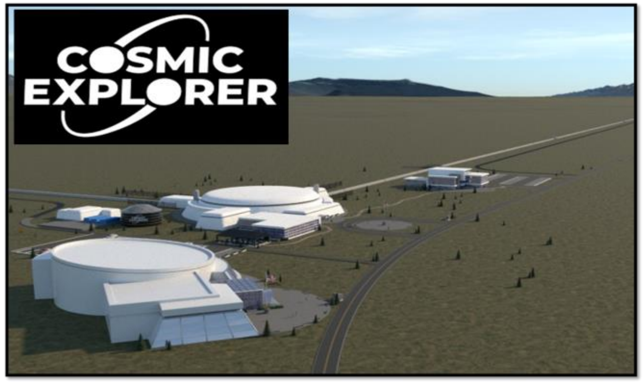
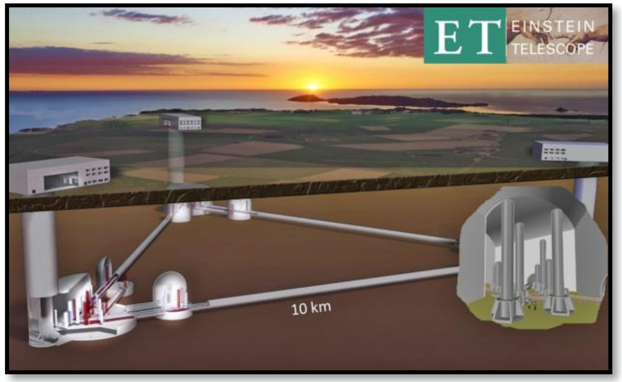
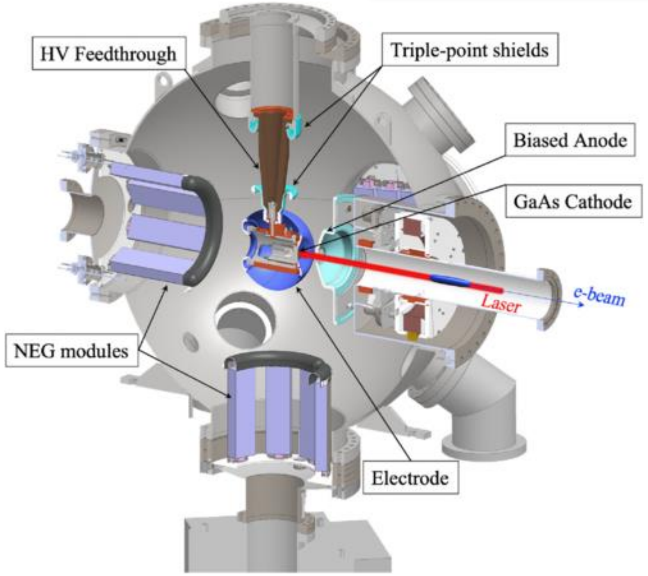
Practical Considerations for Using Low-Carbon Steel for Extreme High Vacuum Applications







Aiman H. Al-Allaq

Md Abdullah Mamun, Matthew Poelker,
Abdelmageed Elmustafa

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Vacuum – why should we work to improve it?

Photogun Lifetime:

- **Improving vacuum from today's -12 Torr to extreme high vacuum (<7.5x10^{-13} Torr)** would significantly reduce ion bombardment and extend the operational lifetime of the photogun.

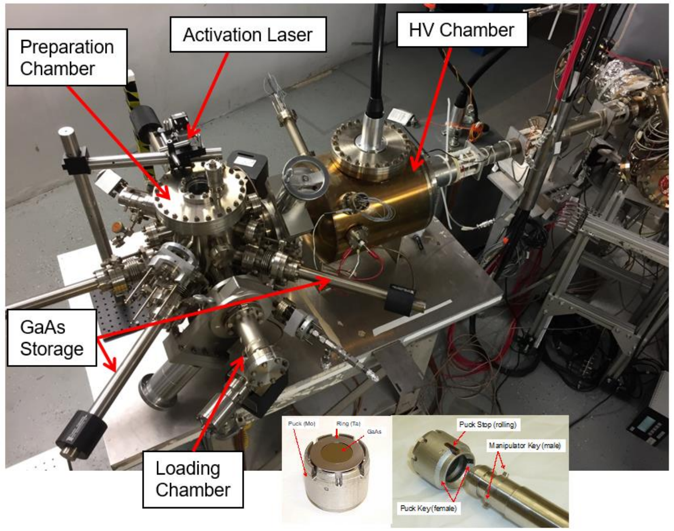
Beam Performance:

- Achieving and maintaining **XHV** ensures that the photogun can deliver longer beam times and support higher beam currents, improving experimental efficiency and output quality.

