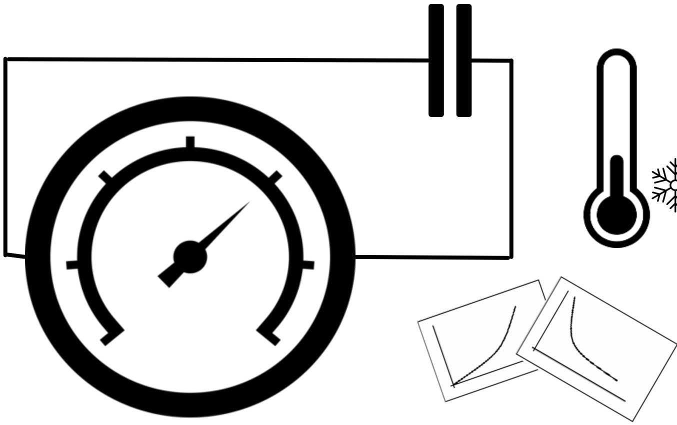
RAZORBILL INSTRUMENTS

APPLICATION NOTE



AP006: GETTING THE BEST OUT OF YOUR CAPACITIVE FORCE & DISPLACEMENT SENSORS

Razorbill instruments stress and strain cells use capacitors to provide a feedback signal of the force or displacement your sample is subject to. This app note covers the expected performance of the capacitors, and how to design your experiments get the best possible performance from them.

App note *AP003: Measuring Capacitance* covers suitable measuring devices and advice on connecting them to your cell, while app note *AP004: Cables and Heat load* contains advice on adding new cables to a cryostat where this is required.

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INTRODUCTION

Razorbill's feedback sensors rely on the variation in the capacitance of a parallel-plate capacitor as the plates are moved towards and away from each other. The design of the capacitor varies slightly, but they typically have an area of a few square mm and an initial spacing of some tens of microns, leading to a nominal capacitance of a few picofarads. To confirm the characteristics of the capacitor in your device, please refer to the datasheet and calibration curve for your specific cell.

As our smallest cells have working ranges of only a few microns, the change in capacitance during an experiment can be quite small. This presents a risk that the desired signal could be of a similar size to other spurious signals. This App note discuses common sources of spurious variation in capacitance, and how to design your experiments to avoid them.

A large signal is always easier to interpret, so we recommend that the experiment is designed, and the sample selected, so that at least half of the cell's design range is used. Where that is not possible, extra care needs to be taken to ensure the data captured is of the best possible quality, as described in this document.

Converting capacitance to force or displacement

Razorbill instruments usually provides a temperature calibration curve in addition to the main force or displacement calibration. For the reasons explained under Calibration below, we recommend applying the temperature calibration as an offset, before calculating the force or displacement.

CALIBRATION

The first aspect to consider is the size of the signal you expect to see, and how that relates to the displacement or force present at the sample. This relationship can be approximated by the parallel plate capacitor equation:

$$C = \frac{\varepsilon_0 \mathrm{A}}{d}$$

Where C is the capacitance, ε_0 is the permittivity of free space (8.854 pF/m), A is the plate area and d the distance between them. The capacitor is not perfectly formed however, so individual calibration is always necessary.

Calibration curves from Razorbill Instruments

All cells are supplied with a calibration curve for their primary response (i.e. displacement or force) and some are also supplied with a temperature calibration. All calibration data is archived at Razorbill Instruments, so if the factory calibration has been lost please contact razorbill instruments with the model type and serial number of your cell and we will be happy to supply a copy. Calibration curves supplied by Razorbill are usually in the form of the equation given above, but occasionally may be a polynomial fit or other equation where this is more accurate.

Methods for on-site calibration

The method of calibration depends on the type of cell. Force and distance calibrations are provided by Razorbill Instruments, and cells can be returned to the factory if updated calibrations are required. If you wish to perform your own calibration for force or displacement, please get in touch with Razorbill Instruments for more information about the parts and equipment required.

