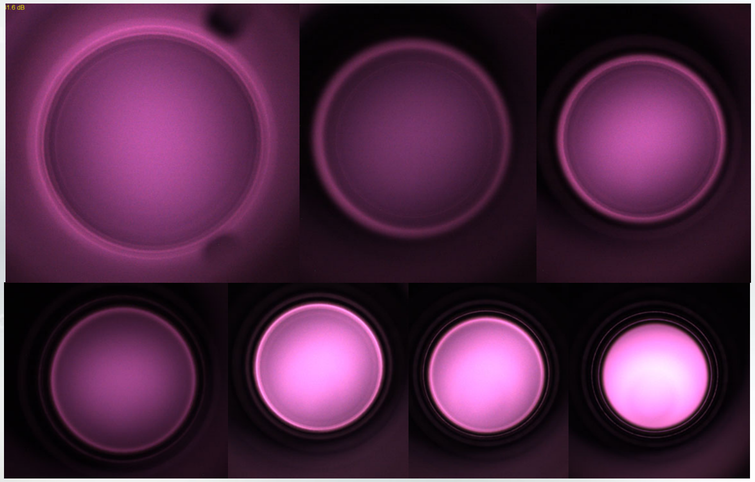
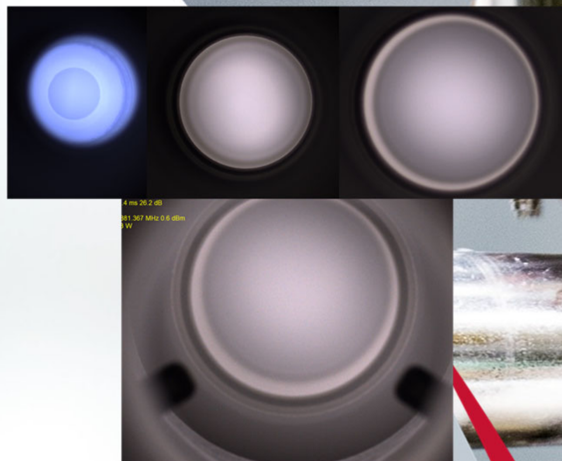




IN SITU PLASMA AND OZONE PROCESSING AT JLAB

2025 NP Accelerator R&D PI Exchange Meeting

Tom Powers

17 Nov. 2025

Jefferson Lab

U.S. DEPARTMENT of ENERGY

Description of the program

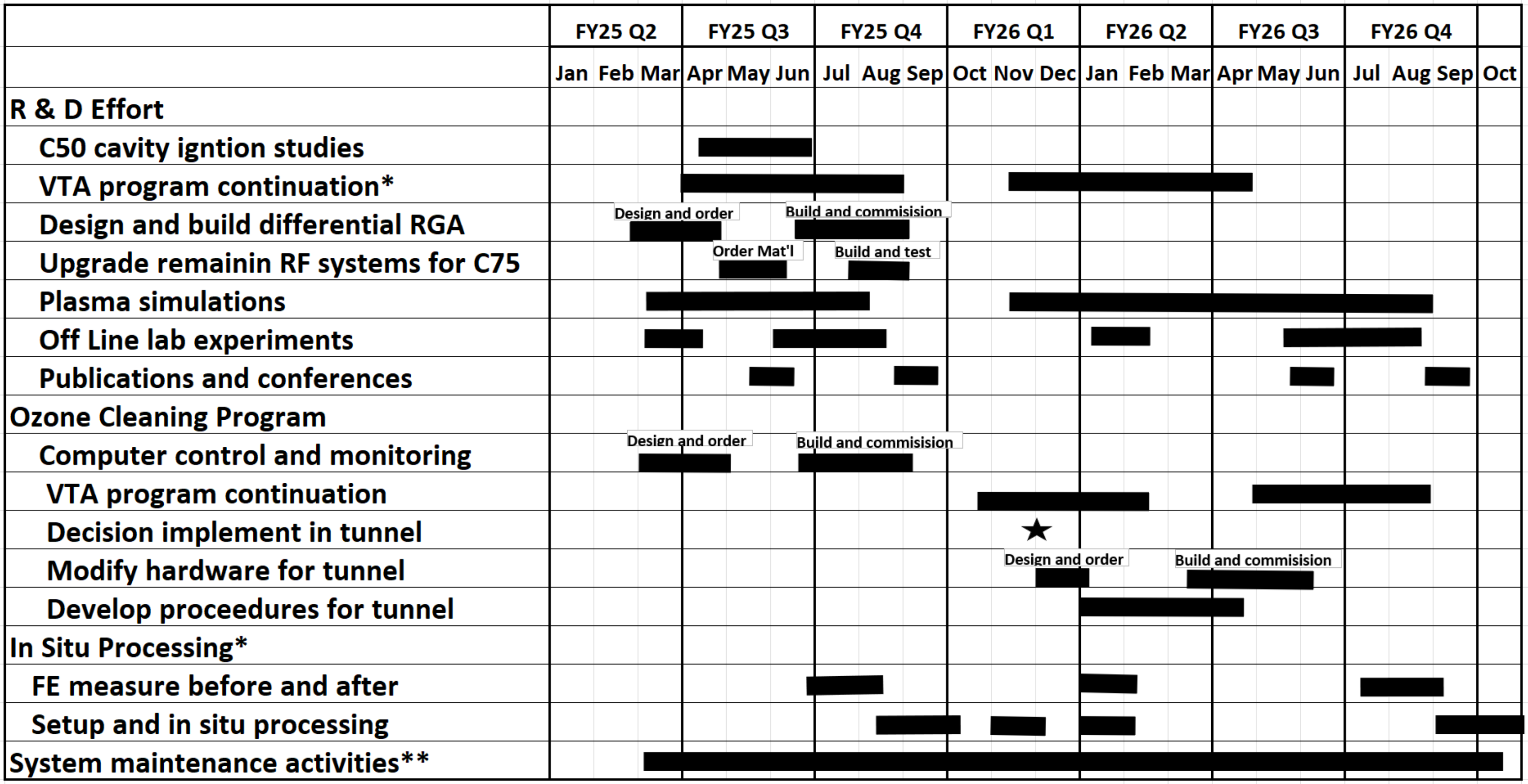
- Work done using JLAB internal accelerator R&D funds prior to this award demonstrated that we could establish and control the plasma generation in a CEBAF C100 cavity on a cavity-cell basis. Additionally, we used DOE R&D funds awarded in FY19, to build up a minimal set of hardware in support of the experimental program. As stated in the FOA proposal, these previously awarded funds were used to supplement this award in FY20.
- The program described in this presentation allowed JLAB to
  - Build up 6 RF test stands, three dedicated clean pump pumping, and two gas supply systems.
  - Maintain a robust vertical test – plasma process – vertical test program for process development.
  - Process 15 C100 and C75 cryomodules, 10 in situ in CEBAF and 5 in the test lab
  - Preform simulations with a goal understanding the gas dynamics of a plasma in CEBAF cavities.
  - Develop methods for in situ processing C100 and C75 cavities.
  - Investigate novel techniques for ignition of plasmas in SRF cavities at room temperatures.
  - To investigate the use of other gas mixtures, and processing techniques.
  - Continue the program through November 2026.

	FY25*	FY26	Totals
a) JLAB allocated	$453,000	$402,000	$855,000
c) Actual costs to date**	$415,000	$37,000	$452,000

Recent progress

- There was a pause in funding between November 2024 and February 2025, which is when the current funding was added to the JLAB budget.
- Completed and ongoing work since March 2025
  - Added a dedicated differential RGA cart for use on “dirty” systems in the off-line facility.
  - Add a second 100 W amplifier and combiner network to 4 more RF racks, which allows us to process up to six C75 cavities simultaneously and improves the diagnostics on C100 cavities.
  - Started the process of training two new engineers as to all aspects of plasma processing.
  - Resume the vertical (cold cavity) testing program.
  - Developed a major revision to vacuum and gas handling procedures for use in CEBAF which added more rigor to process control.
  - Process processed a C75 cryomodule in situ in CEBAF (second one planned).
  - Built up a second ozone generation cart
  - Published a contributed paper on Plasma processing at the North American Particle Accelerator Conference.
  - Published a paper on ozone processing in Phys. Rev. Accel. Beams

Project Time-Line

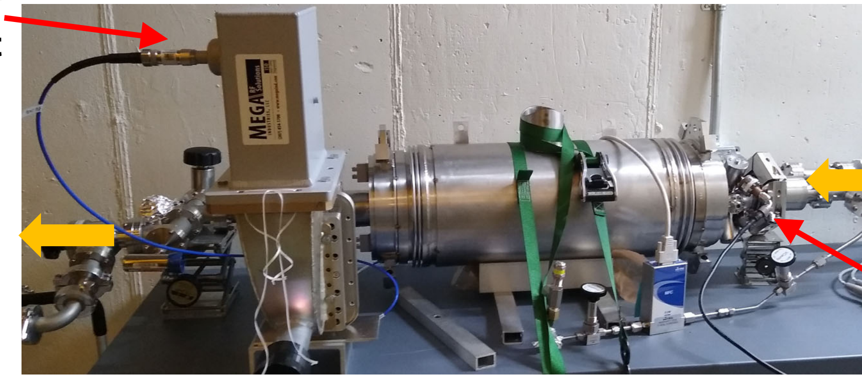


* Note VTA testing and in situ processing makes use of the same hardware. ** System maintenance is on an as needed basis

Reactive oxygen plasma processing

RF Monitor Port

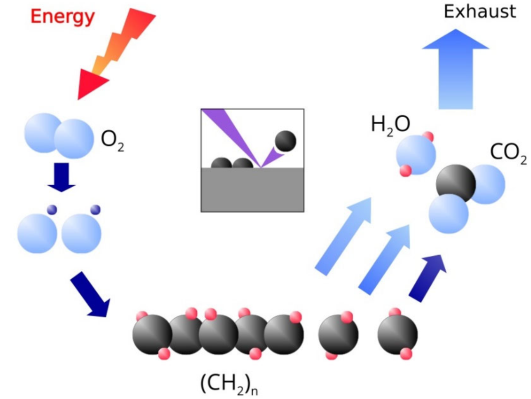
Process Gas With CO_2, CO and H_2O, etc.



