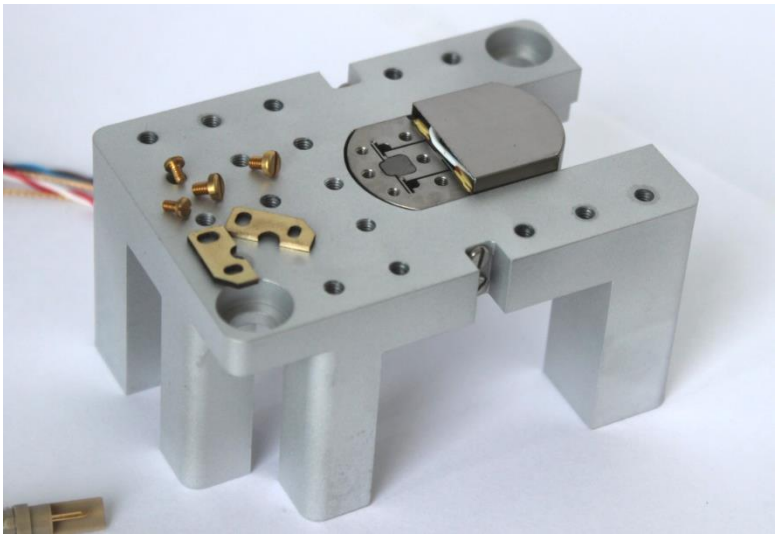
AP005: Mounting Samples



This document describes a practical procedure for mounting matchstick shaped samples of bulk materials for the application of stress and strain at cryogenic temperature. Mounting samples on SR series sample carriers is generally similar to the procedure below but refer to the carrier datasheet for more information.

Thin flakes and 2D materials need different mounting techniques. Contact Razorbill Instruments to discuss the options.

All materials are different, so you may need to experiment with variations on this scheme.

Mounting Procedure

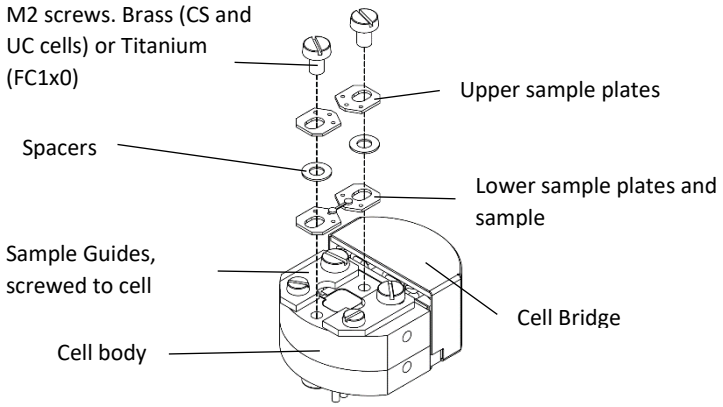


Figure 1: *Exploded diagram of CS100 sample mounting*

The overall procedure for mounting a sample on the CS1x0, CS2x0T and FC100 cells is similar and this guide covers all products (although the pictures used illustrate the process mainly show a CS100 cell).

The aim of the sample mounting process is usually to affix a sample (typically a matchstick shaped piece of ceramic, 1–2 mm long) so that it experiences homogenous uniaxial stresses and strains when the device is operated. The central section of the sample should be clear of epoxy and hence available for probing using *e.g.* Electrical contacts or a scanning probe tip. For most users, it should be electrically isolated from the cell. For others, it should be electrically connected to the cell – in which case the procedure is similar but a conductive epoxy can be used.

To achieve this, the sample must be held firmly as both ends by epoxy. The sample ends should be completely surrounded by epoxy at both ends (not in contact with the sample plates) so that the are electrically isolated, and so that the stress is applied evenly to the sample (not just to the bottom of the sample). The gap

between the sample plates should be as large as possible while being small enough for the sample to straddle it with enough overlap for each end of the sample to be held firmly by the epoxy. The sample plates and the sample should be aligned with the direction of strain.

The following guide briefly describes one possible procedure for achieving this and offers some hints to overcome common problems.

