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## Reliability studies on solid tantalum electrolytic capacitors

Hamed Nassar  
*New Jersey Institute of Technology*

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RELIABILITY STUDIES  
ON SOLID TANTALUM ELECTROLYTIC CAPACITORS

by  
Hamed Nassar

Thesis submitted to the Faculty of the Graduate School of  
New Jersey Institute of Technology in partial fulfillment of  
the requirements for the degree of  
Master of Science in Electrical Engineering  
1984

APPROVAL SHEET

Title of Thesis: Reliability Studies on Solid Tantalum  
Electrolytic Capacitors

Name of Candidate: Hamed Nassar  
Master of Electrical Engineering, 1984

Thesis and Abstract Approved: -----  
Date

Dr. R. P. Misra  
Professor of Electrical  
Engineering and Reliability

-----  
Date

-----  
Date

VITA

Name: Hamed Nassar

Degree and date to be conferred: MSEE, May 1985

Secondary education: Shebin El-Qanater Secondary School

Collegiate institutions attended	Dates	Degree	Date of Degree
Ain Shams University, Egypt	1974-79	B.S.E.E.	May, 1979
New Jersey Institute of Technology	1983-84	M.S.E.E.	May, 1985

Major: Electrical Engineering

Position held: Teaching Assistant

Suez Canal University, Suez, Egypt

## ABSTRACT

Title of Thesis: Reliability Studies on Solid Tantalum  
Electrolytic Capacitors

Hamed Nassar, Master of Science in Electrical Engineering, 1984

Thesis directed and approved by: -----  
Dr. R.P. Misra  
Professor of Electrical  
Engineering and Reliability

The primary objective of this thesis is to determine and analyze the failure mechanisms of solid tantalum capacitors, pinpoint the causes, and suggest the precautionary measures that prevent the occurrence of these failures. To achieve this goal, the study went in two directions, theoretical and experimental. The theoretical part is a comprehensive review of the work done on tantalum capacitors since 1960 up to date.

The experimental part of the study is a life test (1000 hours) of solid tantalum capacitors under humidity and high temperature. To avoid the misconception that could happen by investigating a particular manufacturer's product, units from five international companies are tested. For each unit, three parameters are measured before and after the life test; namely capacitance, equivalent series resistance (ESR), and leakage current. Both humidity and temperature were found to have profound effects on capacitor behaviour.

The study was also able to rank the five major capacitor producers according to their units behaviour before and after the life test.

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I would like to express my deep appreciation and utmost gratitude to Dr. R. P. Misra, under whose supervision this work was undertaken. During the period I worked with him, I benefitted considerably from his wide range of knowledge and invaluable suggestions.

I would also like to thank Dr. S. Pandey, of the Electrical Engineering Department, for the help he provided in the experimental work.

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## CHAPTER I: CAPACITOR

Before going through the performance, reliability, and failure mechanisms of solid Ta capacitor, It is necessary to give a brief review of what capacitor is all about. History and development of electrolytic and Ta capacitor is also given.

### 1-1. Capacitance:

Capacitance is present between any two adjacent conductors. A capacitor consists basically of two metal plates separated by a dielectric material, such as air, gas, paper, ceramic, or an oxide film. When voltage is applied between the two metal plates, the capacitor will become charged, and the amount of the charge will depend primarily on the voltage. The capacitance of the capacitor is the ratio of the charge acquired to the voltage applied, or

$$C = \frac{Q}{V}$$

Where      Q: Charge, coulombs (or amp-sec)  
              V: Voltage, volts  
              C: Capacitance, farad

The farad is the unit of capacitance and a capacitor has a capacitance of 1 farad if its potential rises by 1 volt when it receives a charge of one colomb. This unit is inconvenient for practical use and the pf and  $\mu$ f are rather used.

The energy of the charge is stored up as electrostatic energy in the dielectric and is equal to  $CV^2/2$ . If this energy is absorbed at a uniform rate over a time  $t$ , the power required is

$$P_{av} = \frac{\frac{1}{2} CV^2}{t}$$

Under ac condition the rate at which energy is supplied to and returned by the capacitor is

$$P_{av} = 2 CV^2 f$$

Where  $V$  r.m.s. voltage

When a dc voltage is applied to a capacitor, and the bound electric charges are polarized or displaced from their normal position of equilibrium. Work is therefore done in charging the capacitor. The work done in joules (or watt seconds), which is available as stored potential energy is

$$J = \frac{1}{2} QV \text{ or } \frac{1}{2} CV^2 \text{ or } Q^2/2C$$

The equivalent circuit of a capacitor may be represented as shown in Fig (1).

- Where
- C: capacitance of capacitor
  - $R_s$ : resistance due to leads, contacts, etc...
  - $R_p$ : resistance due to the dielectric
  - L: inductance of leads and the plates of the capacitor

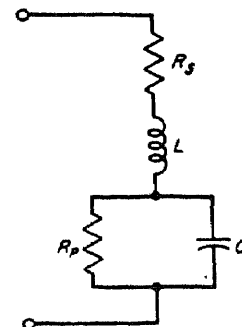


FIG. 1. Equivalent circuit of a capacitor.

1-2. Dielectric Constant:

The dielectric constant, permittivity, or specific inductive capacity of any material used as a dielectric is equal to the ratio of the capacitance of a capacitor using the material as a dielectric, to the capacitance of the same capacitor using vacuum as a dielectric. Dielectric constant of some materials is given in table Fig (2).

Dielectric Constant K

Vacuum .....	1.0
Dry Air .....	1.00059
Polystyrene .....	2.5
Polytetrafluoroethylene (Teflon*).....	2.0
Polyethylene terrephthalate (Mylar*)...	3.0
Impregnated paper.....	4.0 - 6.0
Mica .....	6.8
Tantalum oxide .....	25.0+
Aluminum oxide .....	7.0
Ceramic (magnesium titanate, etc.).....	Up to 20
Ceramic (titania).....	80 - 100
Ceramic (high-K ).....	1,000 and upward

Fig. (2) dielectric constant for different materials

+ Approximate value, with general acceptance.

The high dielectric constant in high-K ceramic capacitors comes from the fact that the electric charges in the molecular structure of the material are very loosely bound and can move almost freely under the polarizing voltage, resulting in high total capacity.

Dielectric can be classified in two main categories: polar and non polar materials. Polar materials have a permanent unbalance in the electric charges within the molecular structure. The dipoles within the structure consist of molecules whose ends are oppositely charged. These dipoles therefore tend to align themselves in the presence of an alternating electric field (if the frequency is not



too high). The resultant oscillation causes a large loss at certain frequencies and at certain temperatures.

In nonpolar materials, the electric charges within the molecular structure are balanced and the dipoles do not rotate under applied fields although they may distort. No sharp loss peaks with frequency and temperature therefore exist. Polyvinyl chloride (PVC) is an example of a polar material, having a dielectric constant of ten at low frequency falling to 3 at a few mega hertz, while polystyrene is an example of a nonpolar material, having a dielectric constant of approximately 2.5 for dc and for ac up to a few megahertz.

### 1-3. Losses in Dielectric Materials:

Losses occur due to current leakage, dielectric absorption, etc. depending on the frequency of operation. The loss-frequency curve for non polar and polar dielectrics is shown in Fig (3), (4).

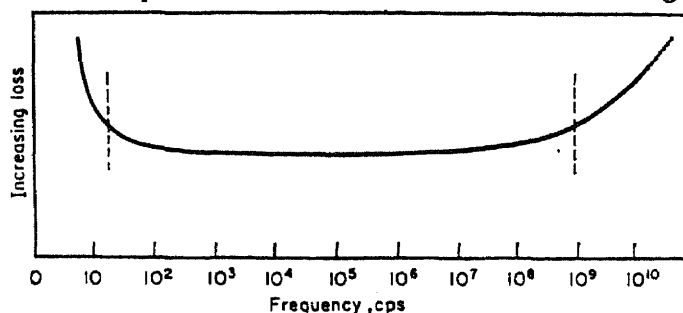


FIG. 3 Loss frequency curve for a nonpolar dielectric.

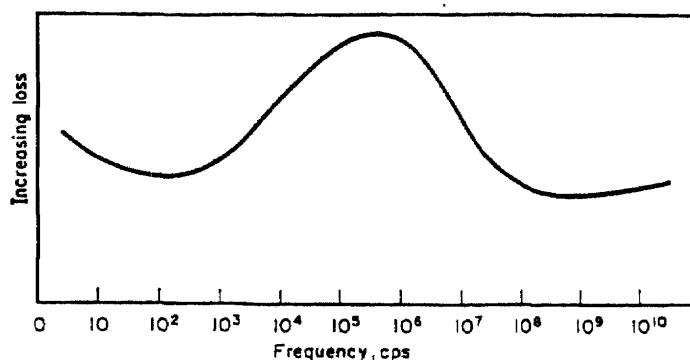


FIG. 4 Loss frequency curve for a polar dielectric.

The variation of dielectric constant with frequency is negligible so long as the loss is low. Increased losses occur when the process of alignment cannot be completed, owing to molecular collisions, and in these regions there is a fall in dielectric constant. Viscous drag in the molecular structure limits the frequency at which full alignment can be carried out. If the applied frequency is comparable with the limit frequency, losses will become high.

The power factor of the capacitor is defined as the ratio

$$\frac{\text{Power wasted per cycle}}{\text{Power stored per cycle}}$$

and for good dielectrics, this is dependent on the frequency.

When ac voltage is applied to a capacitor, the current leads the voltage by a little less than the theoretical  $90^\circ$ , Fig (5).

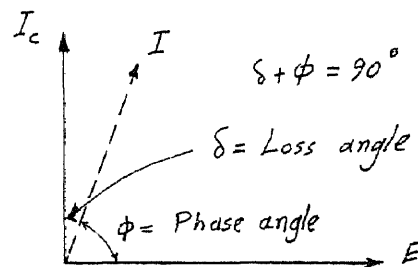


Fig. (5) Vector diagram for a capacitor

The phase angle is termed  $\phi$ , whereas its complement is termed  $\delta$ , the loss angle. The cosine of the phase angle or the sine of the loss angle is the power factor. The dielectric loss is therefore  $EI \cos \phi$

or  $EI \sin \delta$ . The loss angle  $\delta$  is normally so small that  $\tan \delta$ , the dissipation factor, can be considered equal to  $\sin \delta$  or  $\cos \delta$ , since it is easier to measure.

In perfect capacitor with no dielectric loss,  $\delta = 0$ . The dissipation factor is  $\tan \delta = 2\pi fRC$ .

#### 1-4. Dielectric Absorption:

If a capacitor were completely free from dielectric absorption, the initial charging or polarization current when connected to a dc supply would be

$$i = \left(\frac{E}{R}\right) e^{-t/CR}$$

Where       $i$ : current flowing after time  $t$ .  
              $E$ : applied dc voltage.  
              $R$ : capacitor series resistance.

and the polarization current would die off asymptotically to zero. If  $R$  is small, this takes place in a very short time, and the capacitor is completely charged. In all solid dielectric capacitors it is found that, after a fully charged capacitor is momentarily discharged and left open circuited for some time, a new charge accumulates within the capacitor, because some of the original charge has been "absorbed" by the dielectric. This produces the effect known as dielectric absorption. A time lag is thus introduced in the rate of charging and of discharging the capacitor.