Process Gas

RF Into HOM Port

C100 Off-Line Bench Setup



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

- **SRF "Standard" Recipe**
  - Room temperature mix of inert gas (argon, helium or neon) and a few percent oxygen
  - Flow gas through cavity at a few tens of standard cubic centimeters per minute
  - Pressure in the cavity between 50 and 300 mTorr
  - Apply RF (10 to 600 W depending on system, gas species, pressure, cell and cavity type) to ignite plasma in one cell, LCLS II and JLAB C100 via HOM ports, JLAB C20/C50/C75 and SNS via the fundamental power coupler.
  - Move from cell to cell by changing the RF frequency usually with two sources.
  - Maintain the plasma for 30 to 120 minutes in each cell
  - Monitor cracked hydrocarbon residuals of H, CO2, CO and H2O

Vertical test and off-line test facilities

- In addition to being used for process development the systems are being used as part of a staff development program with two engineers that were hired within the past year.

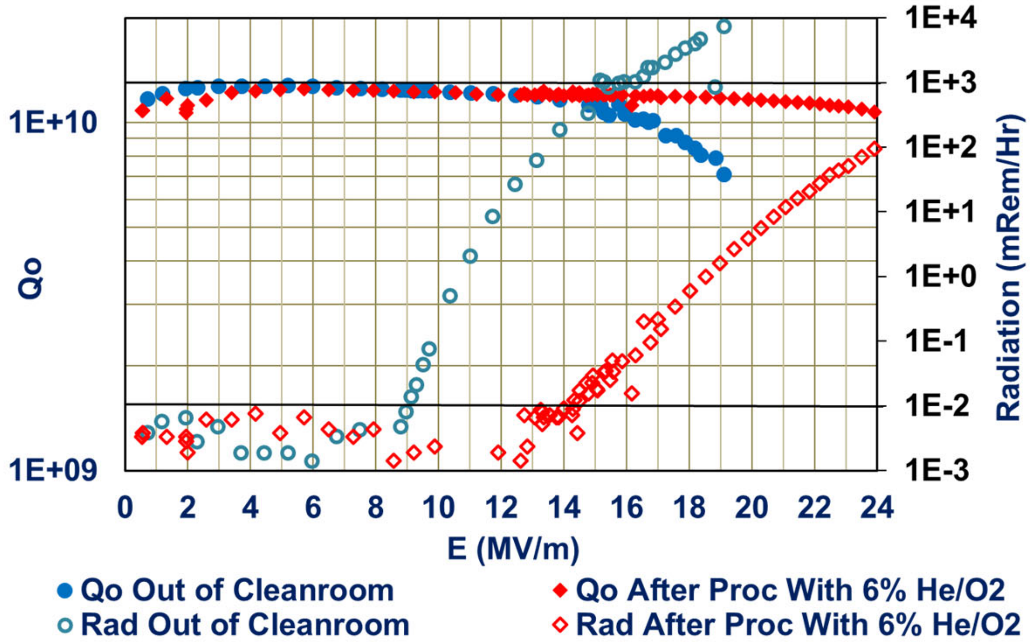
- We are currently experimenting with argon/hydrogen gas mixtures in addition to helium oxygen gas mixtures.

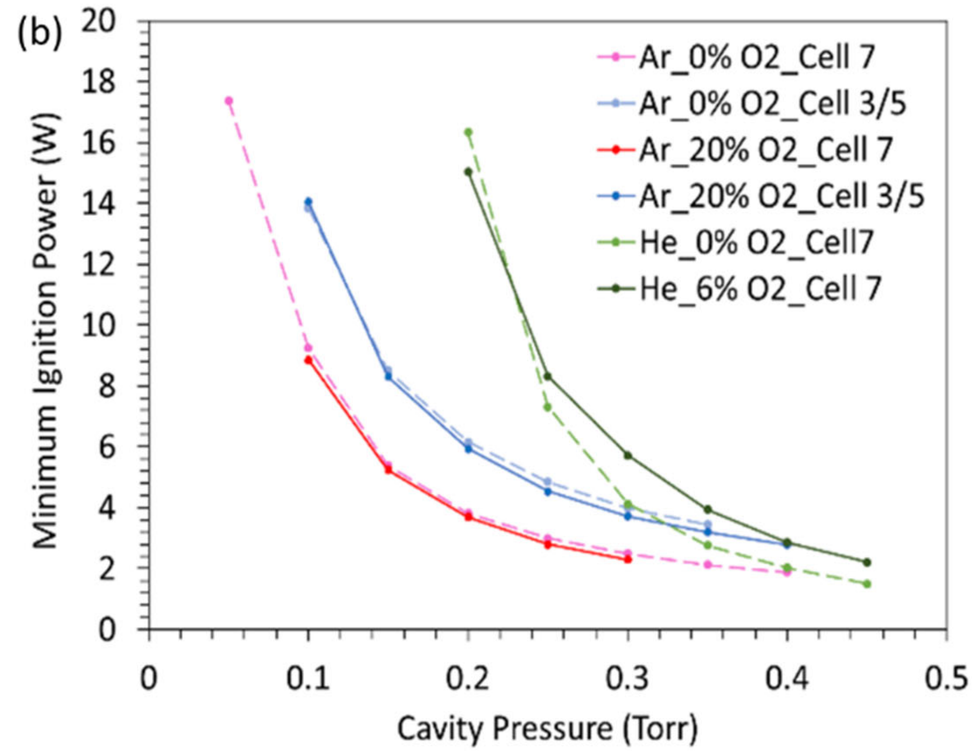
- Part of the effort is to have other individuals come up to speed with the details in the software tools which were developed by the PI over the past several years.

- Once they come up to speed the plan is get back into a one plasma process followed by vertical test cycle per week.

- This once per week cycle is possible because of the unique capabilities of the vertical test area which.
  - 6 shielded test dewars,
  - 4 dedicated low level RF systems covering a frequency span of 100 MHz to 6 GHz.
  - A stand-alone helium refrigerator system that is shared with the cryomodule test facility and the injector test facility.
  - Typically has multiple cavity tests at 2K per day.

Experimental results in VTA and in off line facility





VTA tests led to change from argon / oxygen gas mixture to helium oxygen gas mixture.

High school students did ignition studies in the off-line facilities as part of a summer internship program.

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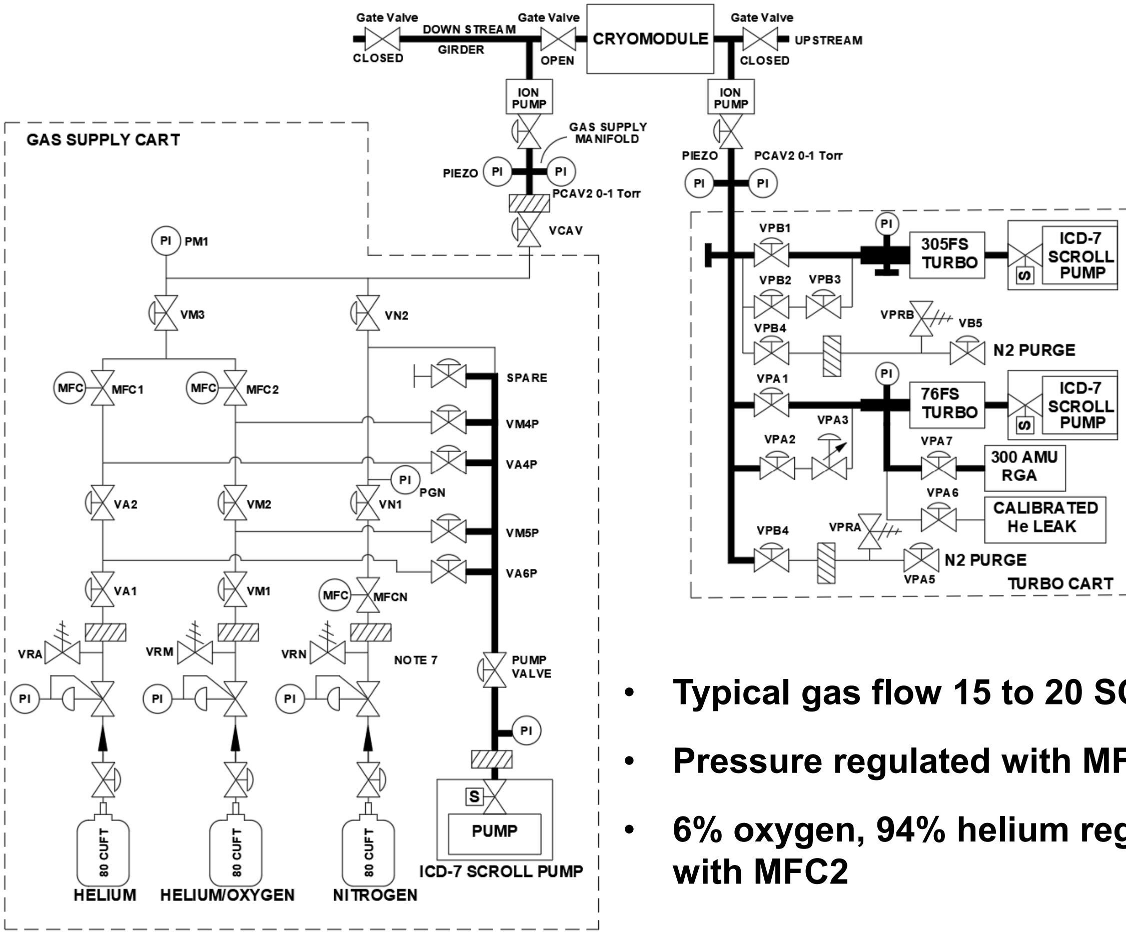
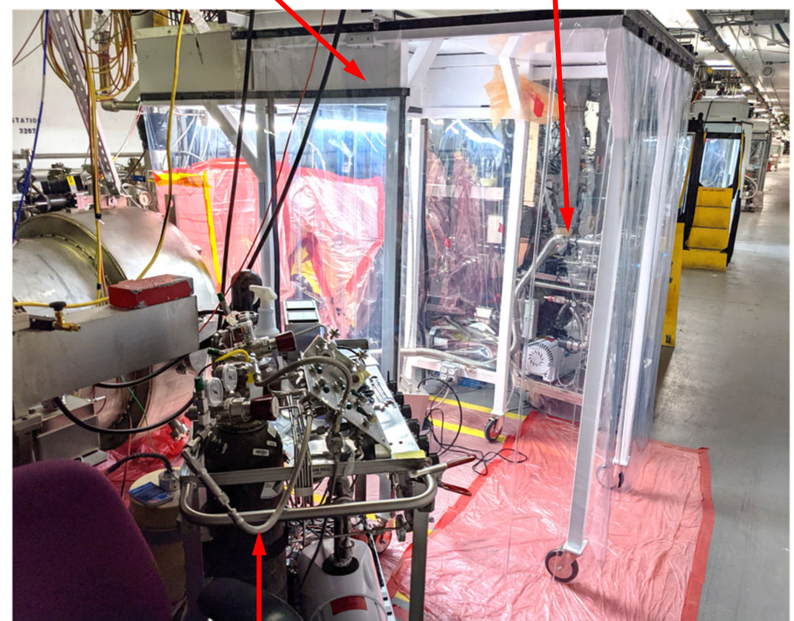
Plasma processing gas supply and pumping systems.



