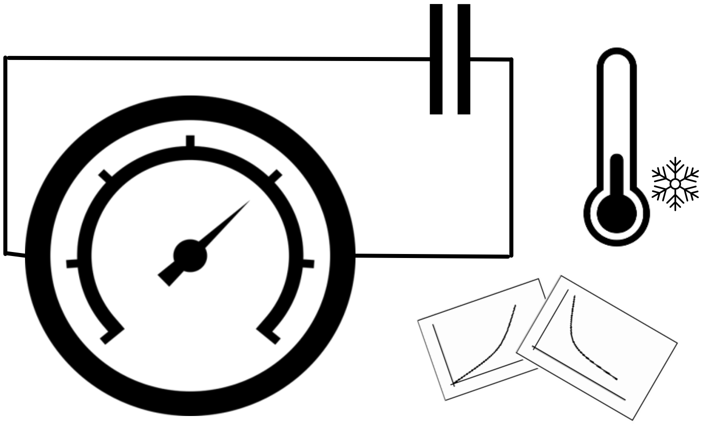
AP006: Sensor Performance



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. App note *AP007 Force or Displacement* covers the merits of the different types of sensors and how the value measured relates to the stress and strain in the sample.

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 around a picofarad. 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 real 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

The capacitance of a parallel plate capacitor is given by:

$$C = \frac{\varepsilon_0 \mathrm{A}}{d_{gap}}$$

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. We slightly modify this equation when drawing our calibration curves, so we use:

$$C = \frac{\alpha}{d + d_0} + C_p \qquad C = \frac{\alpha}{f + f_0} + C_p$$

For force and displacement sensors respectively. Here we have included a parallel capacitance term C_p to account for imperfections in the capacitor, and we have replaced d_{gap} with $d + d_0$, where d_0 is the gap at zero displacement and d is the change in gap that we want to measure. We have also combined ε_0 and A into a single coefficient α for convenience. The second equation is similar, except for force sensors the first term is multiplied by a factor for the stiffness of the spring which converts the force applied to the sensor into a displacement for the capacitor to measure - but this too has been combined into α for convenience. This does mean that for a displacement capacitor, the units of α are given in µm·pF whereas in the second equation it has units N·pF.

The curves and equations provided on the first page of the calibration sheets for each sensor are obtained by fitting the equations above to measured points. These measurements are made at room temperature and in air, but the values can change due to various environmental factors. The rest of this document includes a description of the main factors, and on page 14 there is our recommended method for correcting for them.

All cells are supplied with a calibration curve for their primary response (i.e. displacement or force) and a temperature calibration[1]. 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.

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[1] This applies to all cells sold in 2021 or later, before that only some models came with a temperature calibration.

Methods for on-site (re)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 quite straightforward. On-site calibration allows variation in measurement equipment, cables, or thermometry to be considered, and can be a good way to get familiar with the cell.

Displacement sensing cells should be cooled and rewarmed with the supplied titanium calibration sample attached to prevent any movement of the displacement sensor. The complete procedure is:

- With the cell at room temperature, apply +120V to both tension and compression stacks, then reduce to 0V over about 30 seconds. We call this “zeroing” the cell, and it has a couple of purposes. First, it is a clearly defined history, so it is something you can do before mounting a sample to get close to the same point. Secondly, by giving the stacks the same history as each other, we reduce the difference in thermal expansion coefficient and reduce the actual movement that the stiff sample needs to resist.
- Screw on the titanium stiff sample provided with the cell. There are two types: ones with rounded ends are for the cells with 9mm sample screw spacing (CS1x0) and ones with chamfered ends fit the 11mm cells (CS2x0, UC200).
- Cool the cell, stepping the temperature and holding for sufficient time at each step for the cell to equilibrate. Record the capacitance at each step.
- Rewarm, measuring the capacitance at each step the same way as when cooling. You should get similar values, if not, then you may need to wait longer at each temperature.

- Convert the measured capacitances to displacements, these form our new calibration and should be added or subtracted from the measured displacement when using the cell.

