## **Plasma Processing At Jefferson Lab**



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#### **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 is allowing JLAB to
  - Apply more labor to the project
  - Obtain specific support from ORNL, which is a leader in plasma processing of SRF cavities.
  - To build up the infrastructure necessary for processing multiple cryomodules in the CEBAF accelerator simultaneously.
  - Increase the vertical (cold cavity) testing program.
  - Process cryomodules in the SRF test lab and, as programmatic limitations permit in the CEBAF accelerator.
  - To develop methods for in situ processing C50/C75 cavities which do not have external HOM couplers.
  - To 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 Sept. 2022

	FY20*	FY21	Totals
a) JLAB allocated	\$607,500	\$607,500	\$1,215,000
b) ORNL allocated	\$93,500	\$93,500	\$187,000
c) Actual costs to date*	\$471,274	\$0	\$471,274



#### **Deliverables and schedule**

	FY21 Q1	FY21 Q2	FY21 Q3	FY21 Q4	FY22 Q1	FY22 Q2	FY22 (	<b>Q</b> 3	FY22 (	<b>Q</b> 4
	Oct Nov Dec	Jan Feb Mar	Apr May Jun	Jul Aug Sep	Oct Nov Dec	Jan Feb Ma	r Apr May	Jun	Jul Aug	Sep
Complete existing RF/vacuum										
systems					Modify / Upg	rade				
Continue off-line process										
development and optimization										
Apply argon/oxygen processing										
to C20/C50/C75 cavities										
Investigate novel techniques for										
plasma processing										
SNS/ORNL Small Sample Surface										
Science Studies of JLAB methods										
Process cavities in VSA/VTA										I
Procure and build up RF systems,	,									
for dedicated cryomodule										
SNS/ORNL cryomodule										
experience and lessons learned										
Procure/prepare vacuum										
hardware for processing CM.										
Process cryomodules in Test Lab										
(opportunistic, schedule)										
Process cryomodules in CEBAF										
(opportunistic, schedule)										
Tenative										
publications/conferences										
JLAB, Accelerator R&D PI Exchange	30 Nov. 202	21								

#### **Overall summary, progress since Oct. 2021**

- Built up a second gas supply and pumping system.
- Built up and commissioned a third RF system for use in the vertical test area and for processing cryomodules in the test lab or in the tunnel. Additionally built up two half rack systems which will turn each of the RF systems into 2 channel systems.
- Completed modifications to a vertical test stand and performed 7 full cycles of RF test, plasma process, RF test cycles in the vertical test area.
- Completed an extensive number of bead pull and ignition studies to understand the effects of having an open cable on the second HOM coupler.
- Developed software, for processing multiple cavities simultaneously in a cryomodule. New features
  included automatic plotting of mode shifts, characterizing effects of phase shift of cable on second HOM
  coupler, etc.
- Completed field emission onset and EMAX measurements, plasma processed and did a second set of field emission onset and EMAX measurements on cryomodule C100-10 in the cryomodule test facility.
- Upgraded the main turbo pump on cart 1 to 300 L/s pump and procured a second pump which will be used to
  upgrade the original vacuum system.
- We procured some UV and VUV micro discharge excimer lamps and developed a packaging scheme which includes a nitrogen purge.
- We started procuring ozone production equipment and are developing concepts for application to cavities and warm beamline hardware.



#### **Overall Summary, Issues**

- Remote working and limited staff on site due to Covid restrictions, slowed progress, especially until vaccine was widely available.
- Availability of clean assembly resources is limited due to overall staffing levels, Covid "events" and higher priority projects, e.g. HE, PPU, CPP programs. This limited the VTA testing throughput.
- 10 month delay on-boarding a senior staff member due to covid related visa issues.
- Vacuum failure on a pump cart uncovered a design "weakness" which was rectified. At the same time one of the turbo pumps failed and had to be returned to the factory for repair. Fortunately we had a spare pump on hand.
- The effects of the strong coupling between the HOM antennas and impacts of slight differences in cable lengths within a cryomodule required significant effort to understand.
- The cavity used in the VTA testing program had reduced field emission onset after plasma
  processing the seventh time, leading to a reconfiguration of the test stand and a review of our clean
  assembly protocols.
- Cavity used for vertical testing has yet to perform well after light EP. (Early FE onset.)
- The principle investigator is out for 1 2 months starting Oct. 21 due to surgery.



