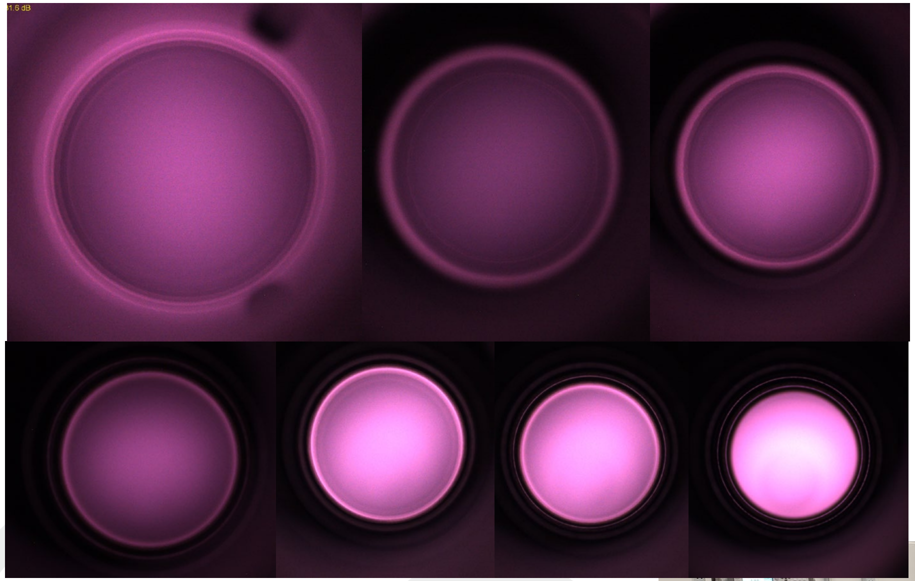
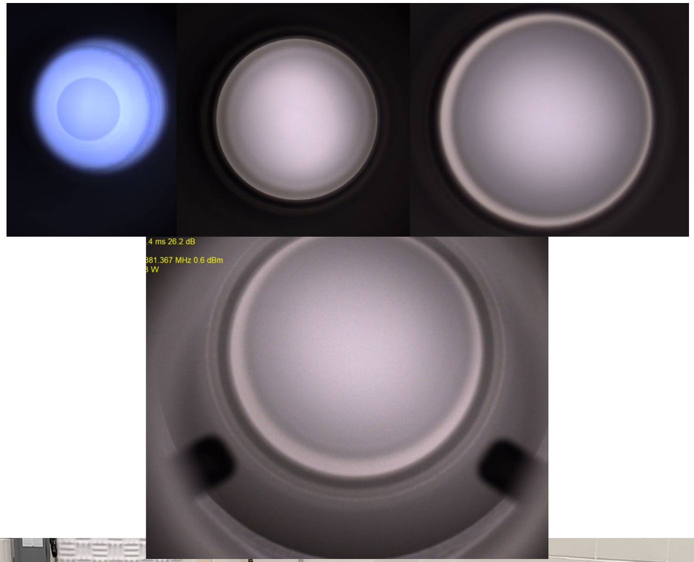
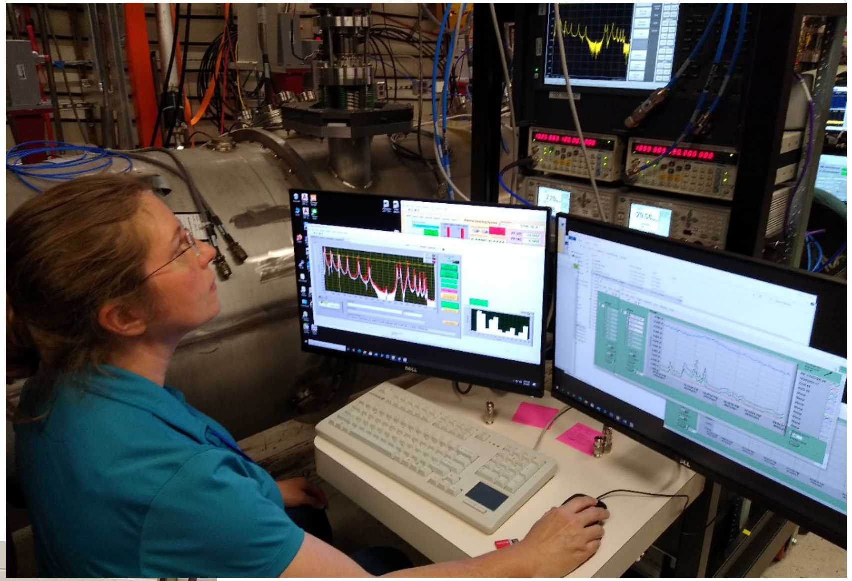
IN SITU PLASMA PROCESSING HARDWARE AND METHODS







Tom Powers and Tiffany Ganey

Plasma Processing Readiness review
28 Feb. 2023

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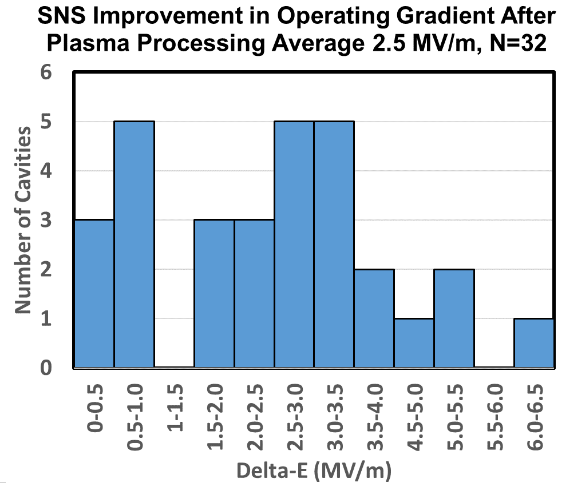
Why use plasma processing

Industial Uses

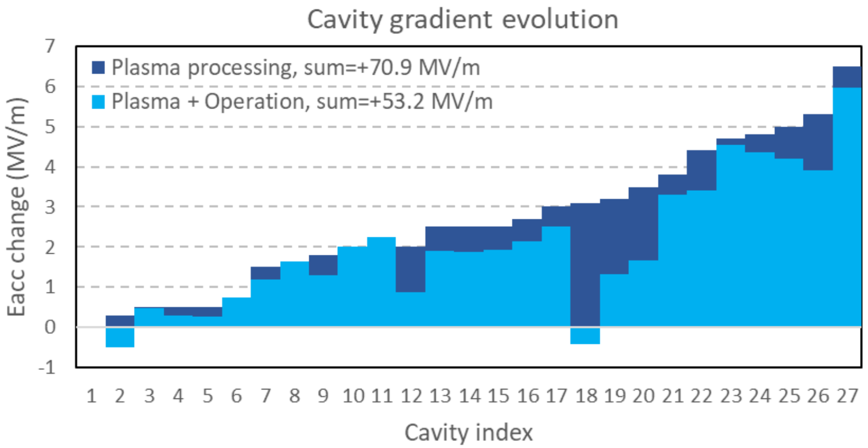
- **Plasma processing is a common technique for removing hydrocarbons from surfaces and improve the wettability of the surface.**
- **It has the capability to treat complex shapes and can be tuned to deliver surface specific properties.**
- **Princeton Scientific Corporation has a line of chamber based plasma processing systems that use the same approach.**



Early SRF Successes

- **2012 Bob Legg, based on the work at JLAB and ORNL, led an effort at the Synchrotron Radiation Center located at University of Wisconsin, the WiFEL SRF gun cathode surface fields improved from 6 MV/m to 26 MV/m.**

- **2015-2018 Marc Doleans at ORNL lead an effort to process 32 cavities in the SNS linac improving the gradients an average of 2.5 MV/m. The work was done during scheduled maintenance periods. After 3 years of operation most of the processed cavities are still doing well.***



*Marc Doleans personal communications

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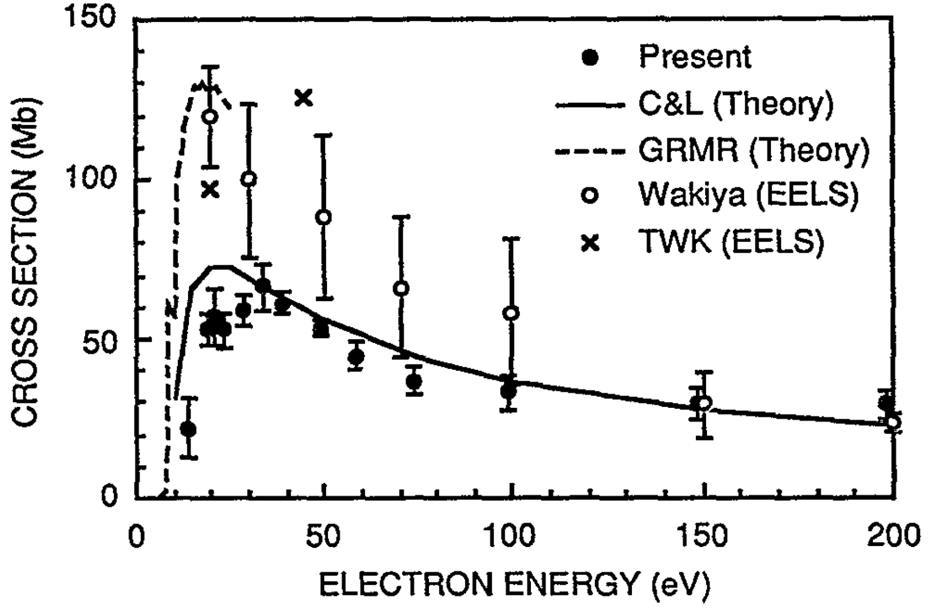
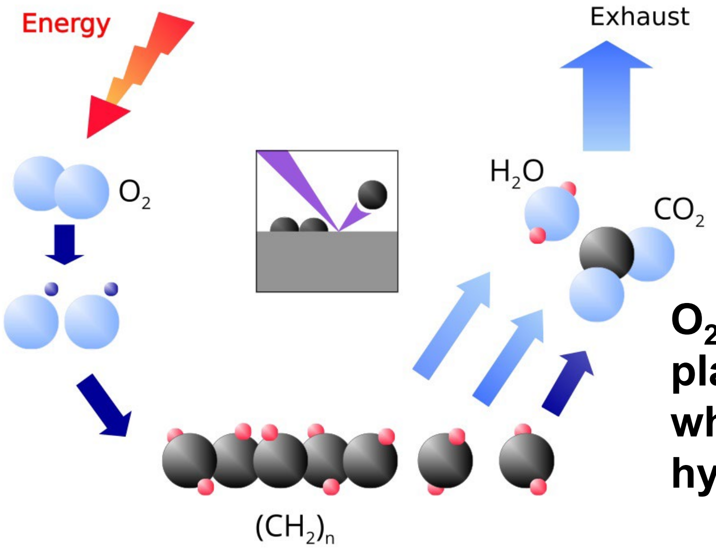
Plasma processing basics, current SRF technique

- Using a room temperature plasma, as is currently done, is not an ion bombardment process like standard 2K or 4K helium processing.
- An RF glow discharge is established with a gas mixture that contains a moderate amount of oxygen.
- Free electrons with an energy in excess of about 10 eV crack the oxygen into free oxygen.
- Free oxygen reacts with the hydrocarbons on the surface cracking it into molecules such as H_20, $C0_2$ and CO which are removed from the cavity with the process gas.
- Removal of hydrocarbons from the surface increases the work function and reduces the secondary emission coefficient both of which improve the field emission properties of the niobium surface.*



Dissociation Cross Sections and excitation cross sections for electron impact on O_2**



O_2 is cracked in the plasma to atomic oxygen which breaks down the hydrocarbons

*Tyagi, et.al., Applied Surface Science 369(2016) 29-35.

