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Characterization of a Fused Silica Capacitance Standard

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Characterization of a Fused Silica Capacitance Standard

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Abstract

An ovenized 10 pF standard capacitor was constructed by the National Institute of Standards and Technology (NIST). The dielectric material used was Suprasil II grade fused silica. This report discusses a temperature coefficient analysis of the capacitor performed at the Primary Standards Laboratory (PSL) of Sandia National Laboratories (SNL).

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Characterization of a Fused Silica Capacitance Standard

Introduction

Investigation of capacitance standards using fused silica as a dielectric was conducted at NIST for a number of years by R. D. Cutkosky and L. H. Lee. The most stable out of a set of twelve standards constructed at NIST are currently implemented as their primary standards. These capacitors have been periodically checked against the NIST calculable capacitor which is the ultimate reference.³

An understanding of what gives a capacitor its capacitance will aid in understanding many of the other characteristics discussed in this report. All capacitors consist basically of two metal electrodes separated by an insulator that is called a dielectric. In the fused silica capacitor, for example, this structure is realized by depositing a metal film on both sides of a ground silica disk and attaching leads. A metalized polycarbonate capacitor consists of two plastic films with metal deposited on one side of each, laid on top of each other, and wound in a spiral to form a tubular shape.⁵

The capacitance of parallel plate capacitors is directly proportional to the relative dielectric constant of the insulator and to the area of the plates and inversely proportional to the spacing between the plates. (see Table 1, Dielectric Constant Table) The capacitance is greater if the separation between the plates is small, and visa versa.

These facts are expressed by the following equation:²

$$C = \frac{\epsilon_0 K A}{d}$$

Where:

C = capacitance in farads, F

***ϵ_0 = a constant, called the permittivity of free space
(8.85×10^{-12} F/m)***

***K = relative dielectric constant of insulator
(4.2 for fused silica)***

A = area of either capacitor plate, m²

d = distance between the plates, m

Since this equation neglects the fringing flux lines at the edges of the plates, the result may be slightly less than the actual capacitance⁷. The fringing effects can be greatly reduced by a guard ring that surrounds the active plates. To increase capacitance for a given size, we could reduce d and, by adding more layers, increase A . This process is limited by the maximum field strength of the dielectric since, for a given voltage, the electrical field increases as the thickness decreases. We have no control over ϵ_0 , but K ,

the dielectric constant, can range from one to several thousand for various insulating materials and is, therefore, an important parameter. Almost all capacitance variations with temperature, frequency, and applied voltage are due to variations in K .

<i><u>Dielectric Material</u></i>	<i><u>Dielectric Constant, K</u></i>
<i>Vacuum</i>	<i>1</i>
<i>Air</i>	<i>1.0006</i>
<i>Teflon</i>	<i>2</i>
<i>Polystyrene</i>	<i>2.5</i>
<i>Mylar</i>	<i>3</i>
<i>Fused Silica⁸</i>	<i>3.78</i>
<i>Paper, paraffin</i>	<i>4</i>
<i>Mica</i>	<i>5</i>
<i>Aluminum oxide</i>	<i>7</i>
<i>Tantalum oxide</i>	<i>25</i>
<i>Ceramic (low k)</i>	<i>10</i>
<i>Ceramic (high k)</i>	<i>100 -- 10,000</i>

Table 1. Dielectric constants of some commonly used materials²

A laboratory can have several values of commercially available standard representations of capacitance. The best types are usually three terminal, hermetically sealed capacitors with nitrogen or fused silica dielectrics.⁴

NIST 10 pF Fused Silica Dielectric Capacitor

The capacitor characterized in this report will be referred to as the SNL - NIST Fused Silica Capacitor (SNL - NFSC). The SNL - NFSC was constructed by grinding a disk of Suprasil II grade fused silica to the dimensions shown in Figure 1; and then firing gold electrodes on the surfaces, and in the guard ring around the edge. The guard ring electrode is connected to ground, and the center electrodes on the two surfaces form the active electrodes of the capacitor. The special grade of fused silica has the advantage of low losses and low frequency dependence of its dielectric constant in the audio-frequency range. Fused silica capacitors can be exceptionally stable, and are far more rugged than air capacitors. The major difficulty encountered in their construction is the elimination of voltage dependencies, which in exceptional cases can cause the capacitances to change by several parts in 10^7 when the voltage changes from a few volts to 100 or 200 volts.

