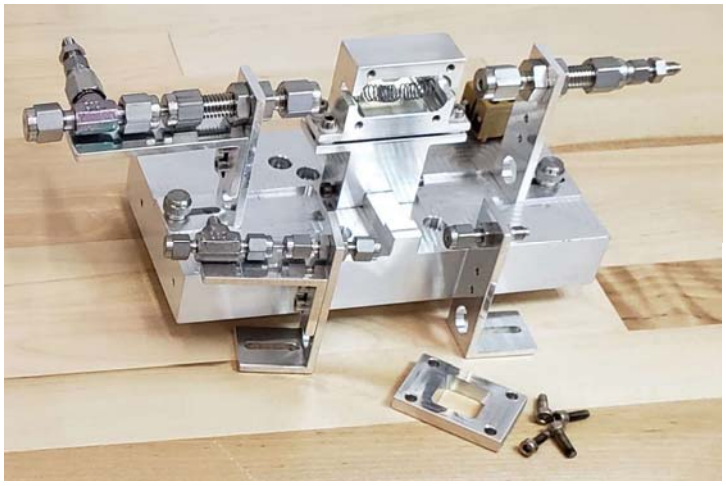
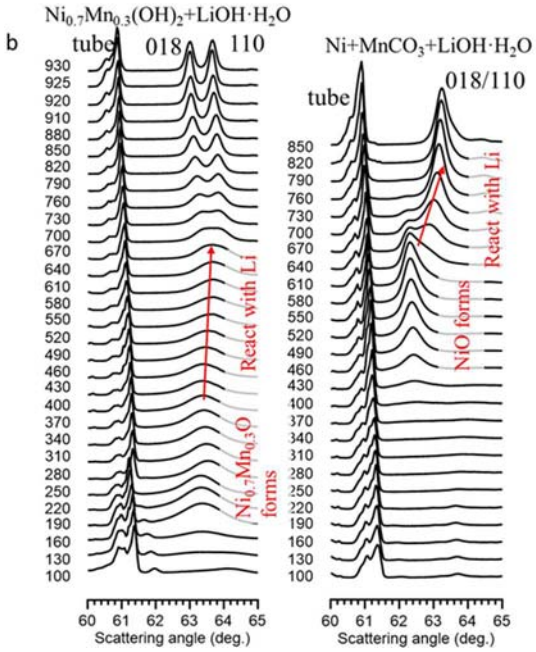
BXDS High Temperature Gas Flow Furnace (25 - 1200°C)

Standard Operating Procedures





N Zhang et al 2023 J. Electrochem. Soc. 2023, 170, 070515

Adam Leontowich, Karim Louca
December 19, 2024

Brockhouse Sector (BXDS) Contact List

Note: First dial "9" to access an *external* line from a CLS phone. For CLS internal numbers, you only need to dial the 4-digit extension number.

Serious emergency (Fire/Ambulance):	**911**
U of S Security:	9-306-966-5555

Beamline and CLS staff:	**Extension**	**Personal cell**
Beatriz Moreno (beamline responsible)	3868	306-241-1999
Adam Leontowich	3555	306-850-0408
Graham King	3760	639-998-1886
Narayana Appathurai	3648	306-514-1384
Karim Louca	3583	226-504-1169
Al Rahemtulla	3530	519-993-9137
Joel Reid	3854	
Garth Steel (controls lead)	3730	
Deborah Nguyen (mech. eng. lead)	3656	
Brian Schneider (elec. eng. lead)	3841	