P.C. Cosby, J. Chem. Phys. **98, 9560 (1993); https://doi.org/10.1063/1.464387

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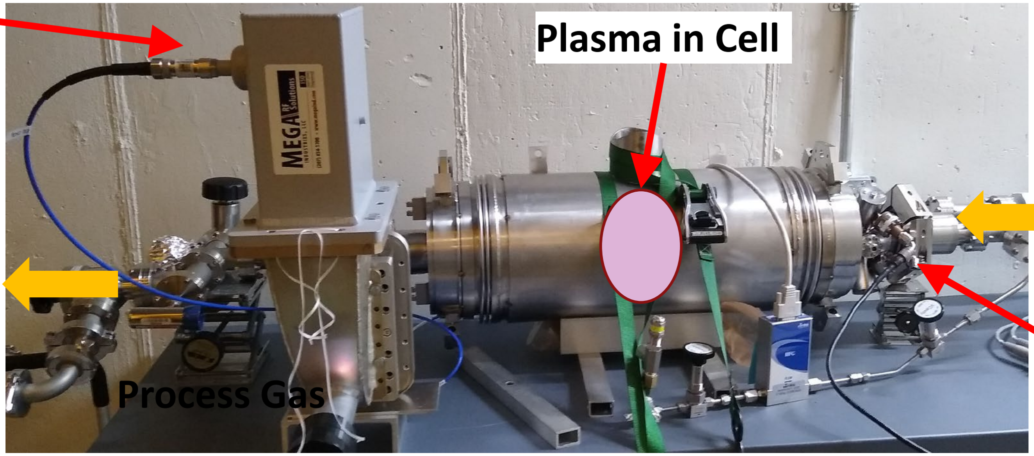
Reactive Oxygen Plasma Processing

RF Monitor Port

Process Gas With CO_2, CO and H_2O, etc.



Process Gas

RF Into HOM Port

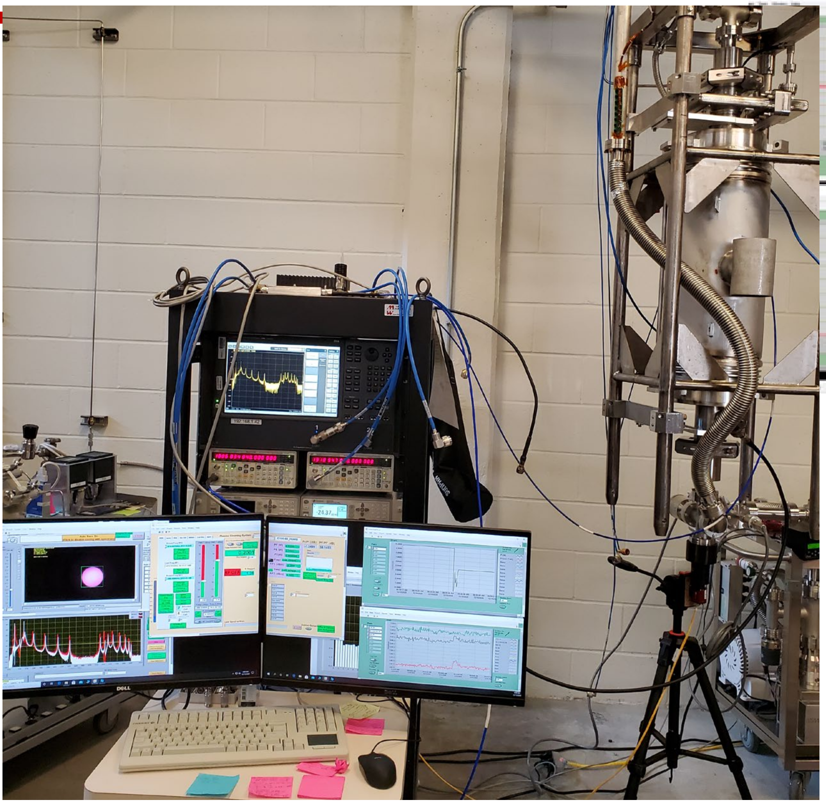
C100 Off-Line Bench Setup

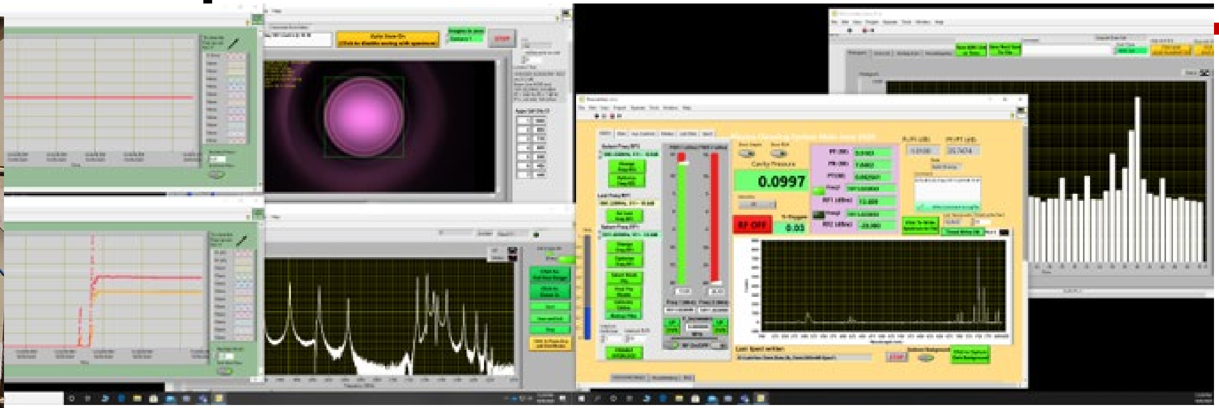
- **SRF “Standard” Recipe**
  - Room temperature mix of helium and 6±1% oxygen gas.
  - Flow gas through cavity between 10 and 20 of standard cubic centimeters per minute
  - Pressure in the cavity 300 mTorr
  - Apply up to 20 W of RF to ignite plasma in one cell, via the lower HOM port
  - Monitor the frequency shifts and RF transmission through the cavity using a waveguide to type-N transition.
  - Move from cell to cell by changing the RF frequency using two sources.
  - Maintain the plasma for 60 minutes in each cell
  - Use interlocks to insure that the RF is turned off automatically if there is a coupler breakdown.
  - Monitor cracked hydrocarbon residuals of H, CO2, CO and H2O

JLAB Recent Experience (Jan. 2021 to Jan. 2023)

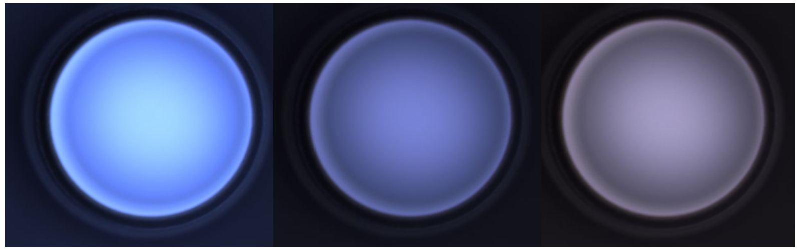
- Completed 31 cycles of vertical test, plasma process, vertical test a single cavity in the VTA.
  - The general process was reactive oxygen process, contaminate with methane argon plasma and reprocess with reactive oxygen, every few months we would do a high pressure rinse and clean assembly.
  - The first 18 months we worked with argon-oxygen gas mixtures
  - The past 8 months we used helium-oxygen gas mixtures.
- June 2021, Processed cryomodule C100-10 with Ar/O^2 after it was removed from the machine.
  - Minimal improvement as this cryomodule had a beam line valve that had a catastrophic failure.
  - Average increase in FE onset was 0.4 MV/m
  - Average increase in 100 mRem/hr FE was 0.3 MV/m
- June 2022, Processed cryomodule C100-5 with Ar/O^2 after it was removed from the machine.
  - Average increase in FE onset was 2.1 MV/m or 11.8 MeV
  - Average increase in 100 mRem/hr FE was 2.3 MV/m or 13.0 MeV
  - Reduced in FE dose at 18 MV/m reduced by a factor of 6.
- January 2023, Processed 4 cavities in cryomodule C100-10R with He/O^2 after the initial acceptance testing.
  - Average increase in FE onset was 3.5 MV/m or 9.7 MeV
  - Average increase in 100 mRem/hr FE was 3.4 MV/m or 9.5 MeV
  - Three of the cavities that were processed went from 30 mRem/hr, 200 mRem/hr and 300 mRem/hr at 18 MV/m to field emission free at 18 MV/m

