1$, $Z(x) = \frac{\beta}{\alpha} \times \frac{10^5}{500} = 0.5 \times \frac{10^5}{10^3} = 0.1\%/K$ hr.

Converting the instantaneous failure rate equation to logarithmic form simplifies further calculation:

$$\log Z(x) = \log \frac{\beta}{\alpha} + (\beta-1) \log x$$

or the failure rate at one hour. A plot for the numerical example is given in Figure 4. The required test time to attain a failure rate goal can be read directly. The entire lot of capacitors would then remain on test until the selected goal had been attained.

As an example, Figure 3 shows a Weibull plot for a lot of solid tantalum capacitors placed on test at 85°C and 52 volts. The rated voltage of these capacitors was 35, so the test voltage was 1.48 times rated. Mil-C-39003 has established exact acceleration factors correlating actual time in test at accelerated voltages to a much longer time at rated voltage, but for this example, lets assume that every hour on test is equal to 1,000 hours under rated conditions. This value is called $A$, the “acceleration factor.” From the Weibull plot, $-\ln \alpha$, the intercept with the principal ordinate, may be read directly as 6.23, yielding a value for $\alpha$ of 500. The value of $\beta$ is determined as the slope of the line:

$$\beta = \frac{2.3}{4.6} = 0.5$$
Figure 1.  KEMET Solid Tantalum Electrical Capacitor

Figure 2.  Effect of Impurities in Anodization of Tantalum

Figure 3.  Weibull Plot for 35 Volt Capacitor Tested at 52 Volts, 85°C.

Figure 4.  Weibull Parameters Determined from Figure 3. Used to display Failure Rate versus Test Time.
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