- Typical gas flow 15 to 20 SCCM
- Pressure regulated with MFC1
- 6% oxygen, 94% helium regulated with MFC2

Telescoping clean room

Turbo pump cart with differential RGA



Gas supply cart

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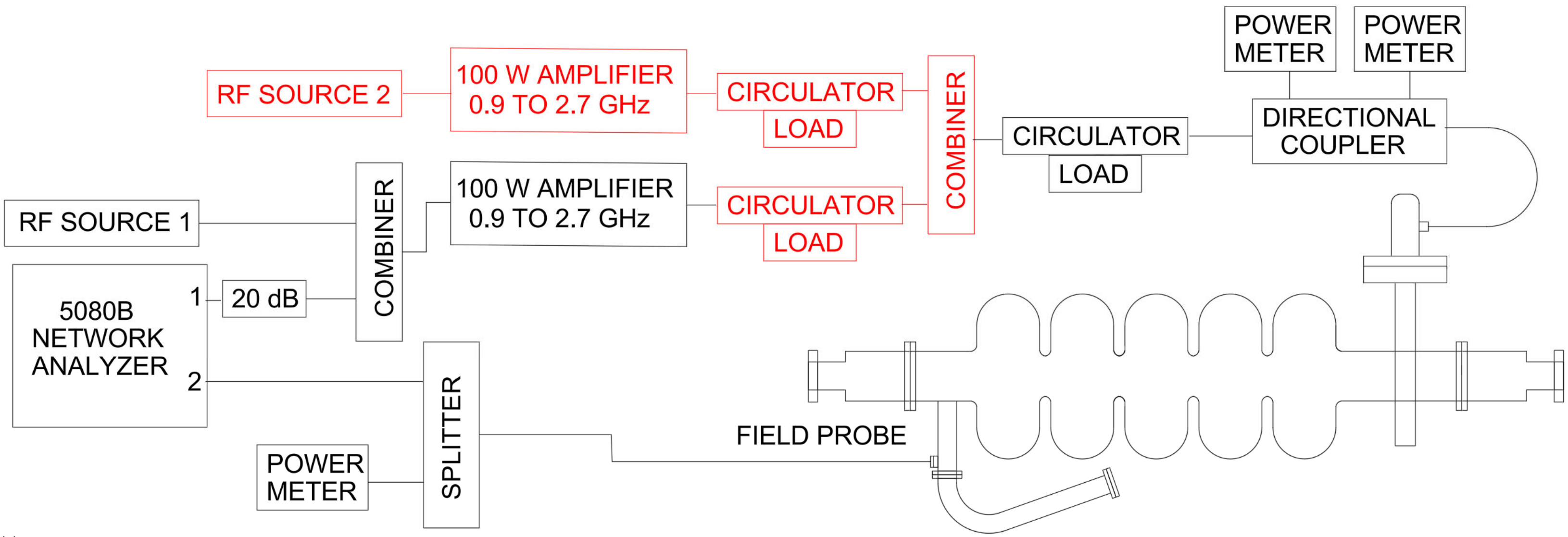
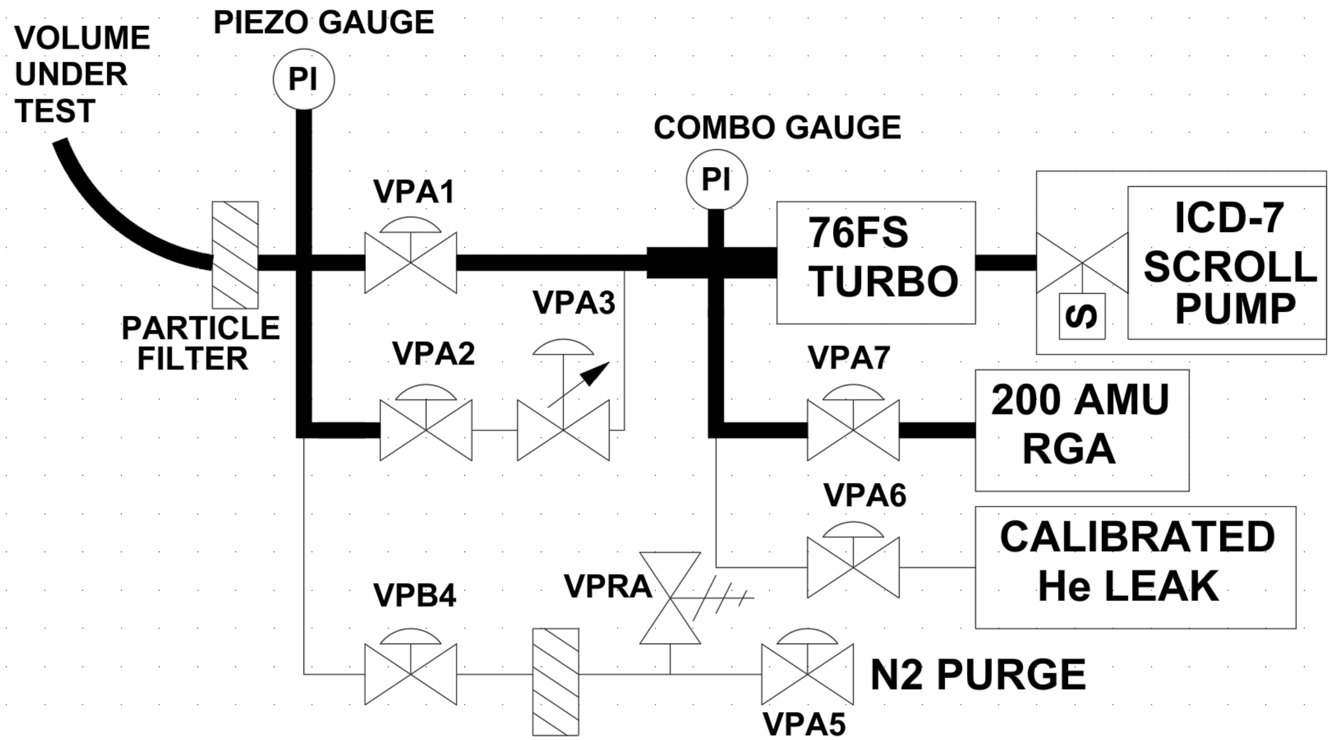
Two amplifier setup required for C50/C75 cavities



- **C100 cavities require about 15 W of RF power at the cavity for processing.**
- **C75 cavities require about 40 W of RF power at the cavity operating with two frequencies**
- **At increased RF power levels saturation effects, specifically intermodulation effects, cause the amplitude of one frequency to be reduced when the amplitude of the second frequency is increased.**
- **Adding a second amplifier eliminated the amplitude "cross talk"**
- **Initially only two systems were upgraded. The remaining systems were upgraded in advance of processing C75 cryomodules in the tunnel.**

DIFFERENTIAL RGA



- **A dedicated differential residual gas analyzer system was developed and built so that we could monitor gas content in the off-line system without having to risk contaminating the pumping systems used for plasma processing.**

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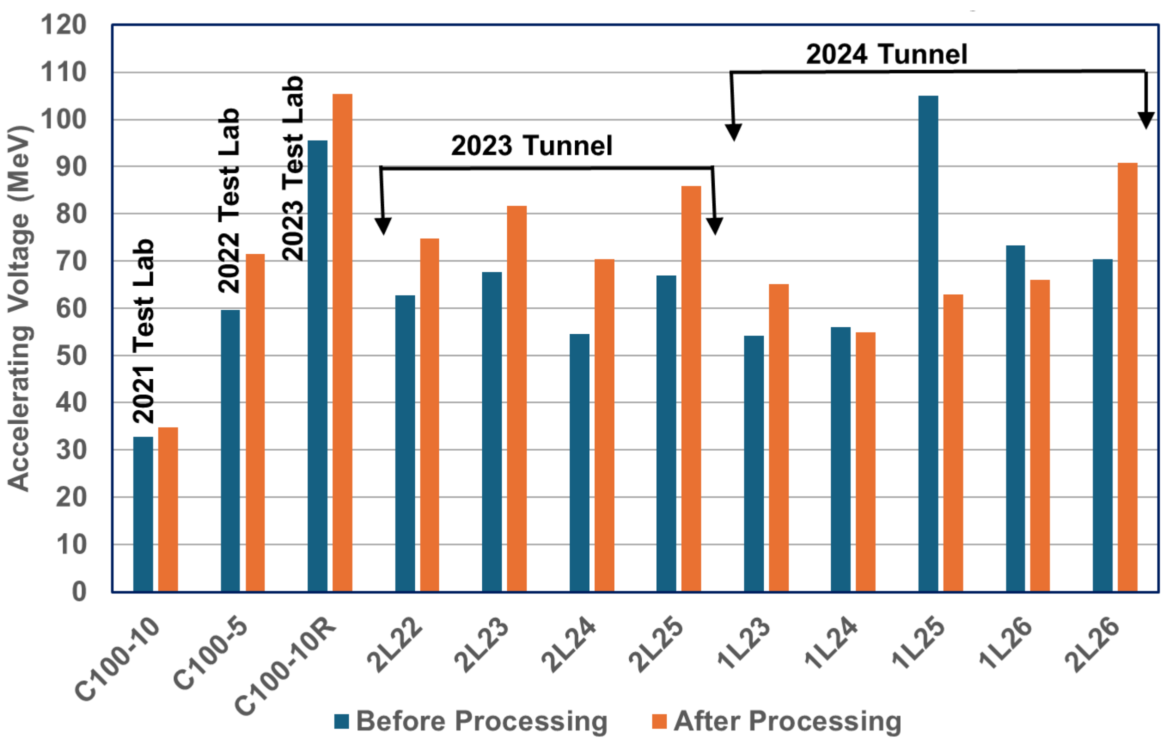
Plasma processing in the tunnel over the years.