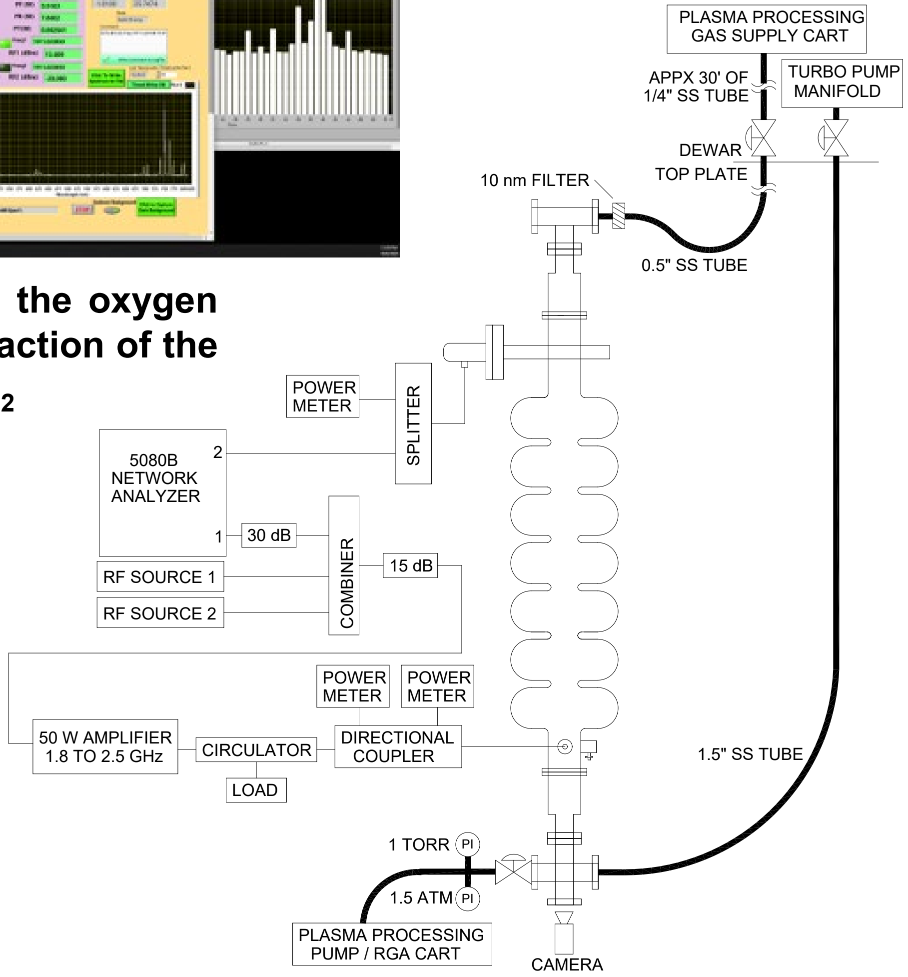
JLAB Vertical Test Stand Setup





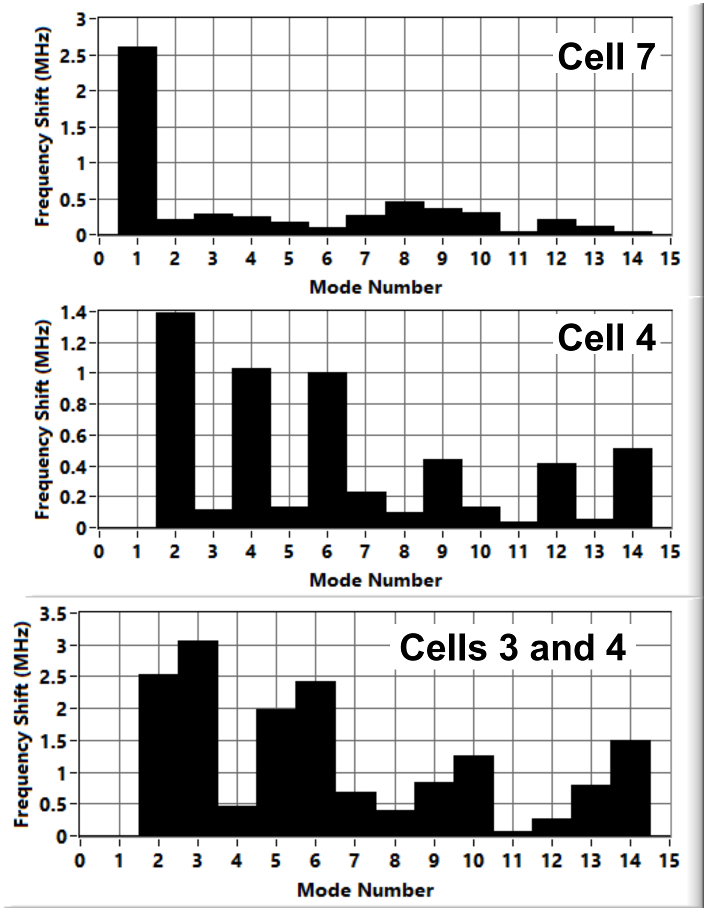
Helium/oxygen discharge changing color as the oxygen content went from 3.8% to 5.6% because a fraction of the oxygen was used to produce H_20, CO and CO_2



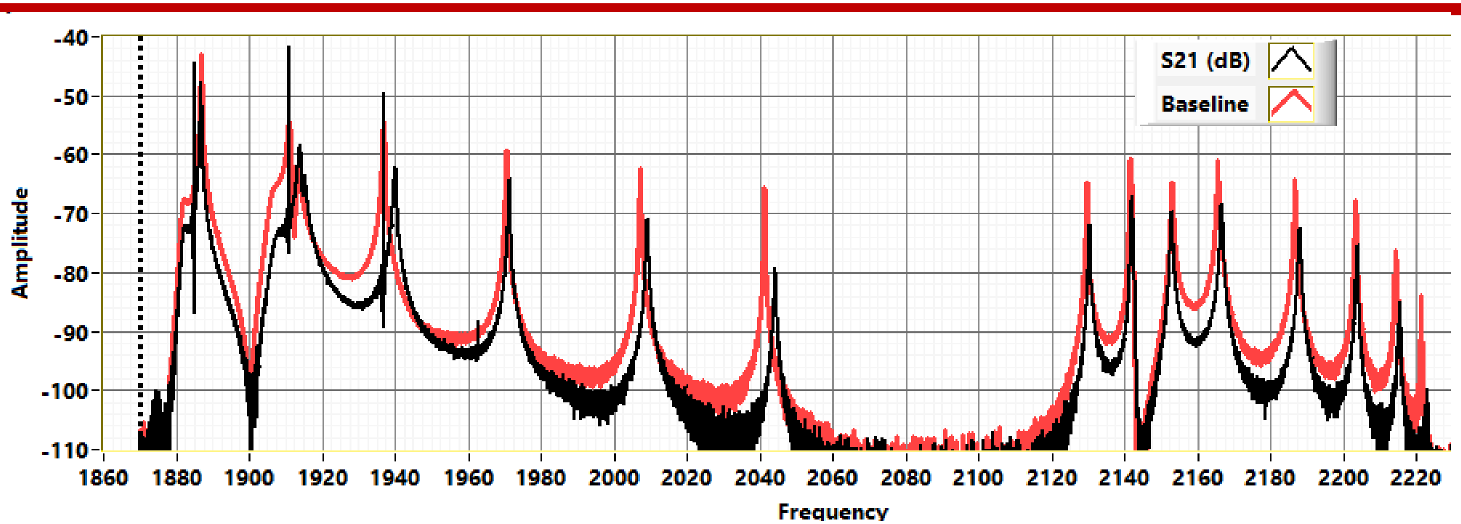


- **Automated software logs pressure, flow, RGA, and RF data multiple times per second as well as network analyzer traces every 30 to 180 seconds depending on user controls.**
- **Plasma processing is done in the vertical staging area while the cavity is still mounted to the vertical test stand.**
- **A camera was used so that we could verify the cells with plasma and gain confidence in the RF based approach for determining plasma location.**
- **Exhaust gas was monitored with an RGA**

Using S21 measurement to characterize and locate plasma



Measured Mode Shifts



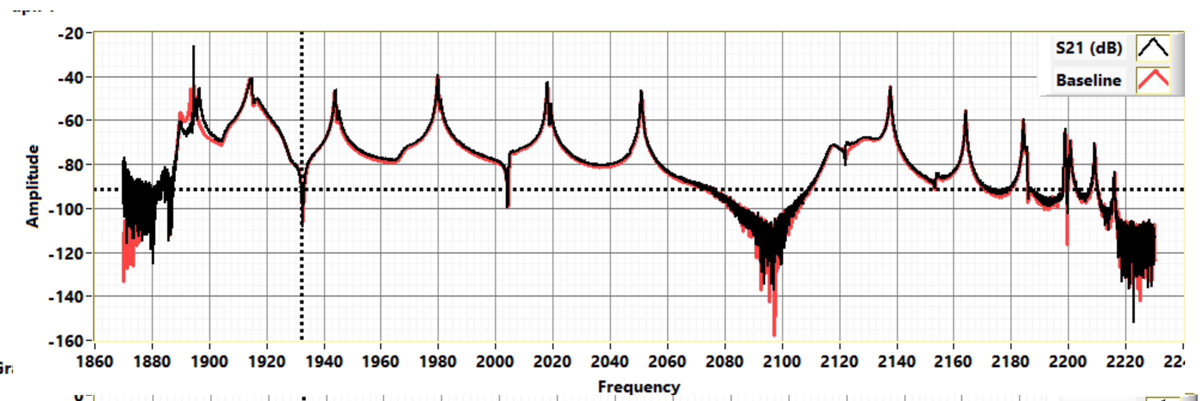
Cavity S21 measurement and frequency shift by mode for cell 3 and 4 ignited.

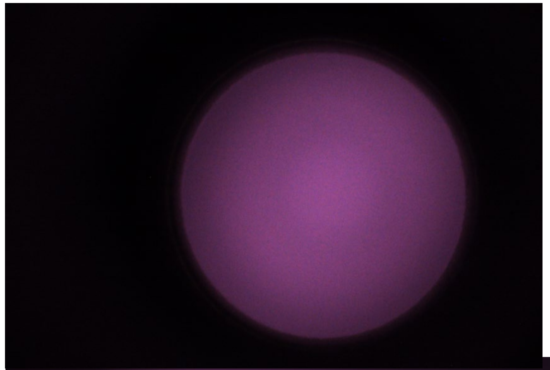
- A low level network analyzer signal is applied to the input of the amplifier and the "probe" signal was fed back to port 2 on the network analyzer.
- The dielectric constant is reduced where there is plasma. The higher the plasma density the lower the dielectric constant.
- Each mode is affected differently depending on the location of the plasma and the mode pattern, e.g. no frequency shift for a mode with no field in the ignited cell.
- The frequency shift per mode is presented live while we are processing.
- This method allows us to confirm the plasma location without a camera.

Detecting coupler breakdown using network analyzer

We have an interlock to protect the cavity from HOM coupler breakdown.