Dielectric absorption is due to the finite time taken to displace the bound charges in the dielectric from their normal positions because of the viscous forces which resist their motion. Polarization may be almost instantaneous or it may take many hours. Even within the same dielectric, a few electrons or ions become free to move only after a period which may be seconds or even days.

The effect of dielectric absorption is to reduce the capacitance as the frequency of operation is increased and also to cause unwanted time delays in certain pulse circuits or in circuits requiring rapid charge and discharge characteristics.

#### 1-5. Leakage Current and Time Constants of Capacitors:

Losses due to leakage currents when a capacitor is being used on direct current prevent indefinite storage capacity being realized, and the charge acquired will leak away once the source is removed. The time in which the charge leaks away to  $1/e$  or 36.8% of the initial value is given by  $RC$ , where  $R$  is the leakage resistance and  $C$  is the capacitance. If  $R$  is measured in  $M\Omega$  &  $C$  in  $\mu F$ , the time constant is in seconds.

Some typical time constants for various dielectrics used in capacitors are:

Polystyrene	several days
Impregnated paper	several hours
Ta-pellet electrolytic cap	1-2 hours
High dielectric constant cap. (ceramic)	several minutes
Plain-foil electrolytic cap.	several seconds

It should be borne in mind that below capacitance values of about  $0.1\mu\text{F}$ , the time constant is generally determined by the structure, leakage paths, etc., of the capacitor assembly itself rather than the dielectric material. Leakage current increases with the increase of temperature (roughly exponentially). In good dielectrics at room temperature it is too small to measure, but at higher temperatures the current may become appreciable even in good dielectrics.

#### 1-6. Insulation Resistance:

The insulation resistance of a dielectric material may be measured in terms of surface or volume resistivity in ohms or ohms-centimeter respectively. Volume and surface resistances can be measured as shown in Fig (6).

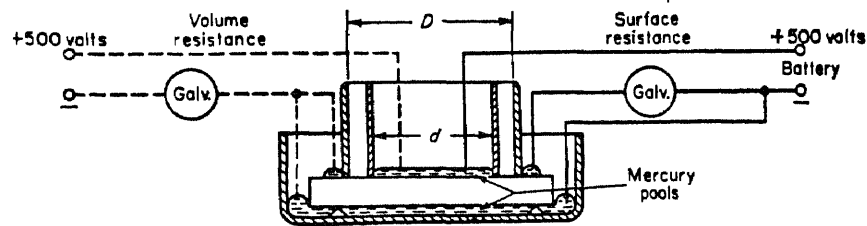


Fig. (6) Typical arrangement of apparatus for surface and volume res. test

The insulation resistance of any capacitor will be lowered in the presence of high humidity (unless it is sealed) and will be reduced when operated in high ambient temperature (whether sealed or not). For perfectly sealed capacitors used under conditions of high humidity there should be no deterioration, but for imperfectly sealed capacitors

the drop in insulation resistance will be roughly inversely proportional to the effectiveness of the sealing.

Under high-temperature conditions, the fall in insulation resistance for most capacitors is given approximately by the formula

$$R_T = \frac{R_t}{e^{-k(T-t)}}$$

Where  $R_t$ : insulation resistance at high temperature T.

$R_t$ : insulation resistance at low temperature T.

K: constant depends on the material used.

#### 1-7. Dielectric Strength:

The ultimate dielectric strength of a material is determined by the voltage at which it breaks down. The stress at which breakdown occurs depends on the thickness of the material, the temperature, the frequency and the waveform of the testing voltage, the method of application, etc. Therefore, comparisons between different materials should ideally be made on specimens equal in thickness and under identical conditions of measurement.

The dielectric strength of a material is always reduced when it is operated at high temperature or if moisture is present. Most dielectrics withstand a much higher voltage for brief periods. With increasing frequency the dielectric strength is reduced particularly at radio frequencies depending on the power factors etc., of the material.

1-8. The Effect of Frequency on Dielectric Materials and Capacitor Assembly:

At very low frequency and also at high frequencies, there is an increase of loss which sets a limit to the practical use of a capacitor with any given dielectric. At very low frequencies, various forms of leakage in the dielectric material have time to become apparent, such as dc leakage currents and long time constant effects, which have no effect at high frequencies. At very high frequencies, some of the processes contributing to dielectric polarization do not have time to become effective and therefore cause loss. The losses might be simply and approximately represented in Fig (7).

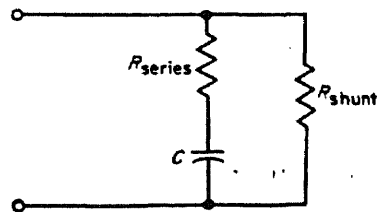


Fig. (7) Series and shunt losses in a capacitor

At very low frequencies the circuit is entirely resistive, all the current passing through the shunt resistance (dc leakage resistance, etc.). At very high frequencies the current passes through the capacitor  $C$ , but all the volts are dropped across the series resistance, and again the circuit is lossy. This series capacitance may be due to the resistance of the capacitance leads, contact resistance, etc, in the capacitor assembly itself. These limit the upper frequency independently of the dielectric material used. Similarly, leakage across the case

containing the dielectric may limit the lower frequency so that not all the useful range of the dielectric itself may be realized.

1-9. The Impedence of a Capacitor:

The current I in a capacitor (when an alternating voltage V of frequency f is applied is

$$I=2\pi fCV$$

and the reactance is given by

$$X_c = \frac{1}{2\pi fC}$$

An ideal capacitor would have entirely negative reactance, but the losses, described previously, due to dielectric, case and leads, preclude this. In addition, inductance is also present in varying amounts, and therefore as the frequency is increased the inductive or positive reactance increases, and above a critical frequency the capacitors behaves as an inductor.



## CHAPTER II: TANTALUM CAPACITOR

### 2-1. Origin and Development:

The idea (2-62) of forming an insulating film on a metal electrode to make a capacitor is generally attributed to Wheatstone in 1855, but the idea of the electrolytic capacitor arose in Germany some 20 years later.

The precursor of the modern electrolytic capacitor, consisting of two aluminium-sheets electrodes immersed in an electrolyte consisting of a solution of ammonium borate in a glycerine-water mixture appears to have originated about 1908, and in that year an important step in the reduction in size of such capacitors was made by constructing the anodes of thin sheets, rolling them up into cylindrical form and placing them in containers of the same shape. However, the more bulky form persisted for many years after this.

Solid tantalum capacitors possess unique features and characteristics which make them invaluable in certain applications. Chemical and physical stability of the dielectric causes tantalum capacitors to exhibit high reliability and long life when compared with many other dielectrics such as aluminium types. Electrical parameters such as low dc leakage currents, stability of performance over a wide temperature range and close capacitance tolerance are some of the benefits available. Frequency characteristics cover an extremely wide range.

## 2-2. Electrolytic Capacitor:

Essential parts of an electrolytic capacitor are:

- a) A metal anode (the positive electrode), made from a film-forming metal. Aluminium, tantalum, zirconium, niobium, hafnium, tungsten, bismuth, and antimony are able to form anodic oxide films by an electrolytic "forming" process. Only aluminium and tantalum are at present used to make electrolytic capacitors on a commercial scale. Extensive research is being carried out on zirconium and niobium, and experimental capacitors have been made successfully, but so far there is no commercial production. The principal problem appears to be obtain metal of a sufficiently high degree of purity. None of the other metals mentioned has yet yielded a practicable electrolytic capacitor.
  
- b) Very thin oxide film, produced electrolytically on the anode, the film constituting the dielectric. The thinness of the film, coupled with its medium permittivity, is the main reason for the high capacitance per unit volume, the basic advantage of electrolytic capacitors.  
The films consist of substantially pure oxide of the anode metal (e.g.  $\text{Al}_2\text{O}_3$  or  $\text{Ta}_2\text{O}_5$ ) with a possible content of adsorbed water or entrapped electrolyte ions in the more porous ones. The films are mainly amorphous and glassy in structure, showing interference colours characteristic of the thickness and refractive index of the film. These compact anode films can be made practically free from defects while still being extremely thin (about 3 Å/volt of formation) and can withstand very high electric stresses (e.g. 500 volts/micron) that in some instance approach the intrinsic dielectric strength of the oxide -  $10^4$  volts/micron.  
The permittivity is about 8.4-10 for aluminium, 25-27 for tantalum and 41 for niobium.  
To develop the oxide film, the thoroughly-cleaned metal slug is placed in a suitable electrolyte and connected to the positive terminal of a source of direct current. Electrolysis of the water in the electrolyte takes place, and the oxygen combines rapidly with the anode metal to form the surface layer of oxide, with nearly 100 per cent efficiency in the case of pure metal anodes. As the oxide film is a dielectric and practically non-conductive material, the current falls rapidly, and as the film thickens, the potential across the film rises until it is approximately the same as that of the supply.
  
- c) An electrolyte suited to the anode metal and characteristics is required. The electrolyte is the true negative electrode. If during its life the oxide film is punctured or cracked,

the voltage across the capacitor causes a local current to flow that in turn leads to a local growth of oxide film, thus giving the capacitor a self-healing property. Both this and the very small steady leakage current cause electrolysis of water in the electrolyte, so that oxygen and hydrogen gases are evolved in proportion to the total electric charge that has passed. Some of the oxygen combines with the anode in the self-healing process, but the remainder, plus the hydrogen, accumulates as a gas and if a suitable vent has been provided, leaks away. If the capacitor is completely sealed, and internal gas pressure may build up, causing the seals to bulge and leading to an explosion in extreme instances if the current is considerable. With modern types of capacitor, and particularly if the voltage is low, the leakage current is so small that the gas pressure is very slight.

### 2-3. Tantalum:

Ta (3-82) atomic weight is 180,9479, atomic number is 73, melting point is 2990°, b.p. 5425 ± 100°C, specific gravity is 16.654, valance 2?, 3, 4?, or 5. Discovered in 1802 by Ekeberg, but many chemists thought niobium and tantalum were two different acids. The early investigators only isolated the impure metal. The first relatively pure ductile tantalum was produced by von Bolton in 1903. Tantalum occurs principally in the mineral columbite-tantalite (Fe, Mn) (Nb, Ta)<sub>2</sub>O<sub>6</sub>. Tantalum ores are found in the Republic of Zaire, Brazil, Mozambique, Thailand, Portugal, Nigeria, and Canada. Separation of tantalum from niobium requires several complicated steps. Several methods are used to commercially produce the element, including electrolysis of molten potassium fluotantalate, with sodium, or reacting tantalum carbide with tantalum oxide. Sixteen isotopes of tantalum are known to exist. Natural tantalum contains two isotopes; one of these, Ta<sup>180</sup>, is present in very small quantity (0.0123%) and

is unstable with a very long half-life of more than 10-13 years. Tantalum is a gray, heavy, and very hard metal. When pure, it is ductile and can be drawn into fine wire, which is used as a filament for evaporating metals such as aluminum. Tantalum is almost completely immune to chemical attack at temperatures below 150° C. and is attacked only by hydrofluoric acid, acidic solutions containing the fluoride ion, and free sulfur trioxide. Alkalis attack it only slowly. At high temperatures, tantalum becomes much more reactive. The element has a melting point exceeded only by tungsten and rhenium. Tantalum is used to make a variety of alloys with desirable properties such as high melting point, high strength, good ductility, etc. It is also widely used to fabricate chemical process equipment, nuclear reactors, and aircraft and missile parts. Tantalum is completely immune to body liquids and is a nonirritating metal. It has, therefore, found wide use in making surgical appliances.

