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Evaluation of Constant Voltage Chamber Modifications

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Introduction

Determining the electrical properties of highly insulating materials can be a challenging task. These materials are designed to greatly resist the flow of electrical current making them useful in the construction of spacecraft. Due to the fact that satellites are isolated from ground, charging caused by the plasma environment found in typical orbital radii (1) is of concern to the designers of modern spacecraft (2). The Utah State University Materials Physics Group Constant Voltage Chamber (CVC) has been designed to measure extremely low currents and low conductivity. Over the last five years, many changes have been made to improve the accuracy and precision of measurements made with the CVC, now allowing currents as low as hundreds of attoamps (3, 4, 5). In developing a data analysis procedure, a program has been written to quickly generate reports of the temperature, current, and conductivity which include standard deviation and statistical analysis of the instrumentation error for the system (5). This has allowed for immediate assessment of the system operation providing a means to more easily improve the quality of data taken with the CVC.

Conductivity Measurements

The Constant Voltage Chamber is a unique apparatus designed to take measurements of the conductivity of various insulating materials. Since many of these thin film polymers and ceramic samples are used in the space environment, the CVC utilizes a high vacuum pumping system to mimic the low pressures found in the space environment. The chamber can operate at a range of temperatures spanning 250 K, from liquid nitrogen temperatures to near the melting point for many of the samples tested.

The goal when analyzing insulating materials using the CVC is to determine the conductivity of the material. An electric field is applied between the electrode and high voltage plate which is directed though the sample (see Fig. 1). This setup allows us to measure the corresponding decay currents, typically in the femtoamp range, that occur as a result of the electric field. To show the relationship between the electric field and measured current, starting with Ohm’s Law: \( V = I \cdot R \), then dividing by sample thickness \( d \) and using the definition of resistance \( R = \frac{\rho \cdot d}{A} \), resistance \( R \) equals resistivity \( \rho \) times sample thickness \( d \) divided by cross-sectional area \( A \) gives:

\[
\frac{V}{d} = \frac{l \cdot R}{d} \quad \rightarrow \quad \frac{V}{d} = \frac{I}{d} \cdot \frac{\rho \cdot d}{A} \quad \rightarrow \quad \frac{V}{d} = \frac{I}{A} \cdot \rho
\]

Since resistivity is the inverse of conductivity, \( \rho = \frac{1}{\sigma} \), and the electric field is defined as a potential voltage over some distance \( \frac{V}{d} \), substituting these values gives:

\[
\frac{V}{d} = \frac{I}{A} \cdot \rho \quad \rightarrow \quad \frac{V}{d} = \frac{I}{A} \cdot \frac{1}{\sigma} \quad \rightarrow \quad E = \frac{I}{A \cdot \sigma} \quad \Rightarrow \quad I = A \cdot \sigma \cdot E \quad [1]
\]

The electric field \( E \) and electrode area \( A \) are constant so by simply measuring the current \( I \) that propagates through the sample, the conductivity \( \sigma \) can be determined using Eq. [1].
Modifications

The CVC chamber has undergone numerous revisions, in both the electronic and hardware configurations. A detailed electrical schematic has been generated (see Appendix A) to more easily identify grounding loops, inadequate shielding, and noise issues associated with improper use of the filtered A/C power strip. This schematic has also helped to better understand the subtle details of how grounds are handled in the data acquisition interface box (NI BNC-2110) allowing for more accurate and responsive data acquisition. Most notably, this meticulous characterization of the electronics helped identify a flaw in the building design where the third prongs in the power outlets were not being grounded; this has been resolved.

As the schematic developed, mechanical systems including vacuum and cryogenic layouts were added allowing a more complete characterization of the system as a whole. This helped to locate and correct leaks in the liquid nitrogen system which caused vacuum system failures at low temperatures by allowing atmosphere to be introduced into the chamber leading to excessive noise in the current measurements. Resolving these issues has allowed more precise measurements over temperatures ranging from 100 K to 350 K.

To insure proper contact between the electrodes and the surface of the measured sample, a spring clamping mechanism has been built to allow for consistent and repeatable sample pressure (see Fig. 2). This setup consists of four springs at the corners of the electrode plate assembly constructed to maintain electrical isolation between the voltage plate and the cooling reservoir. Adhering to ASTM D 257-99 standards (6) recommendations for an applied pressure in the limit of 140-700 kPa, calculations were made to determine the correct spring constant for use in this setup (see Appendix B) exerting a pressure of approximately 400 kPa, the average of the standards recommendation.