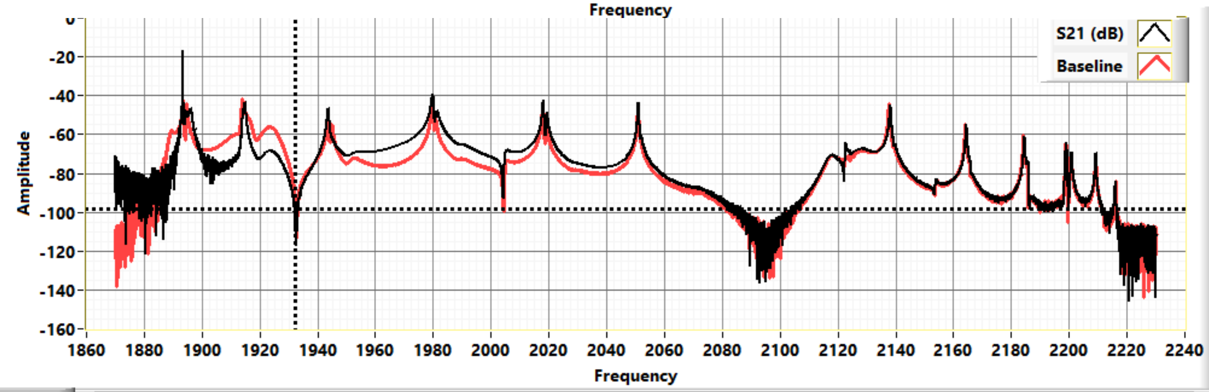
Nominal plasma on/off (black / red) measurements with plasma in cell 7.

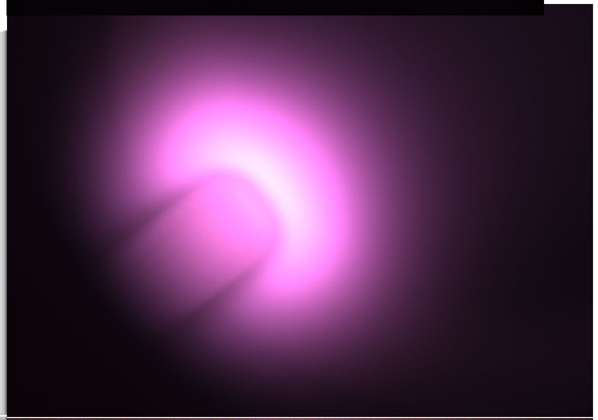




Not a terrible fault mode diffuse discharge at probe tip.

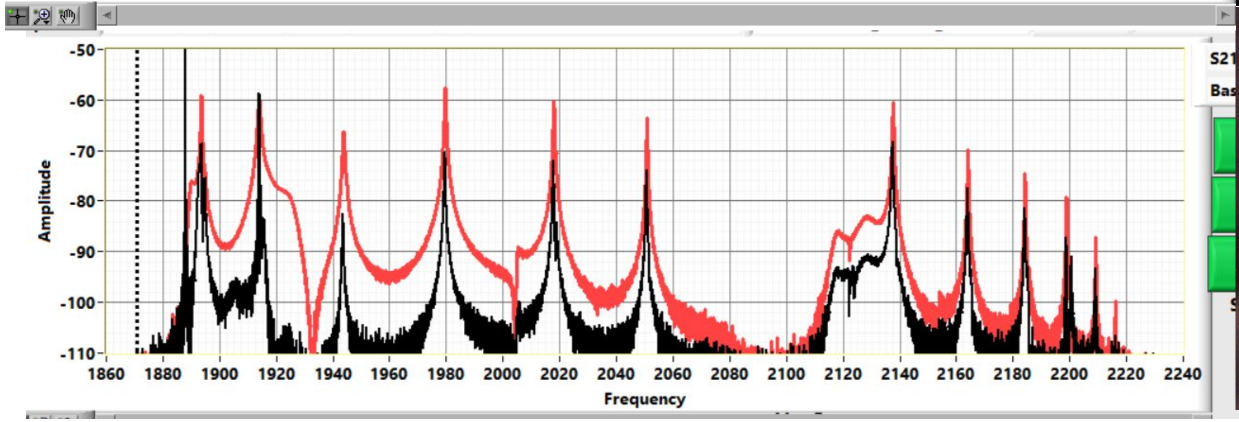
Typical signals for plasma on HOM antenna tip with RF on/off (black / red).

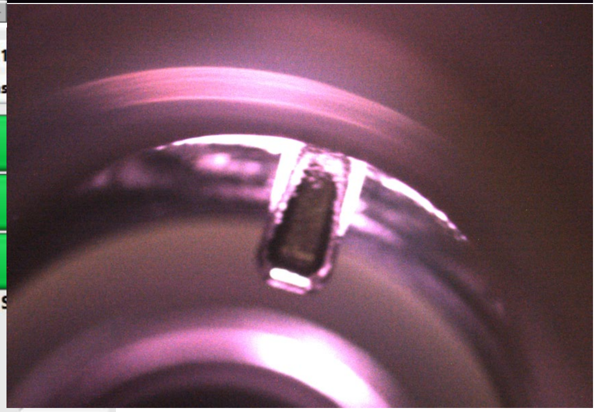




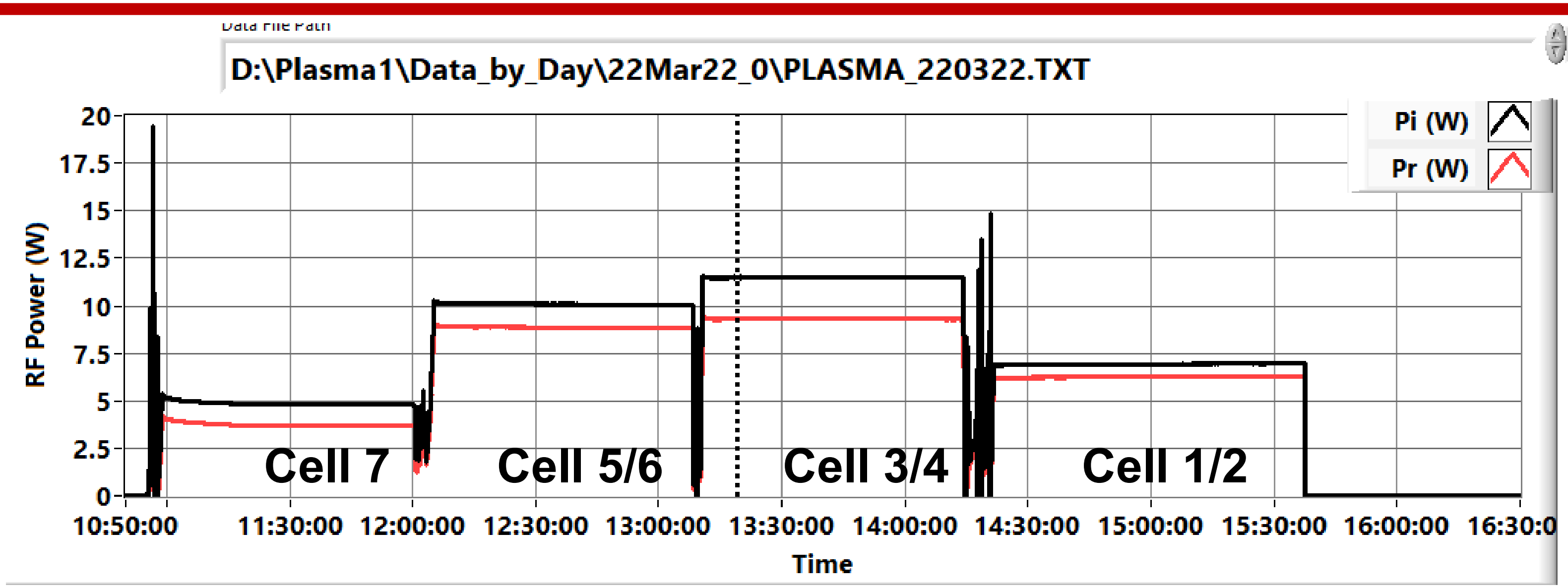
This one is bad as it is an arc like discharge in the tube containing the coupler feedthrough antenna.

Typical RF on/off (black / red) for breakdown within HOM coupler.