Over the other metals (1-71) that can have coherent oxide layer, tantalum has numerous advantages. With tantalum, we are able to prepare a better dielectric by anodization than with aluminum. The result of this is that the capacitor has a better shelf-life than does the equivalent aluminum one.

Tantalum and tantalum oxide (2-62) are much more inert materials chemically than aluminum and its oxide film, and for this reason would be expected to make more stable and reliable capacitor

units. This inertness also makes possible a much wider choice in the range of electrolytes used. One of the common electrolytes being moderately-strong is sulphuric acid (which makes it possible to use such capacitors over a wide temperature range). Others are lithium-chloride solution and variants of the borate solutions used with aluminum anodes. Under equal conditions, the leakage current of a tantalum electrolytic capacitor is generally lower than that of the average-quality aluminum electrolytic capacitor.

For a given voltage (1-71), tantalum gives a thicker oxide than does aluminum. One can quantify this by noting that tantalum gives a thickness of 1.6 cm for every volt applied.

The only disadvantage of tantalum is that it has the cost of silver and the weight of lead and therefore it is not the adequate choice for applications are not worth.

Resistivity of tantalum is  $15.5 \mu\Omega/\text{cm}$ , and the temperature coefficient is 0.0031. Magnetic susceptibility of Ta is  $154 \cdot 10^{-6}$  at  $293^\circ\text{K}$  and  $124 \cdot 10^{-6}$  cgs at  $2143^\circ\text{K}$ . Magnetic susceptibility of  $\text{Ta}_2\text{O}_5$  is  $-32 \cdot 10^{-6}$  at ordinary temperature.

#### 2-4. Types and Construction of Tantalum Capacitor:

There are (1-79, 1-71) three types of tantalum capacitor-tantalum foil, solid tantalum, and wet sintered tantalum-which all use tantalum oxide as the dielectric. This dielectric has a permittivity of about 25 compared with 7 for typical aluminium electrolytic types, so high capacitance can be provided in a relatively small space.

Tantalum foil capacitors are normally used for voltages above 100 volts and they are available up to 450 volts. The foil is either plain or etched. In the latter case, the surface area is considerably increased and therefore more capacitance per unit volume is obtained. However, the situations in which tantalum foil capacitors offer clear advantages are so rare that they need not be considered in any greater detail.

Throughout this thesis, solid tantalum capacitor will always refer to the tantalum capacitor with sintered anode and solid electrolyte, which is usually manganese dioxide.

#### 2-5. Processing and Packaging of Tantalum Capacitor:

To make solid Ta capacitors, accurately weighed quantities of powdered Ta mixed with wax are compacted into small moulds, into each of which a Ta wire has been inserted. The purpose of the wire of course, is to provide one of the external connections. The wax has three purposes; in the initial stages it temporarily binds the particles of Ta powder together, it serves as a lubricant so that particles flow freely during compaction to form anodes of uniform density throughout their volume, and it also fills the interstices, thereby helping to prevent over-compaction. Compacting by the use of presses not only ensures good electrical contact between the metallic particles, but also makes each component strong and firm enough to be lifted from the mould for further treatment.

The next step is the removal of the wax by vaporisation in a vacuum oven. The material is then sintered; the electrical contacts

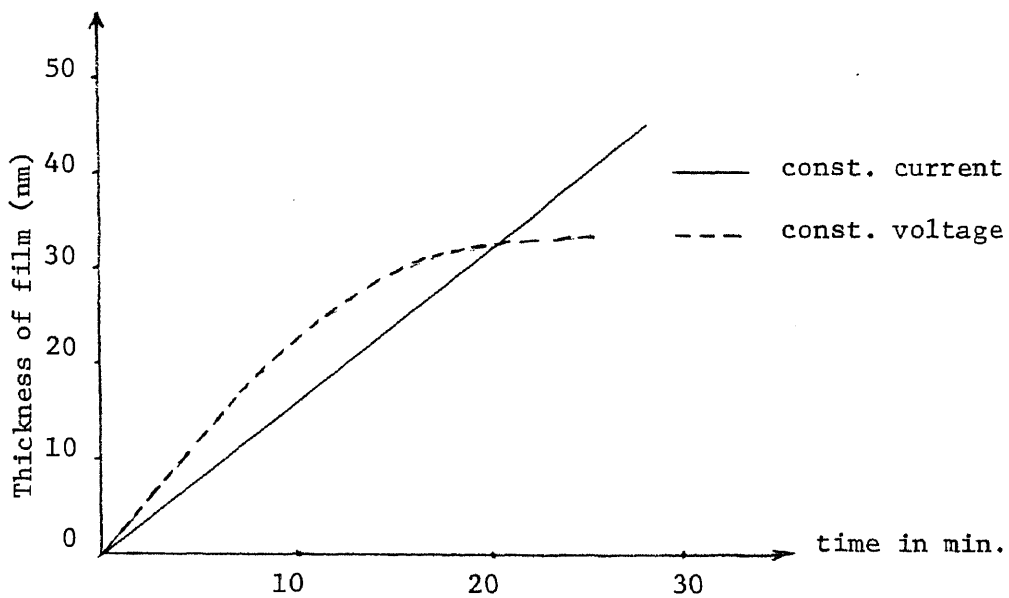
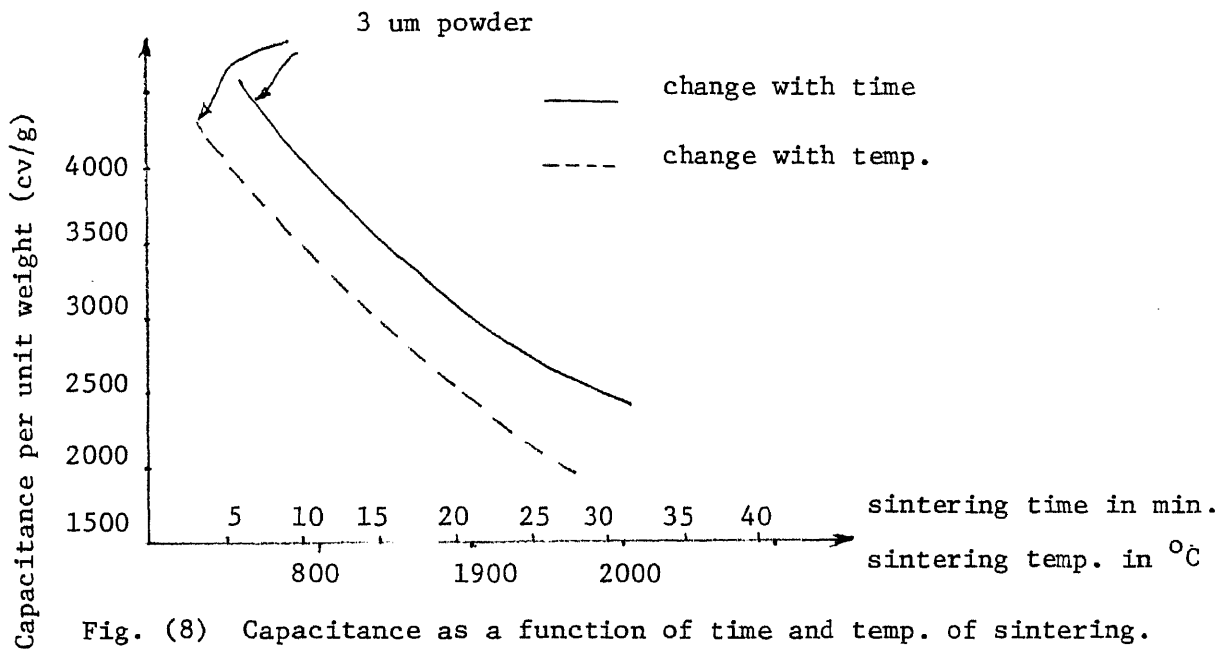
between neighbouring particles and between the Ta and surrounding particles are welded, under high vacuum, in a furnace. The temperature needed for sintering is high enough (1800°C - 2000°C) to vaporise any impurities, but the high vacuum prevents oxidation of the Ta (1-79). The problems (1-71) associated with the preparation of Ta capacitors using sintered powder are partly concerned with the variation of capacitance one gets with sintering conditions. The variation of capacitance as a function of time and temperature of sintering is shown in Fig (8).

The combination of exact proportions of powder and wax, accurate weighing of the mixture, and precision of the compaction and moulding produces (1-79) an open sponge-like mass of size, density, and weight known to within close limits, so that the effective surface area of Ta is also accurately known.

The particles need to be covered with a strong uniform film of dielectric, in order to make possible the small size which is characteristic of these capacitors. To form the dielectric the anodes are dipped into baths of a conductive solution such as aqueous phosphoric acid\*and an electric current is passed through them. This causes oxidation which leaves a film of Ta pentoxide on the surfaces of the particles. Rate of growth of the oxide film for both constant voltage and constant current are shown in Fig (9).

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\*Dr. Misra thinks that there might be some activator in the acid, but he could not determine exactly that trade secret.





Thorough washing removes all traces of the conductive solution and leaves the anodes, now completely covered with dielectric, ready for the next process; the formation of cathodes.

Manganese dioxide, a solid semiconductor, is used as the cathode of these capacitors. It is produced and fixed in position when the anodes are repeatedly (6-20 times) dipped into a solution of manganous nitrate. After each dip in the solution impregnation the anode is converted into solid manganese dioxide by pyrolysis that is decomposition by heat. The effect of the number of impregnation and hence the thickness of the manganese oxide layer is increased but the dc leakage increases mainly due to the effect of the temperature cycling to which the dielectric has been subjected.

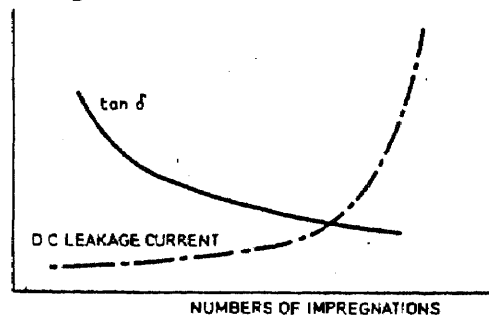


Fig. (10) Effect of impregnations on capacitor loss and leakage current

In this way the interstices between the particles coated with Ta pentoxide are filled completely by cathode material. The original sponge-like anodes are thus converted into solid capacitors with, at this stage, no increase in the original moulded size. However, it is not possible to attach leads directly to the manganese dioxide cathodes. This problem is overcome by dipping the capacitors into a solution of colloidal graphite. As a result, graphite, a low-

impedance conductive material, coats the surfaces and penetrates into the pores to make good electrical contact with all the cathode material. An exterior coat of silver is then added.

Solid Ta capacitors can be classified in five groups, based on size, reliability (resistance to ambient conditions) and price according to their mounting methods and lead-wire take out methods, as shown in Fig (11).

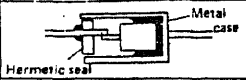




Kinds	Typical structure	Features	Usages
Metal case with hermetic end seal		Superior anti-environmental characteristics High reliability	Military use Computer
Epoxy molding		High reliability	Computer Automobile
Metal case with epoxy resin end seal		Miniature	Mobile telephone Hearing aid
Dipped epoxy coating		Low price	Consumer use Industrial use
Chip		Ultraminiature	Hybrid IC

Fig. (11) Kinds of tantalum capacitors.