Data Analysis

Analyzing data taken with the CVC has been challenging due, in large part, to the immense amount of data acquired with this system. Data runs typically span many orders of magnitude in time (up to 10^5 s duration at 1 s to 10 s intervals) making them difficult to repeat if a problem occurs during a run. A hybrid program (using Labview, Excel, and IGOR pro) has been developed allowing for rapid analysis of the data, as well as the system’s performance. This program uses an adaptive binning algorithm to calculate mean averages for the current measurements. From this, the statistical error is applied to the data spread using Eq. [2] for each npnts bin:

\[
\Delta \sigma_{SD} = \frac{1}{npnts - 1} \sum (Y_i - npnts_{avg})^2
\]

An instrumentation error document (7) has been generated outlining the error associated with each piece of equipment used in the CVC. This document incorporates second order error calculations based on quantities such as the response time of the low level electrometer and operating frequency of the data acquisition card; constant and relative error of the applied voltage associated with the power supply and current measured with the electrometer, as well errors in sample and electrode measurements. The voltage
error Eq. [3], current error Eq. [4], and instrument error Eq. [5] are calculated for each bin and applied to the corresponding graphs (see CVC Error Analysis v1_7 document for a detailed explanation of these equations).

\[
\Delta V = (N_V - 1)^{\frac{1}{2}} \cdot [250 \text{ mV} + 0.1\% \cdot V]
\]  

\[
\Delta I_{err}(I, R, S) = \left[ N_{bin} \cdot (N_I - 1) \cdot \min \left( 1, \frac{100}{T_R(R) \cdot f_I} \right) \right]^{\frac{1}{2}} \cdot \left[ I \cdot (\Delta F_{elec}(R) + \Delta F_{DAQ}) + (10^R \cdot A) \cdot \left[ \Delta I_R \cdot [1.4 - 0.4 \cdot (3 - S)] + \Delta I_D \cdot 10^{(S-2)} \right] \right]
\]

\[
\Delta \sigma = \sigma \cdot \sqrt{\left( \frac{\Delta I_{err}}{I} \right)^2 + \left( \frac{\Delta V}{V} \right)^2}
\]

- \( N_V \) = Number of samples taken for a given voltage data set,
- \( V \) = Measured voltage,
- \( I \) = Current measured by the electrometer,
- \( R \) = Electrometer current range setting,
- \( S \) = Electrometer display sensitivity setting,
- \( \Delta F_{elec} \) = Electrometer range resolution factor at a given range, \( R \),
- \( T_R \) = Rise time (response time of the meter for a current change from 10% to 90% of full scale) at a given range, \( R \),
- \( \Delta F_{DAQ} \) = DAQ resolution factor,
- \( N_I \) = Number of samples taken for a given current data set,
- \( f_I \) = Sampling rate of DAQ card,
- \( \Delta I_R \) = Range resolution,
- \( \Delta I_D \) = DAC card error for least significant bit (LSB).

By comparing the statistical error, \( \Delta \sigma_{SD} \), to the instrument error, \( \Delta \sigma \), a quantitative assessment of how well the chamber is performing can be made.

Results

Three data sets have been chosen for comparison of instrumentation performance and quality of data. All measured samples are 27.4 ± 0.5% \( \mu m \) thick Low Density Polyethylene (LDPE) with an applied electric field of 100 V. The first data run, (LDPE Up to 1000 V 8-14-2007) (3), was taken prior to the modifications outlined in this report. The second and third data runs, (LDPE 27.4 100V RT filter test 3-26-2009; and LDPE 27.4 100V 22hr RT testing 2-5-2009), were taken with the chamber modifications; the latter used a 100 V battery as a highly stable supply voltage. This analysis will identify valid time ranges for comparison of calculated conductivity values, statistical error, and instrument error error.