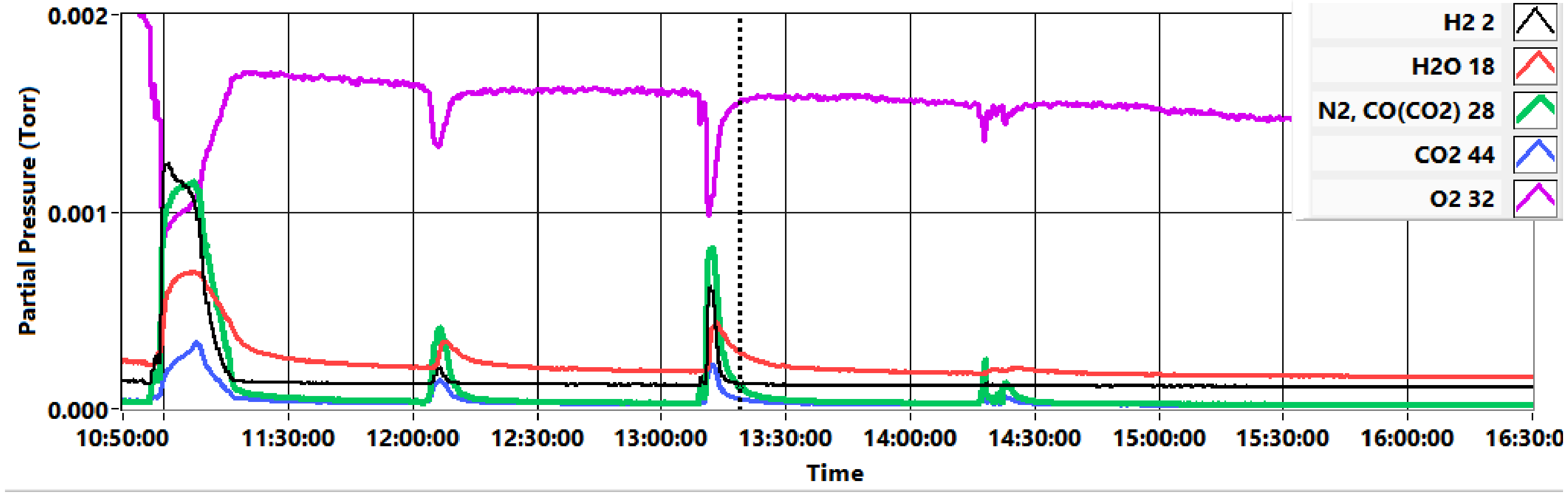


Typical processing cycle in the vertical test area



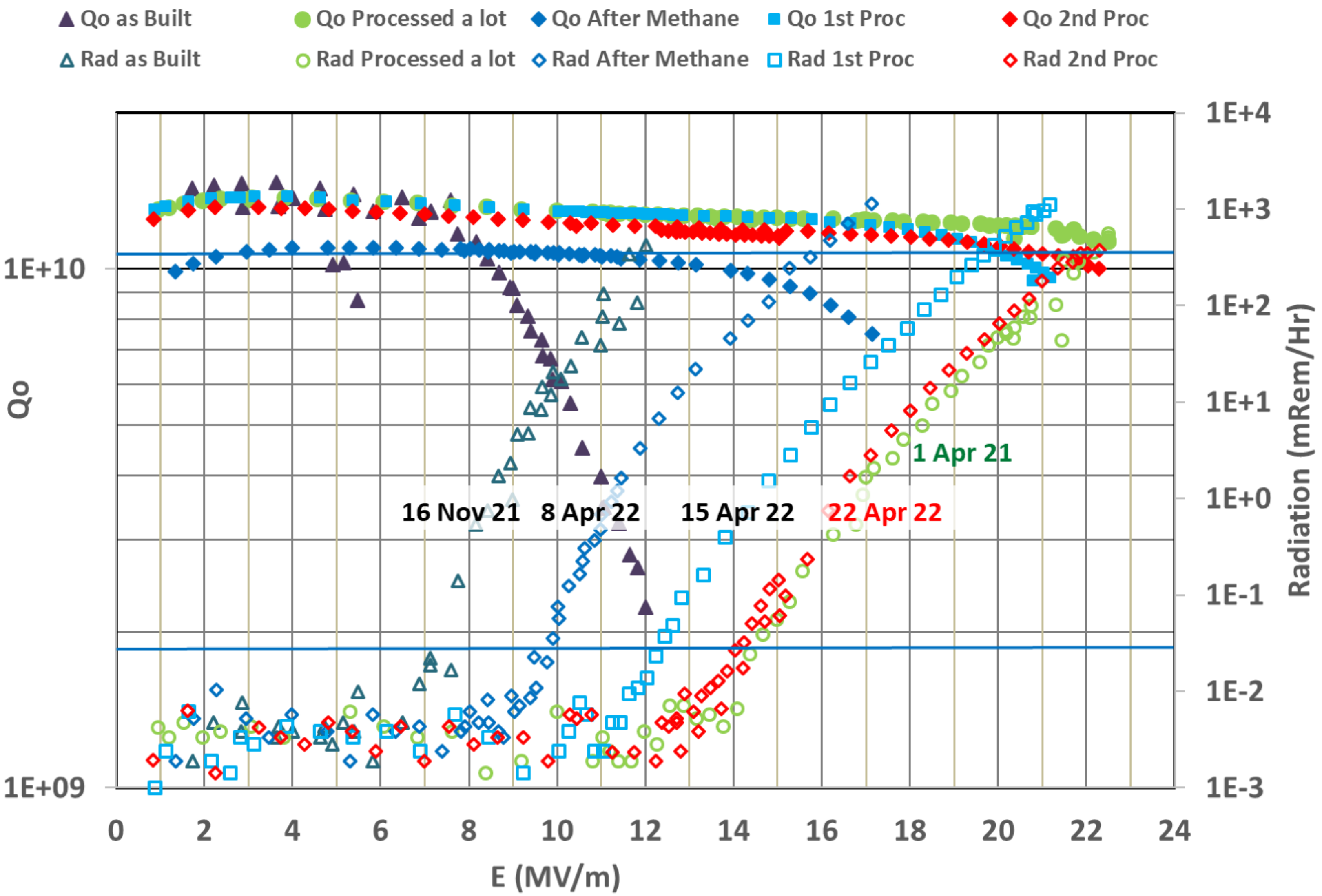
Pi (W)	11.4552
Pr (W)	9.2930
Pt (W)	8.2962E-7
Pf/Pt(dB)	71.4012
CPLR_FLT	0.0000
Amp_SRC1(dBm)	9.6000
F_SRC1	1935.1613
F_SRC2	1908.5640
RF_ON_SRC1	1.0000
RF_ON_SRC2	1.0000
%O2	1.0521
AR 40	3.1080E-5
O2 32	3.2700E-7

- The Upper Plots are incident and reflected power calibrated to the input of the HOM port.
- Processing 2 cells at the same time reduces the processing time by 40%



- The violet trace which is oxygen, the lower plot are the hydrocarbon residuals of hydrogen, water, carbon monoxide and carbon dioxide.
- The partial pressures are scaled to the pressure at the exit of the cavity.
- The oxygen content was reduced as it was used to produce water, carbon monoxide, and carbon dioxide.

Cavity C100-86 improvements after plasma processing with argon/oxygen



- **Field Emission (FE) onset out of the clean room 7.5 MV/m**
- **Processed several times the last time with 20% oxygen gas mixture to get to the 1 April results (Green) FE onset of 14.7 MV/m**
- **Methane plasma used to deposit hydrocarbons on the surface and reset the FE onset to 10 MV/m (8 Apr. results)**
- **Plasma process using 1% oxygen (15 Apr. results) followed by processing with a 20% oxygen gas mixture (22 Apr. results) in order to repeat the results of FE onset at 14 MV/m**
- **Final results is the red data plots FE at the operating gradient of 18 MV/m was improved from >1 Rem/hr to less than 0.008 Rem/hr.**

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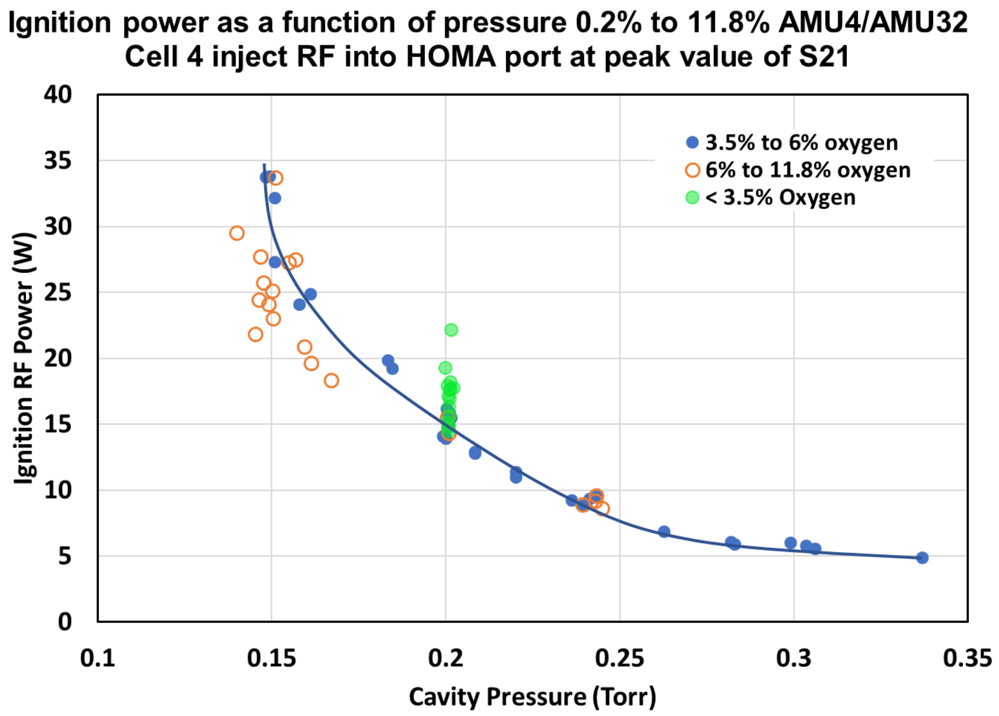
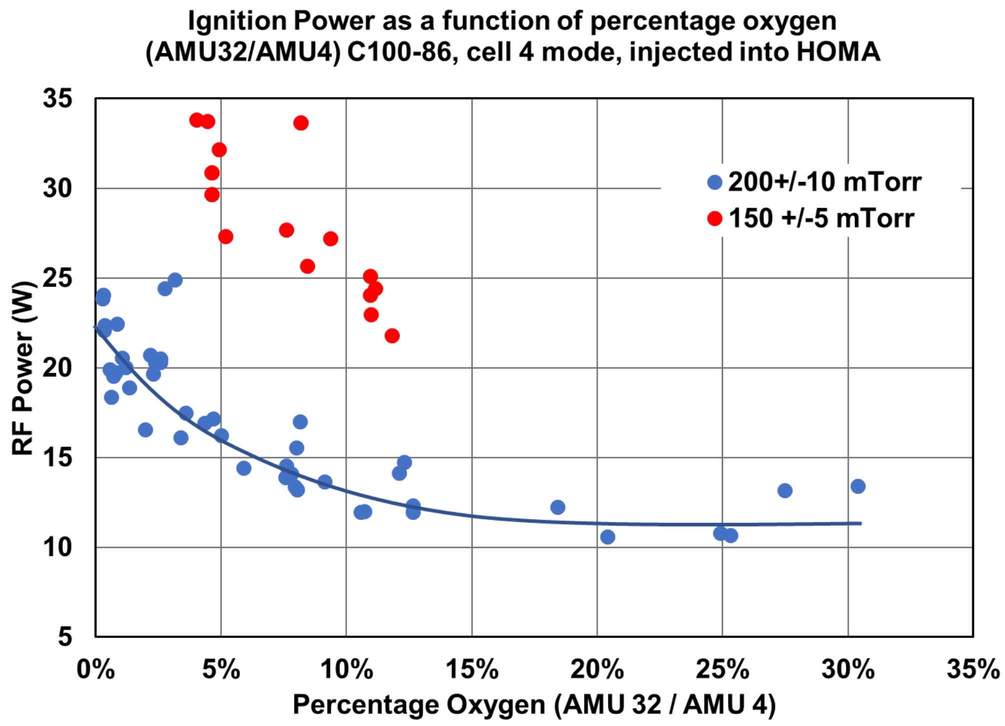
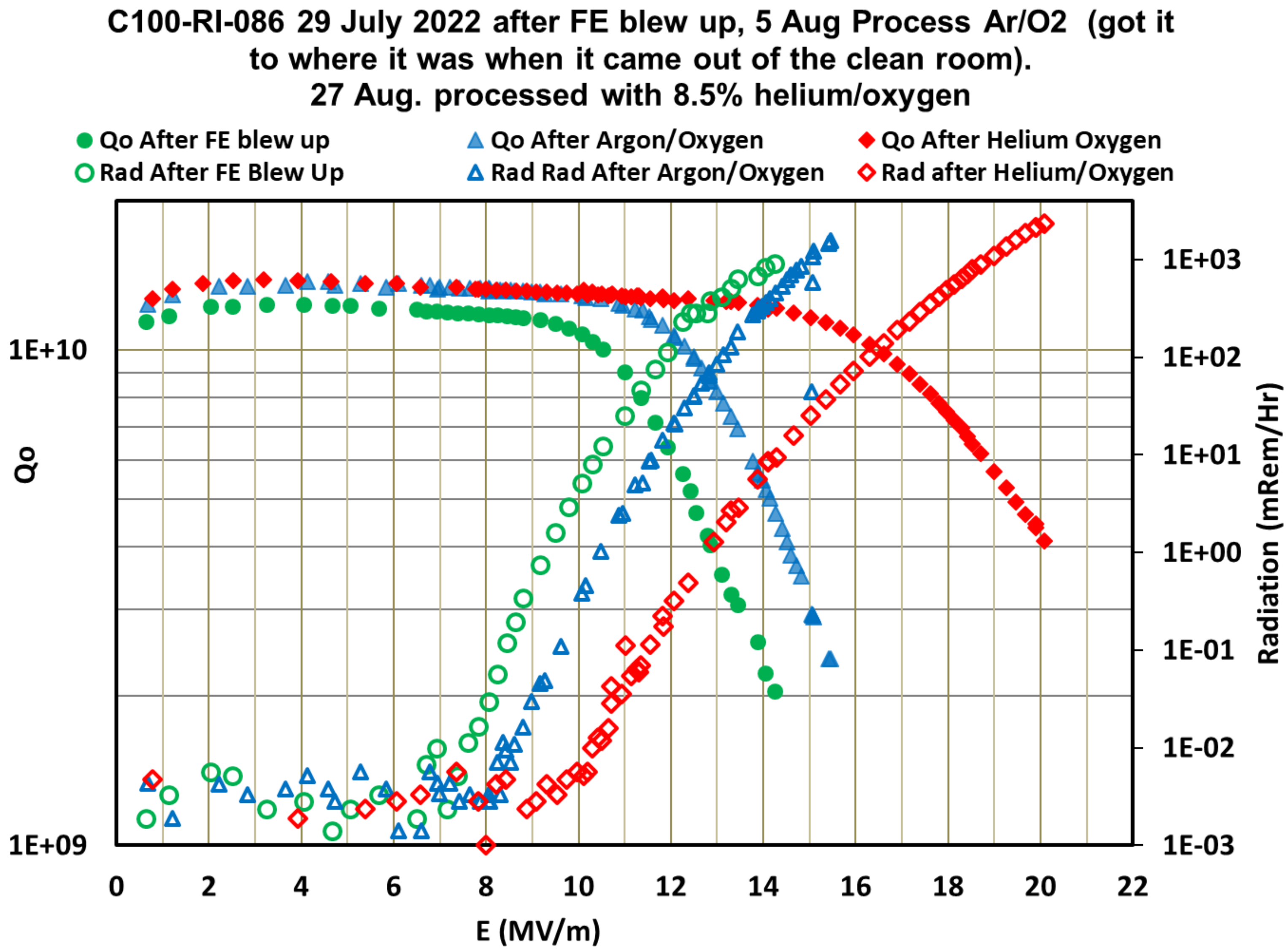
Ignition power as a function of pressure and oxygen content for helium