WARNING! Do not drive the cell to high voltages with the calibration sample mounted. The calibration sample is much stiffer than the samples the cell is designed to work with. Excessive forces will be generated, and the cell may be damaged.

Force-measuring cells can be temperature-calibrated simply by cooling and rewarming the cell with no sample attached, as that guarantees zero force is applied to the force sensor. The complete procedure is:

- Cool the cell, stepping the temperature and holding for sufficient time at each step for the cell to equilibrate. Record the capacitance at each step.
- Rewarm, measuring the capacitance at each step the same way as when cooling. You should get identical values, if not, then you may need to wait longer at each temperature.
- Convert the measured capacitances to forces, these form our new calibration and should be added or subtracted from the measured displacement when using the cell.

If your cell is fitted with both displacement and force sensors, two calibration experiments will be needed to calibrate each sensor separately.

Factors affecting the calibration

There are several factors affecting the calibration of the cells, some of which can be corrected for by adjusting the coefficients in the equations on page 2, and others which cannot be easily corrected so must be avoided instead.

Temperature

Temperature has several effects on capacitance. It causes the plates to shrink, and also affects the gap between and alignment of the plates.

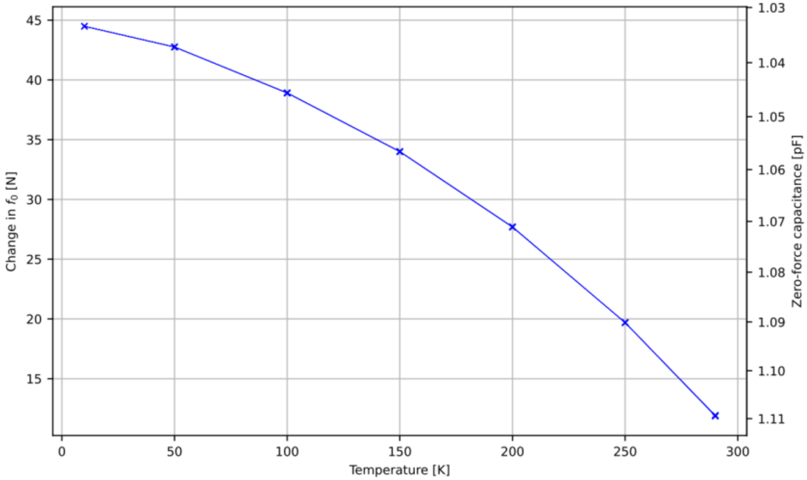

***Figure 1**: An example temperature calibration curve. Total capacitance variation is c. 10 %, which corresponds to a significant spurious force reading. Note that the left axis does not go all the way to 0 as there is no correction for humidity. The left axis is the apparent force measured at each temperature in vacuum, converted from capacitance using the calibration curve measured at room temperature in air.*

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 plate movement. Razorbill Instruments has conducted several experiments to calibrate both force and displacement sensors at low temperature, and we have observed that the calibration curves remain fairly accurate if the change with temperature is treated as an offset in the quantity being measured. This means converting the capacitance measured during temperature calibration to a force or displacement, and subtracting that from the force or displacement measured.

For force sensors, there is an additional contribution which affects the gain of the sensor as well. The sensor works by using a stiff titanium beam to convert the force to a displacement, then measures that displacement. As the titanium gets stiffer on cooling, this changes the gain of the sensor. Razorbill has measured this stiffening on a number of force sensing cells, and the change in α is:

$$\alpha(T) = \alpha_{290K} \times (0.91 + 5 \cdot 10^{-5}T + 9 \cdot 10^{-7}T^2)$$

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 more complex, involving complex thermal gradients as heat flows through the cell 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 and through the cell 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.

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.