Securing the strain cell and affixing the sample plate guide

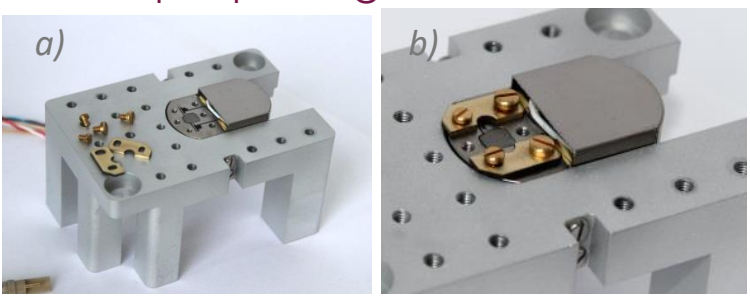


Figure 2: *a) To secure the CS100 strain cell in place, clamp the main body, not the bridge. b) With the sample plate guide installed.*

The cell will need to be held upright to work on. One way to do that is using the work stands sold by Razorbill Instruments, which also fit into the TB100 work table which is useful if you want to use a manipulator to help with mounting. FC1x0 cells come with a stand by default. UC200 and CS2x0 cells don't come with a stand, as they sit horizontal on a flat surface, but stands are available. The CS1x0, as the budget option, does not include a stand but you can buy one from Razorbill Instruments, or improvise one from e.g. a piece of aluminium angle with a hole drilled in it. Do not clamp any part of the cells other than the outer tube of the FC1x0,

and do not support the cells by the bridge as this may damage them.

CS1x0, CS2x0, UC200 and FC150 cells

CS1x0 cells manufactured before August 2019 use a single U-shaped sample plate guide, whereas later models, and all CS2x0T and UC200 cells ship with a two-part sample guide which allows the gap to be precisely adjusted. Either way, the procedure is similar. The sample plate guide is affixed to the top surface using one M2 and one M1.6 (CS1x0) or two M2 (CS2x0T & UC200) screws for each half. The guide is intended to simplify aligning the sample plates in the following sample mounting steps.

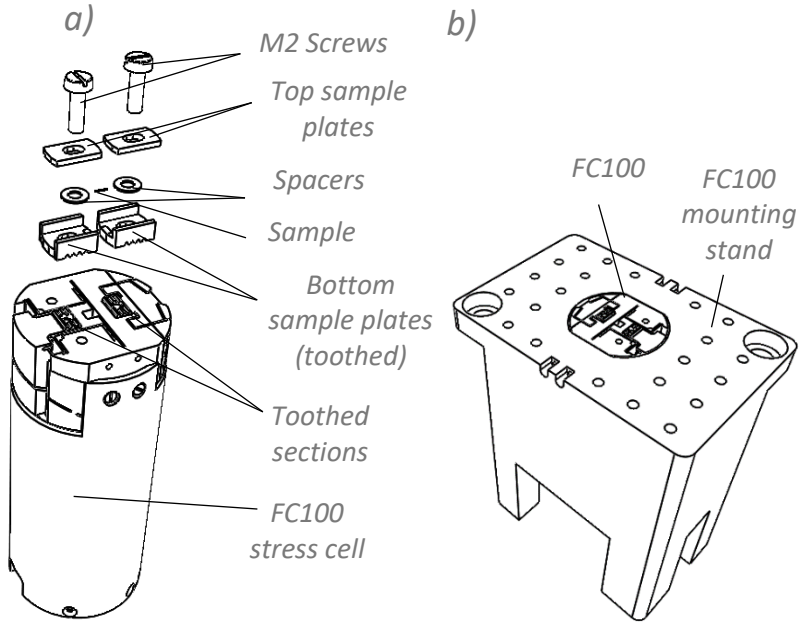


Figure 3: *a) The FC100 cell with its sample plates and spacers labelled. b) the FC100 is provided with mounting stand so that it can be held in position during sample mounting. This stand can be fitted into a TB100 mounting table for extra control during sample mounting.*

FC100

The FC100 has no need for the sample plate guide as the teeth on the bottom of the bottom sample plate mesh with the teeth on the top surface of the FC100 and keep the sample plates well aligned with the direction of applied strain as the bolts are done up (see section 4).

Adding bottom sample plates

CS1x0, CS2x0, UC200 and FC150

In this mounting procedure, each end of the sample is secured with lower and upper sample plates, separated by spacers. Place the lower plates as illustrated. The gap between them can be adjusted to suit the sample, ensuring there is enough overlap with each end of the sample to hold it securely.

