

# Assembling NEG, Ion Pump and Bakeable BNNT Cryopump System to Reach XHV

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## Abstract

New accelerator initiatives require spin polarized electron photoinjectors. The gallium arsenide (GaAs) photoguns must provide high average current ( $\gg 1\text{mA}$ ) and a long operating lifetime. Its performance depends on our ability to improve vacuum inside the chamber. The Center of Injectors and Sources (CIS) at Thomas Jefferson National Accelerator Facility has nearly reached extreme high vacuum (XHV)  $P = 1 \times 10^{-12}$  Torr by combining Non-Evaporable Getter (NEG) pumps and ion pumps(IP) in the Continuous Electron Beam Accelerator Facility (CEBAF) polarized source. Obtaining XHV pressure requires careful material selection and preparation, and appreciation for pump characteristics, capability and limitations. Measuring pressure at XHV is also a challenge so gauges must be used that have low x-ray limits, and the x-ray contribution to the pressure must be measured. In this project we investigated cryopump technology to maintain an XHV on the electron gun to achieve  $P < 10^{-12}$  Torr, using a bakeable cryopump with mechanically attached Boron Nitride Nanotubes (BNNT), and the NEG-ion pump system. We also measured the x-ray limit of the extractor and bent belt-beam (3BG) gauge that we used to measure the pressure. Additionally we used the Monte Carlo simulation software MolFlow+ to model the pressure distribution in the chamber, using the expected outgassing rates, measured temperatures, and expected pump speeds for the NEG, ion and cryopumps. We found that the system reaches a pressure of  $1.6 \times 10^{-11}$  Torr with the cryopump off, and a pressure of  $2.7 \times 10^{-12}$  Torr with the cryopump on. We measured the same pressure as before: This first attempt at using BNNT cryopumping in addition to the NEG/ion pump system previously used at Jefferson Lab showed that we can achieve similar pressures with the prior technology, and that the limitations to ion pump speed at low pressures are not the limiting factor for chamber base pressure.

# Introduction

The Ion back-bombardment is the main cause of degradation of quantum efficiency (QE) of photocathodes in DC photoguns. The rate of QE degradation depends strongly on the rate of ion production which is a function of the vacuum condition within the gun. The CIS is investigating cryopump technology to achieve an extreme high vacuum (XHV) in the electron gun [1]; extending the operating lifetime of the GaAs photogun strongly depends on our ability to improve vacuum. Cryopumping is a potential alternative to Ion pumps and could improve the base pressure when combined with Non-Evaporable Getter (NEG) pumps. The goal is to obtain lower pressure compared to today's level and make a comparative pressure measurements. Not only obtaining XHV is a challenge but also measuring it, because the limit to the lowest measurable pressure may not be caused by the pressure in the system, but by an x-ray effect in the gauges. In order to compare the experimental measured results, we used to model the pumping performance in the Monte Carlo simulation software Molflow+.

# Methodology

## Cleaning

To reduce the surface outgassing rate, all the surfaces and components inside the vacuum chamber need to be rigorously cleaned. The surface of the components may have impurities such as finger oil, traces of lubricants and other air-borne particles from fabrication, manufacturing and handling [2].

Our general cleaning procedure is described below:

Mechanical cleaning using isopropanol swipe to remove any visible dirt. Soak the pieces in detergent for 5 minutes and then put it in a ultrasonic cleaner, for 15 minutes with detergent and another 15 minutes in deionized water to rinse it. Finally mechanical cleaning

using methanol swiipe.

Clean work is a must for ultra-high vacuum, all parts must be thoroughly cleaned before installation and must be installed with grease-free gloves. During the entire cleaning and assembling process, we need to be very careful of not touching the components with bare hands or unclean equipment.

## **Assembly**

Our system consists in a vessel similar in size to an electron gun chamber, with ten NEG pumps inside, an ion pump and a cryopump.

**NEG. The SAES SORB-AC Non-Evaporable Getter Wafer Modules WP 950.** (5600 *l/s* total) These pumps have been designed to maximize hydrogen and active gases pumping speed. Each module consist of a laminated getter-coated metallic strip forming an array of parallel fins which is laminated with reactive elements to make a getter pump. The gettering materials used are a Zr-V-Fe alloy.

The first task for this project was finding a way to mount ten WP 950 getter pumps into a chamber, which required machining a mounting frame then assembling the array inside the vacuum chamber. This had to be done cleanly using gloves and a tyvek cleanroom jacket to avoid getting any oils into the system. The WP950 modules have to be securely mechanically mounted and electrically isolated, since it is necessary to pass current through them to heat and prepare them for pumping.



(a) WP950 modules

(b) Mounting the NEG array

(c) 10 NEG array

Figure 1: Images of the WP950 modules before and after being mounted into the chamber.

**Ion Pumps.**(35  $l/s$  for hydrogen) Provide a clean, simple, low maintenance method for producing and maintaining high and ultra-high vacuum, and are used throughout the CE-BAF accelerator to both maintain vacuum and monitor pressure since the Ion pump current is proportional to the system pressure. Ion pumps work by using an electrical, ionizing discharge which is maintained under vacuum conditions, and chemically active metals, such as titanium. The discharge is called a Penning Discharge, after its discoverer, F.M. Penning in 1937 [3].