Calibrating a cell for temperature dependence requires no extra parts and is strongly recommended, even for cells that come with a temperature calibration from the factory. On-site calibration allows variation in measurement equipment, cables, or thermometry to be taken into account.

Force-measuring cells can be calibrated simply by cooling and rewarming the cell with no sample attached. Displacement sensing cells should be cooled and rewarmed with the supplied titanium calibration sample attached to prevent any movement of the displacement sensor. If your cell is fitted with both displacement and force sensors, two calibration experiments will be needed to calibrate each sensor separately.

Before taking a measurement, the cell should be held at a fixed temperature for a period of time in order to allow all the components in the cell to reach the desired temperature. It is recommended that measurements are taken both on cooling and warming, if hysteresis is observed the hold period was too short. Two hours is suggested as an initial hold period, but this may depend significantly on the design of your cryostat.

EFFECTS OF TEMPERATURE

Temperature has two principal effects on the capacitance. Firstly, it causes changes in area of the plates, and secondly it causes changes in their position (or alignment).

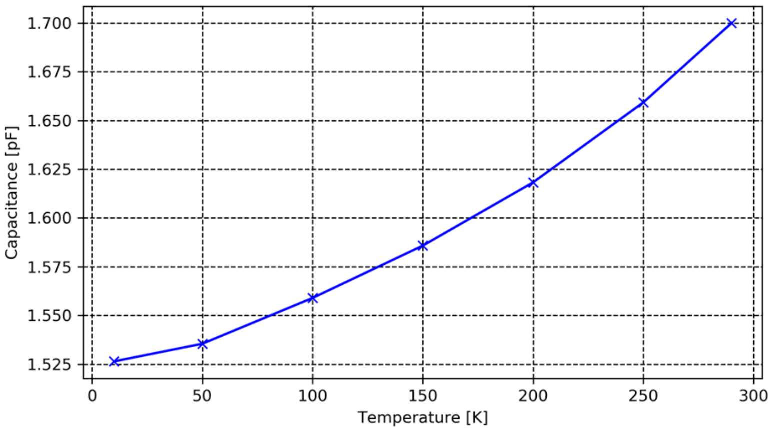

Figure 1: An example temperature calibration curve. Total variation is c. 10 %

As the cell cools, the capacitor plate area will shrink slightly. This effect is easy to quantify, as the thermal expansion coefficient of Titanium is known. Contraction from 293 K to 4 K is 0.151 %[1], so the change in area will be 0.302 %. This change will result in a change in gain in the capacitor.

[1] *Experimental Techniques for Low Temperature Measurements* by Jack W. Ekin, Oxford Univ. Press 2011

The movement of the plates is harder to quantify, being dependent on the deformation of both titanium and insulating parts. Movement of the plates, if in the direction of operation, results in an offset. Rotation or distortion of the plates away from parallel would change the shape of the curve, as would translation across the direction of operation. These effects are thought to be small, but we have not yet been able to fully calibrate the capacitors against an absolute standard at low temperature.

When we consider a typical temperature calibration, we find that the change in capacitance between room temperature and 4 K is typically around 3-10 %. This is much greater than the 0.3 % attributable to change in plate area, so must derive mostly from their movement. Movement is believed to result predominantly in offset rather than gain, so we recommend treating the temperature calibration data as an offset.

EFFECTS OF RATE OF CHANGE OF TEMPERATURE

Rapid changes in temperature lead to erroneous changes in capacitance. This occurs because different parts of the capacitor are at different temperatures. Let us imagine the at a cell is at 300 K and is cooled. Firstly, the titanium body of the cell cools, causing it to contract, and increasing capacitance. Next, insulating layers behind the capacitor plates cool, opening the gap, and decreasing capacitance. Finally, the capacitor plates cool and contract in area and thickness causing further decrease in capacitance. The actual process is probably more complex, but the concept of uneven heating or cooling perturbing the capacitance remains valid. The extent of this effect depends on heat flow within the cryostat which will vary significantly according to the presence or absence of exchange gas, the radiation environment, and the way heat is conducted into the cell.