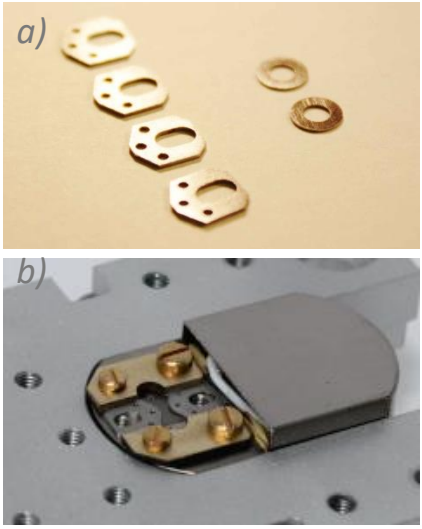


Figure 4:

a) The sample plates and spacers required for the mounting procedure (CS100 plates pictured).

b) The bottom sample plates laid in the correct

FC100

Because of the way the teeth of the FC100 sample plates mesh with the teeth on the top surface of the cell, the spacing between the plates is discrete rather than continuous as in other cells. The

gap may be adjusted by 1 mm by moving the plates one tooth together or apart and by 333 um by turning the plate end-to-end. By rotating both plates and moving them over the teeth, the gap may be adjusted in 333 um increments from 200 um to 2.8 mm. At this stage you will need adjust the gap spacing so that it is suitable for your sample, the sample should be able to straddle the gap resting on each side, with length at least twice its width available for gluing but as much as possible of the sample should be exposed between the plates.

The rest of the mounting procedure may be easier if the lower sample plates are lightly secured with, e.g., GE Varnish.

Adding spacers and attaching the sample

One common problem encountered by our customers is that the sample makes electrical contact with the sample plate through the epoxy, disrupting electrical measurements being performed during the experiment. To prevent this, epoxy thickness spacers (such as silica beads, cotton fibres, cigarette paper or tissue paper) can be added in the epoxy above and below the sample to help set the glue thickness. These should be used in addition to, not in place of, the sample plate spacer. Alternatively, a thin layer of the epoxy can be painted onto the tip of the sample plates where the sample will be laid. This thin epoxy layer can be fully or partially cured before the sample (and additional epoxy) is added. This bottom cured layer ensures the sample does not sink through the epoxy to make contact with the bottom sample plate.

For attaching the sample, we recommend using a two-part epoxy such as Stycast 2850. Firstly, decide on a suitable approximate epoxy thickness: if the epoxy is thicker, the maximum shear stress within the epoxy is lower, and the sample and epoxy are less likely to fail at high applied strains. If the epoxy is thinner, strain is applied to the sample more efficiently, and higher strains are

possible in principle. An epoxy thickness of 30–50 µm is a good place to start.

Sand the spacers down to match the thickness of the sample, plus desired epoxy thickness. E.g. if the sample is 100 µm thick and ~30 µm-thick epoxy layers are desired, sand the spacers down to 160 µm thick. If using epoxy thickness spacers be careful that the sample will not be crushed when the upper sample plate is installed. E.g. if the sample is 100 µm thick and 25 µm epoxy spacers are used, the sample plate spacers might be sanded to 170 µm in thickness, leaving 20 µm margin

Sand the spacers in a way that their upper and lower surfaces remain parallel.

In Fig. 5(a), epoxy spots (white) and epoxy thickness spacers have been laid in the uncured epoxy. The thickness spacers here are 30 µm diameter nylon fibre (too narrow to be visible in the photographs), intended to set the epoxy thickness to ≥ 30 µm.

In Fig. 5(b), the sample has been placed on the epoxy. If desired, the ends of the sample may protrude over the central holes in the lower sample plate: by doing so the ends of the sample will be accessible for contacts for electrical transport measurements. This can be necessary for layered materials with weakly coupled layers, where contacts need to be in contact with the full height of the sample.

In Fig. 5(c), epoxy has been placed on the upper surface of the sample. Further fibres as epoxy thickness spacers have again been added.