**Bake-able BNNT cryopump.** BNNT has a molecular-sieve structure giving the material a high surface area —  $300m^2/g$ . It also has a high thermal conductivity of  $3,000W/mK$ , remains strong in air up to  $800^\circ C$ , and is resistant to thermal oxidation up to  $920^\circ C$ .5 It is

stronger in these aspects than carbon nanotubes. It is being explored as an alternative to the conventional charcoal (from coconut fiber) for the cryosorber surface for cryopumps [4] since it is freestanding and can be mechanically mounted to the cryopump instead of glued like charcoal. This allows the system to be baked out fully and may extend the range of cryopumps into the XHV pressure range.

This is the first test of this material as a cryosorber.



Figure 2: BNNT on the fin array.

Every time we are going to bake a system, we need to be sure that we are using silver plated bolts and gaskets. The silver plated gaskets resist corrosion at the high temperatures of a vacuum bakeout, unlike copper gaskets which oxidize and can contaminate a system with copper oxides. Using silver plated vacuum bolts avoids the welding of similar materials under the heat conditions. Inside the vacuum chamber, stainless steel screws in a stainless steel nut will weld together, but the silver will act as a lubricating layer and prevent this. For the vacuum hardware connecting the conflat flanges, the steel bolts would again react with the water in the atmosphere at high temperature and corrode, with the silver again

providing a dissimilar metal to allow the system to be disassembled following bakeout.

Before you begin assembly be sure you have all the components prepared (gaskets, nuts, bolts and tools). This is our procedure for seal each flange joint:

Try to touch the gasket only around the outer edge (where it will not be in the vacuum). If it touches a dirty surface, throw it away (in recycle) and get a new one. Finger tight a couple of bolts in the bottom of the flange and carefully drop the gasket in until the gasket is properly seated. If the gasket is not in place at the first try(which happens a lot), you can, if you have a rotateable, just turn until get there, if is a no-rotateable, you can wiggle a little bit the joint or use a gasket-pusher(ruler) to get the gasket is properly in.

When the joint is held together by the screws and nuts, we are ready to tighten. You want to make sure that the seal is even all the way around, so you will go around the flange as may times as you feel comfortable. We use a “star-shaped” pattern for tightening, where the next screw you tighten is as far away as possible from the screws you just tightened as shown in figure 2.

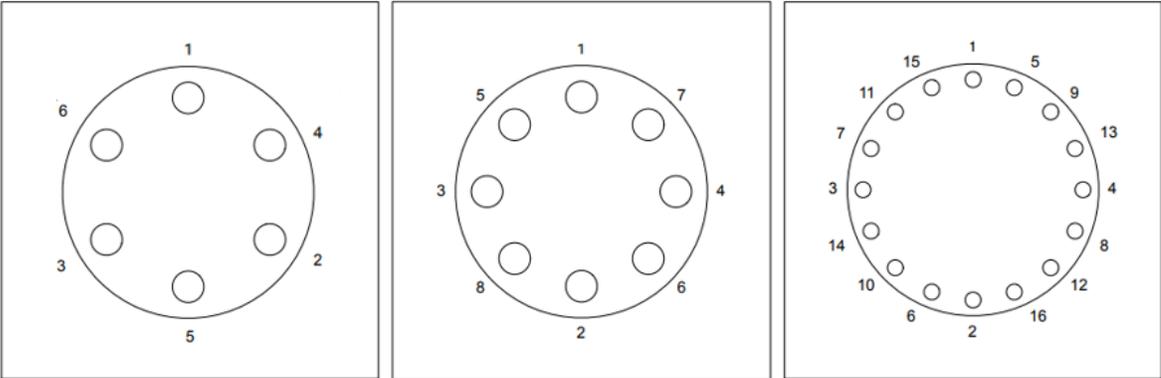


Figure 3: The order in which to tighten bolts on flanges with 6, 8, and 16 screws.

## Bake-out and pumping

In order to obtain UHV, a system needs to be baked because there is residual gas (mostly water) that is adsorbed to the chamber walls and the vapour pressure from the water is so high that the system does not go into the UHV range. The water slowly desorbs and is thus pumped away but this takes a very long time at room temperature, so the bakeout is performed in order to accelerate this process. Bake-out is an artificial acceleration of the process of outgassing.

The system was pumped down with the turbo and the Ion pumps and the vacuum was in the low  $10^{-6}$  Torr range before starting the bake-out. The heating must be even for all surfaces so we used a hot air blower oven in order to heat uniformly. Is best to start bake with Ion pump off and use turbo pump to remove gas during the process. Turbo pumps remove gas from the system, while ion pumps are capture pumps with a finite capacity, so using them to pump water vapor will shorten their lifetime.

The system was baked to 250°C for 155 hours as we show in figure 3. We set the ramps of the heat treatment starting from 25°C to 85°C, then another ramp to 120°C, other one to 200°C and finally one up to 250°C. This is much slower than a typical bake cycle ramp to temperature, but we had to do it this way since we have to have a HEPA filter in the pumping line to prevent any accidental exposure to the BNNT nanoparticles that might be displaced during pumpout of the system. Then we let it set in 250°C for 45 hours and started a cooling down to 120°C and let it there 4 hours to make NEG activation. Then ramp down to 25°C. All the time, the cryopump displacer region was purged with dry nitrogen gas throughout the bake to avoid oxidizing the system.