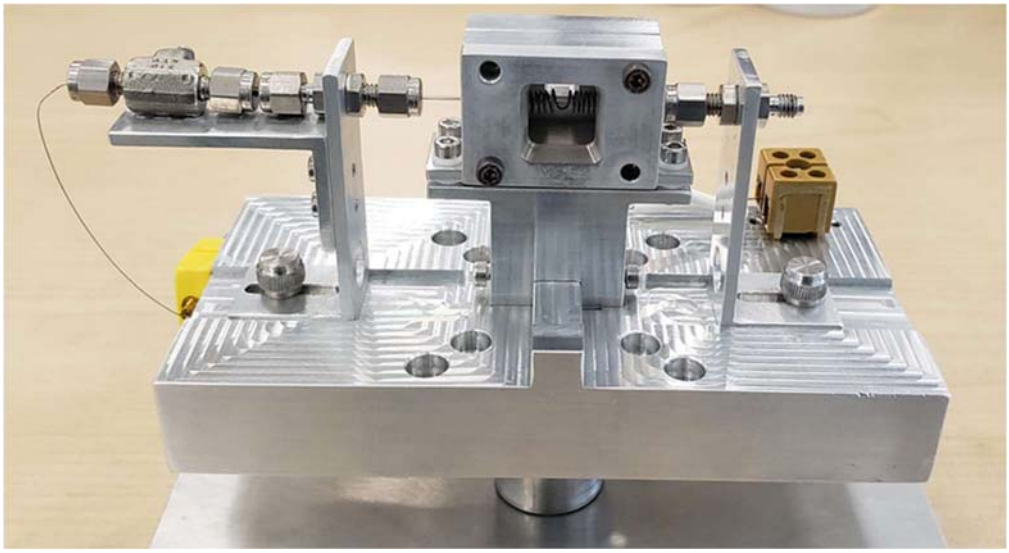
Call the beamlines:

Low energy beamline (SOE-1)	657-3821
High energy beamline (SOE-2)	657-3830
Undulator beamline (SOE-3)	657-3832

Other important contacts:

Floor coordinator	657-3639
Control room	657-3570
User services office (USO)	657-3700
Health and safety (HSE)	657-3663
Front desk	657-3500

Prepare and Test the Furnace *For Your Specific Conditions*



The furnace is very flexible! We have different sets of fittings, ferrules, and thermocouples. It can handle capillaries as large as 3.0 mm OD. The specific conditions of the experiment should be bench tested before attempting to measure with beam.

Test fit ferrules, and drill them out to an appropriate size if needed.

Test fit the thermocouple to see that it is going to the right depth. Mark that perfect depth with marker.

Test and then set and test the gas flow of the system, using the leak valve near the endstation. Set the gas outlet into a cup of water, and adjust until the max flow rate is a few bubbles per second.

For most accurate ramp rates, the downstream cover and actual gas flow should both be on when testing

Note that extended periods above 900 °C may cause the thermocouple to fail.

Tips for Filling Capillaries

Capillaries available at the beamline are
Material: Quartz, OD: 1.0 mm, ID: 0.8mm, Length = 70mm
Material: Quartz, OD: 2.4 mm, ID: 2.0 mm, Length = 100mm

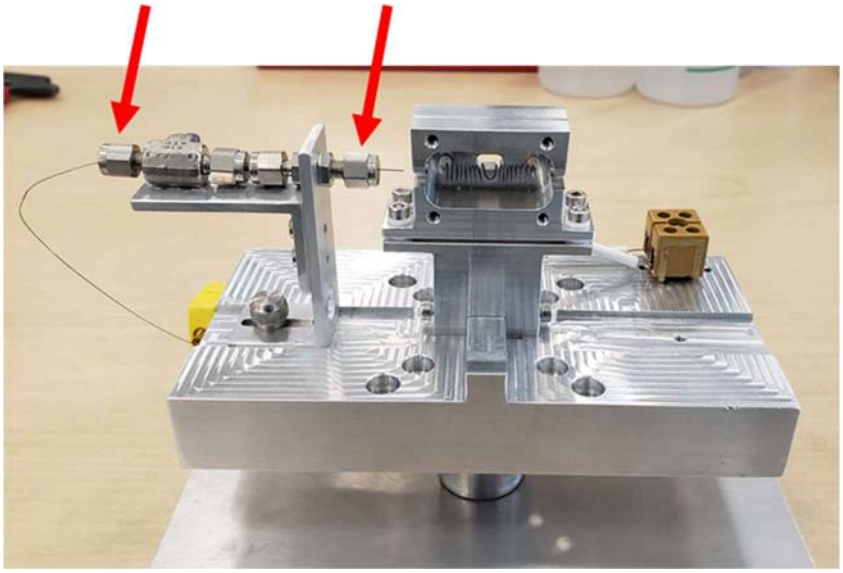
When filling the capillary, you don't need much more than a 4 mm plug of material in the beam location. If the material is a powder, its best to keep the powder packing very loose so that gas can flow through easily.

Put wool on the downstream end only.