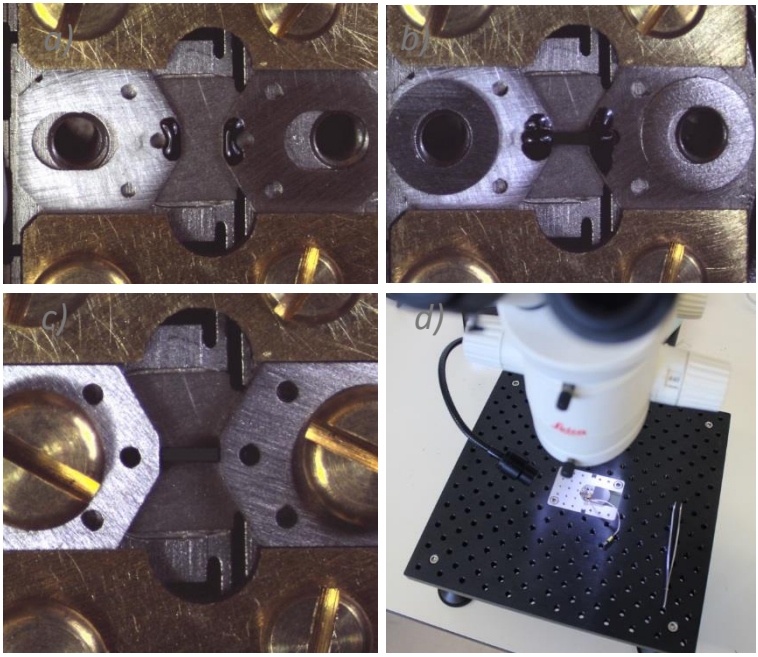


Figure 5: *a) The spacer disks are laid on top of the of bottom sample plates above the tapped holes. Close to the tips of the triangular end of the sample plates, a small daub of epoxy is deposited with a single fibre brush. Onto these droplets are added 30 µm diameter fibres. b) Additional epoxy is added, and the sample is laid so it straddles the two plates. c) Additional epoxy is added and an additional pieces of spacer fibre are added on top of the sample. d) a stereomicroscope and good quality tweezers will make this task much easier.*

Adding the top plate and bolts and removing the sample plate guide

Place the upper sample plates on top of the sample and epoxy. Insert and tighten the M2 sample plate screws and then cure the epoxy.

When curing the epoxy, observe the maximum permitted temperature on the cell datasheet, excessive temperature will damage the piezoelectric stacks or the electrical insulation. If using a high temperature cure connect all drive wires together, (preferably through resistors) to prevent large voltages building up on the piezoelectric stacks. Cells shipped in 2023 or later come with shorting caps for this purpose.

It is also possible to “pre-compress” or “pre-tension” the sample at this stage. Apply a voltage to the stacks to pull the sample mount points together or apart as the epoxy is curing; when the voltage is released the sample will be tensioned or compressed as desired. If you plan to apply more than 10-20V at this stage, make sure the cell is connected to a suitable safety ground and there is no risk of e.g. slipping and pushing a sharp pair of tweezers through the insulation.

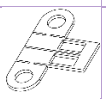
Mounting plates and spacers

All the below sample plates and spacers are available from Razorbill Instruments. Asterisked entries are provided as standard with the purchase of the relevant stress/strain cells. The others are available on request. This list is updated from time to time, download the most recent version of this note to see new options.

If you would like an option which is not listed for your particular cell, or if you have an idea for an alternative sample mounting technique please get in touch. Razorbill Instruments will be happy to advise you and/or make suitable sample mounting hardware for you.

All the following items are manufactured from titanium.

Please note: compatibility with CS1x0 denotes compatibility with CS100, CS110, CS120 and CS130 strain cells. Similarly, CS2x0T covers CS200T and CS220T. Most CS2x0T accessories are also compatible with the UC200.

Name	Part Number	Diagram	Fits Cell	Description
Standard CS1X0 sample plates*	CS100_smplplts		CS1X0	Top and bottom sample plates for CS1X0 cells
Bottom FC100 sample plates*	FC100_SmpPlt		FC100	Toothed, bottom sample plate for FC100 cells.
Top FC100 sample plate*	FC100_SmpCov		FC100	Top sample plate (cover) for the FC100.
Standard CS2x0T sample plates*	CS200_smplplts		CS2X0T	Top and bottom sample plates for CS2X0T cells
Standard spacer*	CS100_spcr		FC100 CS1X0 CS2X0T	Spacer that sets the distance between the top and bottom plates. Initial thickness 250 um.
Narrow spacer	CS100_spcrN		FC100 CS1X0 CS2X0T	Narrower version of above. Initial thickness 50 um.
Horizontal mounting spacer	CS100_spcrH		CS1X0	Can be used instead of a spacer to hold a sample horizontally for transmission mode probes
CS1X0 wide angle sample plates	CS100_widanplts		CS1X0	Bottom sample plates that raise the sample up so that it is the highest point on the top of the CS1X0 cell and hence allows 360° probe access to the sample
FC100 wide angle sample plates	FC100_widanplts		FC100	Same as above for the FC100
CS1X0 small / 2D sample plates	CS100_smplplts_2D		CS1X0	Sample plates held at a 200 um spacing via flexures for small samples and 2D materials

Items marked * are provided with the cell as standard.