#### Why Use Plasma Processing

#### **Industrial Uses**

 Plasma processing is a common technique for removing hydrocarbons from surfaces and improve the wettability of the surface.

Number of Cavities

- 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.\*



SNS Improvement in Operating Gradient After

Plasma Processing Average 2.5 MV/m, N=32

Cavity index \*Marc Doleans personal communications



#### **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 small 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<sub>2</sub>0, C0<sub>2</sub> 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.\*





\*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



#### **Reactive Oxygen Plasma Processing**



C100 Off-Line Bench Setup

- SRF "Standard" Recipe
  - Room temperature mix of inert gas (argon 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 200 mTorr
  - Apply RF (5 to 600 W depending on system, gas species, pressure and cell) to ignite plasma in one cell, LCLS II and JLAB C100 via HOM ports, JLAB 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 60 minutes in each cell
  - Monitor cracked hydrocarbon residuals of CO<sub>2</sub>, CO and H<sub>2</sub>O



#### **JLAB Vertical Test Stand Setup**



PLASMA PROCESSING PUMP / RGA CART

CAMERA

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- the RF based approach for determining plasma location.
- Exhaust gas was monitored with an RGA

#### **Using S21 Measurement to Characterize and Locate Plasma**

We use a method similar to that developed at Fermilab where 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.



Mode Pattern for cells 1/2 and 3/4 ignited.



- 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.
- Initially we looked at a live S21 plot. Then both a baseline and a live plot. Then we added a feature to our system where 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**

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

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

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









#### **Vertical Test Results**



- Initial clean room cycle.
- Initial field emission (FE) onset was poor at 5.5 MV/m
- First processing cycle improved FE onset by 0.5 MV/m and operating gradient at 100 mR/hr by 1 MV/m
- Second processing second test had low Q but similar FE onset. It was later determined that this was due to poor magnetic field control during cooldown.
- Third test of second processing cycle had similar results to the first processing cycle.



- Second clean room cycle.
- Initial FE onset 12 MV/m
- After first processing the FE onset and 100 mR/hr gradients improved by 2 MV/m.
- After second processing FE was eliminated but the quench gradient was reduced from 20 MV/m to 15.5 MV/m.
- Positive Q slope indicates the effects of nitrogen doping from 6 years ago are still present.
- Afterwards the surface was reset using electropolish.



- Third clean room cycle.
- FE onset 11 MV/m.
- No improvement after first plasma processing.
- Severe degradation of Qo and FE onset after second plasma processing. This was interpreted as surface contamination by normal conducting particles.
- The suspicion is that the sintered metal gas filter failed during a cryocycle.
- Test stand reconfigured and filter was moved to the top plate.



#### Why all of the bead pull work

- During the early development stages and in the VTA, all of the plasma processing that we have done has been by injecting RF power into one HOM port with the other port unterminated.
- A cryomodule has RF cables connected to each of the HOM ports. Although possible, it is not practical to disconnect one of the cables prior to processing.
- Unlike LCLS-II / ILC cavities the HOM couplers on C100 cavities are both on the same end of the cavity. This
  means that there is a strong broad RF coupling between the 2 HOM coupler antennas.
- Early on we did an experiment where we put a 10 foot cable on the second port and found that it was difficult to establish a plasma in all of the desired modes.
- The cable and the second HOM port act as a "stub tuner" affecting the RF properties of the cavity.
- The cables used in cryomodules are built with a specified length of 10 feet +/- 1 inch. Using a velocity factor of 0.83 it is 0.4 ns two way travel time. That works out to a range of about 280° of two-way phase shift on the signal that returns to the unused HOM coupler.
- Two approaches considered.
  - Put a phase shifter at the end of the unused cable and optimize the phase shift for a balance of gradients at the different modes.
  - Use a splitter and drive both HOM ports in parallel with a phase shifter in one of the paths.
- Things are complicated by the fact that RF signals might be coupled directly from one HOM port to the other.
- We have to pick the phase setting using only network analyzer measurements, no cameras, no bead pulls on a cryomodule.