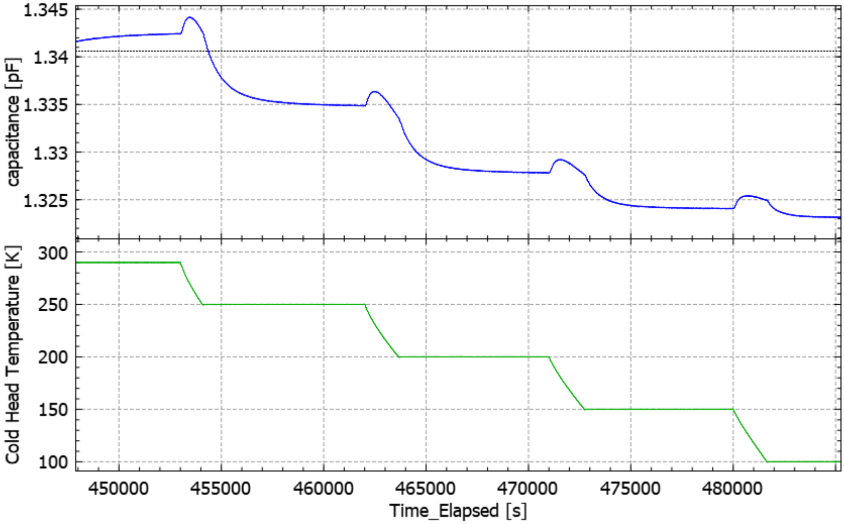

Figure 2: A data sample from our QA process showing capacitance transients caused by rapid temperature changes (2 hour hold periods with 5 K/min ramps). This cell design (CS200T) has a lower temperature dependence than some others, making the transients more visible.

The effects of rate of change of temperature on capacitance cannot easily be predicted and compensated for. It is therefore recommended that important measurements are only taken when the temperature is constant, and the cell has had time to reach thermal equilibrium.

Where it is necessary to take measurements during temperature changes (for example, when using PID control to keep the specimen at zero force during cooling) it is important to change temperature slowly enough that the measurement remains accurate. In the this example, if the rate effect exceeds the differential expansion, the control loop will do more harm than good.

Without intimate knowledge of each and every customer cryostat, we cannot easily recommend a maximum rate of change of temperature. We recommend cooling and rewarming the cell at your desired rate while monitoring hysteresis in the capacitance. If the hysteresis is unacceptably large, the rate must be reduced.