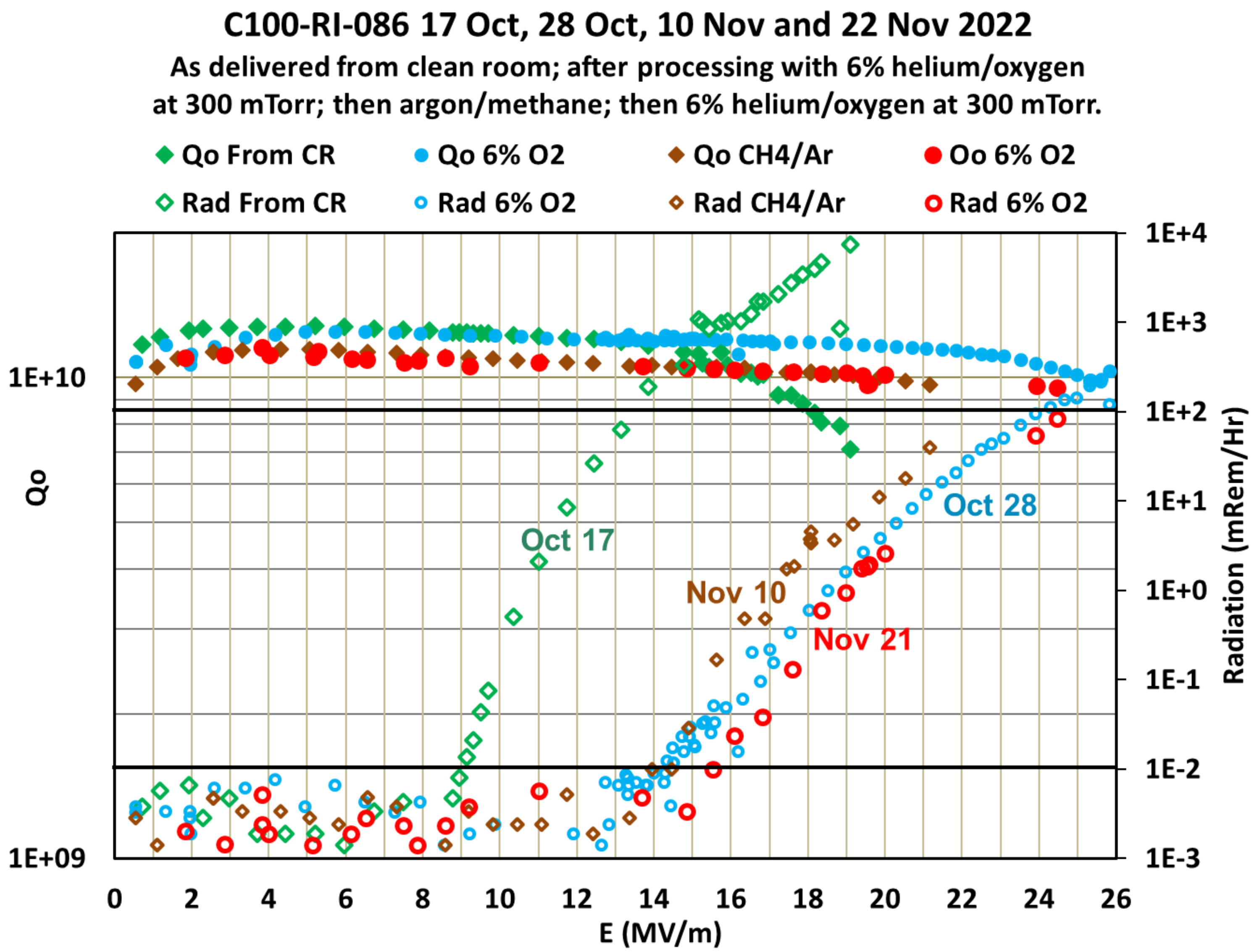
- Data indicated that it took a moderately higher power to ignite the plasma in helium oxygen as compared to argon.
- There is dependence on the oxygen content
- Ignition is statistical in nature, e.g. if you are patient and stay at a given power just below prompt ignition the plasma will often ignite after 30 seconds.
- Once ignited at higher pressure one can lower the pressure, while maintaining the discharge.
- So far we have done processing at 50 mTorr, 100 mTorr, 200 mTorr and 300 mTorr.
  - 50 mTorr - difficult to do and we saw no improvement
  - 300 mTorr, easy to ignite the plasma very few breakdown issues

First results processing with helium oxygen



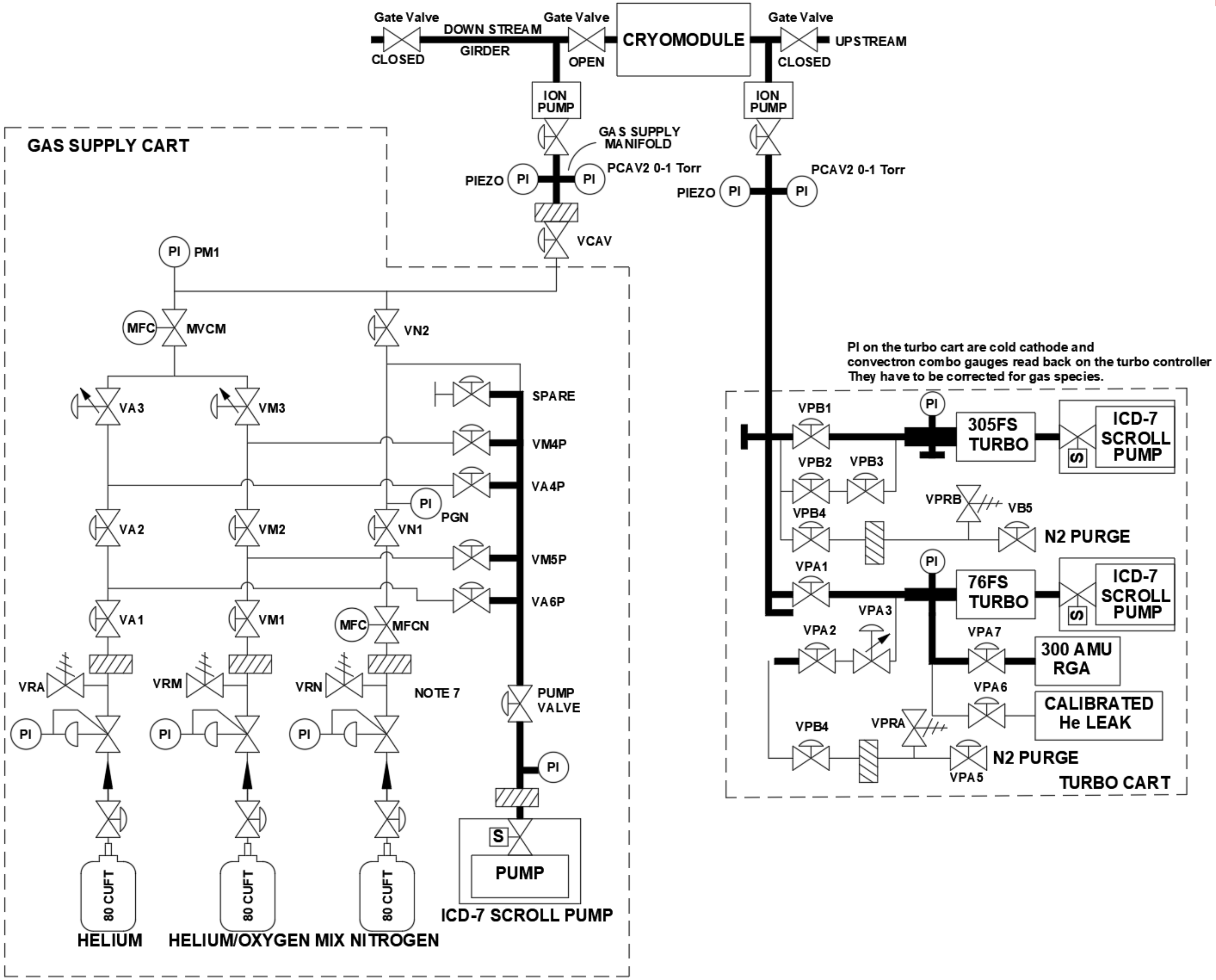
- After we blew up a field emitter (green circles) we processed with the standard 1% to 2% argon/oxygen followed by 20% argon/oxygen and got about 1 MV/m improvement (Blue Triangles).
- We followed this by processing with a 8.5% helium/oxygen gas mixture and improved it another 2 MV/m (Red Diamonds).
- **Helium/oxygen improved a cavity that was not improving any more with argon/oxygen.**
- **Promising first results.**