The CIS previously tested a Leybold pump that required a LN2 chill circuit. The cryopump needed to be fitted with a liquid nitrogen chill circuit that maintained low temperatures

near the fin array during baking, because the epoxy used to coat the fins with cryosorbent cannot undergo baking temperatures above 50°C without melting. Now, the BNNT cohesive fibrous texture allows it to be mechanically attached with a wire, thereby eliminating the problematic chemistry in the Leybold model. Before the bakeout, we removed the displacer from the cryopump since it cannot withstand the temperature of the bakeout, but it is outside the vacuum system, so it can be removed for the bakeout then replaced to run the system after the bakeout.

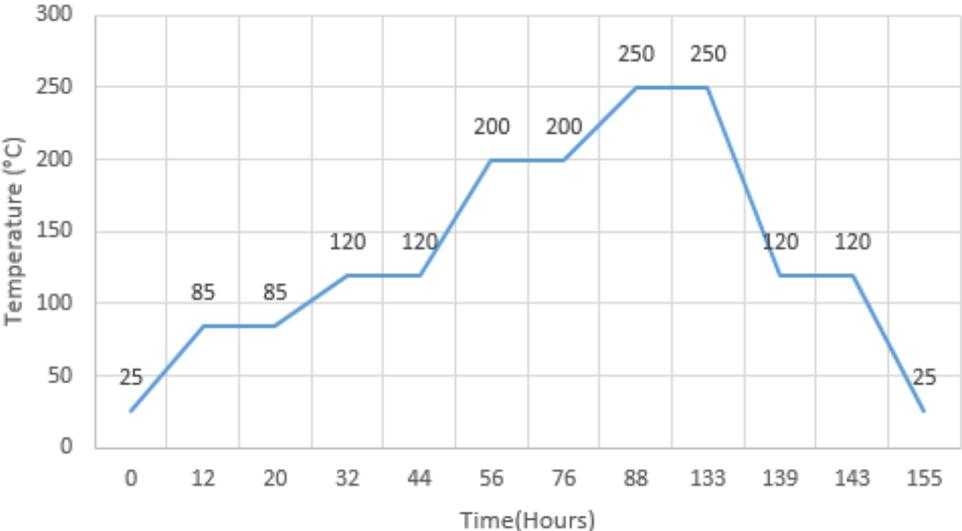


Figure 4: Temperature ramp of the bake-out process.

After the baking, we initiated the cryopumping. Every time the cold head of the cryopump has been opened to the atmosphere, to avoid contaminating the system with air and water vapor, you need to clean up and recharge the cryopump with 99.995% pure helium gas at 300 *Psi*.

## Troubleshooting

Building things is not as easy as people think and even though there are a lot of manuals and books about how to create a vacuum system, in real life there are more much factors to consider while you are working. These are some notes about the troubleshooting we had in the process and how can you deal with it.

- **Closed Valves.**

This was a recurring problem. The first time the valve from the pump cart to the rough line was closed. The second time, the valve to the chamber was closed. The lesson of the day: Always double check the valves you need open are open and the ones that need to be closed are closed before pumping down your system!

- **“Always know where you are.”**

This problem is derived from the previous. As we didn't notice that the valves were closed we thought that the pressure in the pump cart gauge was the pressure of the whole system and we try to turn on the ion pump multiple times. We also wanted to make a leak checking using the RGA but this one won't turn on. The lesson of the day: When you are pumping a system you need to keep in mind that the actual pressure of your main chamber is not the same as you are measuring in the rough line, and can be up to one order of magnitude higher.

- **Ion Pump wasn't working.**

This is a consequence of both previous. We were trying so hard to achieve the correct pressure to turn on the IP that we were turning it on at atmospheric pressure and we end up breaking it down. So we needed to replaced it for a new one. The lesson of the day: always test your pumps before installing it and make sure where are you in terms of pressure all the time, what is your gauge actually measuring.

- **Reconfigure rough line for bake over.**

Getting a vacuum system working can be a slow process, and discovering problems along the way can make it a lot slower. When we initially installed the rough line we didn't think about the baking panels, so the way that was assembled hadn't enough room for setting the baking panels. Lesson of the day: With careful planning you can catch a lot of the problems and design around them.

- **IP won't turn on during baking.**

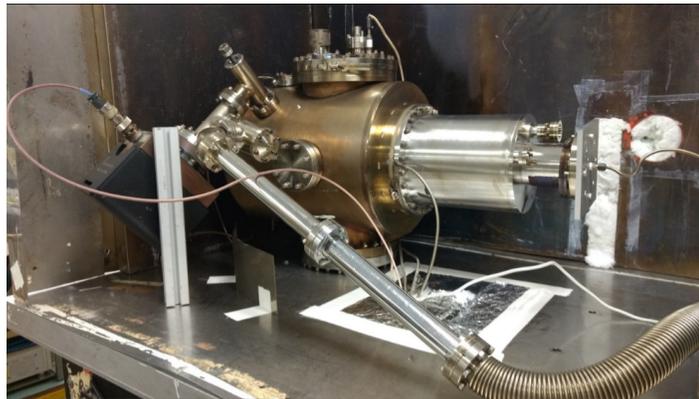
Everything is more complicated if you add oven panels. We tried to turn on the IP during the baking and it won't turn on. As you are not able to look into the oven and see what is actually going on, you can just make assumptions and hope for the best. The lesson of the day: You need to be very confident about your system before you star the bake out because you are not going to see it until it's over.

- **Cable to IP feedtrhough melted.**

It comes up that the IP won't turn on because the cable that was connected to the feedtrhough was melted during the baking. The bakable cable was installed with a connector that can only be baked to 180°C rather than the older style connectors that could withstand temperatures to at least 300°C. This change wasn't noticed until the cable melted, and we now need to check all our cables to see if these lower temperature cables are other places that will cause problems too. The lesson of the day: Always double check the specifications of your equipment.