The construction technique that was employed by NIST is still under development, but the SNL-NFSC changes less than 1 part in 10^9 for voltages below 200 V, and has a drift of a few parts in 10^7 per year.¹

Fused silica capacitors have temperature coefficients of approximately 12 ppm per degree Celsius¹. It is necessary to control the temperature of these capacitors to about 0.01 degrees Celsius if the uncertainties in capacitance are to be smaller than a few parts in 10^7 . A change in capacitance occurs with temperature variations due to thermal expansion of the dielectric causing changes in plate spacing and area. This variation in capacitance in relation to temperature can not only be very small but simultaneously very linear. In the SNL-NFSC, a copper wire resistor that is wound on the capacitor housing is used to sense the temperature. This resistor is connected in a four terminal configuration. The Amphenol connector marked "R" on the capacitor is used for resistance measurement.

Calculation of Capacitance:

$$C = \frac{\epsilon_0 K A}{d} \quad C = \frac{(8.85E-12)(3.78)(8.33E-4)}{2.54E-3}$$

$$C = 10.98 \text{ pF}$$

Figure 1. The SNL-NFSC was constructed by grinding a disk of Suprasil II grade fused silica to the dimensions shown. Gold plating was then applied to form the electrodes and guard ring.

Test Components⁸

Capacitance / Loss Measurements:	Andeen Hagerling Model 2500A
(+/- 5 ppm uncertainty)	Ultra Precision Capacitance Bridge
Temperature Measurements:	Instrulab Model 4221 PRT Indicator
(+/- 0.007 Deg C uncertainty)	Minco 25.5 Ohm Platinum Resistance Thermometer
Resistance Measurements:	Hewlett Packard Model 3458A Digital Multimeter
(+/- 20 ppm uncertainty)	
Environmental Chamber Range:	10 - 50 Degrees Celsius
Instrument Controller:	Hewlett Packard Vectra 12/ES & Hewlett Packard Measurement Coprocessor

Temperature Excursion Test Design

The main purpose of this study is to determine the temperature coefficient of the SNL-NFSC in ppm / deg C (change in capacitance per degree change in temperature). The effects of temperature change on dielectric loss will also be discussed.

The SNL-NFSC with its internal oven disabled was placed in an environmental chamber and subjected to temperatures between 10 and 50 degrees Celsius. Measured parameters varied as the SNL-NFSC equilibrated to the environment of the chamber. The capacitance and loss measurements were taken using an Andeen Hagerling Model 2500A Ultra Precision Capacitance Bridge at 15 volts and a frequency of 1 KHz. The 2500A was certified by the PSL to an uncertainty of +/- 5 ppm.

Capacitance, loss, and temperature sensor resistance measurements were made automatically every 10 minutes using software developed by Michael G. Daniel at SNL for the Automated Capacitance Measurement System. The temperature of the environmental chamber was increased from 10 to 50 degrees Celsius at 5 degree increments. A platinum resistance thermometer was attached to the SNL-NFSC to sense the temperature of its mass. The capacitor was allowed to equilibrate for a period of 24 hours after each increment. It was observed that after approximately 12 hours the measured values begin to

stabilize at the temperatures shown in the following table. Figure 2 shows the capacitance time constant and the tracking resistance values of the SNL-NFSC oven temperature sensor. Figure 3 shows the capacitance values with the corresponding temperatures measured using the PRT attached to the SNL-NFSC during the tests.

Temperature Excursion Test Data

The capacitance in picofarads (pF), loss in nanosiemens (nS), and resistance in ohms measurements taken throughout the temperature cycling were linear as expected. The averages of the last ten measurements at each temperature setting are shown below. Plotted results are shown in Figures 4, 5, and 6.

	Deg C	pF	nS	Ohms
(A)	10.842	9.998295	-0.000376	8.877061
(B)	15.687	9.998820	-0.000368	9.051038
(C)	20.503	9.999369	-0.000341	9.225749
(D)	25.359	9.999937	-0.000302	9.401901
(E)	30.197	10.000519	-0.000257	9.577372
(F)	34.989	10.001110	-0.000212	9.751466
(G)	39.728	10.001708	-0.000170	9.923392
(H)	44.530	10.002330	-0.000129	10.098083
(I)	49.331	10.002962	-0.000091	10.272167

The following data show the averaged values and calculated change per Degree Celsius for the various parameters at each temperature setting. Temperature coefficients for the various parameters are summarized in Table 2.