The test run taken on 8-14-2007 consisted of a series of voltage runs; 25, 50, 100, 200, 300, 400, 500, 600, 700, 800, 900, 1000 V. For each run, the electric field was applied for 3600 s then turned off for the same duration to allow discharge. The 100 V charged run is the focus of this analysis which corresponds to a start time of 14400 s. The pre-exposure to an electric field in this run effectively reduces initial polarization effects which are found in the other data runs used for this comparison. In addition, the output program used to generate the raw data file did not include actual voltage data from the power supply hence a constant 100 V was used to analyze this data set using the CVC analysis program resulting in an unrealistically low instrument error, \( \Delta \sigma \).
The test run taken on 3-26-2009 used a voltage filter in an attempt to dampen power supply fluctuations, in addition to the above stated modifications to the system. The test run taken on 2-5-2009 used a 100 V battery supply designed to operate with minimal drift while maintaining a very steady supply voltage. For the purpose of this comparison, a time range for these two data sets has been determined to be approximately 40000-78000 s since these runs both started with similar initial conditions. The time scale for test run taken on 8-14-2007 is between 1035-3645 s, which allows for a reasonable comparison of dark current values for all data runs. The CVC analysis results Table I summarizes the averages for current measurements taken with the CVC and the conductivity calculations determined by the CVC error analysis program. Values obtained for current measurements show the two 2009 data runs agree within ~ 10%. The average current obtained for the data run taken on 8-14-2007 agrees with the previous measurements within ~ 50%. These measurements vary within a reasonable amount for this type of high grade LDPE sample (typical measurements can yield up to 200% variations for standard samples). The highlighted current statistical error shows a reduction of greater than 90% to that of the other two data runs which equates to roughly an order of magnitude increase in the precision of current measurements obtained with the CVC. The current instrument error for the test run taken on 2-5-2009 shows a 50% increase when compared to the other test runs. This is due to the electrometers sensitivity setting for this run which was set to 1 as opposed to the other runs taken at sensitivity setting of 0; all test runs use the electrometers lowest range setting of $10^{-11} \text{A}$. The current instrument error values of $2 \cdot 10^{-16} \text{A}$ represents the lowest possible current measurement that can be taken with this system, which is on the order of hundreds of attoamps.
To compare these values with common literature for LDPE, and to verify the system performance as a whole, the conductivity calculations will be compared. All conductivity values agree within ~ 50%; again this is very reasonable for high quality samples since typical conductivity calculations for LDPE can vary orders of magnitude (4). The long term equilibrium dark current conductivity value of ~ $9 \cdot 10^{-19} (\Omega \cdot \text{cm})^{-1}$ obtained with the CVC agrees with literature for measurements taken at room temperature (8). The highlighted conductivity statistical error clearly shows a reduction of ~ 90%, when compared to the other two test runs. This order of magnitude increase in the precision of conductivity calculations is the culmination of all the work that has been done to the CVC over the last five years, including modifications to the electronics, hardware, and the addition of the CVC data analysis program. The value of $5 \cdot 10^{-21} (\Omega \cdot \text{cm})^{-1}$ obtained for the instrument error in the 2-5-2009 run is ~ 40% greater than the other test runs. This is due to the sensitivity setting of the electrometer set to 1 as opposed to 0 for the other runs. Since the test runs taken on 8-14-2007 and 3-26-2009 used the most sensitive setting for this setup, the conductivity instrument error of $3 \cdot 10^{-21} (\Omega \cdot \text{cm})^{-1}$ represents the lowest possible limit for conductivity measurements using the CVC system. Using Eq. [6] for the decay time:

$$\tau = \frac{\varepsilon_0 \varepsilon_r}{\sigma}$$

where $\varepsilon_r$ is the dielectric constant for this material, a value for the longest measurable decay time of $\geq 1.5$ years is obtained for conductivity values of $\approx 3 \cdot 10^{-21} (\Omega \cdot \text{cm})^{-1}$. The measured precision of $\approx 1.7 \cdot 10^{-20} (\Omega \cdot \text{cm})^{-1}$ corresponds to decay times of $\geq 0.5$ years. Implementation of an equally stable high voltage power supply would allow voltages of 2000 V. With this applied electric field, the longest measurable decay time increases by ~ 20x; therefore $\Delta I/I$ would decrease by ~ 20x. Assuming that $\Delta \sigma/\sigma$ is dominated by the $\Delta I/I$ term, the mean precision for time decay would decrease to $\approx 4 \cdot 10^{-22} (\Omega \cdot \text{cm})^{-1}$ corresponding to decay times of $\geq 10$ years.

**Future work**

Continued development of the CVC chamber would involve improving the stability of the power supplies, continuing to test materials within the temperature limits of the instrument, and continued development of the CVC analysis program expanding its capabilities. A major contributor to the precision of the CVC can be attributed to the use a 100 V battery power source. Unfortunately, a battery power source is not practical for achieving voltages in excess of 2000 V, high enough for long time duration discharge calculations. The use of the low (10-1000 V) and high (1000-10,000 V) voltage power supplies will need to be employed. To achieve a stable voltage, testing should be done using a portable battery backup system to supply 120 V 60 Hz to the power supplies. This may help stabilize the output voltages although limitations on
run times may be introduced. Some testing has been done, with no apparent success, to drive the input signal for these power supplies using a secondary battery source instead of the PC DAQ card; this needs to be investigated further. In addition, an RC filter has been built to reduce short-term fluctuation in the supply voltage. Initial tests seem to show improvements to the CVC input voltage, although additional testing and fine tuning of this filter should be done. A schematic for the filter has been started in the latest CVC schematic diagram.