Field emission onset before and after plasma processing

- **C100-10 was severely damaged during operations. It was operated for a year after a gate valve had a catastrophic failure.**
- **C100-5 was processed before disassembly it improved 12 MeV.**
- **4 cavities were processed C100-10R after it was rebuilt. It improved by 10 MeV.**
- **The cryomodules that were processed in situ in 2023 on average improved by 15 MeV.**
- **In 2024**
  - **There was a incident that degraded 1L25, 1L26 and to a lesser extent 1L24.**
  - **1L23 improved by 11 MeV.**
  - **2L26 improved by 20.5 MeV.**

***Note. Cryomodules C100-8R and C75-4 were processed in the test lab without determining FE onset prior to processing.**

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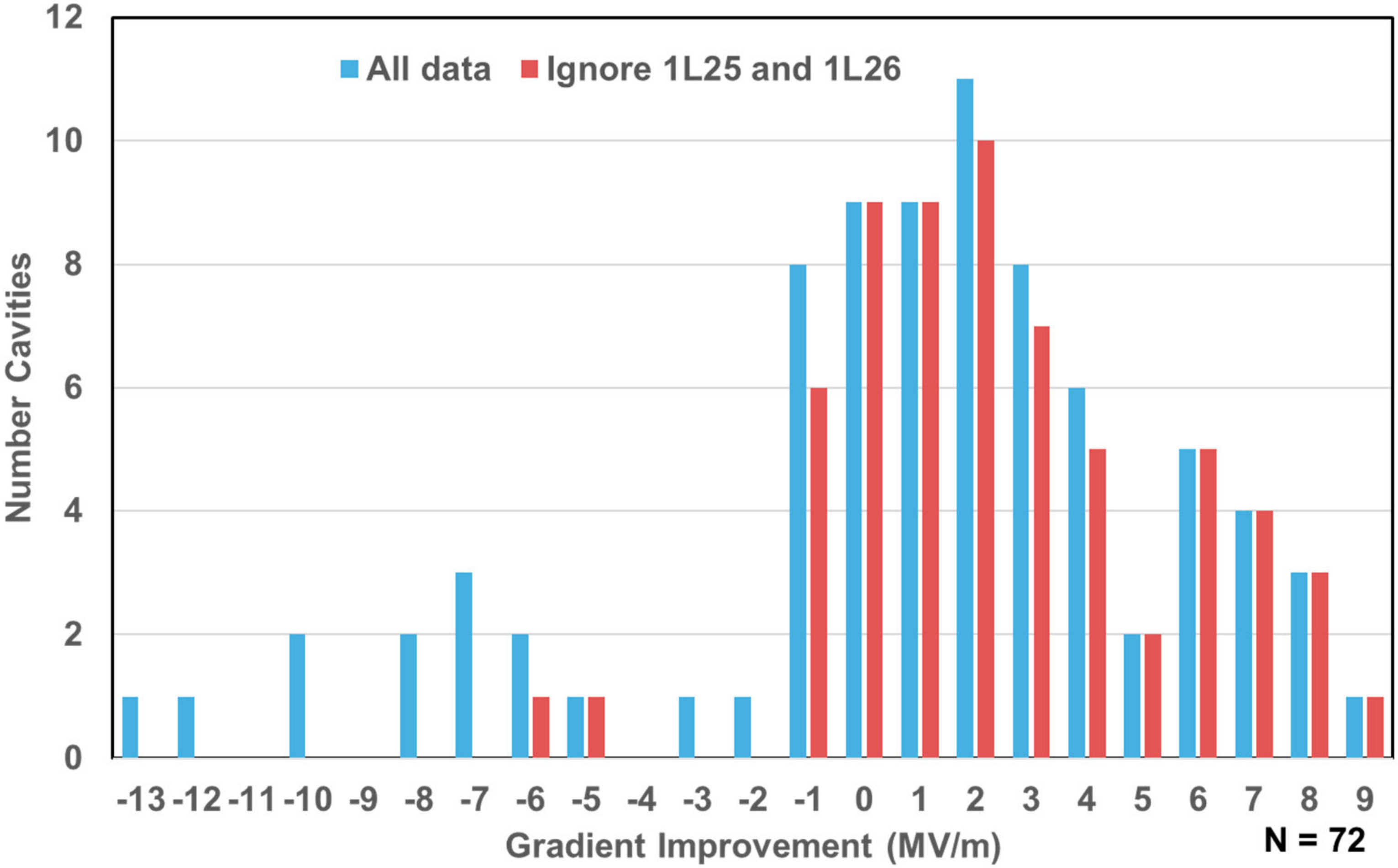
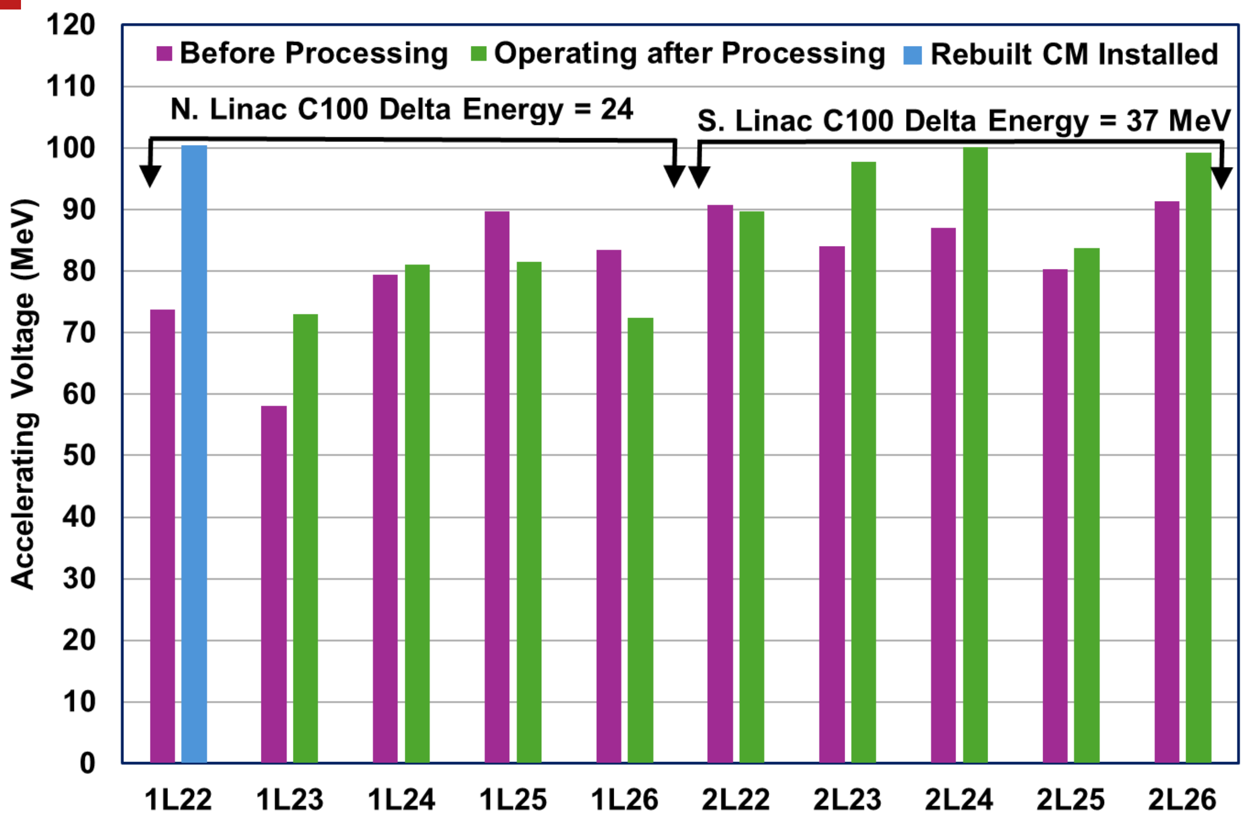
Distribution of FE onset improvement after plasma processing in cryomodules



- **Average improvement all cavities 1.82 MV/m**
- **Average improvement excluding 1L25 and 1L26   2.3 MV/m.**

Operating energy before and immediately after plasma processing



- **Field emission onset is a measurable metric for understanding processing results.**
- **The metrics that are important are cryomodule operating are:**
  - **Operating energy**
  - **Neutron production in the girder**
  - **Cryomodule trip rate**
  - **Cryogenic heat load**
- **The 4 NL CMs the that were processed lost 2.5 MeV.**
- **Including the rebuilt 1L22*, the 5 C100 cryomodules in the NL improved by 24 MeV.**
- **These operating gradients were set using the same neutron or lower dose metrics.**