Calculated Temperature Coefficients:

Oven Deg C (Approx)	Measured Delta pF	Measured Delta Deg C	Coefficient pF/Deg C	
15	0.00053	4.845	0.000108	
20	0.00055	4.817	0.000114	
25	0.00057	4.856	0.000117	
30	0.00058	4.837	0.000120	
35	0.00059	4.792	0.000123	
40	0.00060	4.739	0.000126	
45	0.00062	4.803	0.000129	Average pF/Deg C
50	0.00063	4.800	<u>0.000132</u>	0.000121 (12.1 ppm/Deg C)

Oven Deg C (Approx)	Measured Delta nS	Measured Delta Deg C	Coefficient nS/Deg C	
15	8.3 E-06	4.845	1.72 E-06	
20	2.6 E-05	4.817	5.47 E-06	
25	3.9 E-05	4.856	8.11 E-06	
30	4.5 E-05	4.837	9.23 E-06	
35	4.5 E-05	4.792	9.39 E-06	
40	4.2 E-05	4.739	8.91 E-06	
45	4.1 E-05	4.803	8.53 E-06	Average nS/Deg C
50	3.8 E-05	4.800	<u>7.94 E-06</u>	7.41 E-06

Oven Deg C (Approx)	Measured Delta Ohms	Measured Delta Deg C	Coefficient Ohms/Deg C	
15	0.17398	4.845	0.035911	
20	0.17471	4.817	0.036273	
25	0.17615	4.856	0.036276	
30	0.17547	4.837	0.036275	
35	0.17409	4.792	0.036327	
40	0.17193	4.739	0.036282	
45	0.17469	4.803	0.036372	Average Ohms/Deg C
50	0.17408	4.800	<u>0.036264</u>	0.036248

NIST Fused Silica Capacitor

Time Constant

Figure 2. This plot shows the time constant required for stabilization at each temperature setting from 10 - 50 degrees Celsius. Each 5 degree increment in temperature was held for a period of 24 hours. Resistance measurements of the oven sensor were made using the copper wire resistor that is permanently mounted inside of the SNL-NFSC.

NIST Fused Silica Capacitor

Time Constant

Figure 3. This plot shows the capacitance time constant with the corresponding temperatures measured with the Instrulab 4221 and the PRT attached to the SNL-NFSC.

NIST Fused Silica Capacitor

Temperature Coefficient

Figure 4. This plot shows the temperature coefficient of the capacitance parameter. The response was essentially linear with an average 12.1 ppm per degree Celsius change.

NIST Fused Silica Capacitor Temperature Coefficient

Figure 5. This plot shows the temperature coefficient of the loss parameter. The calculated average change per degree Celsius was 7.41×10^{-6} nanosiemens.

NIST Fused Silica Capacitor

Temperature Coefficient

Figure 6. This plot shows the temperature coefficient of the resistive sensor used to monitor the SNL-NFSC internal oven temperature. The calculated average change per degree Celsius was 36.2 milliohms.

Change / Degree Celsius

Capacitance	12.1 ppm
Loss	7.41 E-6 nS
Resistance	0.036248 Ω

Table 2. Calculated Temperature Coefficients

A linear regression analysis was performed to derive a correlation between pF and the temperature sensor measurements in ohms. This relation is valid only for this particular capacitor with 15 volts applied at 1 KHz.

Formula: **$pF = 0.00335 \times \text{ohms} + 9.96850$**

The following table shows the difference between the actual measured capacitance and the calculated capacitance at various measured resistance values.

Measured Deg C	Measured Ohms	Measured pF	Calculated pF	Difference Calc-Meas pF
10.842	8.877061	9.99830	9.99822	- 0.00008
15.687	9.051038	9.99882	9.99881	- 0.00001
20.503	9.225749	9.99937	9.99939	+0.00002
25.359	9.401901	9.99994	9.99998	+0.00004
30.197	9.577372	10.00052	10.00057	+0.00005
34.989	9.751466	10.00111	10.00115	+0.00004
39.728	9.923392	10.00171	10.00173	+0.00002
44.530	10.098083	10.00233	10.00231	-0.00002
49.331	10.272167	10.00296	10.00289	-0.00007

The calculation error in deriving capacitance values strictly from measured temperature sensor resistance values can be as large as 8 parts per million (ppm). Therefore, this method should not be relied upon for primary level metrology. Another formula that can be used with this particular capacitor is one that converts the temperature sensor resistance readings to degrees Celsius.