(a) First configuration



(b) Configuration with new rough line for baking

Figure 5: Configurations through the process.

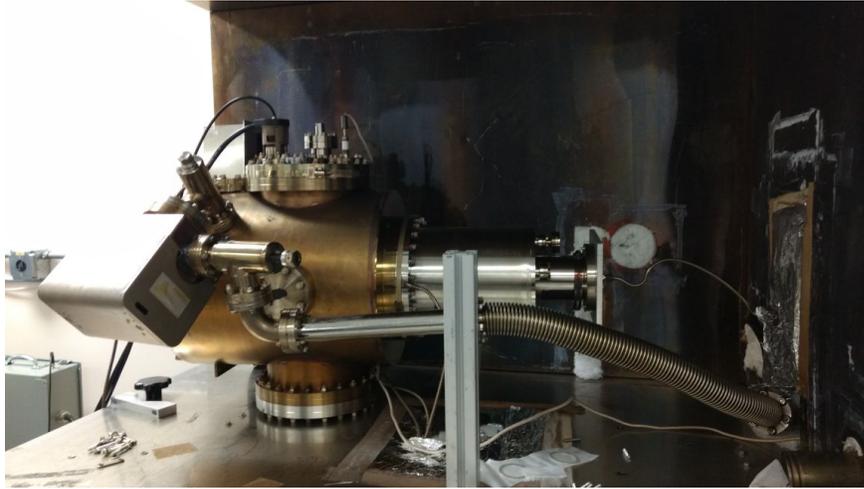


Figure 6: Final configuration of the system.

You'll probably still find a few things you didn't think of or didn't expect, so try to leave some room for flexibility in the plan and be very patient.

## Leak Checking

When working in ultra-high vacuum, it can be important to know the composition of the residual gas in order to monitor and control processes. We used a residual gas analyzer (RGA) in order to do the leak checking.

A RGA used a RF field to be able to separate the components of gas that are left in the vacuum system by mass. In a sealed vacuum system, the residual gasses tend to be hydrogen(mass 2) outgassing from the walls, water(18) if it is not baked, and methane(peaks around 16) which is generated by the RGA itself. Other expected gasses are mass 28, which can be CO or N<sub>2</sub> and 44, CO<sub>2</sub>, which comes from the Ion pumps. Indications of a leak are peaks at mass 32, oxygen, or Argon at mass 40. The oxygen abundant in air, but is usually pumped well, and if it is seen in the RGA, it is probably a leak in the system. Argon is not abundant, but we don't have very good pump speed for it, so it will accumulate if there is a

leak and you see the mass 40 peak.

No hydrocarbons will be found when using turbomolecular pumps. They are very effectively kept out of the chamber due to the high molecular masses and the resulting high compression ratios.

## Measuring

Above the ultrahigh vacuum (UHV) regime it is not possible to measure pressure as a force on a certain area as the definition of pressure indicates. Instead, it turns out that the most practical and economically reasonable indicator for pressure is the ionization rate produced by electrons hitting the neutral gas atoms in a UHV chamber [6].

There are two main types of ionization gauges: hot cathode gauges and cold cathode gauges. Hot cathode gauges use current to heat a filament, then an electric potential to extract electrons to ionize the gas in the system. The ionized molecules are collected, and the current of collected ions is proportional to the pressure in the chamber. Cold cathode gauges work similarly, but rather than using a hot filament to generate electrons, trap electrons in a magnetic field where they circulate and ionize gas molecules. Currently, only hot filament gauges are available to reliably measure pressures below  $1 \times 10^{-11}$  Torr.

## X-ray Limit

At the 1st International Vacuum Congress (IVC) in 1947 Nottingham suggested that the limit to the lowest measurable pressure was not caused by the pumps, but by an x-ray effect in the ion gauge [7], he proposed that soft x-rays, produced by electrons impinging on the anode, released photoelectrons from the ion collector; this photocurrent was indistinguishable

in the measuring circuit from the current due to positive ions arriving at the ion collector. This was soon confirmed by Bayard and Alpert [8].

The x-ray limit of a gauge determines the lowest pressure a gauge can measure. The x-ray limit occurs when an energized electron emitted in the gauge strikes the wall of the gauge with enough energy to generate an x-ray. This x-ray then has a probability of striking the ion collector and causing photoemission of an electron, which will give current the same sign as an arriving ion. This is a small effect, but when the x-ray current is similar to the pressure dependent ion current, the reading on the gauge is not an accurate measure of the pressure in the system.

The two gauges we are using to measure pressure at or below  $10^{-12}$  Torr are the extractor gauge and the Watanabe 3BG gauge, both of which have very low x-ray limits.