#### S11 and S21 Injecting Into One HOM Port, Port 2 Open



- Cryomodule C100-10 cavity 6, with waveguide window. Input HOMA port output FPC 10' cable on HOMB port with phase shifter set to 260°.
- TE111 Modes, 1870 MHz to 2040 MHz, have uniform fields with peak electric fields in the center of the cells.
- TM110 modes, 2120 MHz to 2230 MHz, have two dipole modes each anchored by the orientation of one of the HOM antenna positions. These modes have non-uniform electric fields with peaks at the irises.
- TM110 modes require 2 to 5 times more RF power to for plasma ignition.
- S11 being a large negative number means you are putting power into the system
- S21 being a large positive number means that you have RF fields in the cell nearest the FPC.



#### **Coupling Between HOM Ports Calibrated S11, S21**





# Bead Pull Results\* With RF Splitter and Phase Shifter (Just Phase not relative gradient)





- Three consecutive phase shifter settings about 6 degrees per step.
- 2000 point network analyzer sweep taken at each step.
- As expected because of constructive destructive interference the system is very sensitive to phase setting.
- Difficult to get one phase setting where all modes work well.

\*The amount of phase shift is proportional to the magnitude of the electric field at the location of the bead. Plasma Processing at JLAB, Accelerator R&D PI Exchange 30 Nov. 2021



#### Bead Pull Results With Phase Shifter at the End of "CM" Cable. Maximum and Minimum Mode at About 1886 MHz



Phase and relative gradient as a function of bead position and frequency.



- Plots on the left are phase shift as a function of bead position.
- Plots on the right are relative gradient which includes S11 information.
- Slowly varying modes (peak electric fields) as a function of phase shifter setting.
- Several phase positions where all of the modes have strong electric fields. Thus we can use one phase shifter setting for all modes.
- There are some frequency bands where more than 90% of the power goes into the system and there is no measured fields within the cells.



## Cryomodule CM10 Plasma Processing Setup, June 2021

- C100-10 was removed from the machine for rebuild due to extremely poor performance.
- CM characterized before and after in cryomodule test facility.







- Network analyzer signal added to RF at low level in order to track frequency changes due to plasma.
- We were able to process three cavities at the same time.
- Due to coupling between FPCs we could not process cavities 4 and 5 at the same time.
- Standard protocol was to ignite in cell 3/5 then go to cell 4.
- From 4 we went to:

15 dB

32 dB

RF POWER METER

- Cell 7 then 6 then 5 or
- Cell 3 then 2 then 1

WR650 TO

TYPE-N

• We used the plasma induced frequency shift patterns to determine which cell we were in.



- We removed the gate valves one of which was missing its O-ring.
- Gas injected into cavity 8 end of cryomodule.
- Pump cart is connected to the valve on the ion pump on the cavity 1 end.

## **C100-10 Processing Protocol**

- Record S11 / S21 data as a function of open phase shifter position for the modes used for processing.
  - Do S11 calibration using a dummy cryomodule HOM cable, which is the same type and length cable that is inside the cryomodule.
  - Do S21 response calibration using a single cryomodule HOM cable.
  - Calibrate the phase shifter stepper monitor steps for 5° phase shift.
  - Connect phase shifter and to HOMB port with a short cable.
  - Record S11 and S21 for each 5° position (72 files, 20,000 points each)
- Select phase shifter position to provide "acceptable" parameters for the 5 modes used.