Cost Efficiency:

- Moving from **UHV to XHV** reduces the frequency of maintenance and replacement of sensitive components, saving both time and costs in long-term operations.

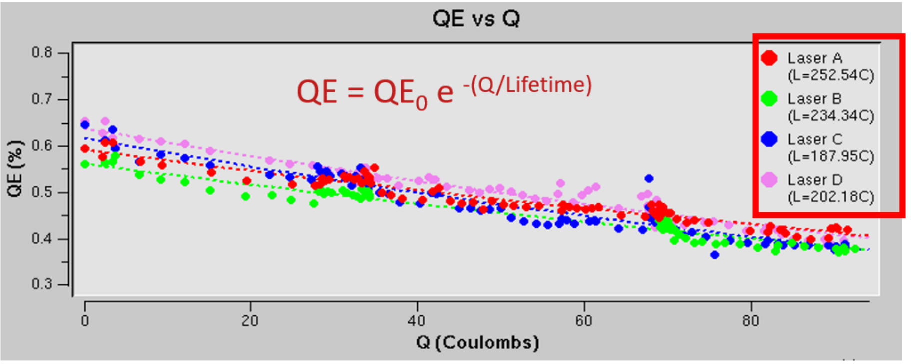
Gun vacuum ~ 1x10^{-12} Torr
Biased anode to +1 kV increased the lifetime by ~50%



P. Adderley *et al.*, Phys. Rev. Accel. Beams **13**, 010101 (2010)

Gun2 Photocathode (Fall 2016 – Spring 2017)

- **Lifetime ~200 C** (σ_{4D} ~ 1mm) with intensity **@ 200 μA**



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What sets the vacuum pressure inside a photogun?

$$P_{ult} = \frac{Gas\ Load}{Pump\ Speed} = \frac{Q}{S} = \frac{q\ A}{S} \qquad Torr = \frac{\frac{Torr \times L}{s \times cm^2}\ cm^2}{\frac{L}{s}}$$

$$Gas\ Load = \theta_1 + \theta_2 + \theta_3 + \cdots \theta_n + Beam\ PipeLeak_{\ Rate}$$

- This study evaluates the **hydrogen outgassing rate** and **ultimate pressure performance** of **AISI 1020 low carbon steel** compared to conventional **SS316 stainless steel**
- Low carbon steel exhibits remarkably low outgassing compared to stainless steel and could provide a means to reliably achieve Extreme High Vacuum (Pressure < 7.5E-13Torr)

Low carbon steel is filthy when it arrives on site

Cleaning and Pre-Baking Treatment:

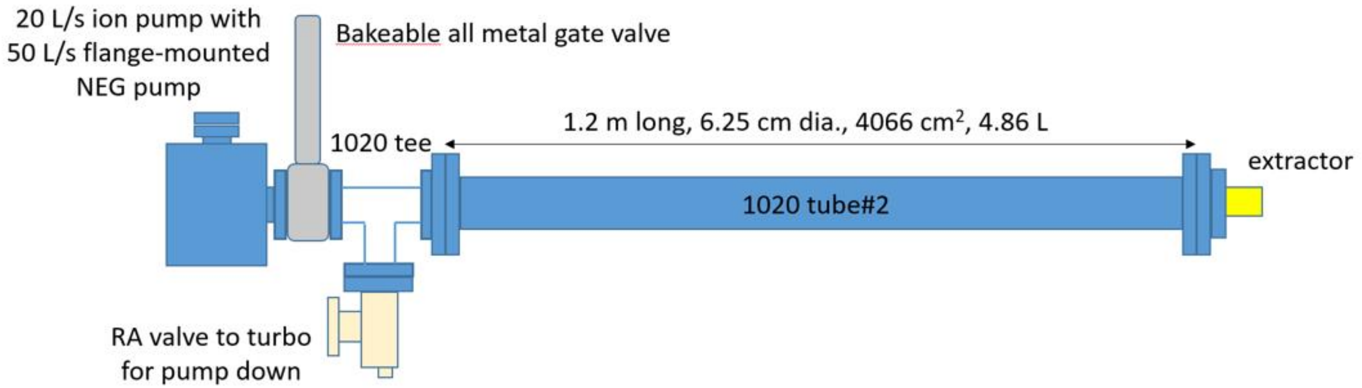
- **AISI 1020 Tubes**:
    1. Interior surfaces were heavily contaminated, requiring **acid etching**.
    2. Tubes were immersed in a solution of **18.25-19% HCl** for **1 minute** to remove oxide layers.
    3. After etching, rinsed with DI water and acetone, then dried with nitrogen gas and sealed in clean room bags.