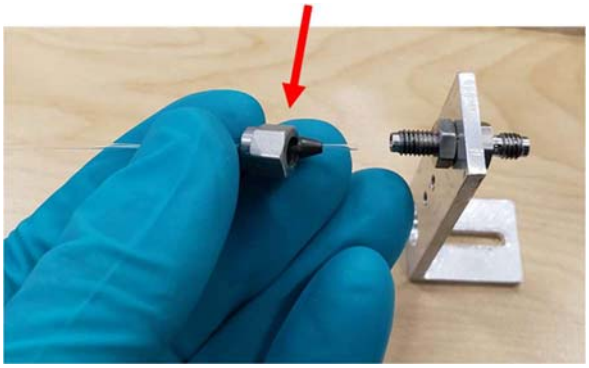
Loading Capillaries and Installing in the Endstation

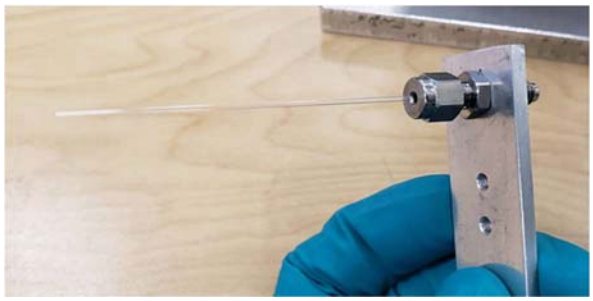
Capillaries are loaded into the furnace on the lab bench, and then the furnace is transferred to the endstation.

1) Nut and ferrule installed over thermocouple, finger tight only. Thermocouple is retracted.

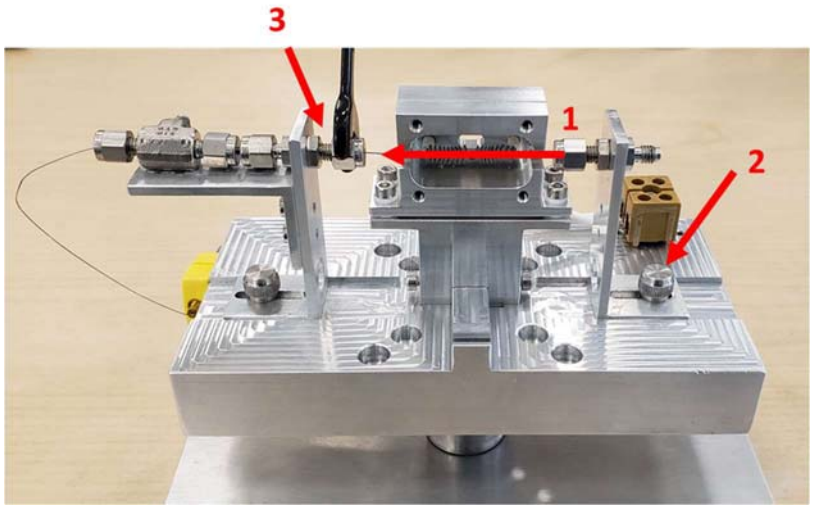


2) Nut and ferrule installed over capillary. Carefully tighten the nut a little at a time, until the capillary can't be pulled out. Then go a little beyond that… not much more than that, maybe a 1/16th of a turn.