Formula: **Degrees C = 27.570440 X ohms + -233.867340**

Measured Ohms	Measured Deg C	Calculated Deg C
8.877061	10.8	10.9
9.051038	15.7	15.7
9.225749	20.5	20.5
9.401901	25.4	25.3
9.577372	30.2	30.2
9.751466	35.0	35.0
9.923392	39.7	39.7
10.098083	44.5	44.5
10.272167	49.3	49.3

At normal operating temperature, the capacitor's oven sensor measures 9.598624 ohms. Using the formula, this resistance translates to a temperature of 30.77 degrees Celsius. This formula can be useful in applying a temperature correction to the measured capacitance value.

For example:

Certified value	10.000593 pF @ 30.77 Deg C +/- 3 ppm
Measured value	<u>10.000590 pF @ 30.75 Deg C</u>
Measured Difference	0.000003 pF 0.02 Deg C

Calculate temperature correction factor:

$$12.1 \text{ ppm} \times 0.02 = 0.24 \text{ ppm}$$

Correct measured value to certified temperature of 30.77 Deg C:

$$10.000590 \text{ pF} + 0.24 \text{ ppm} = 10.000592 \text{ pF}$$

Since this value is within 0.1 ppm of the certified value, this shows the capacitor is functioning correctly and the measured value to be valid

An actual example of temperature correction occurred when the PSL received a certified General Radio Model 1408 Standard Capacitor from NIST. The 1408 is a fused silica dielectric capacitor with an internal oven. General Radio states that 12 ppm \ Deg C is the appropriate temperature coefficient for this standard.

NIST Certified value 100.003458 pF @ 29.88 Deg C

PSL Measured value 100.003257 pF @ 29.69 Deg C

Measured Difference 0.000201 pF 0.19 Deg C

Allowed Difference +/- 0.000080 pF (or 0.8 ppm)

Calculate temperature correction factor:

$$12 \text{ ppm} \times 0.19 = 2.28 \text{ ppm}$$

Correct measured value to certified temperature of 29.88 Deg C:

$$100.003257 \text{ pF} + 2.28 \text{ ppm} = 100.003485 \text{ pF}$$

The corrected value now differs from the NIST certified value by 0.27 ppm which is well within the allowed uncertainty of +/- 0.8 ppm.

In general, the measured capacitance value of gas or solid dielectric standards at a given temperature depend upon the previous temperatures to which the standards have been subjected.³ Figure 7 shows the hysteresis effect of the temperature excursions for this nine day characterization to be minimal. After the temperature excursion test (at B), the SNL-NFSC was allowed to stabilize under the control of its own internal oven at a temperature of 30.77 degrees Celsius. This equilibration period continued for about fifteen weeks (between points B & C). The after excursions value (at C) differed from the before excursions value (at A) by only 1.92 E-7 pF. This difference can be accounted for by normal random measurement deviations.

NIST Fused Silica Capacitor

Hysteresis Effect

Figure 7. This plot shows the hysteresis effect on the SNL-NFSC measured capacitance. (A) indicates 2/23/94, which was the date opening measurements were started before the nine day temperature excursion testing began on 3/1/94 (shown at the upper point of the vertical segment at B). Values obtained during testing were removed from this plot. The lower point of the vertical segment (at B) 3/11/94, indicates the beginning of the post testing recovery period which ended (at C) 7/1/94.

The data obtained from this characterization study, in addition to the accumulated historical data, show that the SNL-NFSC is a viable laboratory standard and possibly a candidate for capacitance map transfers.

Conclusion

The characterization of the SNL-NFSC successfully accomplished the goal of deriving temperature coefficients for the capacitance and loss parameters. The calculated capacitance temperature coefficient of 12.1 ppm per degree Celsius is characteristic of fused silica dielectric capacitors. A time constant of 12 hours was observed for stabilization at each new temperature setting between 10 and 50 degrees Celsius. This information is useful for capacitance map transfers where the SNL-NFSC would be without AC power for extended periods of time. In these cases, before capacitance values are measured, the SNL-NFSC should be connected to AC power and allowed to stabilize for at least 24 hours. Use of the conversion formula to derive degrees Celsius from the temperature sensor resistance readings will be essential in applying temperature correction factors to the standard's measured value. To make meaningful measurements at a level of a part in 10^7 , the temperature must be known to within 0.01 degrees Celsius.⁶ This requires resistance measurements of the temperature sensor to be made within an uncertainty of $\pm 0.1\%$ based on the conversion formula to derive degrees Celsius. An alternative to using the internal copper wire resistor for temperature measurement would be to permanently attach a platinum resistance thermometer to the outside of the hermetically sealed capacitor chamber.

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