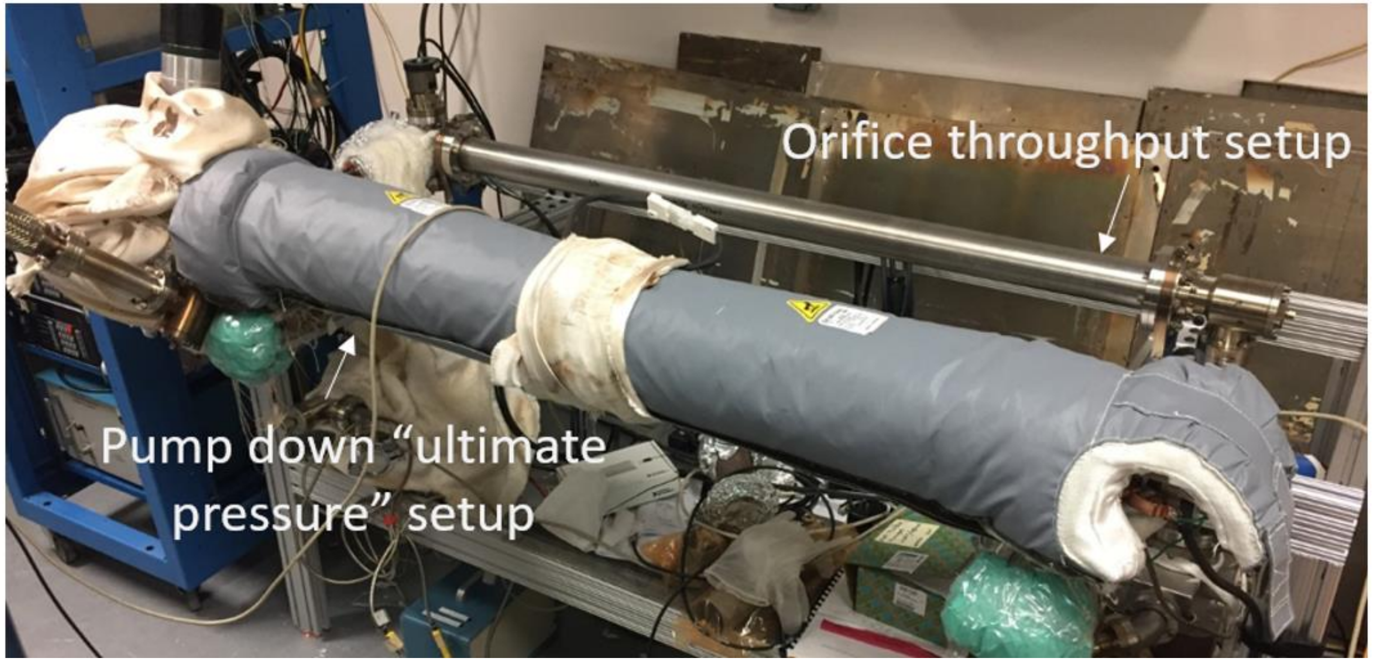
Low carbon steel is magnetic

- For photogun application, we don't think this is a problem (could be a good thing)

Experiment Overview

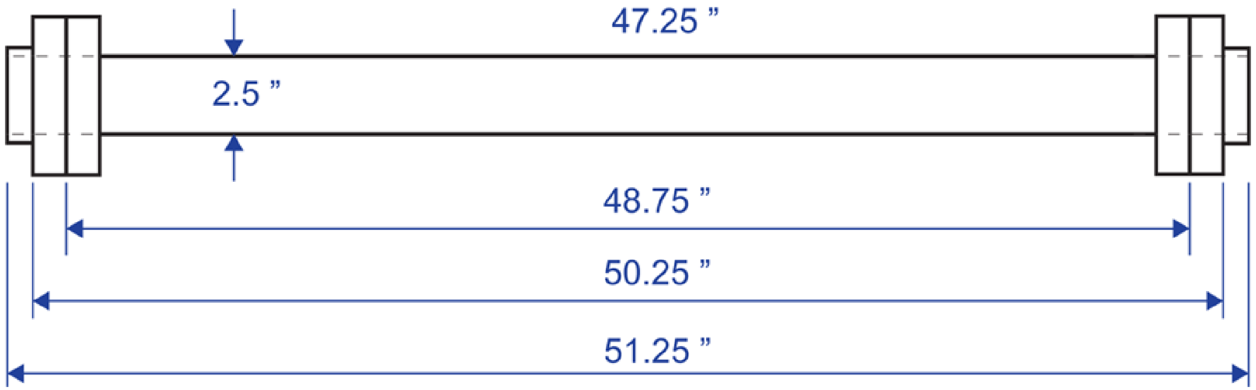
- **Tubes (why? easy to obtain):**
  - Inner diameter ~**2.37 inches**, Length ~**48.05 inches**, Surface area ~**4066 cm²**, Volume ~ **4.856 liters**
  - Surface area-to-volume ratio ~**0.001194 l/cm²**
- **Materials**:
  - **AISI 1020 Low Carbon Steel**:
    - Carbon content: ~0.2%
    - Stainless steel flanges were vacuum degassed at **900°C for 1 hour** to reduce hydrogen outgassing
  - **316L Stainless Steel**:
    - Degreased after welding flanges
- Stainless steel flanges were degassed for the low carbon steel tubes, but not for the stainless steel tubes.