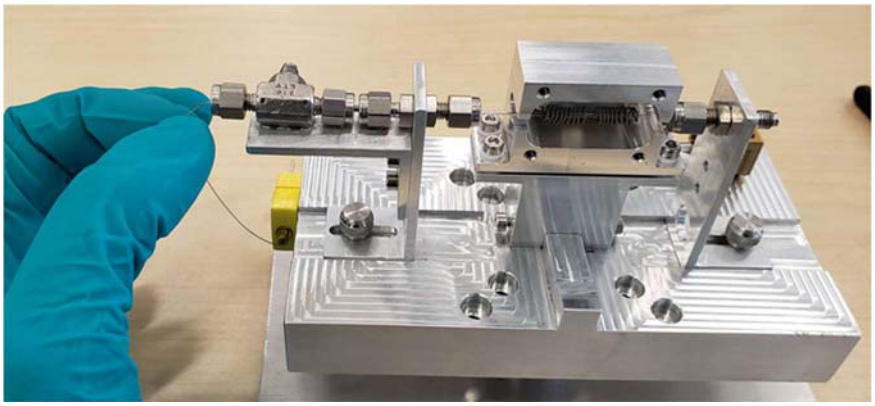




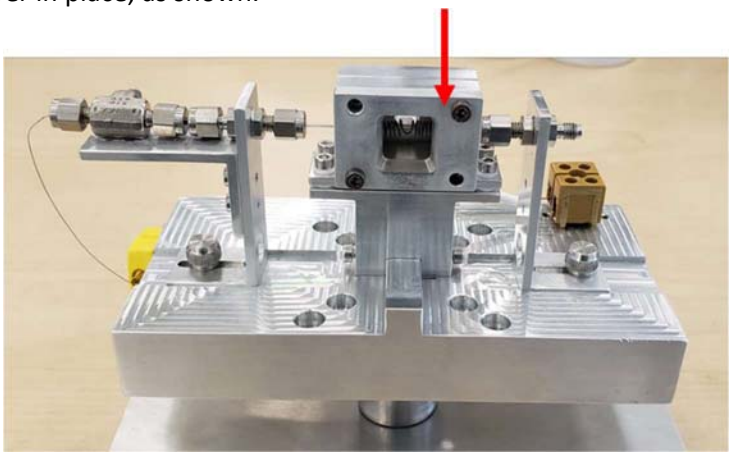
3) Guide the capillary through the heating coil (1), over the thermocouple, and into the ferrule. Finger tighten the thumbscrew for the L bracket (2), then tighten the nut (3) until the capillary can't be pulled out, and not much more than that.



4) Push the thermocouple close to, but not into the sample, and not into the direct beam. Then tighten the nut until the thermocouple can't be easily pulled out, and not much more than that.

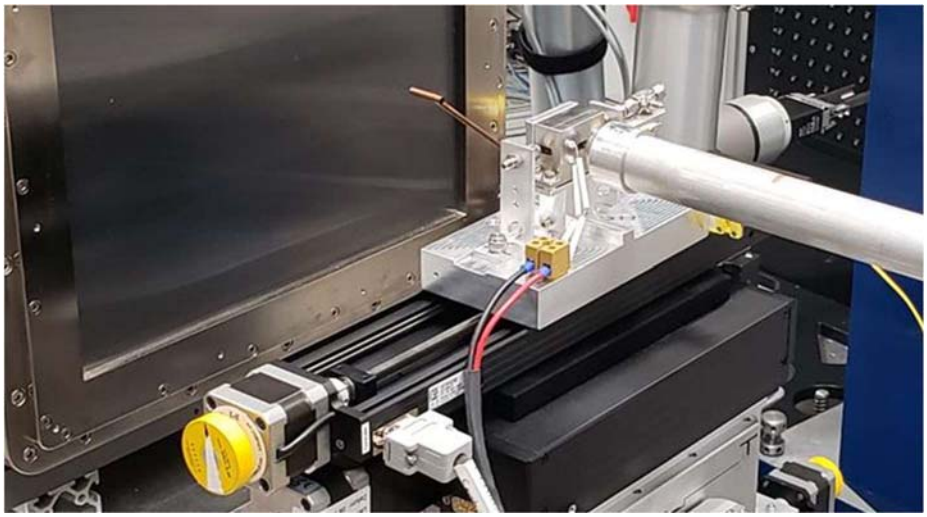


5) Screw the front cover in place, as shown.



6) Transfer the assembly into the endstation: Take care not to bump the collimator, beamstop, and especially the detector!!!

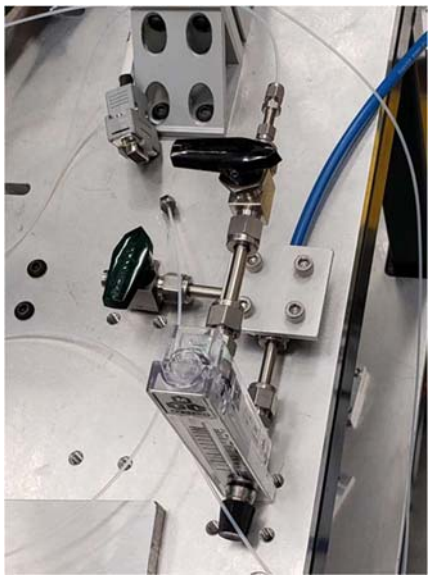
Set the Huber threaded ring, connect power cable, connect thermocouple, connect gas line.