- 1) The metal case type with hermetic end seal is hermetically sealed and is used for high-reliability equipment and equipment used in extreme operating conditions.
- 2) The epoxy-molded type is externally transfer-molded with epoxy resin. This type is somewhat inferior to the metal case, hermetically sealed type in resistance to ambient conditions, but is relatively superior in external dimensional precision. As it is small in size and low in cost, it is used extensively in industrial equipment.
- 3) The metal case type with epoxy resin and seal is not hermetically sealed and has relatively smaller external dimensions and is used extensively in small products such as mobile radio equipment and hearing aids.

- 4) The resin-dip type, dipped in epoxy resin, is inferior in external dimensional precision, and is less resistant to external ambient conditions than the resin-molded type but as it is low in cost, this type is used extensively in consumer products and industrial equipment that is sold at low prices. Of all the Ta capacitor types, the resin-dip type is manufactured in greatest quantities.
- 5) Unlike the four other types, the chip type does not have lead-wires and is intended for insertion in printed circuit boards, and is used mainly for hybrid ICs (2-82).

A symbolic representation of solid Ta capacitor is shown in Fig. (12).

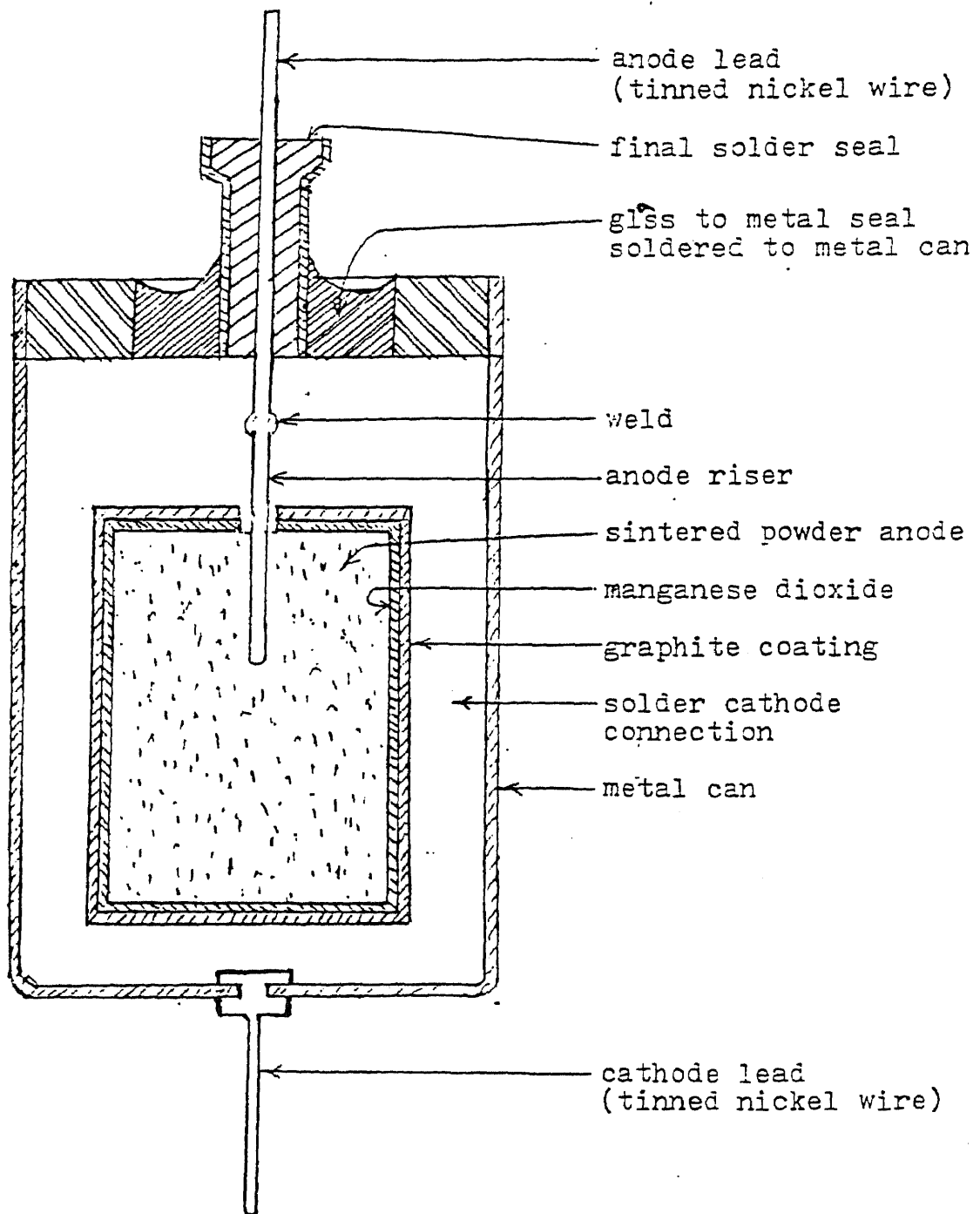


fig (12) Solid tantalum capacitor (2)

## 2-6. Tantalum Capacitors Advantages:

Solid Ta capacitors possess (2-64) the following unique features:

- 1) Chemical and physical stability, which causes Ta capacitors to exhibit high reliability and long life.
- 2) Electrical parameters such as low dc leakage currents, stability of performance over a wide temperature range and close capacitance tolerance.
- 3) Frequency characteristics cover an extremely wide range.
- 4) High capacitance per unit volume.

## 2-7. Losses in Solid Tantalum Capacitors:

All capacitors (5-60) have some energy losses associated with them. The losses in an electrolytic capacitor are due primarily to its internal resistance. Fig ( 13 ) is a simplified equivalent circuit of an electrolytic capacitor. Notice that there are two resistances indicated. The shunt resistance represents the dc leakage current. The series resistance is the primary contributor to the energy loss or heating effect within the unit. In the case of solid tantalum capacitor, this resistance is primarily in the electrolyte-cathode system.

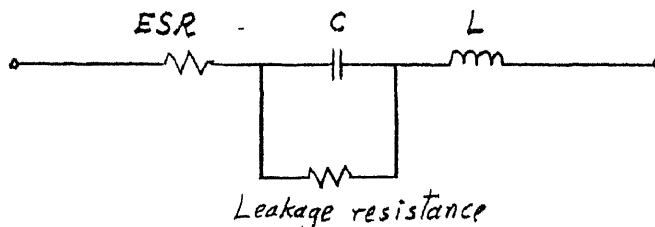


Fig. (13) Electrolytic capacitor eq. circuit.

The high frequency impedance (6-68) and the maximum allowable a.c. ripple of a solid tantalum capacitor is determined almost entirely by the equivalent series resistance of the capacitor. The stability of capacitance with variations of frequency and temperature is also under certain conditions determined by the ESR.

The ESR of a capacitor is normally obtained from a measurement of dissipation factor and is defined as  $ESR = \frac{\text{Dissipation Factor}}{\omega C}$ .

Unfortunately the dissipation factor of a solid tantalum capacitor is not a linear function of frequency and its ESR is not a constant. The total dissipation factor (6-61, 3-68, 6-68) may therefore be conveniently expressed as:

$$\begin{aligned} D.F. &= \omega C (R_1 + R_2) \\ &= \omega C R_1 + \omega C R_2 \\ &= \tan \delta + \omega C R_2 \end{aligned}$$

where C is the capacitance,  $\omega$  is the angular frequency.  $R_2$  is the resistance of the electrolytic ( $MnO_2$ ), carbon layer and leads  $\tan \delta$  is the dielectric contribution to dissipation factor. Fig. (14) shows the details of the ESR.

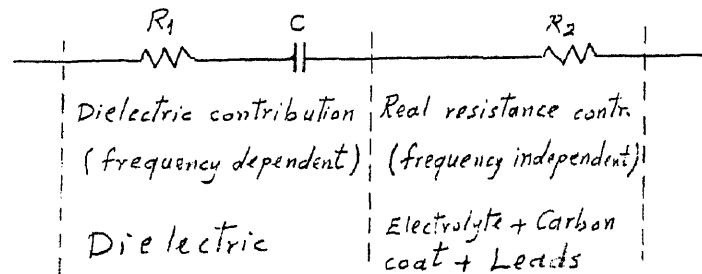


Fig. (14) ESR of solid Ta capacitor.

Considering the dielectric loss produced in a hypothetical R, it is apparant from fig ( 15 ) that at the lowest frequencies  $\tan \delta$  is relatively independent of frequency. This is the region where dissipation in the dielectric predominates. Variation in  $\tan \delta$  in this region is extremely small compared to that which would be produced by a true series resistance such as  $R_2$  or by a parallell leakage path. Furthermore, the values of  $\tan \delta$  at the lowest frequencies are relatively independent of geometry and capacitance and therefore must be a property of the oxide film.

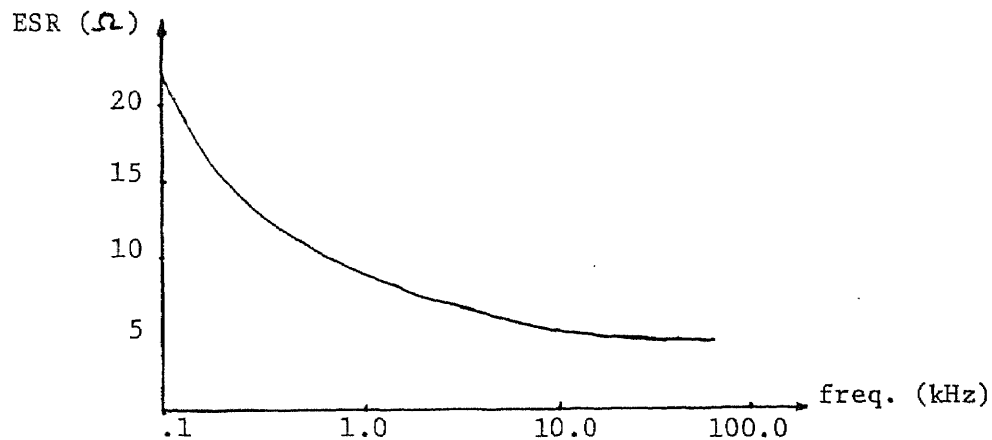


Fig. (15) ESR change with frequency.

The low frequency values of  $\tan \delta$  are, however, somewhat dependent on tantalum purity and structure and on processing steps, as might be expected since these are likely to influence the quality of the dielectric. To have  $\tan \delta (= \omega CR_1)$  independent of frequency, the hypothetical resistance  $R_1$ , must vary inversely with frequency and can be considered as  $k/\omega$  where  $k$  is a constant.

From these considerations it can be seen that the dissipation factor at very low frequency will be a measure of dielectric losses

( $\tan \delta$ ) whereas at high frequency it will be a measure of resistive losses ( $\omega R_2 C$ ). Therefore, a frequency of 1 KHz is usually chosen as a basic frequency to measure the ESR of capacitors, and that gives:

$$ESR = \frac{\tan \delta}{\omega C} = \frac{\tan \delta}{2000\pi C} .$$

Considering now only the resistive part of the dissipation factor, (since it is the main factor determining high frequency characteristics), it is convenient to think of this resistance as composed of two parts:

- a) The resistance of the electrolyte which is within the sintered anode of tantalum and is a distributed resistance in series with the capacitive elements.
- b) The resistance of contact to the electrolyte, leads, etc. which is outside of the anode and consequently is a lumped resistance in series with the capacitive element.