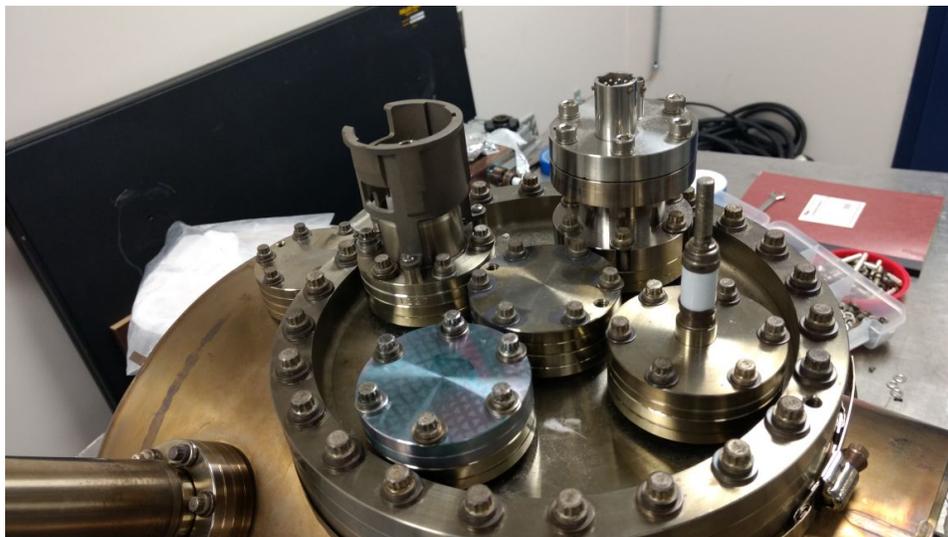


Figure 7: Gauges mounted in the flange. At the bottom, the extractor gauge (right) and Watanabe Bent Belt Beam gauge. (left).

The extractor gauge reduces the x-ray limit from a normal Bayard-Alpert gauge by having a small collector recessed behind a reflector. This reduces sensitivity of the gauge, but also allows it to measure pressures as low as  $8 \times 10^{-12}$  Torr according to the manual. X-ray backgrounds measured at JLab for these gauges have varied between  $0.5 - 2 \times 10^{-12}$  Torr.

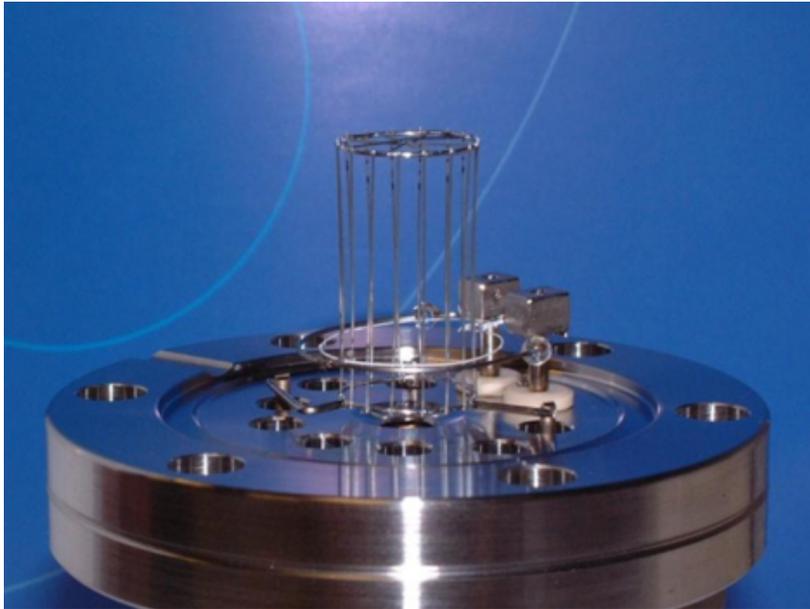


Figure 8: Extractor gauge.

The other gauge being used is the Watanabe Bent Belt Beam gauge. This gauge has been designed specifically for pressure measurement in the XHV regime. It has a 230 degree deflector, which means that the ions have to travel 230 degrees around the gauge between where they are generated at the filament and grid and the collector. This eliminates x-ray currents since there is no way for x-rays generated at the grid to get to the collector through the bend. The signal to noise on this gauge, measured previously at JLab, is still 30:1 at pressures around  $1 \times 10^{-12}$  Torr, so this gauge is quite likely to be able to measure pressures accurately if the pumping combination achieves XHV.

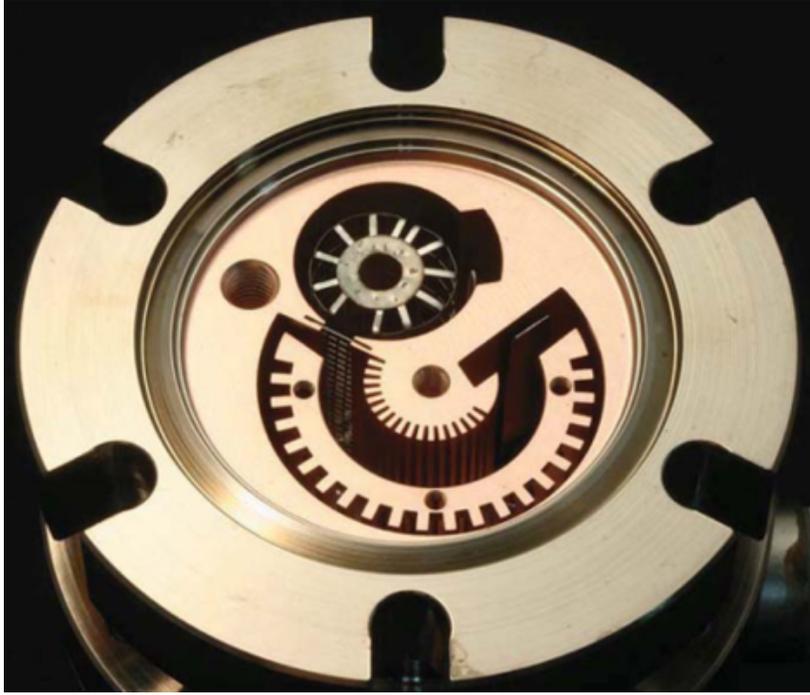


Figure 9: Watanabe Bent Belt Beam gauge.

