Project Description

The initial focus of the project was to outline several options for High-k nanoparticle Based Dielectric materials for super capacitor applications. However, after taking some initial measurements it was clear that the apparatus itself had to be redesigned, which then became the primary aim.

This report briefly summarizes the effort to create a system that can be used to measure the dielectric constant of materials in conjunction with an LCR machine.

Initial Experimentation

Material in use:
Gold nanoparticles deposited on filter paper

Current method:
LCR machine is used to measure capacitance of the material in a sample holder. Distance measurements are then used to calculate the dielectric constant from the equation

\[ C = \varepsilon_r \varepsilon_0 A/d \]

Results:
The LCR was used to measure the capacitance of the filter paper the settings used are mentioned in the table below.

Values of Ag nanoparticles coated filter paper

<table>
<thead>
<tr>
<th>Capacitance (F)</th>
<th>Frequency (kHz)</th>
<th>( \varepsilon_r )</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.63E-09</td>
<td>10</td>
<td>735.79</td>
</tr>
<tr>
<td>5.41E-09</td>
<td>50</td>
<td>413.34</td>
</tr>
<tr>
<td>4.14E-09</td>
<td>100</td>
<td>316.10</td>
</tr>
<tr>
<td>3.09E-09</td>
<td>200</td>
<td>235.99</td>
</tr>
<tr>
<td>2.02E-09</td>
<td>500</td>
<td>154.50</td>
</tr>
<tr>
<td>1.30E-09</td>
<td>1000</td>
<td>99.46</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Constants</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \varepsilon_0 )</td>
<td>8.85E-12</td>
</tr>
<tr>
<td>( E_0 ) (F/m)</td>
<td>8.85E-12</td>
</tr>
<tr>
<td>( V ) (v)</td>
<td>1</td>
</tr>
<tr>
<td>( A ) (m^2)</td>
<td>3.10E-04</td>
</tr>
<tr>
<td>( d ) (m)</td>
<td>2.10E-04</td>
</tr>
</tbody>
</table>
Values for regular paper

<table>
<thead>
<tr>
<th>Capacitance (F)</th>
<th>Frequency (kHz)</th>
<th>Er</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.91E-09</td>
<td>10</td>
<td>298.90</td>
</tr>
<tr>
<td>3.01E-09</td>
<td>50</td>
<td>229.95</td>
</tr>
<tr>
<td>2.46E-09</td>
<td>100</td>
<td>187.98</td>
</tr>
<tr>
<td>1.84E-09</td>
<td>200</td>
<td>140.35</td>
</tr>
<tr>
<td>9.37E-10</td>
<td>500</td>
<td>71.62</td>
</tr>
<tr>
<td>4.40E-10</td>
<td>1000</td>
<td>33.64</td>
</tr>
</tbody>
</table>

Current design of the dielectric cell

Main disadvantages of the design:

- Poor contact between electrodes
- Cannot measure capacitor thickness
- Movement of sample during experiment
Redesign:

Comments:

Disadvantages:
- Too complex
- Both plates need not move
- Electrical connections are a problem
- Thickness measurement not included

Advantages:
- Guard electrode to stop fringe fields
- Tight sample hold, good contact
- Stable
- Easy measurement of thickness
Second Design

Note: A new part was added for use as a liquid test cell

Initial CAD designs for the system
Comments

- Add facility for temperature variation (heating)
- Remove excess rods
- Add a fixed support for the micrometer
- Add facility for BNC connectors
- Fix the upper guard electrode to the upper electrode
- Electrical connections need to be finalized
- Model needs to be made accurately with measurements
- Define material requirements
- Liquid cell portion

Third Design

Comments

- Make sure the dimensions of the insulating cover allow to fit easily
- Include raw dimensions of the ceramic insulator for the cover
- Edit the guard electrodes so they accommodate a range of samples
- Consider the Options for insulating the upper electrodes from the rest of the assembly
- Thermal coating for upper electrode/shaft contact
  - Glue
  - Ceramic
  - Thermal tape (wrapping it around)
- Make exact measurement based drawings for the individual parts matched to their
threaded standards
- Reduce the rods to the new welded design
- Set width to 1.4 cm for supporting rods
- Decide what thread standards are needed for the various parts

Final Design
**Apparatus Manufacture Stage**

This section includes an overview of the individual parts comprising the final system. Technical specifications and design notes are also mentioned, along with images of the original and final parts.

**Basic Overall Design**

![Diagram of the apparatus](image1)

**Insulation Cover**

- Material: Ceramic should be able to handle 400 degrees
- Split into exact half cylinders, the band clamp will be there to secure the setting during heating

![Diagram of insulation cover](image2)

Raw material used:
Cool dried clay, cut and drilled to size

Note: band clamp was removed as it was deemed unnecessary
Main Shaft Holder

- Material: Brass
- Thread Standard: M14 X (1-1.25) (Female to the Main shafts male)

![Image of Main Shaft Holder](image1.png)

Main Shaft

- Material: Brass
- Thread Standard: M14 X (1-1.25) (Female to the Main Shaft Holder)

![Image of Main Shaft](image2.png)