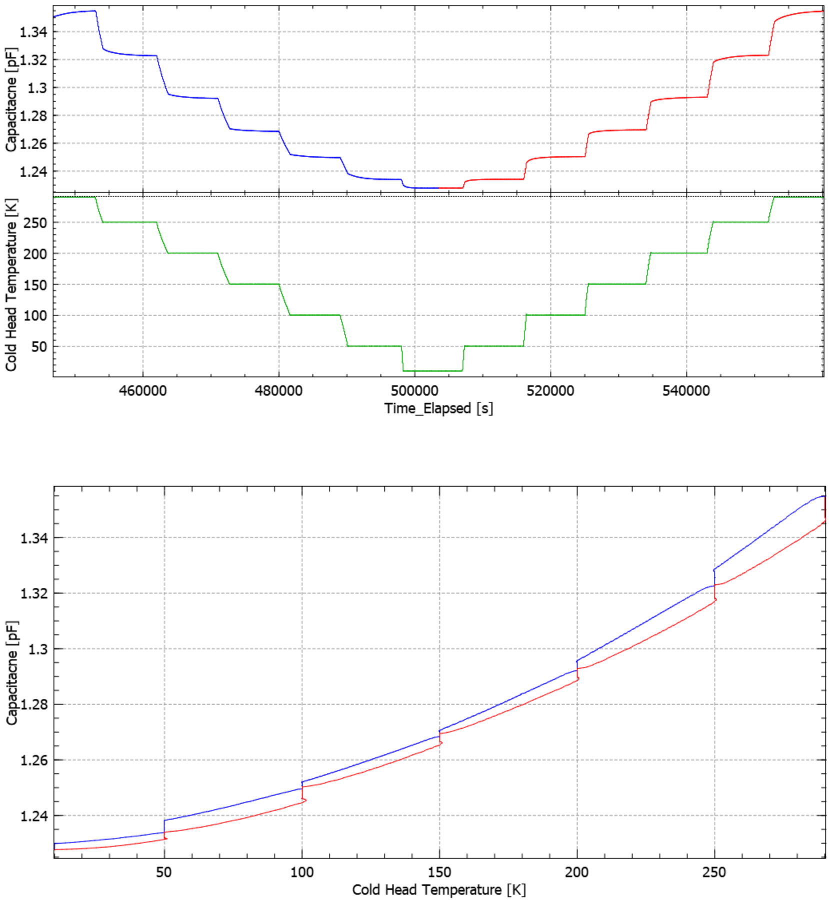

Figure 3: temperature-capacitance hysteresis for a cell in our QA cryostat. Hysteresis of c. 1 % during 5 K/min temperature ramps causes the blue (falling temperature) and red (rising temperature) curves to separate. Holding each temperature step for 2 hours causes rising and falling curves to meet. This data is used to generate the temperature calibration curves such as the one in Figure 1.

EFFECTS OF EXPOSURE TO ATMOSPHERE

The capacitors show some dependence on atmospheric conditions, believed to be related to the adsorption of moisture from the air. When transferred into the cryostat, the capacitors become more stable. In addition, the permittivity of air is slightly higher than that of vacuum.

When the cell is exposed to vacuum, the capacitance will drop slightly. This drop may take some time to stabilise fully. We recommend that 24 hours is allowed between pumping out the cryostat and starting experiments. To minimise this effect, it is recommended that the cells are stored in a dry environment in their original cases when not in use. It may also be worth baking the cells at 60-90 °C for an hour before loading into the crysostat, if the sample allows this.

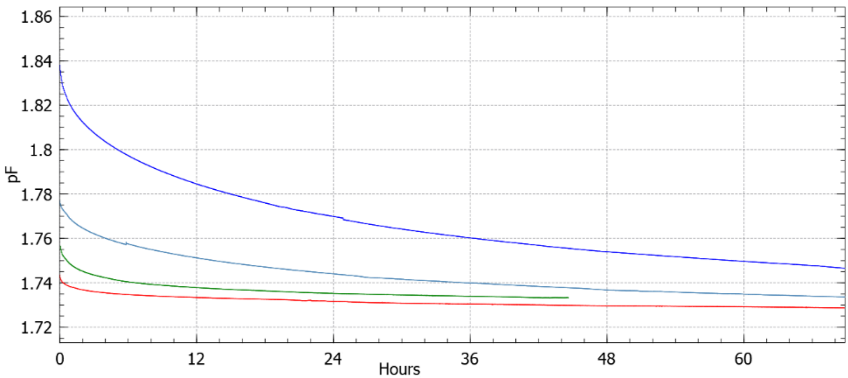

Figure 4: repeated cryostat loading and evacuation on the same cell. The capacitance initially varies according to atmospheric conditions, but after 24 - 48 hours in vacuum the variation is much reduced

If the cryostat vacuum is poor, or exchange gas is contaminated with air, water ice may form on the cell. Even very small quantities, if forming in the capacitor, will affect the measurement. If spikes in capacitance or other unusual behaviour are observed around 274 K, check the cryostat for leaks and confirm that good vacuum has been maintained. Similar issues can occur if other gasses, such as nitrogen, are allowed to condense in the cryostat, though the effect is smaller as their dielectric constants are much smaller than liquid water.

HYSTERESIS

The displacement sensors show no detectable hysteresis. It should be remembered however that piezoelectric stacks do show hysteresis, so if the cell is held at constant drive voltage some creep of the measured value will be observed. This corresponds to genuine change in sample strain.

The Force sensors show some slight hysteresis. This is worse when the cell is taken to it's maximum load than when used at lower force. Consult the individual product datasheet for more information.