Of the two, the electrolyte resistance is normally the greater.

Well recognized advantages of solid electrolytic tantalum capacitors over other electrolytic capacitors (6-68) are that the ESR of this type is relatively low and that it remains low over a wide temperature range. These advantages are utilized in applications where it is required to pass relatively high AC currents through the capacitor or where a low capacitor impedance is required at high frequencies and/or lower temperatures.



### CHAPER III: RELIABILITY OF SOLID TANTALUM CAPACITORS

#### 3-1. How Reliability of Electronic Parts is Guaranteed:

Any study of electronic parts reliability (1-72) should be approached by looking at individual parts, as dependability factors vary from one part to the next. In fact the word "reliability" itself has different shades of meaning and has no true, clear-cut meaning. In examining reliability, one should ask whether he is using the word to define electronic parts themselves of the systems or devices they form a part of.

Usually mechanical and electromechanical parts of electronic systems, such as connectors and printed circuit boards raise dependability problems. Particularily troublesome are the factors of the reliability of contacts and connecting elements (soldering, wound terminals, welding), although these are not classified as electronic parts in the strict sense of the word.

Resistors, capacitors, and other passive parts on the other hand pose very few reliability problems in design and manufacture, due to the technical expertise developed over the long period since they were first produced. Among these passive parts stands solid tantalum capacitor which is a dielectric type device. Although they offer outstanding reliability compared to other types of capacitors, serious research has to be done for further improvements.

It has been found (7-64) that reliability of dielectric type devices is a function of:

- E Electric Field
- S Surface
- P Package
- T Temperature & Temperature Gradient
- A Ambient
- SF Special factors, encountered by the device under consideration such as thermal cycling, mechanical vibrations, etc.

Therefore in studying solid tantalum capacitor's reliability, these factors should all be investigated.

### 3-2. Life Tests and Failure Rates:

Life tests have been used extensively to predict capacitors reliability. Most manufacturers (5-61) have some form of life test that is used in tandem with a statistical analysis to predict or guarantee the customer a certain failure rate over a fixed period of time. The most accurate single life test point, of course, would be to test a group of units at rated temperature and voltage, for the guaranteed life of the capacitor. This method is not practical because of the time involved. However many leading capacitor producers are approaching this accuracy by running a slightly accelerated life test (in the order of 1.5 times rated voltage) for a shorter period of time.

For a given operating temperature, the life of a capacitor is known to be inversely proportional to some power "N" of the applied voltage. Accelerated life tests, to avoid great outlays of time, are based on this relationship.

One of the most important factors that should be considered when determining capacitor reliability or the degree of accuracy that can be obtained when predicting the life of a particular capacitor type, is its failure rate and how it varies with time (called sometimes failure rate pattern). The complete failure rate pattern is normally composed of two types of failures; chance or random failures and wear-out failures.

Random failures are early failures caused by inherent weaknesses in the materials and by damage incurred during the manufacturing processes. The random failure rate pattern is characterized by a high initial failure rate followed by a leveling off period.

Wear-out failures in capacitors are thought to be caused principally by the slow electrochemical deterioration of the dielectric. The presence of contaminants such as rosin or moisture within the capacitor have shown to accelerate this deterioration. The wear-out failure rate pattern is usually bell-shaped and is often normal.

The actual failure frequency distributions are caused by both wear-out and random failures and therefore have a characteristic shape of their own.

### 3-3. Failure Modes of Capacitors:

Generally, a capacitor is said to be failed (3-62) if it exhibits one or more of the following features:

- 1) Short circuit
- 2) High leakage current
- 3) Open circuit
- 4) Appreciable change in capacitance
- 5) Appreciable change in equivalent series resistance (ESR)

The first two modes are related to each other. However, the difference is that when the capacitor is short circuited it can not function as a capacitor anymore. The third mode is reported (1-70) to account for the largest number of failures of solid tantalum capacitors. The two reasons for that were a poor weld from the tantalum risen to the external lead and poor slug anchorage to the can.

The above mentioned failure modes can occur suddenly or gradually. When a failure occurs suddenly, it is called catastrophic failure. Whereas when it occurs gradually, it is called degradational failure. Furthermore, three parts are responsible for any failure that occurs to the product, namely, the manufacturer, the designer, and the user.

### 3-4. Common Causes of Failure of Solid Tantalum Capacitors:

The most common causes of failure of capacitors in general and solid tantalum capacitors in particular can be summarized as follows:

- 1) Seal failure, which allows moisture and other contaminants to infiltrate and hurt the capacitor.

- 2) Dielectric failure, which could be either gradual growth in thickness of the oxide film leading to reduced capacitance, or field crystallization of the essentially amorphous dielectric oxide. Field crystallization occurs in tantalum capacitors at high (e.g. over 100°C) temperature and high electric stresses. Starting from minute nuclei, the amorphous oxide film is replaced by crystalline tantalum pentoxide, see fig. ( 16 ). This process is accompanied by increased leakage current. It has been found (2-60, 2-62, 5-64, 1-77, 5-80) that dielectric crystallization is the basic failure mechanism of solid tantalum capacitor.

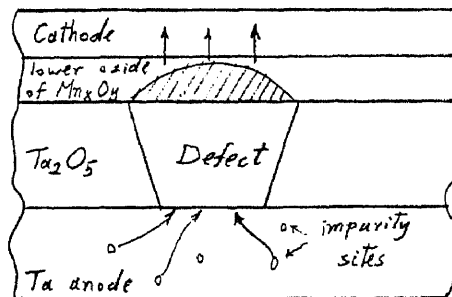


Fig. (16) Migration of impurity molecules.

- 3) Corrosion of electrodes, As a matter of fact this was the case in all failure cases of a supplier whose units were covered in this study.
- 4) Bulging or bursting due to internal evolution of gas.
- 5) Formation of oxide film at one of the metal-to-metal joints leading to a rising series resistance increasing the value of  $\tan \delta$
- 6) Mechanical failure, in contacts, welds.
- 7) Thermal runaway, since oxides of manganese are semiconductors with a negative (5-80) temperature coefficient of resistivity, therefore excessive temperature in a dielectric zone can cause loss of control of leakage current.
- 8) Air gaps creation in the interfaces, specially between tantalum and tantalum oxide because of the radical difference of their coefficients of thermal expansion\*.

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\* Dr. Misra thinks that this is the most likely cause of failure but still needs some more research.

Since an airgap in series with tantalum oxide will have 25 times as much electric field as that in the tantalum dioxide ( $\epsilon_r$  for  $Ta_2O_5 = 25$ ). Meanwhile the breakdown voltage for air is much less than that for  $Ta_2O_5$  which means a breakdown inside the air gap is inevitable which in turn leads to a whole destruction of the capacitor.

CHAPTER IV: FACTORS AFFECTING RELIABILITY OF SOLID  
TANTALUM CAPACITORS

4-1. Effect of Temperature:

Temperature (5-80) is a critical factor since the migration rate of impurity molecules, from the tantalum dioxide to the cathode, is directly affected (see fig.17 ):

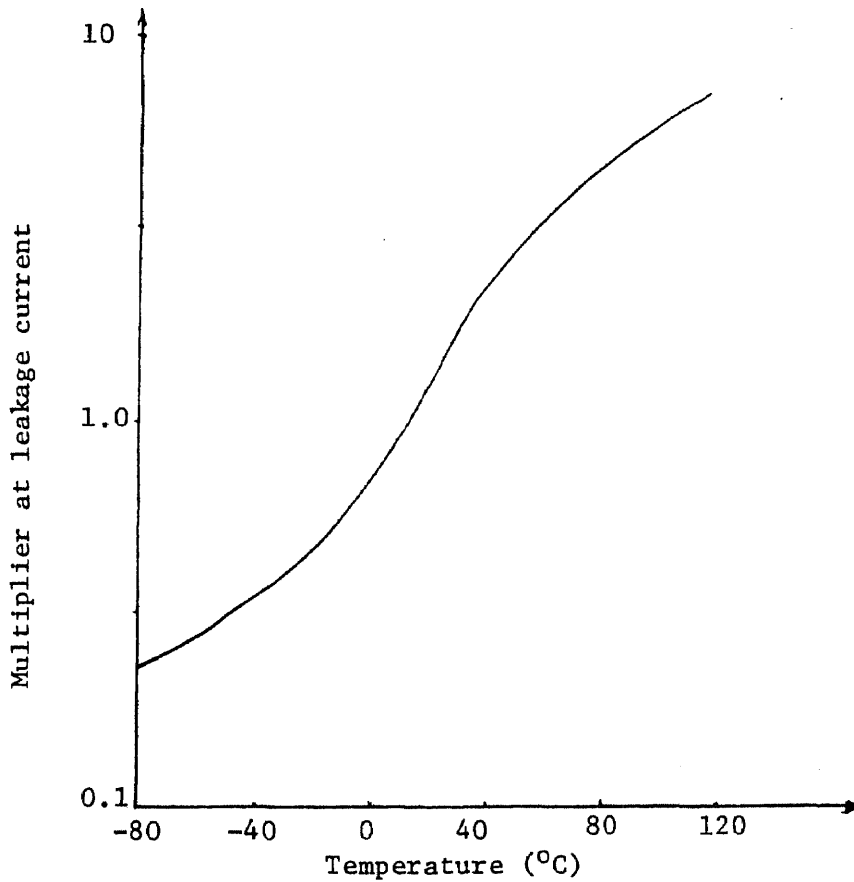


Fig. (17) Typical effects of temperature upon leakage current

It is believed that a certain proportion of capacitors have impurities that create defects which are too large to heal, but as a rule these become obvious during manufacture and are screened out at this point. In others they migrate to the surface some time later.

The oxides of manganese are semiconductors with a negative temperature coefficient of resistivity, therefore excessive temperature in the defect zone can cause loss of control of leakage current. This leads to thermal runaway and catastrophic failure.

#### 4-2. Effect of Humidity:

Infrequently (5-64) failures due to increases in capacitance and/or dissipation factor are found. A defective hermetic seal which allows moisture to enter the unit is the most common cause for such failures. Since more of the available tantalum oxide surface is contacted by the moisture than by the  $MnO_2$ , the capacitance shows an increase. The increase in dissipation factor results from an increase in the resistivity of the  $MnO_2$  due to the presence of moisture.

When environmental factors were examined (1-77), only water vapour was found to have any effect on the leakage current/time plots of the capacitors on life test. The effect of water vapour was examined more closely by life testing capacitors in capsules attached to a vacuum line so that known pressures of water vapour could be introduced to, or removed from, the capacitor at will during the test. It appeared from the observations that two independent processes were occurring. Firstly there was the basic



degradation mechanism which caused the leakage current to rise steadily and irreversibly with time, and which was unaffected by water vapour.

Secondly, superimposed on this steady increase in current short term reversible decreases could be made to occur by introducing water vapour. This effect was in contrast to the effect of liquid water where leakage current rises sharply because water makes electrical contact with damaged areas of oxide previously uncontacted by manganese dioxide. It appears that water vapour at 125°C probably enters the manganese dioxide structure and increases its resistivity, thus lowering leakage current.

In another study (4-73), solid tantalum capacitors were measured for capacitance and dissipation factor under various conditions of relative humidity. The capacitance increased monotonically with relative humidity while the dissipation factor had a maximum in mid relative humidity.