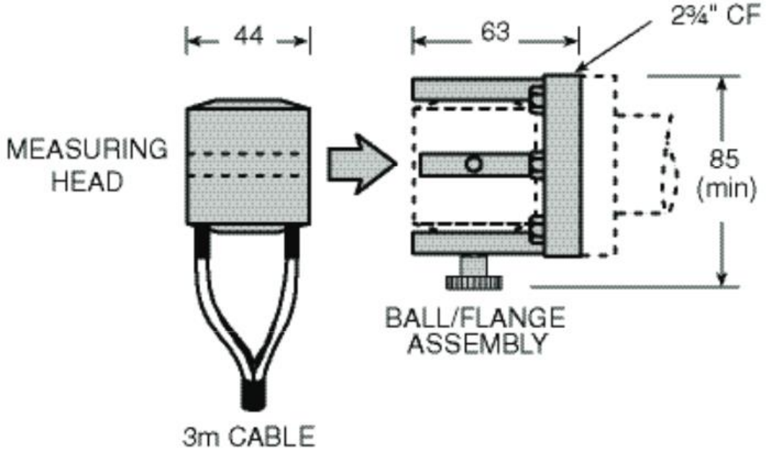




Hydrogen Outgassing Measurements

- **Outgassing Measurement Method**: **Rate-of-Rise (ROR) Method**: Measures the initial pressure rise in a vacuum system to determine the outgassing rate.
- **Procedure**:
  - Tubes were evacuated to UHV, then isolated from the pumping system.
  - The initial rate of pressure rise was recorded and used to calculate the hydrogen outgassing rate.
- **Recommended Practices** (Based on Redhead, 2002):
  - **Gauge Calibration**: Non-pumping gauges (e.g., spinning rotor) used to avoid errors.
  - **Temperature Control**: Bakeouts performed with a custom jacket to ensure uniform heating at precise temperatures.
- **Outgassing Rate Calculation**: $q_A = \frac{\left(dP/dt\right) \cdot V}{A}$ Where dP/dt is the rate of pressure rise, V is the volume, and A is the surface area.

