Dielectric Properties of Thin Tantalum Oxide Layers at Solid Tantalum Capacitors

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***Abstract*—In this paper MIS (Metal-Insulator-Semiconductor) Ta_2O_5 capacitor has been studied in terms of dielectric relaxation with a low frequency dielectric spectroscopy. The results acquired for Ta_2O_5 show a relaxation peak in the temperature and frequency range available, 187 K – 385 K, 1 Hz – 10 MHz. The loss peak frequency follows the Arrhenius law dependence with the activation energy of 0.048 eV. In conductivity spectra, Ta_2O_5 film exhibits a steady–state value at low frequencies and a monotonous increase at high frequencies depending on temperature. The observed conductivity followed a slightly superlinear power law.**

Keywords-thin oxide film; dielectric properties; Havriliak–Negami (HN) equation; electrical conductivity

I. INTRODUCTION

Solid tantalum capacitors are increasingly being used in electronic circuitry because of their high volumetric efficiency, reliability, and temperature stability over the range – 55 °C to + 125 °C [1]. The construction of tantalum capacitor begins with a fine tantalum powder from pure elemental tantalum metal. The anode of the capacitor is formed from tantalum powder by pressing it into a pellet around a tantalum wire. This is anodized at ~ 85 °C to produce a thin dielectric film of amorphous tantalum pent-oxide (Ta_2O_5). For a 1 µF capacitor (manufactured by AVX) with a rated voltage of 50 Vdc the dielectric thickness is ~ 289 nm. Between the tantalum anode and the Ta_2O_5 dielectric a very thin semiconductor layer of tantalum monoxide (TO) forms and this gives the capacitor its polar characteristics [1]. The second electrode is cathode material. The pellet is dipped into an aqueous solution of $Mn(NO_3)_2$ .The pellet is heated till 250 °C to dry the solution and convert it to manganese dioxide (MnO_2). The silver paint is applied to the top of the MnO_2 to allow a low resistance external connection to the capacitor. Before the application of silver, a coating of graphite solution is applied to eliminate interfacial resistances due to the contact and silver oxide formation at interfacial region [2]. The last step is to enclose the capacitor in epoxy resin.

For measurement of the dielectric properties of capacitors dielectric spectroscopy were used. Alpha-A analyzer with the Quatro Cryosystem were used. Quatro Cryosystem is designed for easy, safe and fully automatic operation enabling computer controlled experiments over several days without supervision as required for low frequency measurements with high resolution and accuracy. These features make them ideal for testing low frequency component such as solid tantalum capacitor.

The dielectric relaxation response was analyzed by the Havriliak–Negami (HN) relaxation function which is a frequency-domain function.

$$\hat{\varepsilon}(\omega) = \varepsilon_{\infty} + \frac{(\varepsilon_s - \varepsilon_{\infty})}{[1 + (j\omega\tau_0)^{\alpha}]^{\beta}} \qquad (1)$$

where α and β represent the width (symmetry) and the skewness (asymmetry) of the dielectric loss number ε″(ω) when viewed in the log(ε″) – log(ω) plot.

II. EXPERIMENTAL METHOD

Figure 1 shows a detail of the measurement setup. For temperature measurement, 1 µF solid tantalum capacitor was lined with copper wire to ensure good contact between capacitor and sample cell BDS 1200; the sample cell BDS 1200 was inserted into Cryostat BDS 1100. Quatro controller BDS 1330 consists of four circuits controlling the sample temperature, the gas temperature, the temperature of the liquid nitrogen in the dewar and the pressure in the dewar. Alpha-A analyzer with a test signal of amplitude 2.2 V_{rms} and frequency range from 1 Hz to 10 MHz was used to measure capacitance and dissipation factor. After gas passes through the gas heater, it flows directly through the vacuum insulated sample by a 2 stage rotary vane vacuum pump, providing thermal isolation by low vacuum (< 10 µbar) cell mounted in a cryostat.

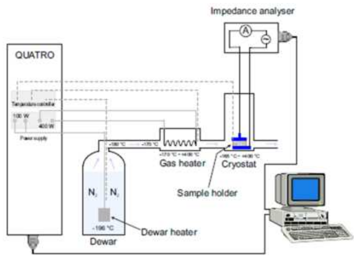

FIGURE I. THE EXPERIMENTAL SETUP [3].

III. RESULT AND ANALYSIS

The relative permittivity versus frequency at different temperature range curves for 1 µF /50 V_{dc} tantalum capacitor are shown in Figure. 2. At low frequency, relative permittivity increases slightly with increasing temperature. This suggests that the low frequency behaviour could be attributed to the accumulation of charges at electrodes due to an electrode polarization. The relative permittivity is 27 at 120 Hz and 304

K. At higher frequency above 1 MHz and specified temperature 187 K, parasitic effects start to dominate.

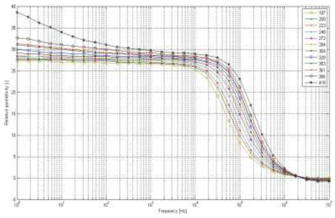

FIGURE II. RELATIVE PERMITTIVITY VS FREQUENCY AS A FUNCTION OF TEMPERATURE.