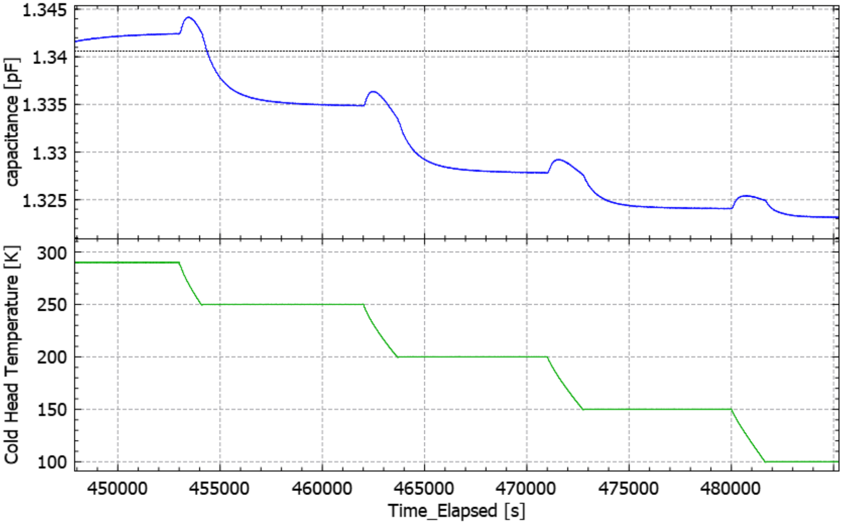

__Figure 2:__ A data sample from our QA process showing capacitance transients caused by rapid temperature changes (2 hour hold periods with 5 K/min ramps). This cell design (CS200T) has a lower temperature dependence than some others, making the transients more visible.

Where it is necessary to take measurements during temperature changes (for example, when controlling the drive voltage to keep the sample at zero force during cooling) it is important to change temperature slowly enough that the measurement remains accurate. Practically we find it is best to change the temperature in steps of e.g. 30K, and then wait for thermal equilibrium and adjust the voltage at each step. Otherwise, if the (spurious) rate effect exceeds the genuine change due to 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.

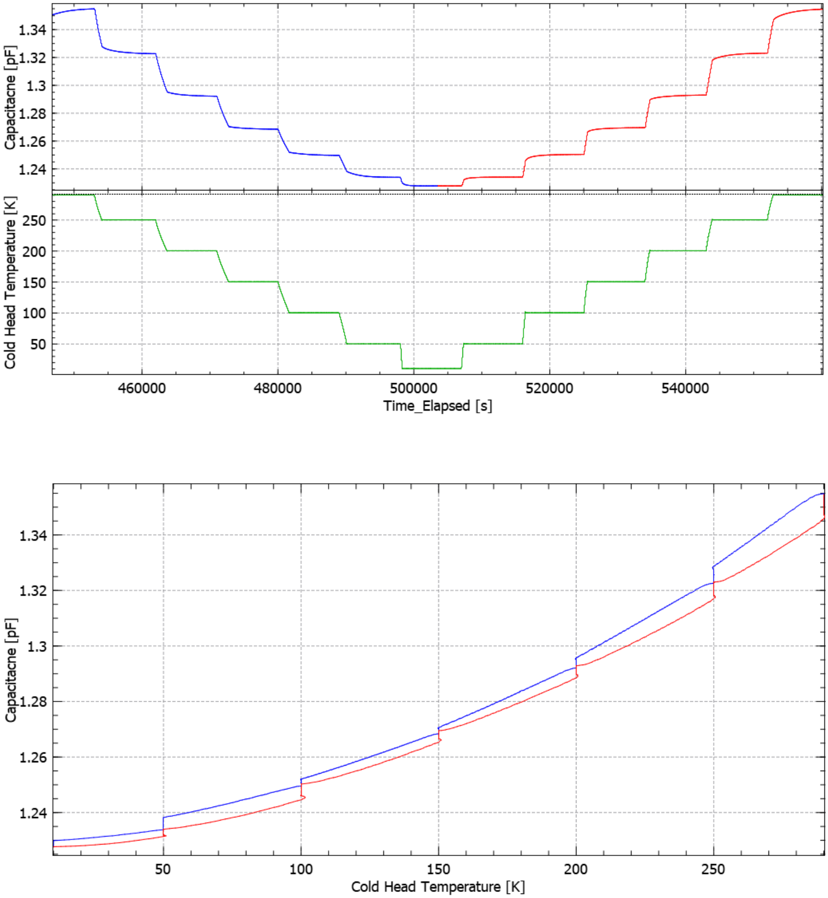

Figure 3: *temperature-capacitance hysteresis for a cell in our QA cryostat. Hysteresis of c. 1 % during 5 K/min temperature ramps causes the blue (falling temperature) and red (rising temperature) curves to separate. Holding each temperature step for 2 hours causes rising and falling curves to meet.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

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. There is also a small change as 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. Storing the cells in a vacuum chamber or dessicator can also help desorb moisture.