## Comparing Re(S11) and S11(dB)



- The peak on the gradient scaling function  $(1 Re(\overrightarrow{S11}))$  is more-or-less at a constant frequency.
- The peaks in S11 that are changing in frequency do not have a significant value for the gradient scaling function.
- Expanding the vertical scale on the minus S11 plot and reducing the frequency range shows that there is a moderate amount of energy going into the system at the phase settings and frequencies where the gradient scaling function is largest.
- The minus S11 6 dB-ish values at "constant" frequencies and peak gradient scaling factors occur between 0 and about 35 phase shifter steps. After that the mode with a changing frequency takes over.



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## **C100-10 Processing Protocol**

- Process cells in the following order 7, 5/6, 3/4, and 1/2.
- Each cell or pair of cells was processed for 1 hour.
- Except for cavity 1 each cavity was processed twice with > 48 hours between processing cycles.
- Cavity 1 was processed continuously while we were doing the other cavities.
- In general hydrocarbon residues were released when cell 7 was processed with a lesser amount on the other cells.
- Cavity 8 was an exception in that cells 5/6 had an increase in hydrocarbon release.
- When looking at the data remember that we were processing multiple cavities at the same time, e.g. H2, CO, CO2 lines at 14:20.



![](_page_20_Picture_9.jpeg)

## C100-10 Results

#### Results

- Validated procedures and trained three staff members.
- We demonstrated that, with two channels of RF, we can process a cryomodule in 2 shifts and one shift if we use 4 channel. Further this could be done with one person on duty at a time.
- Processed 7 cavities two times.
- Processed cavity 1 six times.
- The average increase in field emission onset was 0.4 MV/m.
- The average increase in 100 mRem/hr FE levels was 0.3 MV/m.
- Cavities 5 and 7 had a moderate increase in FE onset.
- A number of cavities had moderate improvement in maximum gradient.
- The performance of cavities 1 and 8 was degraded.

#### Disclaimer

- This cryomodule was degraded when Viton O-rings on two valves had catastrophic failures. One was 2 m and the
  other was 30 cm away from cavity 1.
- After the second failure cavities 1 and 2 could no longer be operated stably in CEBAF. The cryomodule was operated for another year in this state.
- The plan was to remove the cryomodule from CEBAF and rebuild it after plasma processing was complete.

Cav	FE onset Before After		FE 100	mR/hr	E Max		
			Before	After	Before	After	
1	4.3	3.6	4.7	4.1	6.5	5.3	
2	5.1	5.3	5.7	5.9	8.0	7.8	
3	6.3	6.8	6.9	7.0	10.9	11.5	
4	5.1	5.3	6.3	5.7	7.8	8.1	
5	6.6	8.2	7.2	9.0	11.5	15.0	
6	6.5	6.4	7.1	6.8	9.3	9.9	
7	7.2	9.1	7.8	10.1	9.6	15	
8	5.6	5.0	6.2	5.5	6.9	8	

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#### What are we doing in the mid term future

- In VTA we will be experimenting with different percentages of oxygen as well as using the TM110 modes.
- We will try to schedule another C100 cryomodule before rebuild. This is complicated by the tight schedule in the energy reach program.
- We are starting the process of investigating ozone processing of vacuum surfaces.
- In the off line system we will be
  - Investigating the HOM tip glow phenomena
  - Trying to better understand all of the implications of the "modes" where the peak losses are a function of unterminated cable phase, e.g. where is the best phase to operate.
  - Use some methane / argon mix gas to lay down a layer of hydrocarbons so that we can have a controlled surface to experiment with. Once we get it to work we can use it as part of the VTA cycles to experiment with different methods.
  - Use an excimer micro discharge VUV lamp at 172 nm (9.6 eV) to seed the discharge or produce free oxygen to process the cells.
  - Continue to prepare to process a cryomodule in the tunnel during the summer down or in the test lab depending on the machine plans and the cryomodule refurbishment program schedule.

![](_page_22_Picture_11.jpeg)