Most resent results processing with helium oxygen



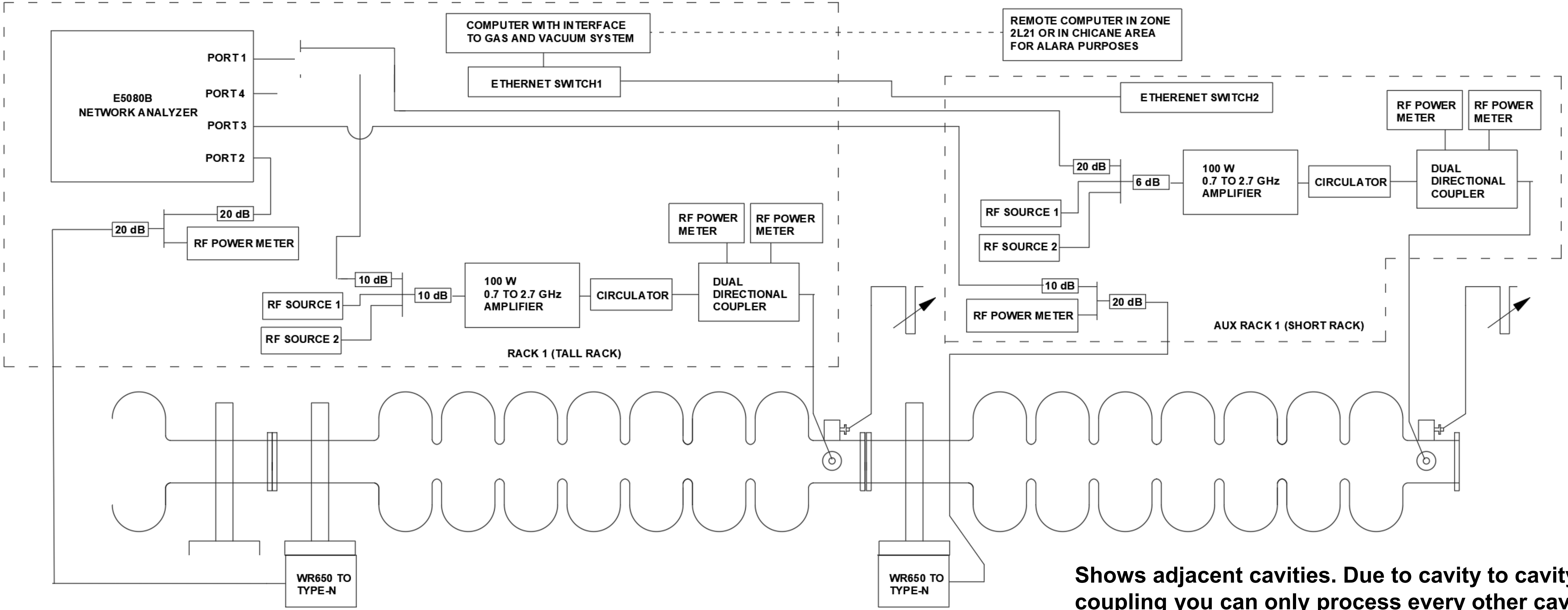
- We sent the cavity back through the clean room and it came back with a field emitter that turned on a 9 MV/m (green diamonds).
- We processed with 6% helium/oxygen at 300 mTorr and improved FE onset by 5.25 MV/m (blue diamonds)
- We tried to degrade the cavity with a methane/oxygen plasma without much success as FE onset only went down by 0.25 MV/m (brown diamonds).
- We processed it again with 6% helium and it improved to 15.5 MV/m or 6.5 MV/m better than when it came out of the clean room (red circles).
- From an operational standpoint field emission radiation at 18 MV/m, which is the nominal operating gradient of a C100 cavity, went from 4 Rem/hr to 0.0004 Rem/hr.
- Note the drop in Qo is due to residual magnetic fields.

Plasma Processing gas supply and vacuum setup for tunnel.



- **Any leaking gate valves will be replaced and beam line vacuum restored prior to connecting plasma processing gas supply and pumping systems.**
- **We have two identical gas supply and pumping carts.**
- **We have a spare 300 L/s turbo pump, 70 L/s turbo pump and two spare scroll pumps.**
- **As part of the preparation work for this effort we will have replaced the tip seals on all of the scroll pumps.**
- **The gas supply and pumping manifolds will be preassembled and leak checked prior to leaving the test lab.**
- **Pump cart will be connected to the valve on the ion pump on the cavity 1 end of the cryomodule.**
- **Gas will be injected into the down stream girder at the valve on the “A” ion pump which is on the down stream end.**

RF system block diagram for processing cryomodule in the tunnel



Shows adjacent cavities. Due to cavity to cavity coupling you can only process every other cavity.

- **Same general setup as was used for vertical testing except:**
  - **4 Port network analyzer and second RF equipment rack used to process 2 cavities at once using one computer.**
  - **Phase shifter added to second HOM port because of the effects of coupling from one HOM port to the second HOM port.**
  - **Using a two channel system we can process 8 cavities in two 10 hour shifts.**
  - **If the zones are co-located, one operator can process 2 zones (4 cavities) at once.**
  - **We have two identical RF systems.**

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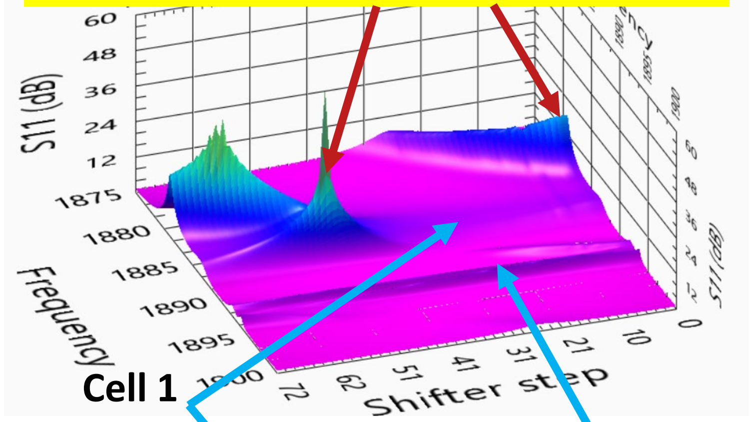
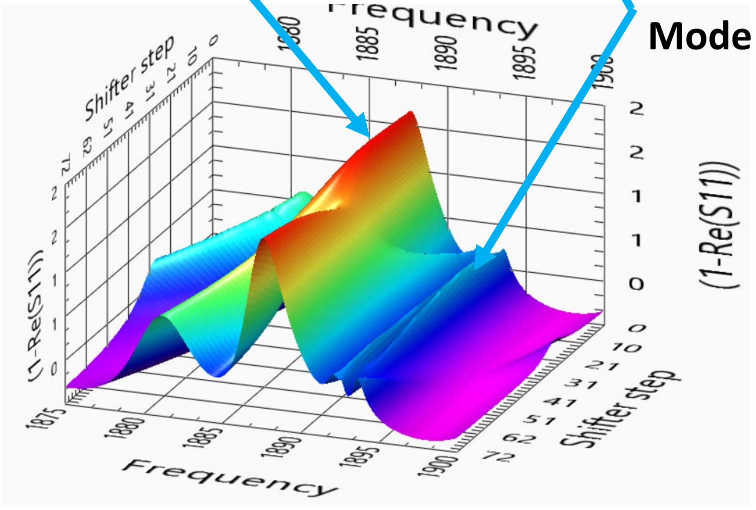
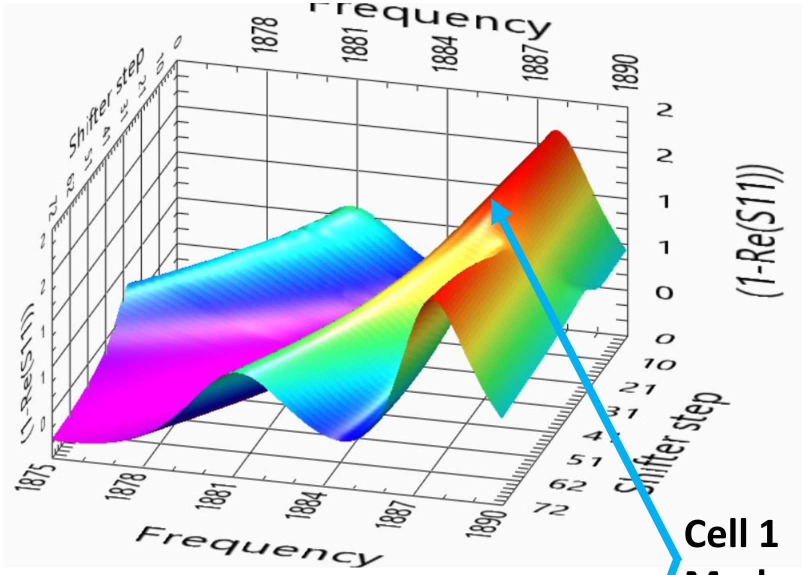
Why is the phase shifter necessary

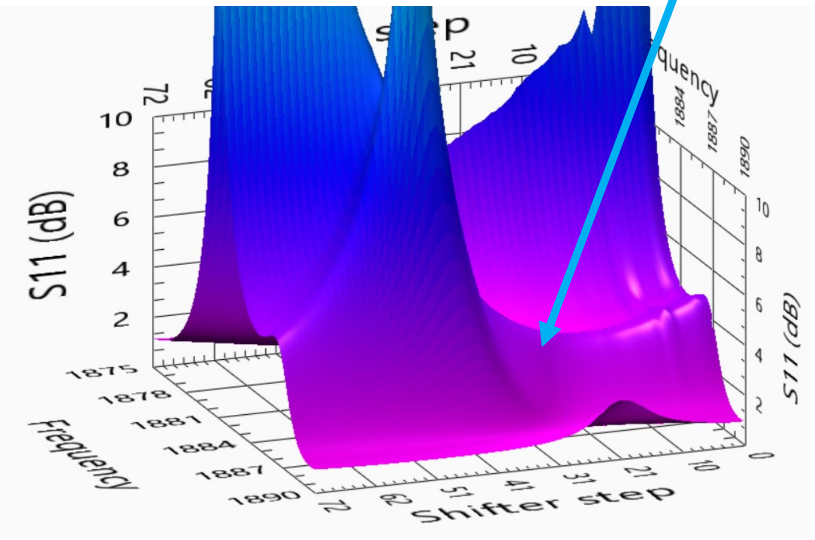
Based on bead pull data, these peaks do not create fields in the cells





- The cables between the HOM couplers and the connectors on the vacuum vessel are 10’ +/- 1”. This amounts to a 270° randomness in phase.
- There is strong coupling between HOMA and HOMB couplers in the TE111 frequency band.
- The coupled signal goes to the end of the unused cable and is reflected back and tries to drive or suppress the mode because of the fixed but random phase length of the cable.
- After extensive bead pull experiments we decided to use an open circuit phase shifter on the unused port, measure the S11 and S21 parameters of the system and choose a phase that provides favorable RF properties for exciting the different modes.
- One of the main issues is the Cell 1 mode. If one tries to operate at the phase settings with large losses that do not couple into the cells the couplers will experience breakdowns without establishing a plasma in the cells.