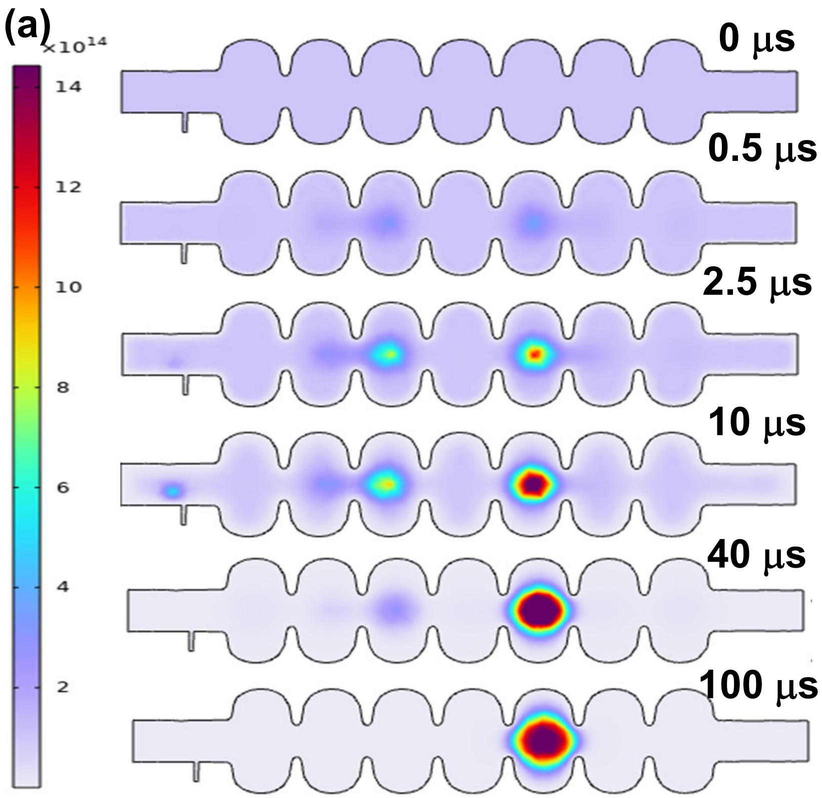
***Note: 1L22 is C100-8R which was plasma processed in the test lab prior to installation.**

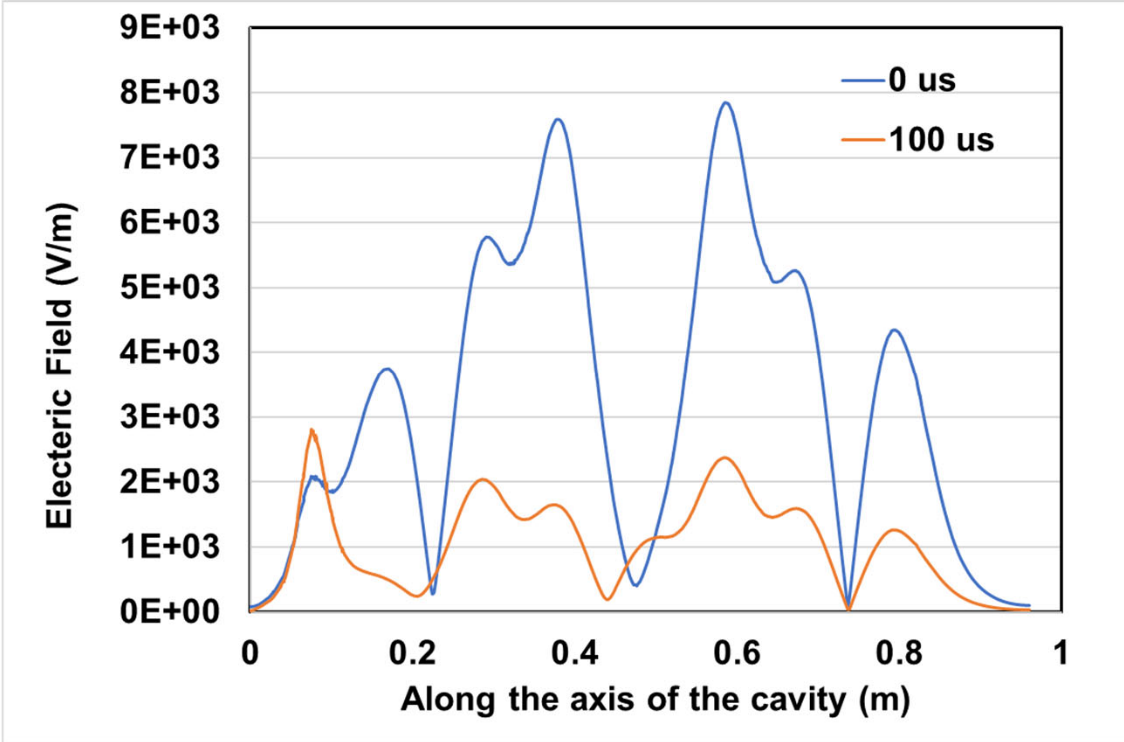
Corrective Actions

- The matter was reviewed both internally and as part of an external review.
- A number of changes in protocols, procedures, etc. were implemented in order to substantially reduce the probability of similar problems in the future.
  - Administrative tagging system (same level of control as lock out tag out without safety implications) applied to all beam line valves that were required to be open or closed for plasma processing.
  - Valves that were required to be opened had the pneumatic gas supply tubes swapped so that they could not be closed without swapping back.
  - Procedures were modified to require a visual inspection to ensure that all tags are in place and all valves are in the required states prior to introduction of gas into the system.
  - Procedures included a check list for all critical parameters.
  - An interlock was implemented in the mass flow control software to ensure they could not be opened unless the beam line valves were in the proper state.
  - The revised procedures included specific instructions regarding actions to be taken in off normal conditions.
- Procedures and processes were reviewed prior to restart of plasma processing in the tunnel and staff were trained to the new procedures.

Using COMSOL Multiphysics software with plasma and RF options

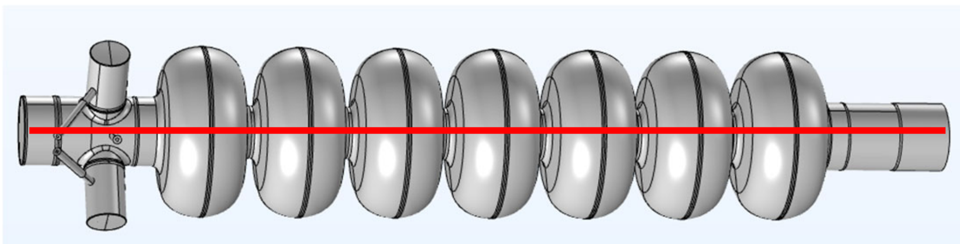
Changes in electric fields due to the presence of plasma

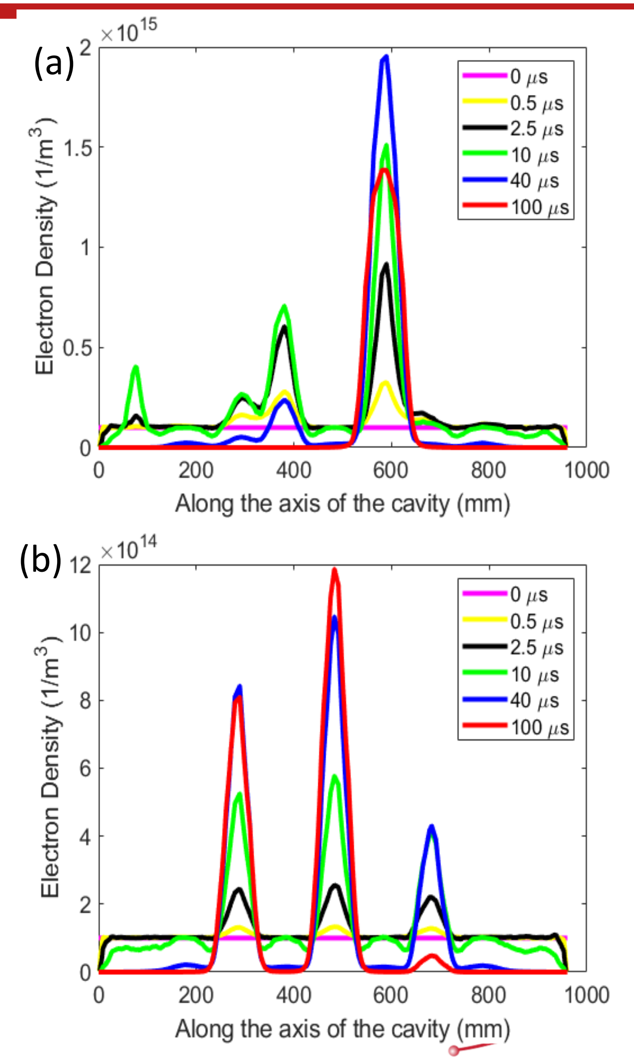




Electron Density as a function of time.