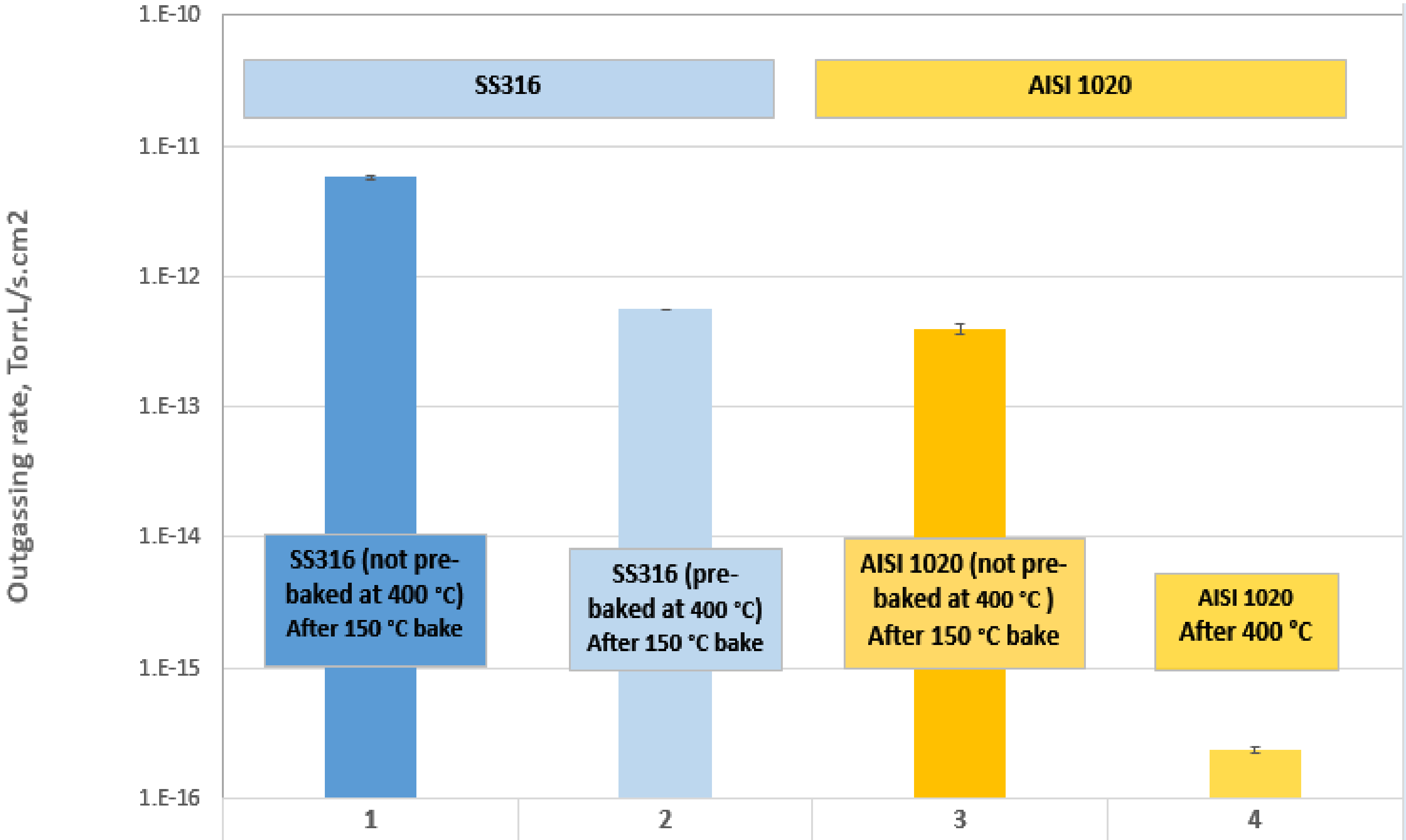




Hydrogen Outgassing Measurements

Rate-of-Rise Method (Accumulation):

- **Key Data Points**:
  - **SS316 (not pre-baked) baked at 150C**: Initial outgassing rate of **5.75×10^{-12} Torr L/s/cm²**.
  - **SS316 (pre-baked at 400°C) baked at 150C**: Outgassing rate reduced to **5.6×10^{-13} Torr L/s/cm²** after treatment.
  - **AISI 1020 (not pre-baked) baked at 150C**: Initial outgassing rate of **3.97×10^{-13} Torr L/s/cm²**.
  - **AISI 1020 (400°C Bake)**: Further reduced to **2.35×10^{-16} Torr L/s/cm²**—almost **1000x lower** than SS316.
- **Activation Energy**:
  - Calculated from temperature-dependent outgassing rates: **AISI 1020** shows a low activation energy of **~27.015 kJ/mol** compared to SS316.