The relaxation peak starts to appear at frequency about 10 kHz as shown below. The relaxation peak moves slightly towards the higher frequency with the increasing temperature. The relaxation peak shows almost no amplitude increase with temperature. This behaviour has already been reported for Ta2O5 due to the stability of tantalum raw material and its oxide [4].

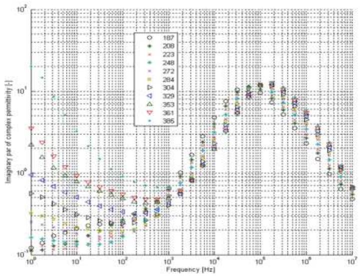

FIGURE III. RELAXATION PEAK VS FREQUENCY AS A FUNCTION OF TEMPERATURE.

In order to evaluate the change in the dielectric loss (dissipation factor) curve we obtained values $\Delta\varepsilon$, α_{HN}, β_{HN} and τ_{HN} as a function of frequency by fitting the observed values to HN equation.

TABLE I .HAVRILIAK–NEGAMI PARAMETERS OF 1 µF / 50 V_{DC} AT DIFFERENT TEMPERATURES: FROM 187 K TO 418 K.

T [K]	α_{HN} [-]	β_{HN} [-]	$\Delta\varepsilon$ [-]	τ_0 [s]
187	0.985	0.710	26.88	4.198×10^{-6}
208	0.980	0.724	27.14	3.370×10^{-6}
223	0.969	0.756	27.27	2.817×10^{-6}
248	0.965	0.769	27.65	2.303×10^{-6}
272	0.971	0.766	27.98	1.965×10^{-6}
284	0.974	0.764	28.15	1.829×10^{-6}
353	0.955	0.852	28.76	1.164×10^{-6}
361	0.950	0.873	28.84	1.105×10^{-6}
385	0.928	0.965	28.94	9.057×10^{-6}
418	0.912	1	29.72	7.231×10^{-6}

Figure 4 show the loss peak frequency follows an Arrhenius law with the activation energy of 0.048 eV.

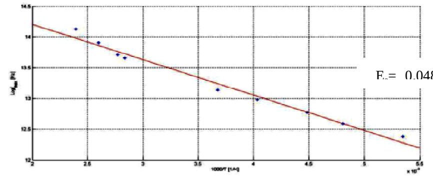

FIGURE IV. ARRHENIUS PLOT OF THE FREQUENCY VALUE OF RELAXATION PEAK.

The capacitance – temperature curves for 1 µF / 50 V_{dc} tantalum capacitor are shown in Figure. 5. This figure clearly shows the decrease of the relative permittivity with increased frequency; this is in keeping with specification data of solid tantalum capacitor [1]. This is the main reason for limiting these types of capacitors to low frequency applications.

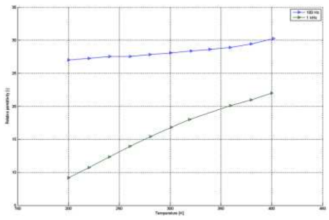

FIGURE V. RELATIVE PERMITTIVITY AS FUNCTION OF TEMPERATURE AND FREQUENCY.

The dissipation factor was 6 % at temperature 300 K and frequency 100 Hz. At this frequency, losses are at an acceptable level; for the temperature 418 K, i.e., above the normal working temperature, the loss increases considerably. At higher frequency and low temperature dissipation factor increased considerably.

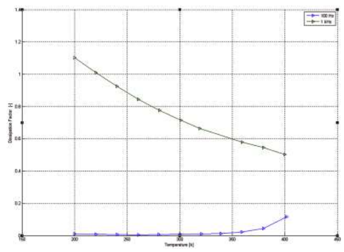

FIGURE VI. DISSIPATION FACTOR AS FUNCTION OF TEMPERATURE AND FREQUENCY.

Figure 7 shows the frequency dependence of electrical conductivity at various temperatures for 1 µF / 50 V_{dc} tantalum capacitor. The electrical conductivity depends on frequency according to the “universal dynamic response” [4] and can be related as

$$\sigma(\omega) = \sigma_{dc} + A\omega^n \qquad (2)$$

where A is the temperature dependent parameter and the exponent n is a characteristic parameter representing the many body interactions of the electrons, other charges and impurities. It varies from 0 to 1. For all temperatures, the conductivity exhibits a steady increase in low frequency regime. Above a characteristic frequency, the conductivity increases with increase in frequency with the characteristic ω^n dependence.

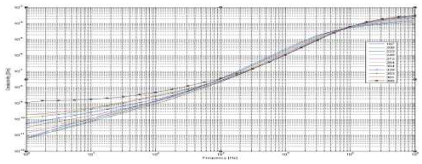

FIGURE VII. THE REAL PART OF THE AC CONDUCTIVITY AS A FUNCTION OF FREQUENCY AT DIFFERENT TEMPERATURES.

IV. CONCLUSIONS

1. The low frequency behaviour of the relaxation peak could be attributed to an electrode polarization mechanism related to the accumulation of mobile charges at electrodes.

2. The relaxation peak in Ta_2O_5 follows the Arrhenius law dependence with the activation energy of 0.048 eV.

3. Capacitance and dissipation factor as function of temperature performance curves for tantalum (1 µF / 50 V_{dc}) capacitor clearly show the capabilities and limitations of tantalum capacitors at low temperature and high frequency.

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