The cause of change of dissipation factor is the change of dielectric properties of the interface between tantalum pentoxide dielectric film and manganese dioxide counter electrode by the effect of the amount of adsorbed water.

The forming voltage of a capacitor plays an important role as to what extent the moisture affects that capacitor. In high forming-voltage capacitors, the capacitance and the dissipation factor changed mainly with the change of dielectric dispersion frequency caused by the change of conductivity of dielectric surface with water molecules absorbed. In the case of low

forming-voltage capacitors, the capacitance changed mainly with the change of series capacitance in the interface, while the dissipation factor scarcely changed. These changes were observed to be reversible, because they are apparently due to physical adsorption.

#### 4-3. Effect of Circuit Impedance:

Commercial experience suggests that solid tantalum capacitors are more stable in service if they have an impedance in series with them than if they have voltages applied from a low voltage source. To check this and investigate the mechanism of circuit impedance effect, life tests were performed (1-77) with an added circuit impedance. The addition of a circuit impedance resulted in a much lower and more stable leakage current which increased very slowly over a long period of time. The morphology of the oxide surface before and after life test showed different characteristics. With a high circuit impedance, bumps in the film, although increasing in number during life test as in the low circuit impedance case, do not generally crack. A further important feature was the initial absence of large electrical discharge sites, only one of these being found. It appears that an added circuit impedance not only prevents the development of large electrical discharge sites, but also the growth of uncracked bumps into somewhat larger cracked bumps.

#### 4-4. Effect of Impurities of Tantalum Anodes:

It is generally believed that the dc leakage of a sintered-anode tantalum electrolytic capacitor is affected highly by the

chemical purity of the tantalum metal from which it is made. To substantiate this belief it is necessary to correlate the dc leakage of the capacitor with the impurity content of the sintered anode rather than with that of the original tantalum powder since impurity levels change during sintering to an extent dependent on sintering conditions. The impurities found (2-60) in tantalum anodes included O, C, N, Na, Al, Cr, Cu, Fe, Mo, Nb, Ni, Si, and Ti. On the other hand, leakage current was found to be a direct function of quantity and quality of contaminants. High leakage due to copper contamination has occasionally been observed in other powders. Field crystalization as discussed in chapter III stem always from where these impurities are.

Sintering was found to change appreciably impurities such as oxygen, carbon and sodium, while other contents change to a lesser extent.

## CHAPTER V: EXPERIMENTAL WORK

### 5-1. Procedure and Description:

Solid tantalum capacitors were randomly obtained from five different manufactures. The suppliers are called in this study, 1, 2, 3, 4, and 5 according to their standing after the life test, e.g. Supplier #1 is the company whose capacitors showed the least amount of failures, due to the test, and Supplier #5 is the company whose capacitors showed the highest amount of failures. Out of the samples gathered, which ranged between 150 - 250 units each, fifty units of each sample were introduced to the life test.

The life test was performed in a sealed humidity chamber and lasted for one thousand hours. Throughout the test, the relative humidity and temperature were kept at  $(96 \pm 3) \%$  and  $95^{\circ}\text{C}$  respectively. Three parameters were measured for each unit before applying the life test, capacitance, equivalent series resistance, and leakage current. The same parameters were measured again immediately after the 1000-hour life test was over. Then the units were kept in room temperature and humidity for 48 hours when the three parameters were measured for the third time. The values @ 0 hours and @  $1000 + 48$  hours are the values on which the calculations in this study are based. The values @ 1000 hours are disregarded to avoid any misleading transient or dynamic effects, influences. However, the values @ 1000 hours give some idea on the selfhealing behaviour of solid tantalum capacitor.

The leakage current measurements were done in a shielded room to avoid any stray currents which might have affected the tiny, actual values.

The simple circuit shown in Fig ( 18 ), was used to avoid waiting the charging time, after which the leakage current reaches a steady state, for every individual capacitor.

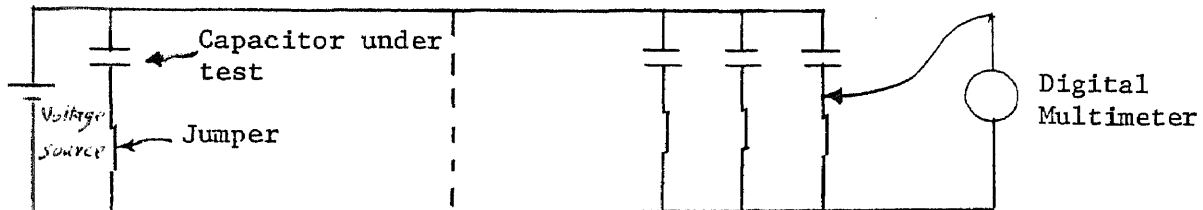


Fig. (18) Circuit used for leakage current measurements.

The charging time ranged between five (supplier # 5) and twenty minutes (supplier #1). To avoid intruppting the current, the ammeter is connected first across a jumper in series with the capacitor, then the jumper is opened and the reading is recorded.

Capacitance and equivalent series resistance (ESR) were easier to measure. Digital meters were used in both cases and the values were taken instantaneously.

## 5.2 Experiment Results:

The following tables show the measurements for each capacitor supplied by the five Suppliers. The unit number is the actual number given to that particular unit before the test; it is not a serial number. At the bottom of each column, the mean and standard deviation for that column are given. As I mentioned in the previous sections, calculation for measurements @ 1000 hrs. are not given to avoid any misleading, transient effects.

To understand the tables, however, the following notations are interpreted as follows:

C1: Capacitance in  $\mu\text{F}$  measured @ 0 hours

C2: Capacitance in  $\mu\text{F}$  measured @ 1000 hours

C3: Capacitance in  $\mu\text{F}$  measured @ 1000 + 48 hours

ESR1: Equivalent series resistance in ohms measured @ 0 hours

ESR2: Equivalent series resistance in ohms measured @ 1000 hours

ESR3: Equivalent series resistance in ohms measured @ 1000 + 48 hours

$I_L1$ : Leakage current in nA measured @ 0 hours

$I_L2$ : Leakage current in nA measured @ 1000 hours

$I_L3$ : Leakage current in nA measured @ 1000 + 48 hours

$\bar{x}$ : Arithmetic mean

$\sigma_n$ : Standard deviation of the entire population

$\sigma_{n-1}$ : Standard deviation for a sample of the entire population (our case)

Table 1  
 SUPPLIER #1  
 22  $\mu$ F: 16V  
 Failure Rate\* = 10%

Unit No.	C1 ( $\mu$ F)	C2 ( $\mu$ F)	C3 ( $\mu$ F)	ESR1 (OHMS)	ESR2 (OHMS)	ESR3 (OHMS)	I <sub>L</sub> 1 (NA)	I <sub>L</sub> 2/10 <sup>4</sup> (NA)	I <sub>L</sub> 3/10 <sup>4</sup> (NA)
2	Fail								
3	22	22.6	22.6	.49	3.44	3.37	1.8	6.4	1.24
4	21.2	21.7	21.7	.54	4.72	4.99	0.36	6.10	.88
5	21.4	21.9	21.8	.55	3.39	3.49	6.40	2.7	1.37
6	21.7	22.4	22.3	.55	5.13	5.73	.27	2.3	.64
7	20.7	18.4	16.1	.64	3.42	3.77	15.	14.78	.43
8	21.9	22.4	22.5	.57	4.46	4.67	.65	3.2	1.
9	22.2	22.5	22.3	.54	4.38	4.87	.56	15.9	.3
10	20.9	12.9	13.2	.54	5.25	5.8	1.2	15.75	15.65
11	22.1	22.6	22.5	.51	2.37	2.46	2.	8.	1.7
13	21.4	22.0	21.9	.53	4.4	4.68	2.5	6.3	1.6
14	21.9	22.4	22.3	.57	6.11	6.33	.81	5.2	1.4
15	22.3	22.8	22.0	.59	4.54	4.79	1.2	9.5	4.38
17	21.8	22.4	22.2	.54	4.8	5.58	1.1	5.6	1.16
19	21.7	22.5	22.4	.54	5.32	5.61	.65	5.	1.18
20	22.2	22.7	22.7	.54	5.4	5.72	1.6	3.3	.69
21	21.7	22.4	22.3	.53	4.25	4.44	1.3	4.8	.71
22	21.7	21.8	22.5	.50	4.93	6.09	6.3	5.7	.5
23	21.8	22.4	22.4	.49	2.62	2.79	3.7	2.82	.63
24	21.2	21.6	21.6	.50	2.57	2.69	2.3	3.1	2.
25	21.1	22.6	21.7	.51	6.3	5.15	3.1	2.27	.9
26	22.3	22.8	22.7	.48	2.23	2.3	6.5	3.7	.77
27	21.7	21.8	22.3	.50	4.5	4.57	2.7	15.77	1.79
28	21.6	22.8	22.4	.5	4.35	4.53	1.8	8.	1.89
29	21.8	22.4	22.2	.51	4.52	5.04	1.40	0.94	.67
30	21.1	22.8	22.7	.51	7.78	7.99	2.9	5.6	.86
31	Fail								
32	21.5	22.2	22.2	.66	2.0	2.04	27.0	6.4	1.28
33	21.2	21.8	21.7	.61	3.28	3.69	25.3	8.4	1.13
34	Fail								
35	21.6	22.2	22.3	.63	3.36	4.04	36.	4.3	4.3
36	21.9	22.7	22.6	.51	2.22	2.32	3.4	7.5	1.72
37	21.7	22.3	22.2	.57	2.	2.19	9.19	6.8	1.42
39	21.8	22.5	22.4	.58	2.47	2.02	33.0	3.34	.195
40	21.9	22.5	22.4	.68	2.84	3.17	5.8	3.7	.34
41	Fail								

\*It should be noted that Supplier #1 failures are all loss of lead(s).