For characterize UHV/XHV gauges you need to consider all the ionization gauge current contributions:

$$I^+ = I_{real} + I_{x-ray}^- - I_{inv,x-ray}^- + I_{ESD} \quad (1)$$

where:

$I_{real} \equiv$  pressure dependent gas phase ions.

$I_{x-ray}^- \equiv$  X-ray effect. X-ray induced electron desorption from collector.

$I_{inv,x-ray}^- \equiv$  Inverse x-ray effect.

$I_{ESD} \equiv$  Electron stimulated desorption. Ions arriving at collector from ESD of molecules on the grid.

## Molflow+ Simulation

Molflow+ is a test particle Monte-Carlo code developed at CERN by R. Kersevan and M. Ady [11], has been modified to calculate pressure in a direct way from the mass and velocity of the molecules. The geometry of each pump was simulated in AutoCAD and Blender to approximate real surface areas [12].

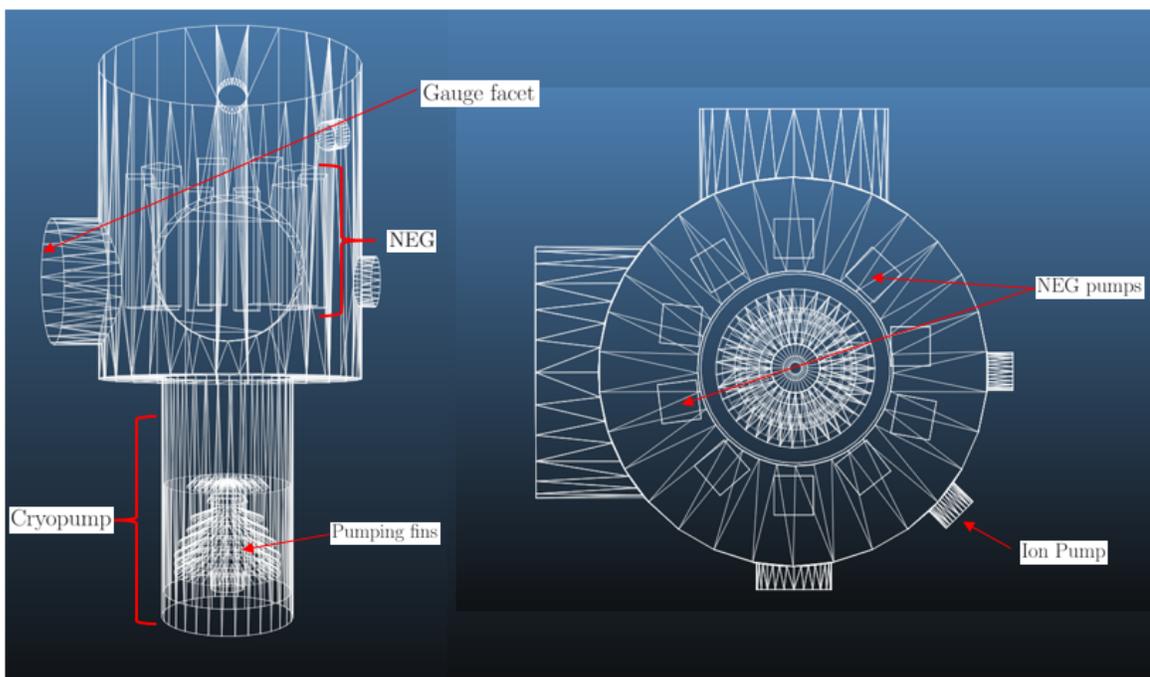


Figure 10: Geometry of the System. NEG, Ion and Cryopumps.

At very low pressures the rate of flow of gases is limited by the frequency with which the molecules strike the walls and may thus be thrown back in the direction of incidence. This type of flow is called “molecular flow”. A gas molecule on striking the surface is reflected in a direction which is totally independent of the direction of incidence, and the distribution of directions of an infinitely large number of molecules after a reflection from a surface follows Lambert’s cosine law [13] furthermore, most of the technical surfaces used in UHV are Lam-

bertian reflectors. For this reason we used cosine desorption in the simulation, this leads to a greater likelihood of desorbing molecules perpendicular to the facet's face.

Molflow+ is a direct simulation Monte Carlo program designed for finding pressure distributions in vacuum systems, taking into account pump speeds, outgassing rates, temperatures, and conductance between gas sources and pumps.

We simulated the expected pressure both with the cryopump on and off, using data from previous tests of this cryopump to determine the effective sticking coefficient. The manufacturer's quoted pump speeds for the Ion and NEG pumps have been measured and are accurate, so they are used here. We have also measured the outgassing rate of the stainless steel chamber and use that number for the simulations.

The results show that the pressure in the system is better in the system with the cryopump on, as expected, but these simulations indicate that the pressure might not reach the XHV pressures. However, we can do further careful calculations to see what effect each of the pumps have and then compare to the measurements we are taking.

The outgassing rate from the BNNT material is our largest unknown for the system with the cryopump off, and the pump speed and sticking coefficient for the cryopump is the largest unknown for the system with the cryopump on. We can compare the simulations with the measurements of pressure in the two states to better determine the outgassing rate of the BNNT and the cryopump pump speed from the pressures we measure, so this model is more going to be used as a description of the system rather than a prediction.