Note: A hanger was added to the main shaft holder to free the main shaft from the upper guard electrode, making it easier to lower the upper electrode accurately (see above image)
Ceramic ring for upper electrode plate
- Material: Ceramic

Note: The manufacture of this ring was dropped in favor for a circular ring of flexible mica

Upper Electrode plate
- Material: Brass
**Lower Electrode plate**

*Technical details:*

- Material: Brass
- Thread Standard: M2.2 X 0.45 (Can be adjusted to M3 X 0.5, Female to be screwed into the base, the horizontal bore as in image)
- The thinner portion of the cylinders diameter is 14mm

![Diagram and Image of Lower Electrode Plate]

Note: The design was slightly altered from the original to include:

- The lower portion of the electrode was designed to accommodate a certain amount of movement to allow complete fit with the upper electrode surface (nut bolt fixture)
- The electrode is isolated from the base using thermal tape and flexible mica washers
- There are two electrical connections, one for the heater (isolated) and one for measurement (taken from the base of the device)
Base

- Material: Stainless Steel
- Note the legs can be cylindrical with a leather/rubber pads

![Base Image]

Note: The main faceplate was included with the base design and was used in place of the two front legs

Main Shaft Support

- Material: Stainless steel
- Thread Standard: M8 X 1.25 (Male to be bolted into the base)
- The bend mustn't be bigger/smaller than that shown in diagram + exactly 90 degrees
- Inside arc length - 21.2mm
- Overall length - 105.1mm (unbent)
- Total height - 82.3 mm

![Main Shaft Support Image]

Note: Due to issues with scheduling the welding process, and to allow for a more flexible assembly
Micrometer Rod

- Material: Stainless steel
- Thread Standard: M6 X 1 (Male to be bolted into the base)
- Note: (Needs to be grooved at regular intervals for micrometer z motion)

Guard Electrodes

- Material: Stainless steel
LOWER PRIORITY PARTS:

Liquid sample Container

- Materials:
  - Green = Teflon
  - Silver = Stainless Steel/Brass
  - Dark Green = plug (insertion of liquid via syringe)

Liquid sample Guard Electrode

- Material: Stainless Steel
Front Panel

- Material: Stainless Steel

Note: Faceplate was incorporated into the overall base design
Apparatus Instructions

The following are suggested instructions for a simple temperature dependant measurement of dielectric constant of a solid material.

1. **Thickness measurement:** Make sure the main shaft is fully tightened and the electrodes are in full contact
2. Lower the micrometer so the measuring tip is flat with the shaft surface, and take a measurement
3. Fully retract the micrometer
4. **Sample Set up:** Remove the Upper electrode connector

5. Unscrew the main shaft for the Upper Electrode
6. Secure the upper guard electrode on the hinges below the main shaft holder

7. Place sample on the lower electrode
8. Place the Lower Guard electrode around the lower electrode
9. Screw the Upper electrode shut ensuring the sample is within the area of the electrodes
10. Make sure the lower guard electrode does not touch the lower electrode
11. Release the upper guard electrode from the hinges and place it on the grooves of the lower guard electrode
12. Reconnect the upper electrode contact
13. Connect the BNC cables from the LCRs output (IH, PH, IL and PL contacts of the LCR machine respectively) to the Upper and Lower Electrode connection points on the faceplate of the apparatus

14. Program the LCR for desired measurement and execute
15. **Heating the Sample:** Place the ceramic insulation chamber around the electrode assembly
16. Connect the thermocouple meter and the heater connection to the dedicated variac
17. Slowly increment the voltage until the desired temperature is reached on the thermocouple meter
18. Repeat step 14 to take readings

# The area of both electrode surfaces is 490.87 mm² (from r = 12.5mm)
Testing for Electrical Measurement (No Heating)

BNC cables were designed to be compatible with the Quadtech 1920 Precision LCR meter. The cable designed was based on a circuit diagram taken from the component measurement accessory of the LCR machine (see image below).

![BNC Cable Connections Diagram]

Key:
- IL: Current Low
- PL: Potential Low
- PH: Potential High
- IH: Current High
- G: Ground

Confirmation of the electrical continuity of the electrodes and isolation of the base was confirmed with a multimeter. The LCR was used to take several measurements successfully.

Heater Testing

The apparatus was connected to a voltage variac via 3.5mm audio jack for a simple and safe connection. The voltage was stepped up slowly from 0 to 50 volts while the temperature was monitored by the onboard thermocouple (black lead as seen below).
Future Additions to Apparatus

As mentioned in the design evolution section of the report there are several additions to be made to the device for different applications. This section will outline the design for those accessories.

1. Measuring the dielectric constants for liquid materials:

These parts would be required in order to take measurements on liquid samples. The materials to be used are mentioned in the design evolution section of the report.

2. Measuring the dielectric constants for powders/composites

For powdered samples the situation is a little more complex. Premade pellets cannot be used as they would be crushed by the electrodes upon contact, and their size most probably will be too small.

The remaining option would be to create a small pellet maker (similar to the above liquid accessory) designed specifically for the apparatus where by the diameter would be the same as that of the electrodes. The entire pellet maker assembly would then be inserted between the electrodes to take a measurement (see the diagram below)
Finalized Apparatus Image Gallery