Table 1 (cnt'd)

Unit No.	C1 ( $\mu$ F)	C2 ( $\mu$ F)	C3 ( $\mu$ F)	ESR1 (OHMS)	ESR2 (OHMS)	ESR3 (OHMS)	I <sub>L</sub> 1 (NA)	I <sub>L</sub> 2/10 <sup>4</sup> (NA)	I <sub>L</sub> 3/10 <sup>4</sup> (NA)
42	21.9	22.5	22.4	.53	1.49	1.55	7.0	1.9	.43
43	22.1	22.5	22.6	.54	2.31	2.33	100.0	8.9	3.6
45	21.0	22.4	21.6	.54	2.97	5.74	10.0	2.1	.195
46	21.7	21.6	21.0	.63	5.45	3.37	35.0	6.0	3.0
47	21.9	22.4	22.4	.53	1.8	1.82	2.6	6.6	.465
48	21.1	21.7	21.7	.5	1.67	1.72	2.7	6.0	1.85
49	20.8	21.4	21.4	.49	1.81	1.83	6.2	4.7	.72
50	22.0	22.5	22.9	.51	1.22	2.12	33.0	15.6	.86
52	22.4	22.9	22.6	.5	2.12	2.3	2.4	4.5	.29
56	21.0	21.6	21.5	.63	2.56	3.4	3.4	3.5	.70
57	21.6	22.3	22.2	.5	2.79	3.1	20.	6.2	1.2
58	21.3	20.2	21.6	.57	2.95	2.8	9.3	11.37	9.6
59	Fail								
60	22.	22.4	22.5	.52	3.91	1.27	.7	7.8	1.37
$\bar{x}$	21.64		21.85	.54		3.68	9.54		1.76
$\sigma_{n-1}$	.43		1.65	.05		1.6	16.97		2.64



Table 2

SUPPLIER #2

20  $\mu$ F: 20V

Failure Rate = 18%

Unit No.	C1 (uF)	C2 (uF)	C3 (uF)	ESR1 (OHMS)	ESR2 (OHMS)	ESR3 (OHMS)	I <sub>L1</sub> (NA)	I <sub>L2</sub> /10 <sup>4</sup> (NA)	I <sub>L3</sub> /10 <sup>4</sup> (NA)
1	21.5	21.8	21.7	.39	.49	.49	60	.21	.04
2	23.	23.	23.1	0.42	.53	.53	20	.21	.20
3	21.9	22.2	22.1	.37	.40	.4	100	2.4	.8
4	21.9	22.3	22.3	.41	.824	.8	200	.4	.18
5	Fail								
6	22.8	23.	23.	.4	.44	.44	150	.95	.46
7	23.4	24.	24.	.43	.52	.53	130	.95	.99
8	22.3	22.6	22.5	.4	.49	.49	70	.68	.21
9	Fail								
10	22.7	22.9	22.8	.42	.45	.45	200	.38	.49
11	23.5	23.7	23.7	.43	.40	3.5	100	1.1	.17
12	22.4	22.8	22.8	.41	.45	.45	20	.229	.66
13*	22.4	22.7	22.7	.44	.66	.66	160	.04	.02
14	21.8	22.	22.	.39	.43	.43	30	.16	.06
15	21.5	21.8	21.8	.39	.4	.4	30	.71	.16
16	Fail								
17	22.3	22.6	22.6	.4	.50	.5	80	.073	.03
18	22.7	23.	23.0	.39	.43	.42	25	.68	1.4
19*	22.1	22.2	22.2	.38	.45	.45	250	.26	.03
20	22.2	22.4	22.4	.37	.37	.37	50	2.2	.95
21	22.8	23.1	23.	.4	.43	.43	60	.38	.2
22	21.7	22.	22	.39	.37	.36	50	2.5	1.27
23	Fail								
24	23.1	23.2	23.2	.62	.87	.88	45	1.5	1.42
25	22.	22.5	22.5	.4	.41	.41	10	.39	.14
26	22.7	23.2	23.2	.34	1.13	1.05	50	.98	.22
27	22.5	22.6	22.7	.38	.4	.41	35	.76	14.6
28	21.2	21.4	21.4	.45	.38	.38	40	.23	.08
29	23.2	23.5	23.5	.38	.39	.34	35	.53	.38
30	22.1	22.3	22.3	.39	.33	.34	55	.13	.04
31	Fail								
32	Fail								
33	21.7	21.7	21.7	.38	.41	.56	7	3.	.59
34	21.4	21.6	21.6	.38	.41	.41	10	1.14	.45
35	22.5	22.6	22.6	.4	.63	.62	80	1.2	.35
36	Fail								
37	21.8	22.2	22.2	.39	.4	.4	200	1.00	.57
38	22.	22.6	21.7	.48	.44	.44	40	.83	17.0

\*Capacitor with no change in characteristics.

Table 2 (cnt'd)

Unit No.	C1 (uF)	C2 (uF)	C3 (uF)	ESR1 (OHMS)	ESR2 (OHMS)	ESR3 (OHMS)	I <sub>L1</sub> (NA)	I <sub>L2</sub> /10 <sup>4</sup> (NA)	I <sub>L3</sub> /10 <sup>4</sup> (NA)
39	22.3	22.6	22.7	.4	.41	.41	40	.63	.2
40	Fail								
41	22.6	23.2	23.1	.33	.64	.63	10	.4	.17
42	22.2	22.1	22.7	.36	.39	.39	30	.47	.22
43	21.9	22.1	22.1	.42	.68	.68	80	1.1	.20
44	23.1	23.2	23.2	.42	.50	1.25	100	.47	.05
45	22.2	22.7	22.8	.4	.46	.43	110	.50	.07
46	23.	22.2	23.3	.44	.49	.49	200	.16	.05
47	23.1	23.4	23.4	.42	.43	.52	40	.71	.27
48	Fail								
49	22.3	22.6	22.5	.43	.41	.42	25	.77	.4
50	21.5	21.7	21.7	.38	.37	.37	25	.57	.1
$\bar{x}$	22.23		22.55	.41		.58	74		.71
$\sigma_{n-1}$	.56		.62	.05		.51	63.4		2.26

Table 3

SUPPLIER #3

22  $\mu$ F: 20V

Failure Rate = 50%

Unit No.	C1 ( $\mu$ F)	C2 ( $\mu$ F)	C3 ( $\mu$ F)	ESR1 (OHMS)	ESR2 (OHMS)	ESR3 (OHMS)	I <sub>L</sub> 1 (NA)	I <sub>L</sub> 2/10 <sup>4</sup> (NA)	I <sub>L</sub> 3/10 <sup>4</sup> (NA)
1	21.9	22.1	22	.52	2.14	2.72	.2	1.16	.83
2	22.5	22.7	22.6	.43	.98	1.0	71	.6	.212
3	22.7	23.	23	.45	2.04	2.02	12	2.52	1.86
4	22	24.8	22.3	.46	1.81	1.76	89	8.9	3.04
5	Fail								
6	Fail								
7	22.7	23	22.9	.4	.887	.9	20	1.04	11.01
8	Fail								
9	22.6	22.8	22.7	.5	1.6	1.64	.22	1.94	1.31
10	Fail								
11	22.1	22.2	22.2	.48	2.58	2.47	9.4	.314	.22
12	Fail								
13	22.3	22.5	22.5	.6	10.09	9.74	7.3	4.15	2.59
14	Fail								
15	Fail								
16	22.7	23	23	.5	2.26	2.11	40	4.6	3.45
17	Fail								
18	Fail								
19	Fail								
20	Fail								
21	Fail								
22	Fail								
23	22.6	22.8	22.7	.35	1.68	1.67	2.5	2.09	1.7
24	Fail								
25	22.6	23	22.9	.52	2.09	2.14	13	3	1.8
26	22	22.1	22.1	.48	1.9	1.72	70.	.13	.046
27	22.8	23	23	.42	.66	.67	6	3	1.34
28	21.8	21.9	21.9	.53	3.68	3.53	53	3.3	3.01
29	22.6	22.7	22.7	.61	6.41	6.23	9.8	1.77	1.2
30	22.8	23	23.1	.48	1.65	1.61	26	1.28	.62
31	Fail								
32	Fail								
33	21.9	22.	22.	.41	.94	.98	10	.09	.033
34	22.4	22.7	22.7	.5	2.46	2.4	3.2	6.16	2.18
35	Fail								
36	Fail								
37	22.5	23	22.5	.47	.67	4.51	1.3	1.68	2.02
38	Fail								
39	Fail								
40	Fail								

Table 3 (cnt'd)

Unit No.	C1 ( $\mu$ F)	C2 ( $\mu$ F)	C3 ( $\mu$ F)	ESR1 (OHMS)	ESR2 (OHMS)	ESR3 (OHMS)	$I_{L1}$ (NA)	$I_{L2}/10^4$ (NA)	$I_{L3}/10^4$ (NA)
41	22	22.3	22.3	.49	1.6	1.49	2.6	.36	.24
42	22.4	22.5	22.6	.43	1.095	1.1	280	.091	.017
43	22.8	23.2	23.2	.65	6	5.92	1790	3	1.77
44	Fail								
45	22.4	22.6	22.6	.42	.89	.9	.06	.18	.056
46	Fail								
47	Fail								
48	22.7	22.9	22.9	.48	2.1	2.01	9.7	.08	.04
49	Fail								
50	22.4	22.8	22.8	.52	1.88	1.87	79	2.17	1.36
$\bar{x}$	22.4		22.6	.48		2.5	104		1.68
$\sigma_{n-1}$	.32		.37	.068		2.1	356		2.2

Table 4  
 SUPPLIER #4  
 100  $\mu$ F: 20V  
 Failure Rate = 68%

Unit No.	C1 ( $\mu$ F)	C2 ( $\mu$ F)	C3 ( $\mu$ F)	ESR1 (OHMS)	ESR2 (OHMS)	ESR3 (OHMS)	$I_{L1}$ (NA)	$I_{L2}/10^4$ (NA)	$I_{L3}/10^4$ (NA)
1	105	109.4	109.3	.16	2.79	1.77	33	1.44	.68
2	Fail								
4	Fail								
5	Fail								
6	105.7	110.2	110	.16	.747	.542	11	1.5	.73
7	Fail								
8	Fail								
9	Fail								
10	Fail								
11	104.8	108.7	108.6	.16	1.84	1.634	9.4	1.4	.54
12	Fail								
13	Fail								
14	Fail								
15	Fail								
16	Fail								
17	Fail								
18	Fail								
19	Fail								
20	104.6	109.1	109.1	.16	.84	.668	26	.27	.15
21	106	110.3	110.2	.16	1.37	1.04	8	2.2	.88
22	Fail								
23	Fail								
24	104.8	108.6	108.6	.3	.596	.448	9	5.8	.37
25	105	109.4	109.8	.17	1.274	.923	7	.1	.11
26	Fail								
27	Fail								
28	105.5	109.4	109.3	.16	2.09	1.719	6	.86	.44
29	Fail								
30	Fail								
31	104.7	109.3	109.3	.16	1.7	1.26	15	.72	.3
32	Fail								
33	Fail								
34	Fail								
35	106.1	111.2	111.1	.16	.387	.308	2	1.2	.52
36	Fail								
37	Fail								
38	Fail								
39	106.5	111	111	.17	.186	.129	47	.46	.18
40	105.5	110.9	110.8	.16	.266	.16	5.3	2.54	1.48

Table 4 (cnt'd)

Unit No.	C1 ( $\mu$ F)	C2 ( $\mu$ F)	C3 ( $\mu$ F)	ESR1 (OHMS)	ESR2 (OHMS)	ESR3 (OHMS)	$I_{L1}$ (NA)	$I_{L2}/10^4$ (NA)	$I_{L3}/10^4$ (NA)
41	Fail								
42	Fail								
43	Fail								
44	105.1	110.	109.9	.15	.84	.676	3.2	2.4	.75
45	Fail								
46	104.7	109.8	109.7	.20	.757	.568	3.8	.64	.17
47	Fail								
48	Fail								
49	105.1	109.2	109.1	.15	1.32	1.17	5.6	1.85	.9
50	Fail								
51	106.6	110.9	110.9	.15	.67	.55	3.2	.85	.78
$\bar{x}$	105.4	109.8	109.8	.17		.81	12		.5
$\sigma_{n-1}$	.65		.82	.036		.5	12.6		.27