Electron density as a function of time along the axis



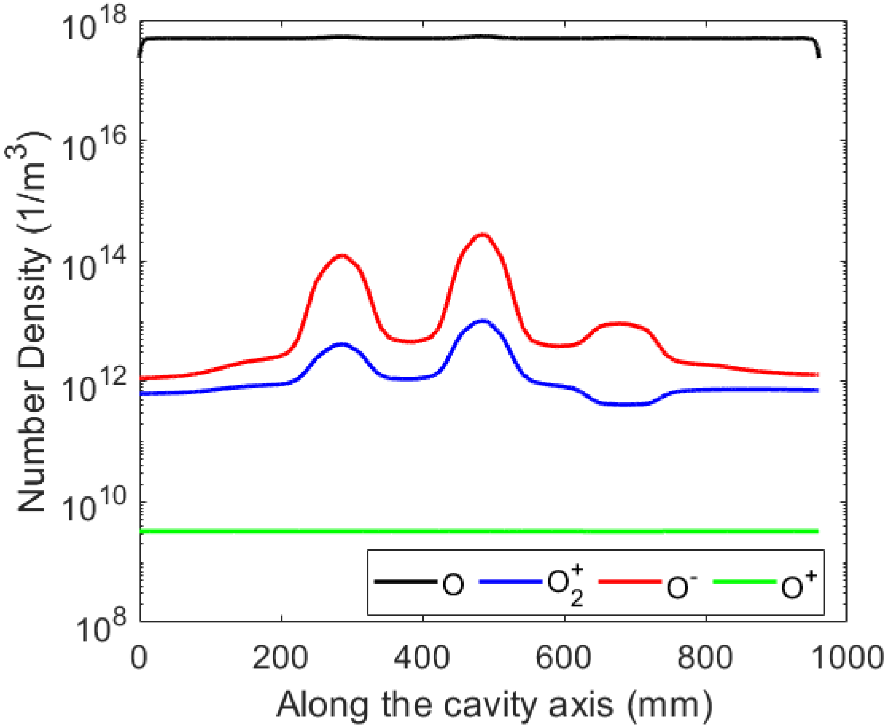


Electron density at plasma ignition with time along cavity axis
(a) cell 3/5, 1947 MHz and

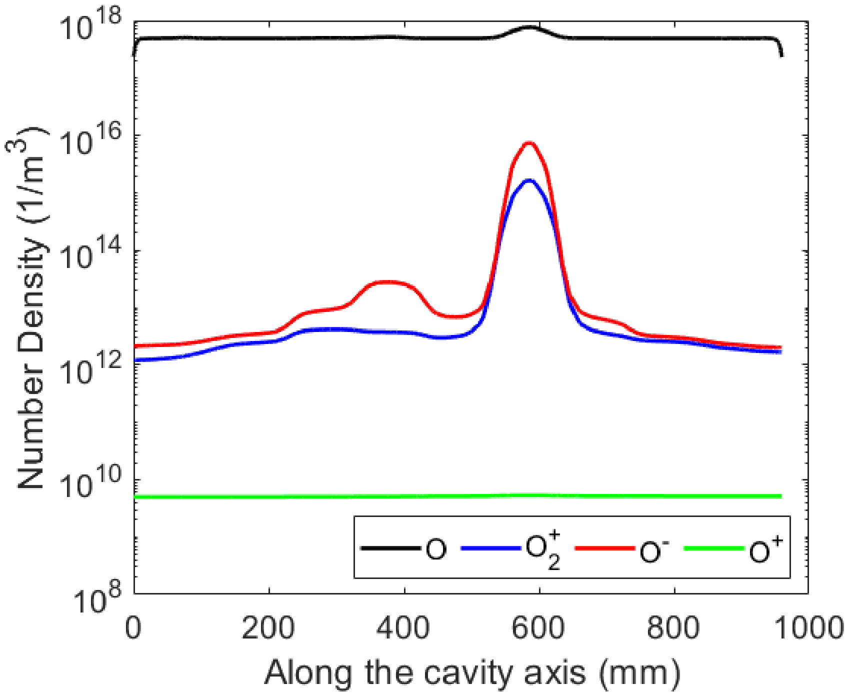
(b) (b) cell 2/4/6, 1980 MHz

(ignition power 5 W, 6 % O2 and gas pressure 0.15 Torr).

Oxygen species along the axis.



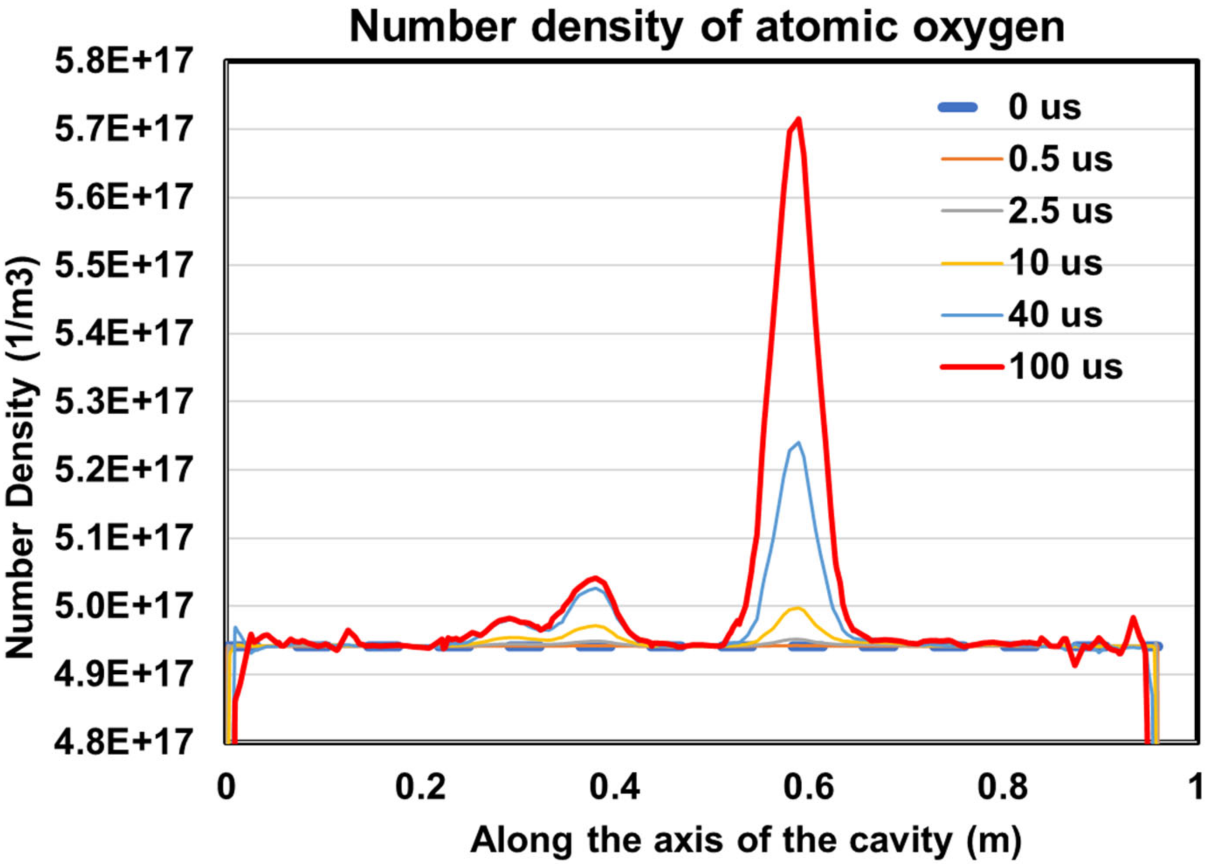
Cell 2, 4, 6 and 5 mode



Cell 3 and 5 mode

Free oxygen looks amazingly well distributed.

Maybe not something is goofy with the simulation



- It seems that the initial conditions had to much atomic oxygen.
- We tried adjusting it and a few other species downward and the initial part of the simulation crashed.
- **To be continued.**
- This will be part of a PhD student's research project.