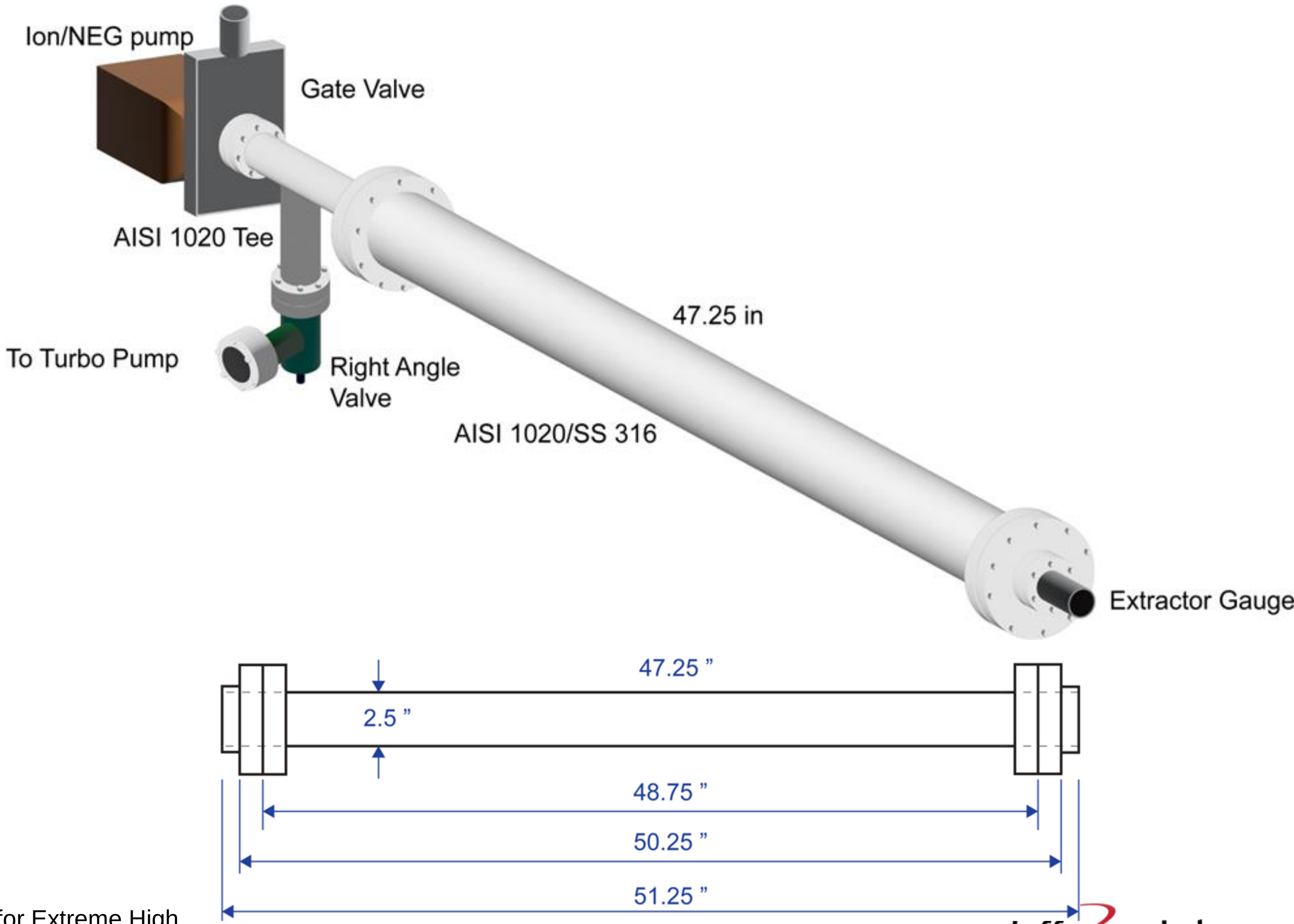


	1	2	3	4
q, Torr.L/s.cm2	5.7490E-12	5.5667E-13	3.9693E-13	2.3487E-16

Will pressure inside a photogun made of low carbon steel be **1000** times lower than inside a photogun made of stainless?