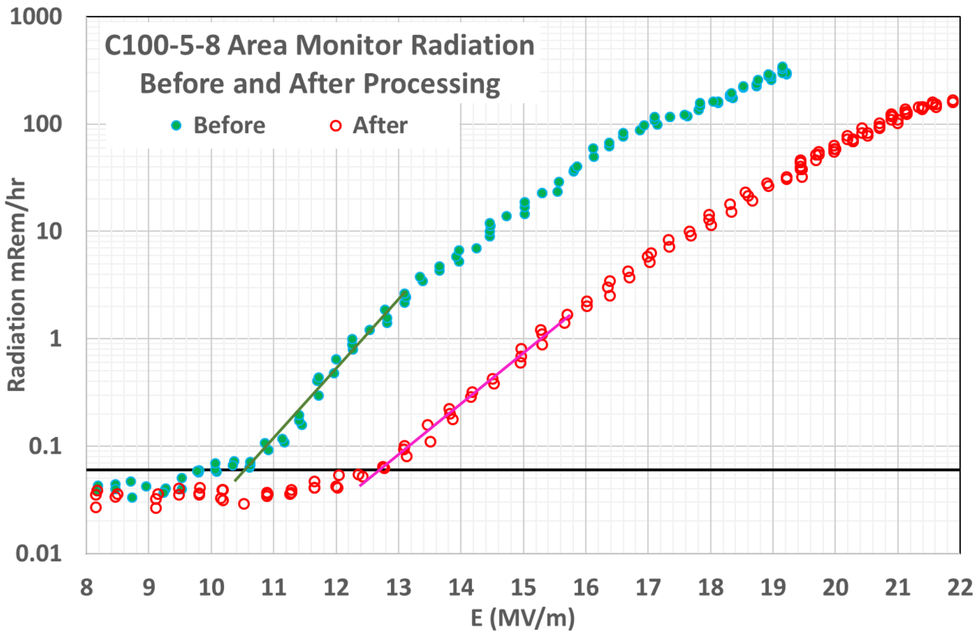
Summary Regarding Hardware and Methods

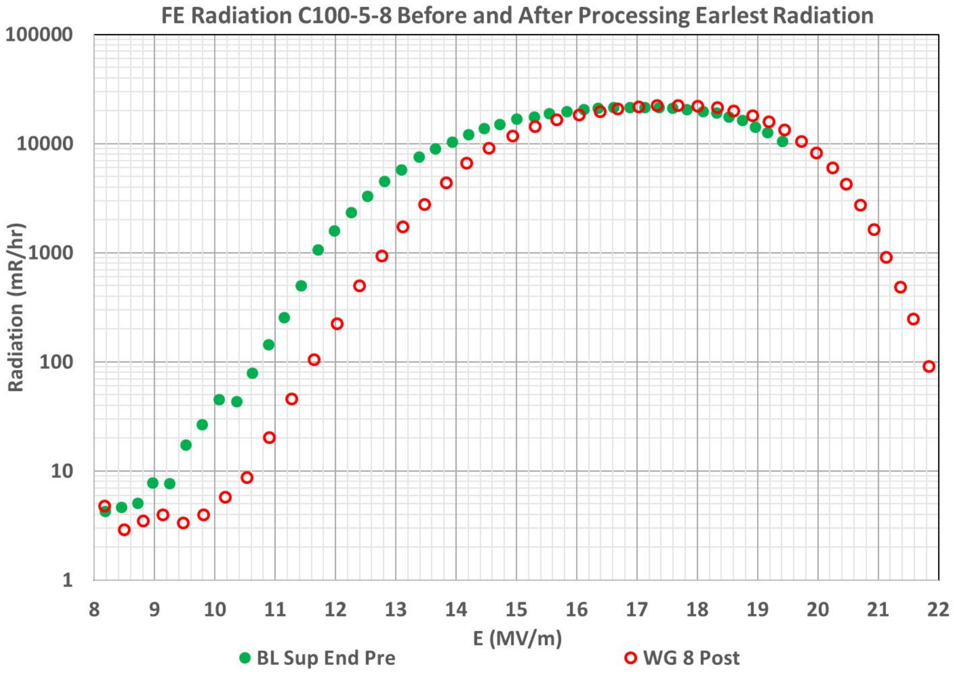
- We have extensive experience plasma processing cavities in the vertical test area.
- We processed 3 cryomodules in the test cave.
- The first cryomodule was not very successful because the cryomodule was severely degraded.
- We were successful processing two of the cryomodules.
- We have the following equipment ready to go or on order** and due prior to the SAD.
  - Two 2-channel RF systems each with a computer that controls and monitors the RF and beam line vacuum as well as logs process data.
  - Two gas supply carts
  - Two pumping carts with differential RGAs.
  - Two particle counters
  - Two ionized nitrogen blow down guns with nitrogen gas tanks and regulators.
  - Multiple laptop computers so that we can operate the systems outside of the radiation area.
  - **Four telescoping portable clean rooms. Telescoping so that that one can be rolled past another without disturbing the clean room that is over the beamline.
- We plan on using 94% helium, 6% oxygen for processing during the 2023 SAD.

Backup Slides

C100-5 reduction in radiation at 18 MV/m

- **While the Geiger Muller tubes in the decarad system are very good for determining radiation onset because of the large number of channels and the directionality of the bremsstrahlung radiation, it tends to saturate at higher radiation levels.**
- **The area monitor which is an ion chamber was much better for comparing radiation levels at higher gradients.**
- **While the two systems gave slightly different onset values on a cavity by cavity basis, the overall improvement results were within 10% of each other.**





Average reduction in radiation at nominal operating gradient was a factor of 6.6

Area Monitor Data (mR/hr)			
CAV	Before Radiation at 18 MV/m	After Radiation at 18 MV/m	Reduction at 18 MV/m
1	9	0.04	0.4%
2	50	25	50.0%
3	1300	200	15.4%
4			
5	2000	300	15.0%
6			
7	4000	60	1.5%
8	150	13	8.7%
		Average	15.2%

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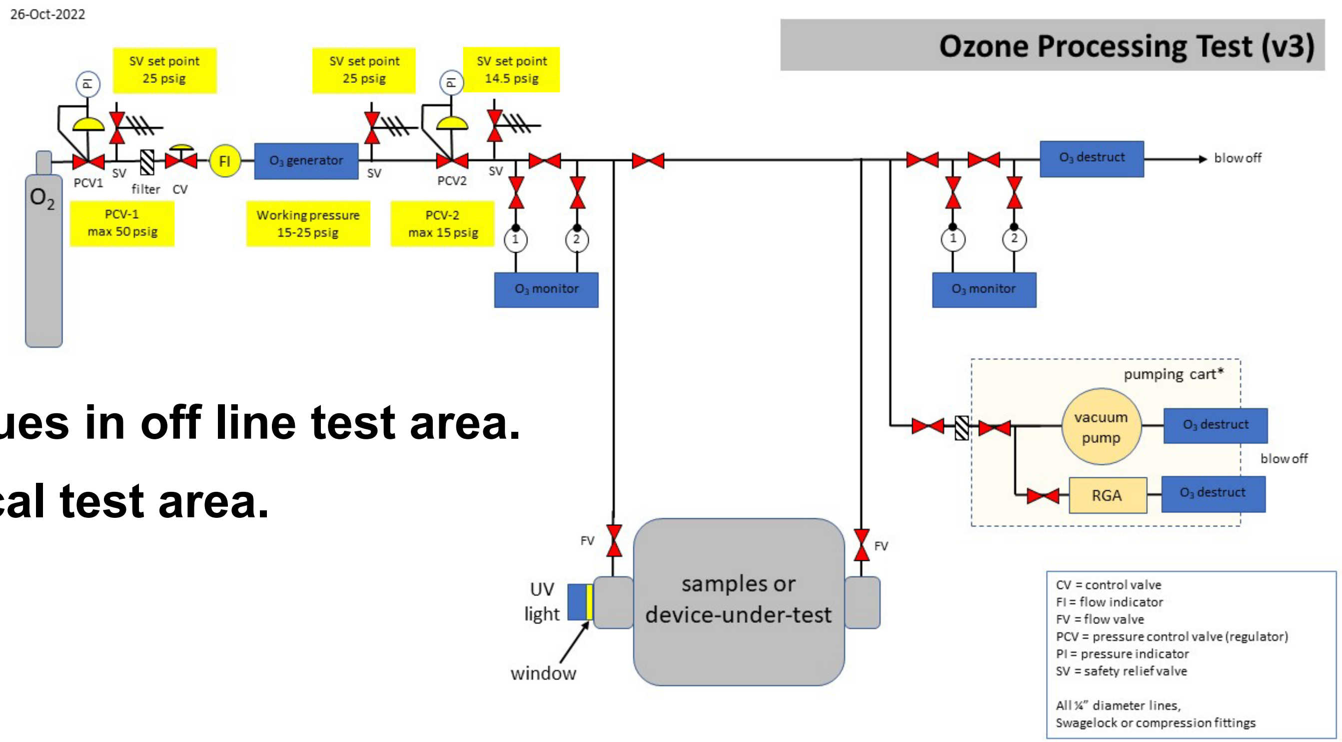
Ozone processing

Issues

- **Improve safety:**
  - **area monitoring ozone sensor,**
    - **to ensure automatic generator is switched-off if the area ozone level exceeds authorized level,**
    - **parts out of stock at vendor, delivery expected in December;**
  - **inline ozone sensor after test volume,**
    - **to ensure safe opening by verifying system purge with pure oxygen after switch-off ozone generator,**
- **Improve stability ozone concentration:**
  - **increase oxygen pressure in generator,**
    - **oxygen compatible regulator out of stock, no delivery time given, on back order.**



Future work

- **Repeat sample tests;**
- **Test on cavity with inline RGA to monitor residues in off line test area.**
- **Test on a cavity followed by RF testing in vertical test area.**