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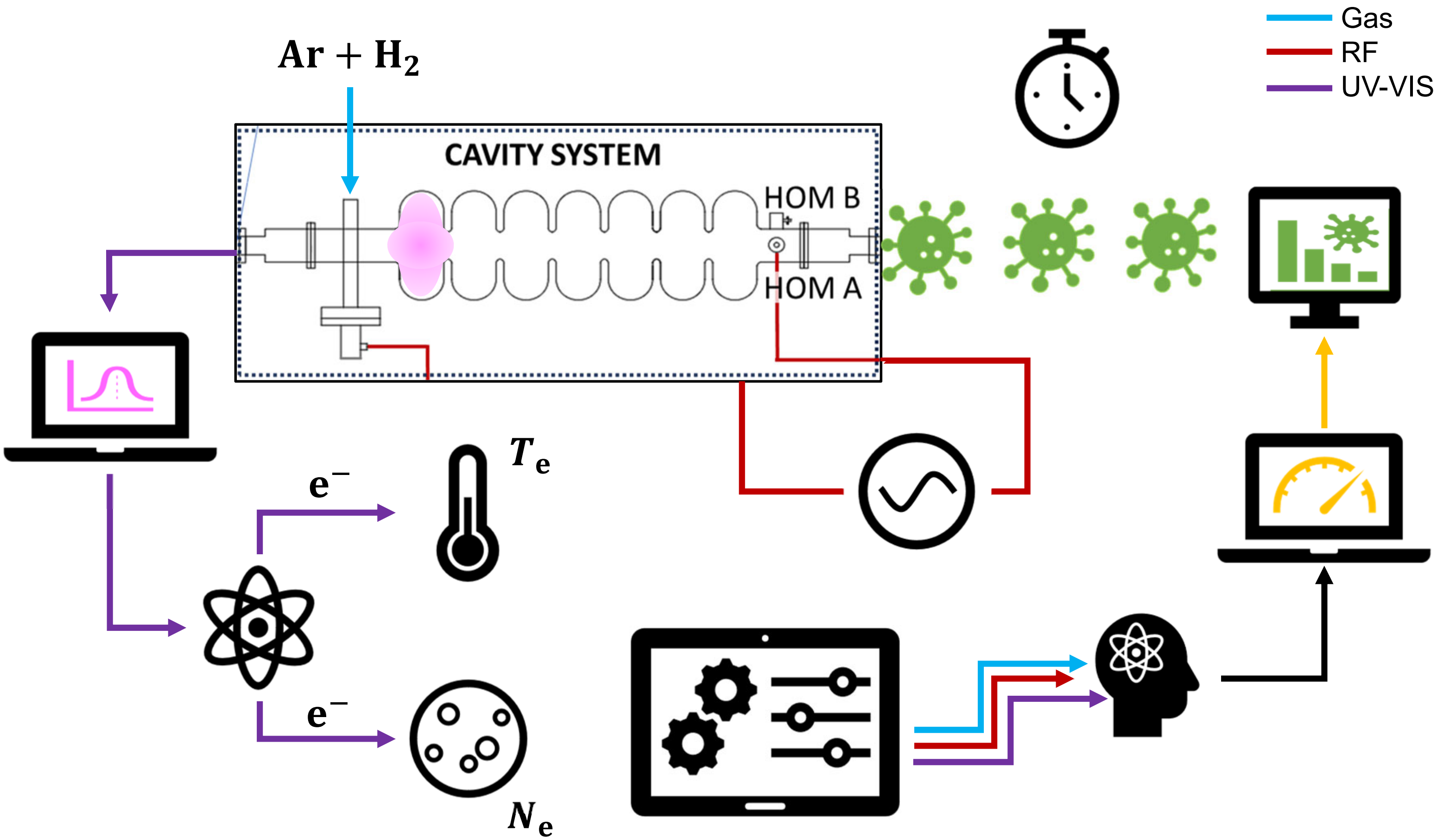
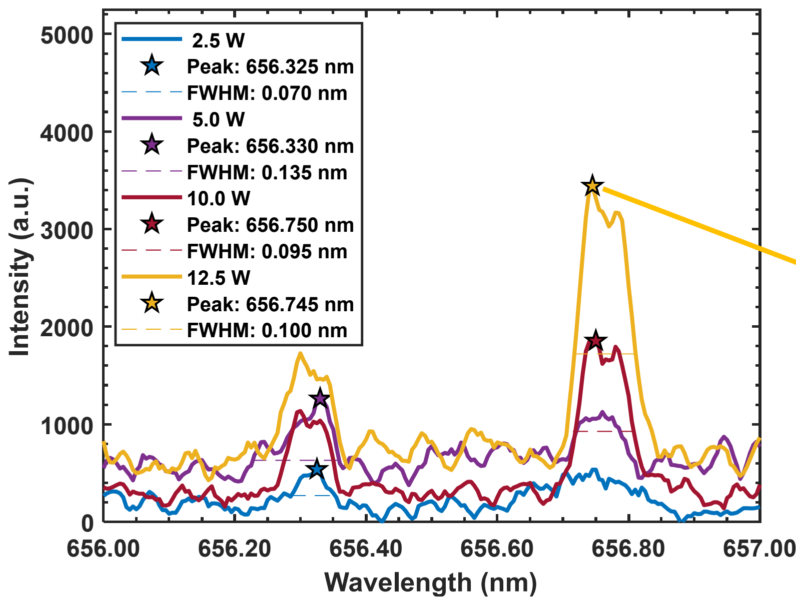
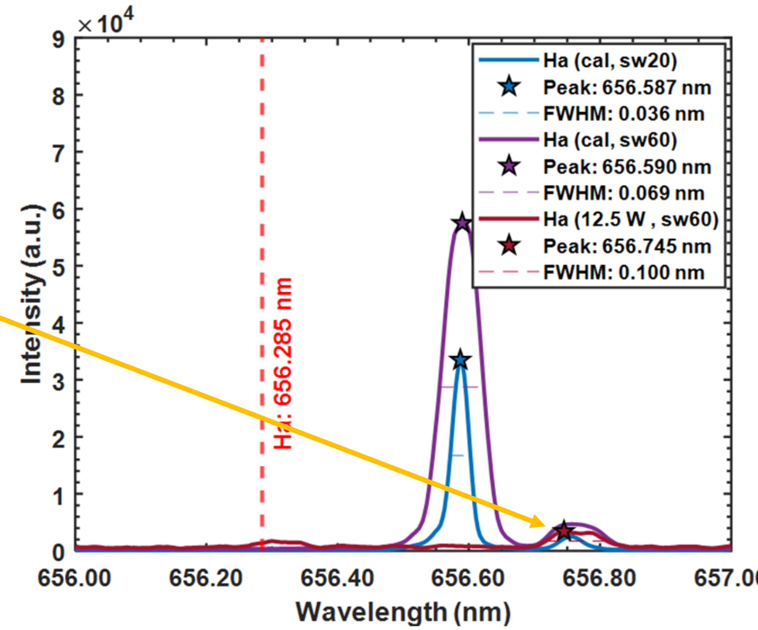
Masters degree research project.

- ❑ Generation of Ar + H_2 cavity plasma
- ❑ Identification of Balmer lines (H_α , H_β)
- ❑ Deconvolution of spectra (Gaussian, Lorentz)
- ❑ Decomposition of spectral broadening
- ❑ Derive e^- density and temperature
- ❑ Long term goal plasma processing optimization

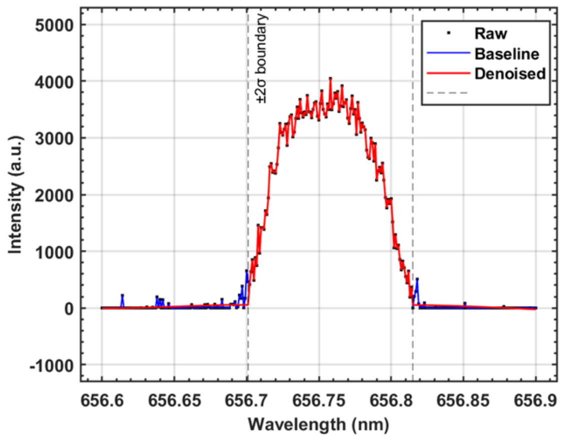


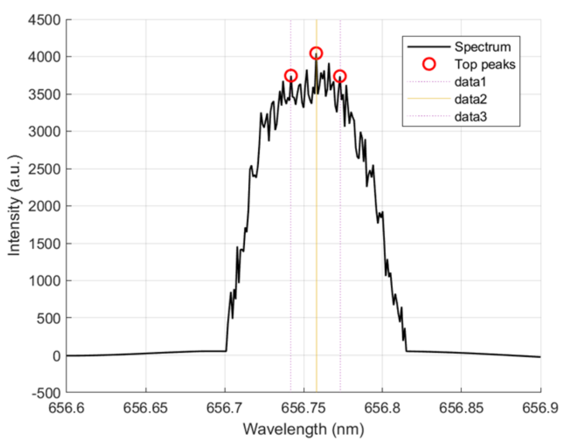
Line Identification: Balmer H_{α}

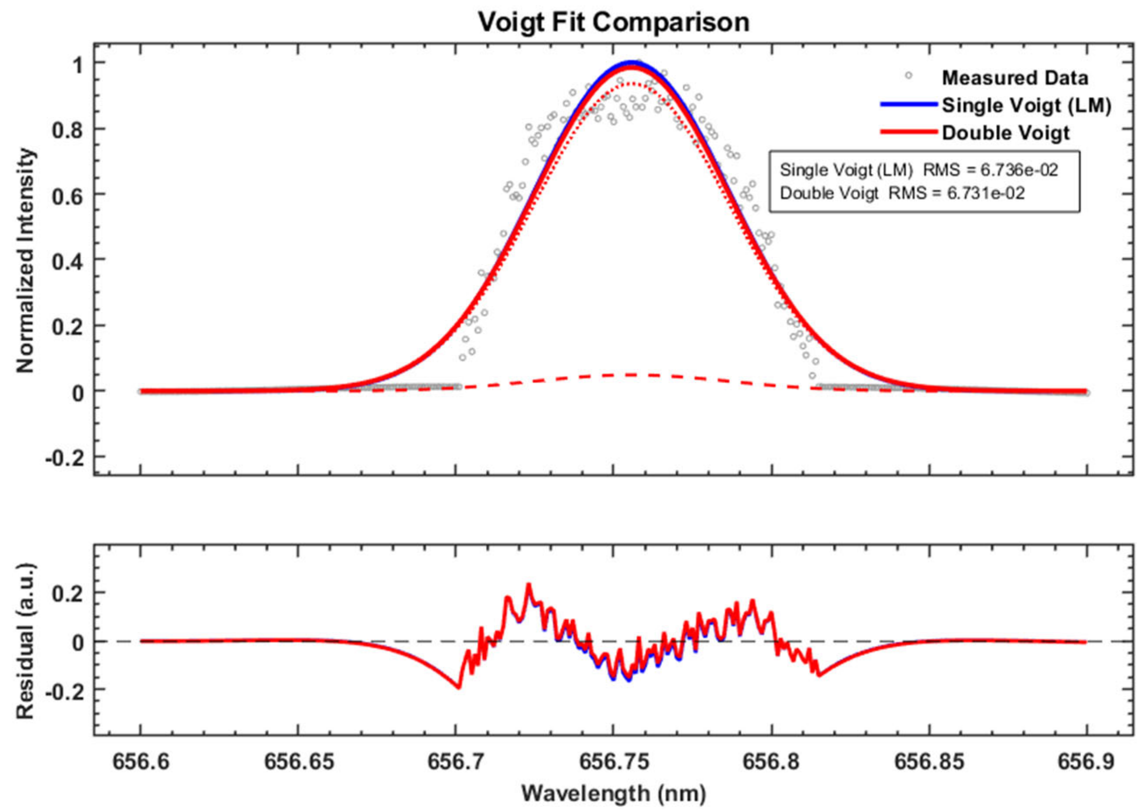




Process and Fitting: Balmer H_α

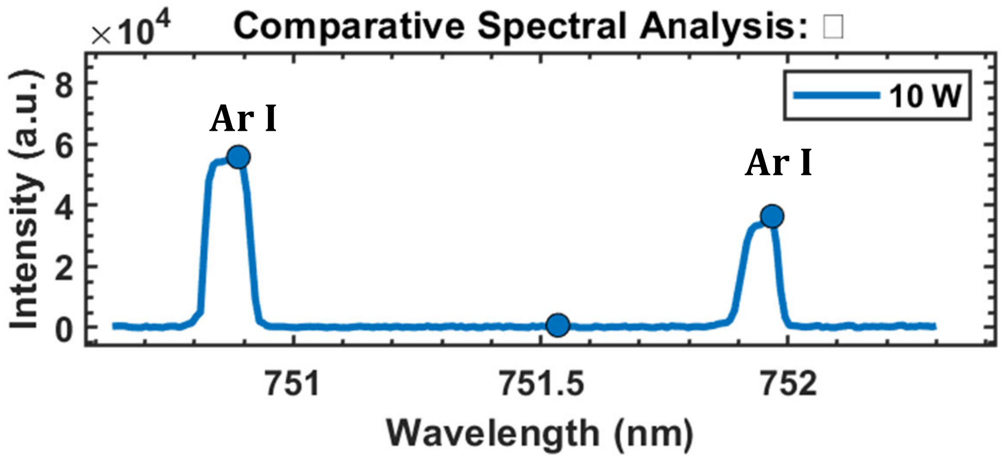






Approximated Gas Temperature

Line-Intensity Ratio Method
(Boltzmann Relation)



$$\frac{I_1}{I_2} = \left(\frac{\lambda_2}{\lambda_1}\right)\frac{g_1 A_1}{g_2 A_2}\exp\left(-\frac{E_1 - E_2}{kT_g}\right)$$ [1]

$$T_g = \frac{E_1 - E_2}{k \ln\left(\frac{I_1 \lambda_1 g_2 A_2}{I_2 \lambda_2 g_1 A_1}\right)}$$