EFFECT OF DISPLACEMENT ON A FORCE SENSOR

Due to the miniaturised nature of the cells, and their flexure-guided construction, the operation of the piezoelectric stacks can cause a small distortion in the force-sensing area of the cell. If a force-sensing cell is operated at its maximum stroke, a small change in capacitance my be observed, but this is limited to about 0.1 %[2] of the full scale range.

In a real experiment, the stroke of the cell is limited by the stiffness of the sample, and the desired signal is much larger than the displacement-related one, so this phenomena is unlikely to be a issue except in cases where an extremely soft sample is used.

EFFECT OF FORCE ON A DISPLACEMENT SENSOR

The displacement sensor is positioned as close as reasonably practical to the sample but does not directly measure the extension of the sample itself. Extension of the sample mounting plates and epoxy are included in the measurement, and will inevitably correlated with force. The force transmitted through the cell's body will cause some distortion, and in extreme cases of overloading the capacitor may be forced away from

[2] Older FC100 cells may be up to 1 %

parallel. Without knowing the position and geometry of the sample, it is difficult to estimate the impact of this effect on the measurement.

In addition, the sample strain is calculated by dividing the displacement by the sample's effective length, which may be difficult to know accurately for a sample mounted in epoxy.

For these reasons, the capacitor should not be relied upon to provide an accurate measure of sample strain. Where accurate knowledge of strain is required, it is recommended that the relationship between indicated displacement and sample strain is established by another method, such as digital image tracking.

If external strain measurements are not possible, the following steps will help minimise the difference between sample ΔL and the displacement reported by the cell.

- The sample should be as long (and thus soft) as practicable
- The sample cross section should be small (and thus soft) relative to the sample plates or mounting arrangements
- The force should be kept within the maximum rating given in the cell datasheet, ideally below one third of the rating.

OTHER SOURCES OF CAPACITANCE CHANGE

The FC100 shows some dependence on the tightness of the sample mounting screw. For more information refer to the FC100 datasheet. Other cells are not affected in this way.

ADVICE ON PLANNING EXPERIMENTS

- Select your sample geometry to use at least half of the cell's design range, if possible
- Keep the sample stiffness within the design envelope of the cell. For displacement-sensing cells, lower is better.
- Ensure a good vacuum is formed in your cryostat, or, if using exchange gas, that the gas is not contaminated with air
- Allow for a period of stabilisation after the cryostat is pumped out, 24 hours are recommended where practical

- Allow for a period of stabilisation after a temperature change, 2 hours are recommended
- Keep the rate of temperature change under 4 K/min, or 0.5 K/min if the cell is driven in a closed loop configuration

Finding the location of zero

Even with the careful application of calibration data, it can be difficult to know the capacitance that corresponds to zero sample strain.

On a displacement sensing cell, differential thermal expansion between the Titanium calibration specimen, actual specimen, and epoxy will disturb the zero point. Best practice is to compare the measurements taken on a strained sample with those taken on an unstrained specimen.

On a force sensing cell, it is sometimes helpful to deliberately break the specimen at the end of the experiment (by applying the maximum tension voltage). This allows a clear no-load condition to be recorded with the minimum possible opportunity for error.

TROUBLESHOOTING

The following symptoms occur from time to time, and can sometimes be easily solved:

Small steps in capacitance (less than 5 %)

Small steps that are not associated with real sample behaviour are usually caused by changes in grounding arrangements. As it is common to use low thermal conductivity cable in cryostats, the cable braid and capacitor guards are often separated from ground by 3-10 Ω. If the coax connectors of the cell touch each other or the cryostat body, the AC potential of the braids and shields may change. In some configurations, capacitance between the braid and core of the cable leads to charge flow from the excitation conductor, via the weakly grounded braids, to the sense conductor.