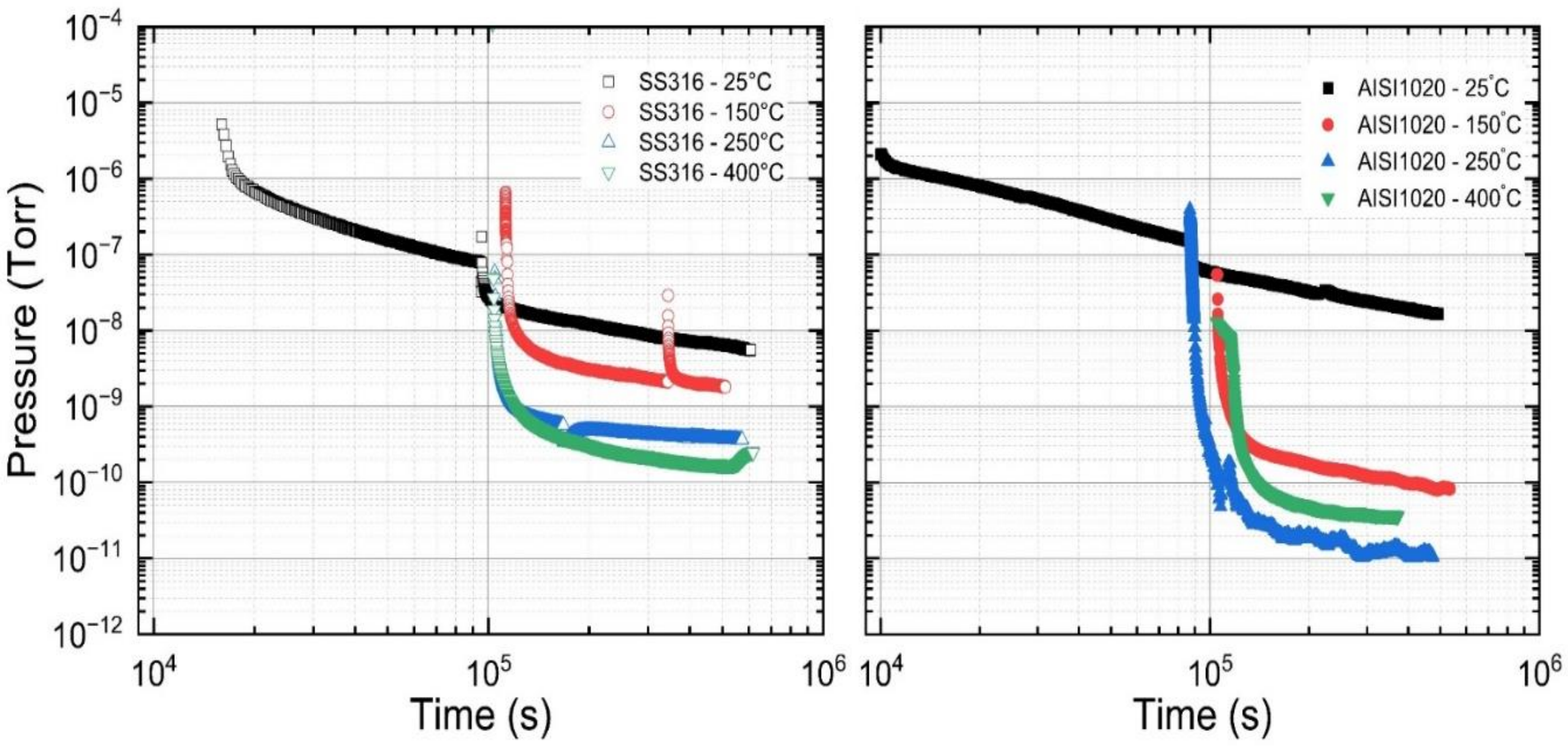
Ultimate Pressure Measurements

1. **Pump Preparation**: Ion pump baked, **NEG activated**, gate valve closed during venting/heating.
2. **Venting**: Temperature set, system vented for 1 hour.
3. **Pumping**: 24-hour **pump down** with turbopump at the set temperature.
4. **Isolation & Cooling**: System cooled, valve to **NEG/ion pumps opened**.
5. **Pressure Measurement**: Gauge activated below 50°C.
6. **Data Logging**: Pressures recorded at **25°C, 75°C, 110°C, 150°C, 250°C, and 400°C**.