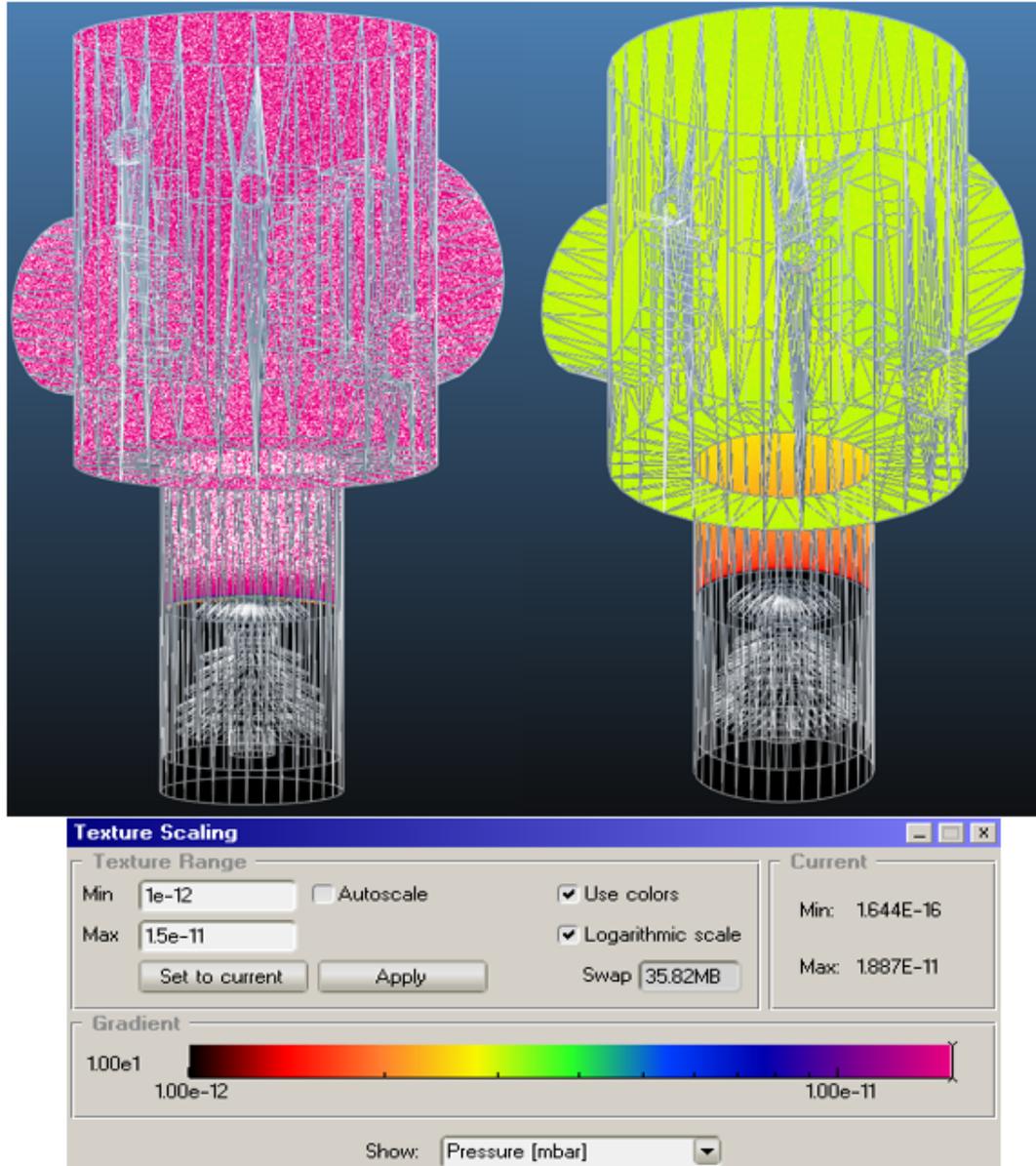
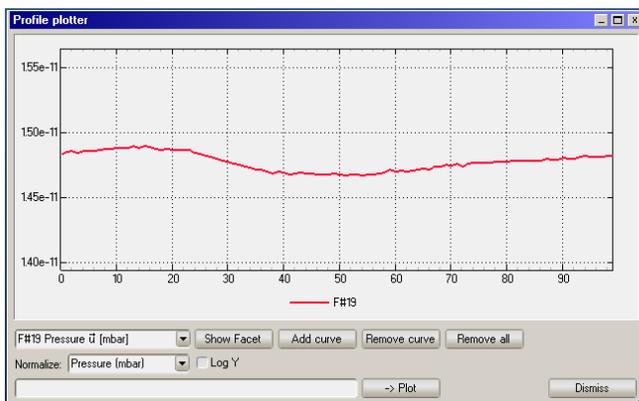


Figure 11: Simulations of the flow in the system. Cryopump off (left) and cryopump on (right).



(a) Cryopump off



(b) Cryopump on

Figure 12: Pressure distribution in a side of the chamber.

## Results

We found that the system reaches a pressure of  $1.6 \times 10^{-11}$  Torr with the cryopump off, and a pressure of  $2.7 \times 10^{-12}$  Torr with the cryopump on.

The x-ray limit of the extractor gauge is  $1.5 \times 10^{-12}$  so the real pressure inside the chamber is  $1.2 \times 10^{-12}$ .