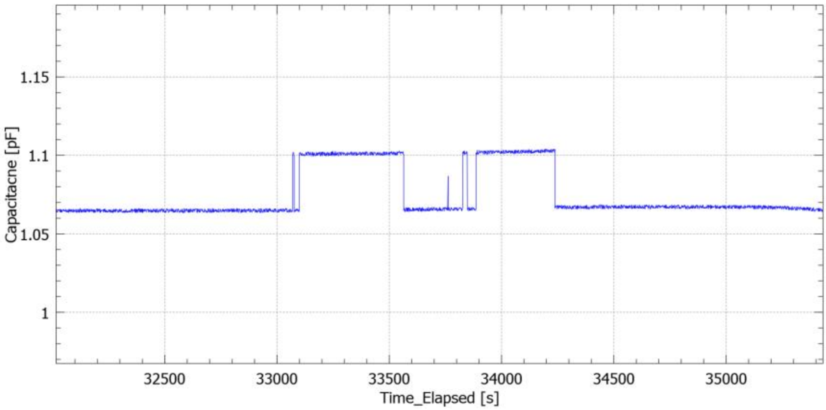

Figure 5: Steps in capacitance caused by intermittent changes in grounding. In this case the cable braid was going open circuit at the MMCX connector attached to the cell.

Potential solutions include insulating over the coax connectors with Kapton or Teflon tape, or, if the electrical noise levels are acceptable, wiring the connectors to the cryostat metalwork.

The above-mentioned effect is exacerbated if one of the braids loses its connection to ground. Cables should be carefully inspected where they join the connectors, and if frayed or appearing loose then the connector should be replaced. If a connector is suspected of poor mating, it is helpful to wrap it in uninsulated copper wire. If this resolves the issue, the connector and/or it's partner should be replaced.

Measured capacitance falls to zero

It is helpful to check the excitation voltage. If this has also collapsed close to zero a short circuit between the excitation and ground or sense has occurred. A multimeter may assist in finding the short circuit, but it's best to avoid directly probing the MMCX connectors as they are easily damaged. Check for stray conductive material in the connectors. A short circuit can be caused by foreign material entering the capacitor, and it is sometimes possible to clean it out, contact Razorbill Instruments for advice.

If the excitation voltage remains at its normal level, there is probably an open connection at some point. Inspect the connectors for damaged centre pins – damage occurs easily if a probe or other object has been inserted.

Capacitor doesn't respond to changes in piezo voltage

This can have several causes:

- On a force-sensing cell, the sample is broken or detached
- Moving parts of the cell have been accidentally glued together
- The piezos are damaged or unplugged
- The same voltage in the same polarity is applied to all the piezoelectric stacks – the cells work on differential voltage.

Hysteresis

Hysteresis with respect to temperature, if not originating from actual specimen behaviour, is best addressed by reducing the rate of change of temperature or increasing the hold time before a measurement is taken.

Hysteresis of displacement or force with respect to applied voltage is usually genuine, and originates in the piezoelectric stacks. Treat the displacement or force as accurate and adjust the applied voltage accordingly.

Hysteresis of sample properties with respect to force or displacement should be small. If it is larger than expected, it may be a property of the sample or the epoxy used to secure it.

Locating a fault

Before attempting a repair or returning a cell to Razorbill it can be helpful to confirm if the fault is in the wiring or in the cell itself. This can be done by substituting the cell for a dummy load. A 1 pF NP0 or C0G capacitor is a suitable load for cryogenic use. Additionally, the cell may be tested outside the cryostat and connected directly to your capacitance meter.

Helping us to help you

If you are struggling to get good measurements, the team at Razorbill Instruments can give advice. It helps us to have as much information as possible. In particular, please try and capture the problematic behaviour on a datalogger or computer. Alongside capacitance it is helpful to record the quadrature component of the measurement and the excitation voltage.

It is also helpful to measure the resistance between components with a multimeter – ideally one that can measure impedances in the MΩ and higher. With the cables disconnected from the capacitance meter measure resistance for all positions in the following grid;

	Cryostat ground	Excitation core	Excitation shield	Sense core
Sense shield				
Sense core				
Excitation shield				
Excitation core				

All these measurements should be open circuit. Please measure on a SMA or BNC connector, as pressing a multimeter probe against the middle pin of the small MMCX connector can permanently damage the connector.