Table 5

SUPPLIER #5  
 40  $\mu$ F: 20V  
 Failure Rate

Unit No.	C1 ( $\mu$ F)	C2 ( $\mu$ F)	C3 ( $\mu$ F)	ESR1 (OHMS)	ESR2 (OHMS)	ESR3 (OHMS)	I <sub>L1</sub> (NA)	I <sub>L2</sub> /10 <sup>4</sup> (NA)	I <sub>L3</sub> /10 <sup>4</sup> (NA)
1	40.7	41.4	41.2	.29	.49	.51	53	.45	.28
2	Fail								
3	42.2	43.1	42.9	.31	.59	.59	26	.12	.44
4	Fail								
5	37	37.9	37.8	.28	.55	.57	23	2.7	3.4
6	Fail								
7	Fail								
8	36.1	36.9	38.7	.38	.63	.65	5	1.3	.88
9	Fail								
10	Fail								
11	Fail								
12	Fail								
13	Fail								
14	Fail								
15	Fail								
16	Fail								
17	34.6	36.6	35.4	.43	35.6	.65	20	3.7	3.9
18	Fail								
19	Fail								
20	Fail								
21	Fail								
22	Fail								
23	Fail								
24	Fail								
25	Fail								
27	Fail								
28	Fail								
29	Fail								
30	Fail								
31	Fail								
32	Fail								
33	Fail								
34	Fail								
36	Fail								
37	39	27.6	28	.26	.46	.46	30	.67	6.8
38	Fail								
39	38.2	38.9	36.7	.45	.66	.71	5	.41	.17
40	Fail								

Table 5 (cnt'd)

Unit No.	C1 ( $\mu$ F)	C2 ( $\mu$ F)	C3 ( $\mu$ F)	ESR1 (OHMS)	ESR2 (OHMS)	ESR3 (OHMS)	$I_L1$ (NA)	$I_L2/10^4$ (NA)	$I_L3/10^4$ (NA)
41	39.3	39.5	39.3	.43	.58	.59	10	.27	.07
42	Fail								
43	Fail								
44	Fail								
45	Fail								
46	Fail								
47	Fail								
48	Fail								
49	Fail								
50	Fail								
51	38.2	38.9	38.7	.3	.6	.61	21	1.1	.94
52	27.4	38	37.9	.32	.45	.46	2	2.2	.94
$\bar{x}$	38.26		37.66	.345		.58	19.5		1.8
$\sigma_{n-1}$	2.1		3.95	.069		.09	14.8		2.3



5-3. Capacitance Analysis:

Capacitance change before and after the test for the same supplier, along with a comparison of the same parameter among the different suppliers are given in Table 6.

Supplier #	Before			After			ca/cb
	$\bar{c}$	$\sigma$	$\sigma/\bar{c}$	$\bar{c}$	$\sigma$	$\sigma/\bar{c}$	
1	21.64	.43	.02	21.85	1.65	.075	1.01
2	22.23	.56	.025	22.55	.62	.027	1.01
3	22.4	.32	.014	22.6	.37	.016	1.01
4	105.4	.65	.006	109.8	.82	.007	1.04
5	38.25	2.1	.055	37.66	3.95	.010	0.98

Table 6

Capacitance mean and standard deviation before and after the test.

A look at ca/cb gives an idea on the change in capacitance for the same supplier. Theoretically, as mentioned in Chapter IV, capacitance is supposed to increase with humidity and temperature. Humidity infiltrates and eliminate series capacitances in the interfaces inside the capacitor, giving rise to increasing the overall capacitance. Temperature effect in this study is not really profound since most capacitors are designed to work without change in the range 30°C - 150°C. Therefore, Ca/Cb reflects mainly the effect of humidity which coincides with the previous work on tantalum capacitors. From a commercial point of view, it reflects in this study the sealing effectiveness of different companies. From Table 6, if we want to rank capacitor sealing of different suppliers, it would be:

<u>First</u>	<u>Second</u>	<u>Third</u>	<u>Fourth</u>	<u>Fifth</u>
#5	#1	#2	#3	#4

5-4. ESR Analysis:

ESR has usually been overlooked by different researchers, even though it gives good understanding on capacitor design and future performance. The figures given in Table 7 are the ESR values for the five suppliers included in this study.

Supplier #	Before			After			ESRA/ESRB
	ESR	$\sigma$	$\sigma$ /ESR	ESR	$\sigma$	$\sigma$ /ESR	
1	.54	.05	.092	3.68	1.6	.434	6.81
2	.41	.05	.12	.58	.51	.88	1.41
3	.48	.07	.145	2.5	2.1	.84	5.2
4	.17	.04	.23	.81	.5	.61	4.76
5	.35	.07	.2	.58	.09	.155	1.66

Table 7

ESR mean and standard deviation before and after the test.

The effect of putting capacitor in high temperature for a long time (1000 hrs). The effect of temperature on real resistors is known to be reversable, that is it goes when the temperature goes. That means there is no difference between the real resistance contribution to ESR before and after the test because the value, after the test, was taken after allowing the unit to cool down for 48 hours. What might be there is a permanent effect on the dielectric loss produced through letting the capacitor in a high temperature for one thousand hours. Also humidity effect theories stated by other researchers may not be taken as final. The results obtained for ESR in this study contradict with the work done by previous researchers, as given in Chapter IV. While those researchers said that humidity effect is to decrease ESR beyond mid freq., the units of the five different suppliers

showed all obvious increase in ESR. Therefore there are two possibilities: either the previous work was not accurate enough, or the factor of temperature is very influential on ESR. Considering that ESR represents two different resistances: hypothetical one representing the dielectric loss and a real one representing the resistance of  $MnO_2$  and leads, we should find the increase in ESR can be interpreted as that humidity has radical effect on the polarizability of  $Ta_2O_5$  which governs the dielectric loss, the main contribution to ESR at low frequencies (the ESR meter used had 1kHz internal frequency).

5-5. Leakage Current Analysis:

Leakage current is the most crucial factor when capacitors are to be evaluated. A direct look at Table #8 shows the differences in leakage current among the different companies before the test. However, this look is misleading and such a simple judgement would be totally erroneous.

Supplier #	Before			After			ILA/ILB
	$\bar{I}_L$	$\sigma$	$\sigma/\bar{I}_L$	$\bar{I}_L/10^4$	$\sigma/10^4$	$\sigma/\bar{I}_L$	
1	9.54	16.97	1.78	1.76	2.64	1.5	1845
2	74	63.4	.86	.71	2.26	3.18	96
3	104	356	3.42	1.68	2.2	1.30	162
4	12	12.6	1.05	.5	.27	.54	416
5	19.5	14.8	.76	1.8	2.3	1.28	923

Table 8  
Leakage current mean and standard deviation before and after test

Leakage current is actually a function of both rated voltage and capacitance of a certain capacitor.

The proof for that is as follows:

$$I_L = \frac{V_{\text{rated}}}{R}$$

$$R \propto \frac{d}{A}, \quad C \propto \frac{A}{d}$$

$$\text{i.e. } R \propto \frac{1}{C}$$

$$I_L \propto V_{\text{rated}} \cdot C$$

where R, d, and A are resistance, thickness and area of dielectric respectively. Therefore, a normalization of the current values we got should be done before any comparison between the different suppliers. The leakage current for each supplier will be divided by the multiplication of  $\bar{C}$  V rated, and the result will reflect how good the manufacturing is. Table 9 shows the leakage ratio for the five suppliers. The smaller the ratio the better the design and manufacturing processes.

Supplier	$\bar{C}$ V rated nA	$\bar{I}_L$ nA	Leakage Ratio = $\frac{I_L \times 100}{\bar{C} \text{ v rated}}$
1	22 X 16 = 352	9.54	2.71
2	20 X 20 = 400	74	18.5
3	22 X 20 = 440	104	23.64
4	100 X 20 = 2000	12	.6
5	40 X 20 = 800	19.5	2.44

Table 9  
Leakage Ratio

From Table 9 we find that supplier #4 is the best (before applying the life test) as far as leakage current is concerned followed by #5, #1, #2, then #3.

To summarize the experimental work, Table 10 shows an overall view of all units. The rank of each supplier is shown as a digit from 1 to 5 which can be interpreted as follows:

- $I_L$ : 1 means the least leakage current
- P: 1 means the least failure rate
- $\Delta c$ : 1 means the least change in capacitance
- $\Delta ESR$ : 1 means the least change in ESR due to test
- $\Delta I_L$ : 1 means the least change in  $I_L$  due to test
- $\frac{\sigma}{\bar{c}}$ : 1 means sample values are closest to the mean
- $\frac{\sigma}{ESR}$ : 1 means sample values are closest to the mean
- $\frac{\sigma}{\bar{I}_L}$ : 1 means sample values are closest to the mean

Supplier #	P	$I_L$	C	ESR	$I_L$	$\frac{\sigma}{\bar{c}}$	$\frac{\sigma}{ESR}$	$\frac{\sigma}{\bar{I}_L}$
1	1	3	1	5	5	3	1	4
2	2	4	1	1	1	4	2	2
3	3	5	1	4	2	2	3	5
4	4	1	2	3	3	1	5	3
5	5	2	3	2	4	5	4	1

Table 10  
Overall evaluation of study units

It should be noted that  $I_L$ ,  $\frac{\sigma}{\bar{c}}$ ,  $\frac{\sigma}{ESR}$ , and  $\frac{\sigma}{\bar{I}_L}$  refer to the values before the test. An average for each supplier should have been calculated in Table 9 except for the importance of different factors are not equal. For instance, leakage current and failure rate

are always of primary concern when capacitors are to be evaluated, whereas change in ESR is not as important.

Therefore, the order of suppliers used in this study reflects to a great extent their superiority among each others.

As to failure, humidity is not a primary cause as long as good sealing is provided. That was quite obvious because of the fact that Supplier #1 did not show any failures if we exclude the loss of lead cases.

On the other hand, since temperature is not prevented by good sealing, it seems that up to 100°C does not have any effect on units failure.

## CHAPTER VI: CONCLUSIONS

As the name of this study indicates, the ultimate goal is to obtain a reliable solid tantalum capacitor. In order to do that, common failure modes have to be analyzed so that the reason of failure can be pinpointed and the precautionary measures can be suggested. Some of the reasons of failure are pertaining to the manufacturer, while others are pertaining to the designer or the operator. Thorough investigation of the previous work, besides experimental work covered by this thesis have come out with the following summary.

Failure mode 1: Short circuit or high leakage current.

Possible reason(s):

- 1) Field crystallization of the originally amorphous dielectric. This is a manufacturing defect and can be avoided by using highly-purified tantalum anodes.
- 2) Bulging or bursting due to an internal evolution of gas. This is a manufacturing defect and can be eliminated by improving anodization and impregnation processes.
- 3) Thermal runaway, due to the negative coefficient of resistivity of  $MnO_2$ . This is an operational defect can be eliminated by not operating the capacitor in high temperature condition and also by avoiding over voltages.
- 4) Air gaps created in the interface between Ta and  $Ta_2O_5$  due to the difference in their coefficients of expansion. This is a manufacturing defect and can be avoided by improving the anodization process so that a greater adhesion between Ta and  $Ta_2O_5$  can be obtained.

Failure mode 2: Open circuit.

Possible reason(s):

Mechanical failure in contacts or welds. This is a manufacturing defect and can be eliminated by adopting better packaging techniques.

Failure mode 3: Change in capacitance

Possible reason(s):

- 1) Seal failure leading to moisture infiltration which increases capacitance. This is a manufacturing defect and the solution is improving sealing techniques.
- 2) Gradual growth in thickness of the oxide film leading to a reduction in capacitance. This is a manufacturing defect and can be avoided by improving anodization process.

Failure mode 4: Change in ESR

Possible reason(s):

- 1) Seal failure and moisture infiltration (as in mode 3) the effect here as shown in the experimental part of this study is a drastic increase ( 300%) in ESR. A better sealing process is recommended.
- 2) Formation of oxide film at one of the metal-to-metal joints leading to a rising ESR: This is an operational defect and can be avoided if the capacitance is kept away from moisture.



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