This cryopumped chamber did not reached the pressures previously measured in JLab NEG/ion pump chambers with the cryopump at room temperature, this was due to the the outgassing of the large surface area inside the chamber. However, when the cryopump was turned on, the chamber achieved pressure near  $1 \times 10^{-12}$  quickly.

The figure 13 shows the pressure evolution when the cryopumping was initiated, cooling the fin array to 10K and the temperature shield to 40K as measured by silicon diodes, and how cryopumping lowered the pressure.

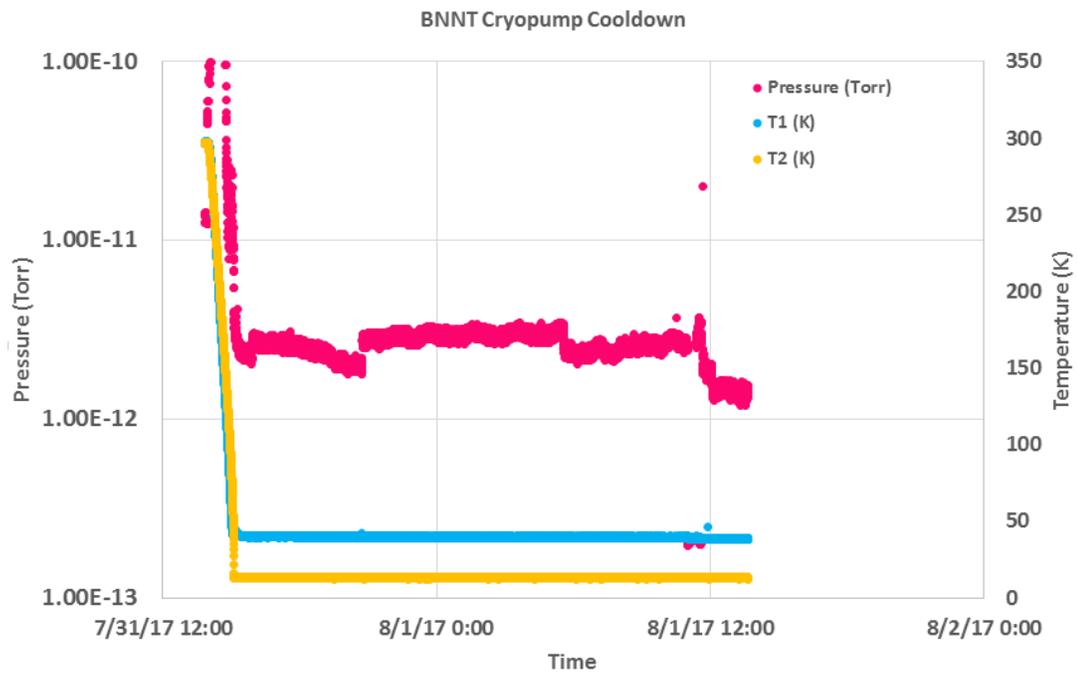


Figure 13: The pressure evolution in the system as measured by the Extractor gauge plotted with the temperature evolution.

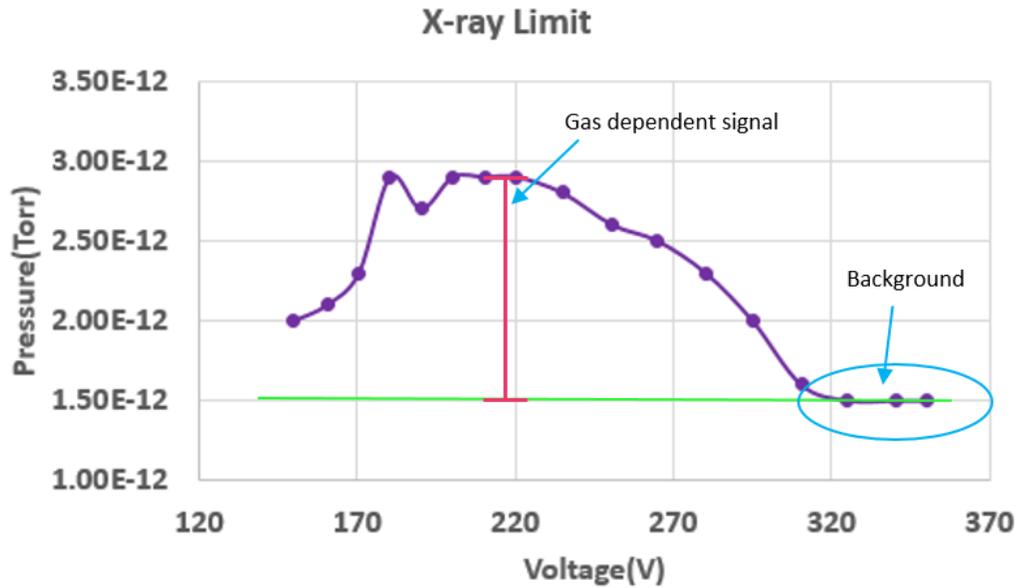


Figure 14: X-ray Limit of the extractor gauge, pressure measured varying the voltage.

## Conclusions

The first attempt at using BNNT cryopumping in addition to the NEG/ion pump system previously used at Jefferson Lab showed that we can achieve similar pressures with the prior technology, and will continue to be used to test the limitations of pumping systems for reaching XHV.

## Forthcoming Research

This is a prototype of using BNNT for a cryopump cryosorber. The amount of BNNT and the mounting should be optimized to improve the performance. This shows potential for a good alternative to the traditional cryopump.

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