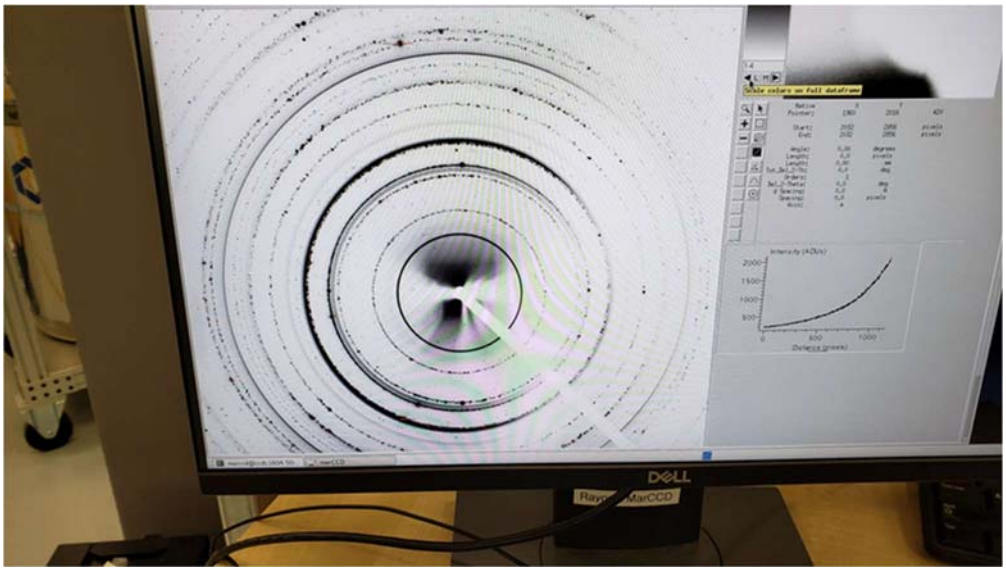
Setting Gas Flow and Beam Alignment

7) Open the leak valve and test the gas flow by putting the outlet hose of the furnace in a cup of water. Once gas flow has been established, turn down the flow as low as possible while still observing bubbles from the exit, about 1 bubble per second is enough. This low flow prevents movement or blowouts of the material at elevated temperature.

Sometimes gas flow cannot be established with the leak valve opened all the way. In this case the capillary will need to be remade. Or you could try making the Swagelok connections on the tube and thermocouple just little bit tighter (assuming they are leaking).



8) Alignment in the beam: Perform a vertical scan using the beamstop photodiode as feedback to find the center of the capillary and move there. Then do the same in the horizontal. Now look at the detector images. You can tweak the vertical and horizontal position to reduce shadowing by the coils, and/or move to a region with more or less sample material.



Making Measurements with Temperature Ramps

To set the temperature, use the following commands in SPEC
Staff will check that the eurotherm counters and micromotor are enabled.

umv m_euro xx # where xx is the target temperature
ct # will show reading of the current temperature

Heating ramp macro
Bond_temperature_scan ("test", 1, 850, 10, 5, 1)

Where "test" is the file name
1 is the exposure time in seconds
850 is the maximum temperature in degrees C
10 is the step size in degrees between measurements
5 is the hold time at the end at max temperature in minutes
1 is the acquisition frequency at the end at max temperature in minutes

The ramp rate is hard coded. Going from 10 to 5 was more like going to 2.5degrees/min.
When rate=13 its about 10 C/min
When rate=20 its about 20 C/min

As the ramp progresses, observe that the material stays in the capillary over the ramp, and doesn't get blown out. This can be a very finicky experiment, good luck!

Troubleshooting

Eurotherm controller reading “SBr” where the temperature value should be	Thermocouple is broken/bad. Ask the beamline staff to replace the thermocouple
Heating element is hot/red when the temperature is set low	Either the thermocouple position is not correct or thermocouple is bad. Switch the power supply power off before troubleshooting.
Controller reads room temp all the time	Heating element might be broken, contact beamline staff

The BXDS High Temperature Gas Flow Furnace is an elaboration/evolution of a device described here: P. J. Chupas et al., “A versatile sample-environment cell for non-ambient X-ray scattering experiments”, J. Appl. Cryst. 41, 822-824 (2008). The paper contains a description of the experimental setup as well as some of the consumables used.