First estimate for initial guess

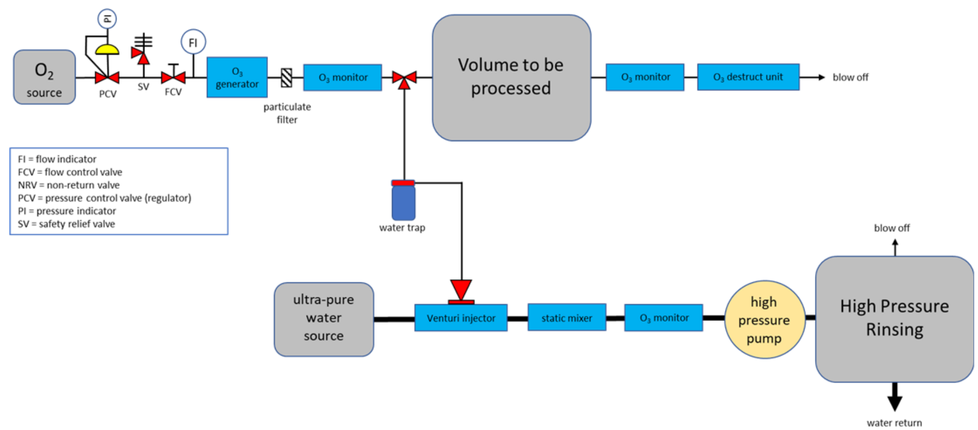
$$\boldsymbol{T_g \approx 7800\ \mathrm{K} \quad (0.62\ \mathrm{eV})}$$

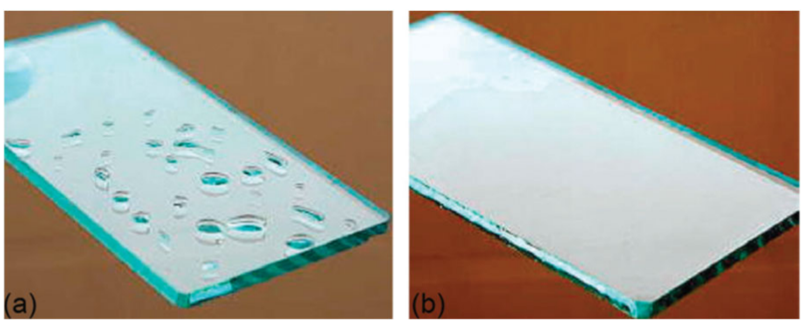
[**NIST** reference values]

Ozone Cleaning

R. Ruber, *Reducing field emission in superconducting cavities with ozone*, Phys. Rev. Accel. Beams 27, 122001.

- **Well-established technique** for removing biological material, hydrocarbons
  - first mentioned in the 1970's
  - using UV radiation in air or low-pressure oxygen (<mTorr)
  - UV also desorbed gases from the vacuum chamber walls
  - used at Brookhaven Nat. Lab. for NSLS-2 vacuum chambers
- **Ozonized water cleaning** used in semiconductor industry since 1990's
  - remove organic impurities from the wafer surface





Glass plate before (a) and after (b) UV-ozone cleaning for 1 min. No water beads on cleaned surface.
R. Kohli, *UV-Ozone Cleaning for Removal of Surface Contaminants*, In Developments in Surface Contamination and Cleaning, Vol. 8 (2015).

First Results of Ozone Processing

C75-RI-032 Ozonized High-pressure Rinse

- O3HPR: 4 h rinse, 49 °C, 2 ppm ozone

C75-RI-032 In Situ Ozone Processing

- In situ O_3: 6 h, 7 wt%, 1 l/min, 84 °C

Postulate

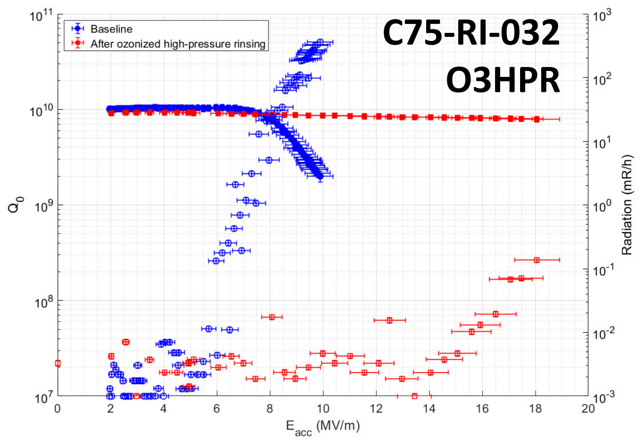
- Ozonized HPR and in situ O3 processing are effective in removing hydrocarbons

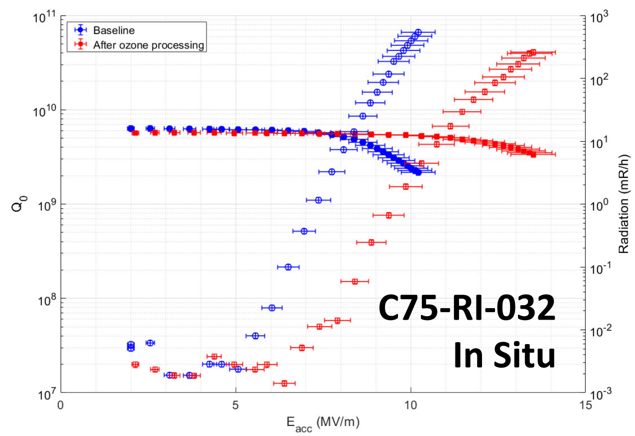
C100-RI-086 In Situ Ozone Processing

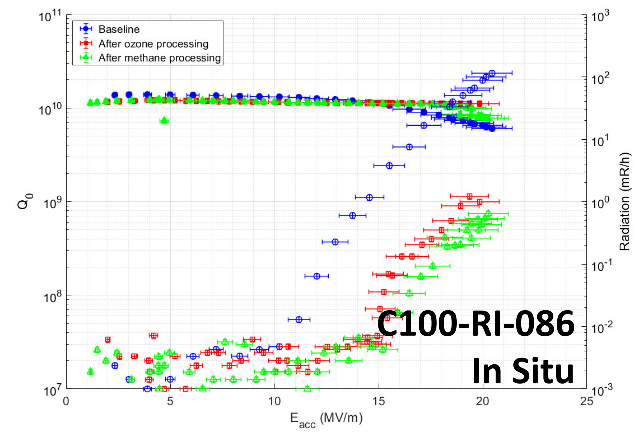
- In situ O_3: 6 h, 6 wt%, 1 l/min, room temp.
- Methane processing (Ar/CH_4)
- **Methane processing was not effective after ozone processing**

Hypothesis

- Ozone reacts with NbO_x to form a Nb_2O_5 surface impeding adsorption of hydrocarbons







Accomplishments Since November 2024

- **Published and presented results**
  - R. Ruber,
    Reducing Field Emission in Superconducting Cavities with Ozone,
    Phys. Rev. Accel. Beams 27, 122001 (2024).
    DOI: 10.1103/PhysRevAccelBeams.27.122001
  - seminar at JLab
  - invited talk at TTC 2025, Daejong
  - contributed oral at SRF 2025,
    - *cancelled due to rejected travel application*

- **Next steps**
  - test new cart, new ozone area monitors
  - continue sample studies
  - continue tests on cavities and cryomodule parts

- **Continuing studies**
  - with summer student, examined effect of ozone cleaning of niobium samples contaminated with hydrocarbon layer (thin oil residue)
    - results published in JLab Tech Note
  - testing effect on
    - ion pumps
    - ferrite used in HOM loads

- **Preparing 2nd ozone cart**
  - procured parts and assembled
    - digital flow controller and pressure sensors (1st cart all manual valves/analogue sensors)
  - passed pressure system authorization
  - upgrading ozone area monitors

Summary

- **Ozone treatment.**
  - **Is an effective cleaning agent removing organic, hydrocarbons, and some inorganic, e.g. sulfur**
  - **Similar effectiveness as plasma processing.**
  - **It seems to have a global effect on cavity's interior**
  - **Simple and promising cleaning method for all cavity shape and other vacuum chambers**

- **In situ plasma processing**
  - **Is a proven method to improve cavity performance in SRF cavities.**
  - **There was a setback in 2024 due to a vacuum incident.**
  - **The procedures and processes were modified in order to apply more rigor to the process and to include engineered controls in the systems.**

- **Effort in the CEBAF and for cavity production is transitioning from R&D funding to nuclear physics operations funds.**

- **Plasma and Ozone R&D and training of new staff will continue under the existing grant through Oct. 2026.**

Acknolegements

JLab staff, especially SRF Ops, SRF S&T, ES&H;

Ozone Cleaning:

Eric Lechner, Volker Ziemann, Anne-Marie Valente-Feliciano, Mauro Taborelli, Sergio Calatroni, Yoshiyuki Morita, Tafafumi Okada, Michiru Nishiwaki;

Plasma Processing:

JLAB – Plasma team, Tom Powers, Harshani Senevirathne, Bashu Khanal, Eugene McGough

JLAB – VTA support, Tommy Doyle, Anthony Malave-Colon, Justin Kent

JLAB – Machine vacuum support, Frank Humphry, Josua Thomason, Tony Sessoms, Greg Marble

JLAB – Operator support, Eric Dier, James Howard, Katheryne Price, Ben Schaumloffel

JLAB – SRF clean room and cavity assembly team.

ODU – Zach Caudell, Professor Chunqi Jiang

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