Continued temperature dependant testing should be carried out, since a procedure for cooling and heating the chamber during the same run has been developed. Liquid nitrogen cooling should yield good results now that many of the main electrical vacuum feedthrough gaskets have been replaced. Once the chamber has reached room temperature, the heating elements may be used to raise the temperature up to 80 C. (Beyond this the integrity of parts in the chamber may become compromised.)

Improvements to the CVC analysis program will be an ongoing process. Plot adjustments and fine tuning may be done as new information about the system is needed. The program is commented (in red) very well so generating an understanding of how it works is possible. IGOR has many curve fitting options making it a useful tool for further analysis and model testing. Preliminary curve fitting has been applied to test runs taken on 3-26-2009 and 2-6-2009 using the time dependant conductivity model (9) shown in Eq. [7]:

$$\sigma_{model}(t) = \sigma_{pol} e^{\left(\frac{t}{\tau_{pol}}\right)} + \sigma_{SC} \cdot t^{(-\alpha)} + \sigma_{DC} \quad [7]$$

where the $\sigma_{pol} e^{\left(\frac{t}{\tau_{pol}}\right)}$ term represents the polarization effects, the $\sigma_{SC} \cdot t^{(-\alpha)}$ term represents the diffusive behavior related to space charge, and the $\sigma_{DC}$ term is the constant long timescale equilibrium dark current. Note the polarization term is attempting to fit data points not seen in this scale (an artifact of the binning algorithm used for
smoothing, which has a minimum of 6 data points for any bin), hence the deviation for the initial part of the curves.

A comparison of the fitting parameters shows very good consistency with the fitting parameters with one major exception, the polarization time term, $\tau_{pol}$. For the run taken on 3-26-2009 shown in Fig. 4, a small value for $\tau_{pol}$ ($\sim 20$ s) yields a longer exponential decay time. The run taken on 2-6-2009 shown in Fig. 5 has a $\tau_{pol}$ ($\sim 50$ s) that implies a much faster polarization response in the system. The details of this analysis will need to be investigated further.

Conclusion

The CVC has undergone modifications which improve the precision of conductivity measurements by nearly an order of magnitude. Uncertainties in measured values of current and conductivity are consistent with detailed error analysis of the system, reflecting the increased precision due to those modifications. The measured values taken with the system agree well with literature for conductivity calculations of LDPE. Conductivity values obtained with the CVC show good promise for reliable knowledge of decay times for LDPE which is used extensively in the construction of modern spacecraft. The improvements made to the chamber will prove beneficial to future measurements taken with the system although more can be done to reach the instruments theoretical limit.

References


Appendix A - CVC Schematic
**Electrode spring calculation.** Used to determine the amount of force needed to add to the weight of the electrode assembly to comply with ASTM D 257-99 standards for pressure applied to sample.

- **Mass of electrode assembly:** \( M_{\text{ass}} = 1350 \text{ gm} \)
- **ASTM D 257-99 recommended pressure applied to sample:** \( P_{\text{min}} = 140 \text{ kPa} \)
- **Area of each electrode:** \( A_{\text{el}} = \pi \left( \frac{0.6\text{-in}}{2} \right)^2 = 1.824 \text{ cm}^2 \)
- **Pressure recommended variables:** \( P_{\text{min}} = \frac{(M_{\text{ass}} + M_{\text{add.min}})g}{4 \cdot A_{\text{el}}} \)

Solving for \( M_{\text{add.min}} \) gives the equivalent mass needed to add to the electrode plate assembly:

\[
M_{\text{add.min}} = \frac{M_{\text{ass}} \cdot g - 4 \cdot A_{\text{el}} \cdot P_{\text{min}}}{g} = 9.067 \text{ kg}
\]

And in lbs:

\[
M_{\text{add.min}} = 19.988 \text{ lb}
\]

The springs used in the CVC excerpt:

- **Spring rate:** \( \text{Spring}_r = 260 \text{ lbf/in} = 4.553 \times 10^4 \text{ N/m} \)
- **\( \Delta X \):** 1.58 mm
- **\( F_{\text{spring}} \):** \( \text{Spring}_r \cdot \Delta X = 71.942 \text{ N} \)

The actual equivalent mass for each spring is:

\[
\text{P}_{\text{CVC}} = \frac{(M_{\text{ass}} \cdot g + 4F_{\text{spring}})}{4 \cdot A_{\text{el}}} = 412.532 \text{ kPa}
\]

And in atmospheres:

\[
\text{P}_{\text{CVC}} = 4.071 \text{ atm}
\]