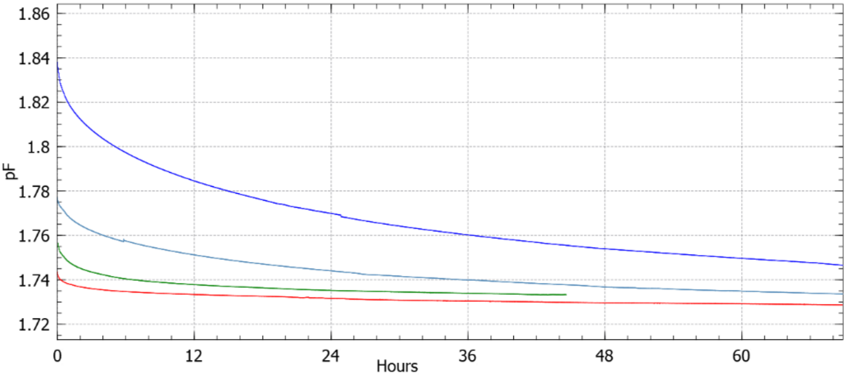

Figure 4: *repeated pump-downs on the same cell, starting from ambient each time. The capacitance initially varies according to atmospheric conditions, but after 24 - 48 hours in vacuum the variation is much reduced.*

Once the cell is cooled below about 250K, the slow reduction in capacitance due to water outgassing stops, and the sensor becomes more stable and repeatable. The remaining offset should be treated as an offset in force or displacement, not in capacitance.

If the cryostat vacuum is poor, or exchange gas is contaminated with air, water ice may form on the cell. Even very small quantities forming in the capacitor will greatly affect the measurement. If spikes in capacitance or other unusual behaviour are observed around 250-275 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, generally less than 0.5% of their full scale range[2]. 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

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[2] Applies to all cells except some early model FC100s, where it may be up to 3% FSR

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 %[3] of the full-scale range.

In a real experiment, the stiffness of the sample is such that the real signal is much larger, so this is rarely an 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 can be as large as the extension of the sample. The force transmitted through the cell's body will also cause some distortion, and in extreme cases of overloading the capacitor may be forced away from parallel. Without knowing the position and geometry of the sample, it is difficult to estimate the impact of this effect on the measurement. You can read more about the limitations of displacement sensing compared to force sensing in app note AP007.

The loss in accuracy due to these effects can be significant, so where possible it is advisable to calibrate the strain in the sample with an external measurement, such as the strain visible in a microscope image. Where 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

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

- 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 for CSxx cells.

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 experiments

To keep the effects mentioned above as small as possible, and to get the best achievable accuracy, we recommend you:

- 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, depending on cryostat design.
- 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.

Converting capacitance to displacement or force, correcting for temperature and other factors.

Taking all of the information in the above sections into account, the recommended procedure for converting the measured capacitance to force or displacement is:

1. Where an accurate zero point is required, consider baking out the cell (max 90°C) or storing it in a vacuum desiccator for 48 hours before loading into the cryostat.
2. Load the cell into the cryostat and pump for as long as practical, we suggest 24 hours.
3. Note the capacitance and convert it to force or displacement using the equation on the calibration curve. This will be your zero-point offset for this experiment.
4. When conducting your experiment, record the capacitance and convert it to force or displacement by:
    a. Using the formula on the calibration sheet. If it is a force sensor, use the temperature dependent α value described above.
    b. Look up the change in d_0 or f_0 between the temperature you are at and the temperature at which you completed step 3. If your calibration curve does not have d_0 or f_0 (curves produced before 2023),

convert from capacitance using the equation on the first page of the sheet.

c. Subtract the change from 4b and the value from 3 from the value calculated in 4a.