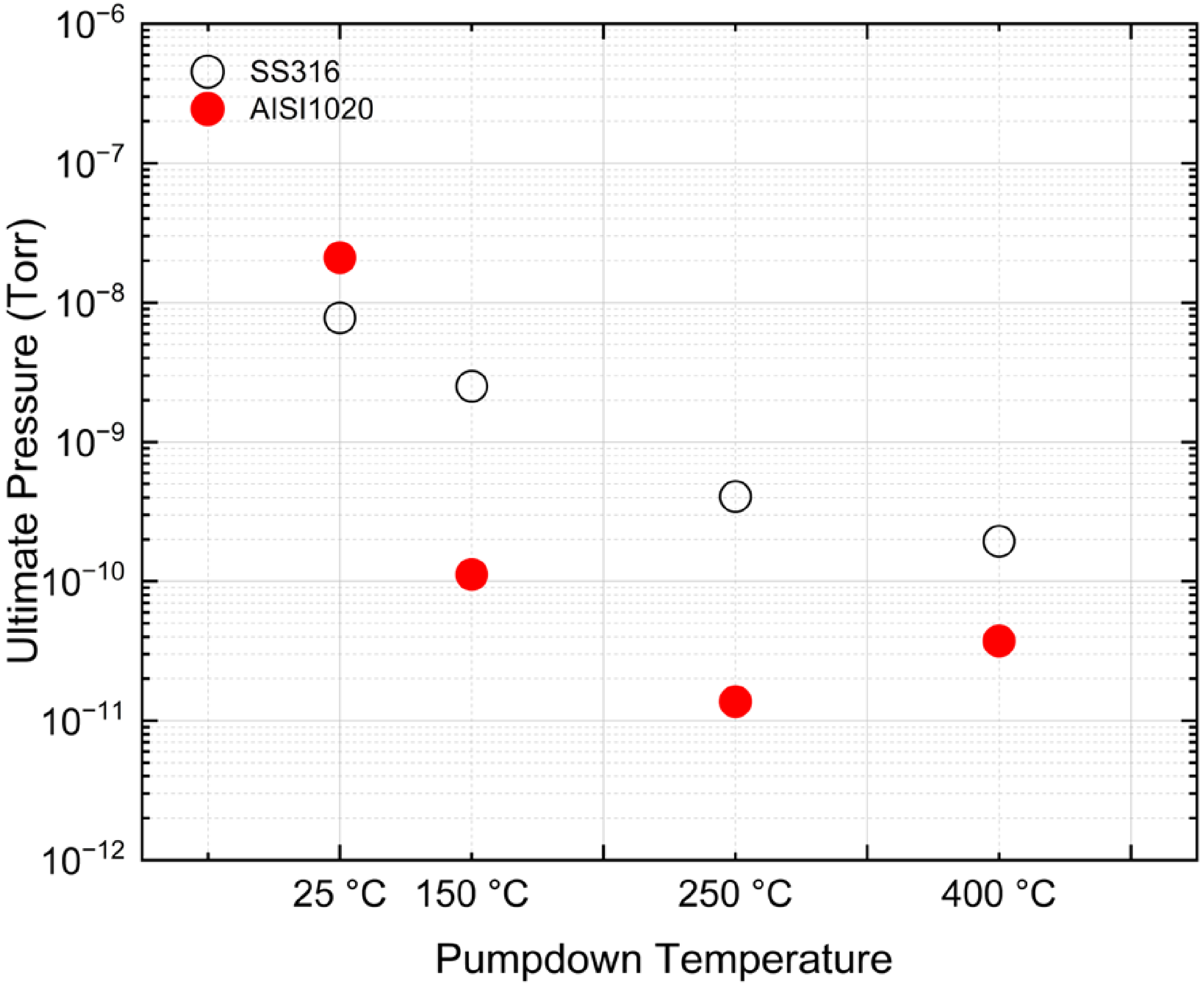
Ultimate Pressure Measurements

- **SS316**:
  - Ultimate pressure after 150°C bake: **2.5x10^{-10} Torr**
  - After 400°C bake: **8.6x10^{-11} Torr**
- **AISI 1020**:
  - Ultimate pressure after 150°C bake: **1.6x10^{-11} Torr**.
  - After 250°C bake: **1x10^{-11} Torr**.
  - After 400°C bake: **7.4x10^{-11} Torr** (Degradation noted)
- **Degradation at 400°C**:
  - Increased ultimate pressure suggests surface degradation, potentially linked to overexposure of the extractor gauge and oxidation effects
  - Also, it suggests degradation of the vacuum gauge. Exposing the sensitive extractor gauge to air at elevated temperatures likely contributed to the degradation.



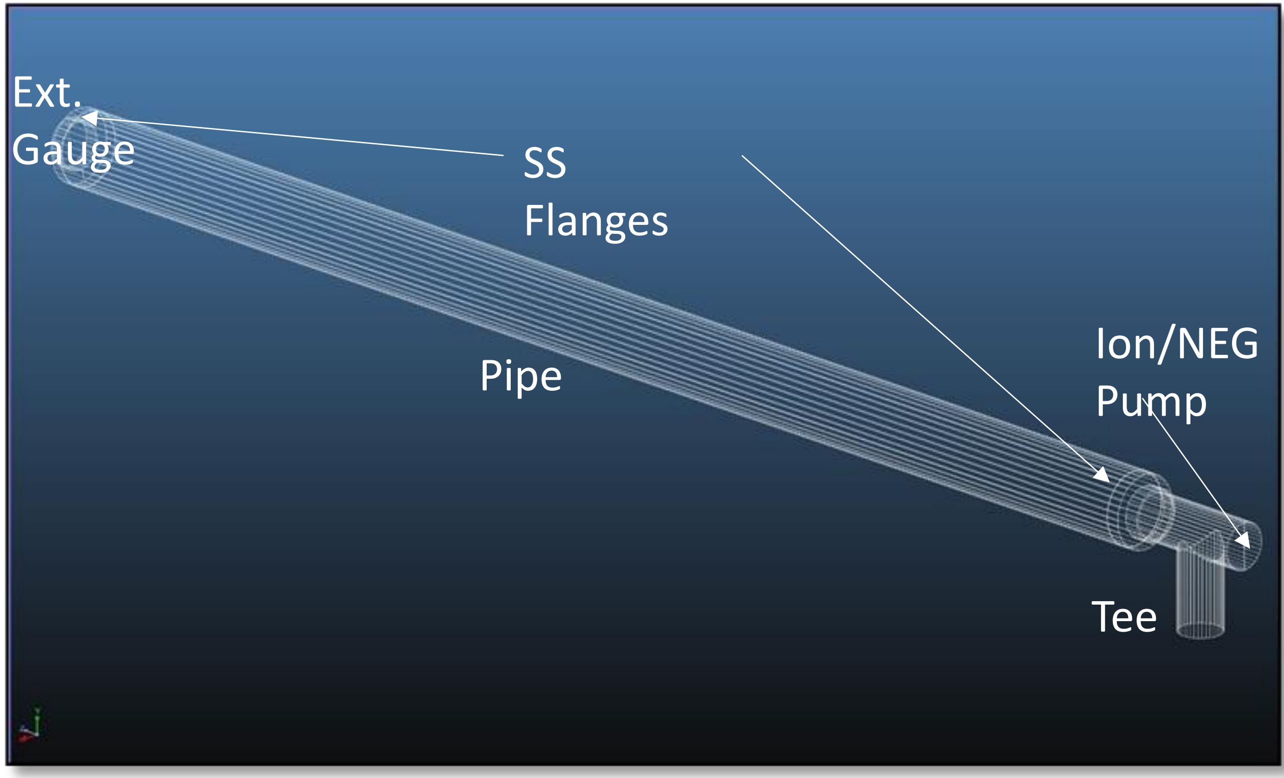
Ultimate Pressure Measurements

- **Low carbon steel** provided **lower** pressure
- But not **1000** times lower
- Is something wrong?
- Time for **MolFlow+** simulations



MolFlow+ Simulations

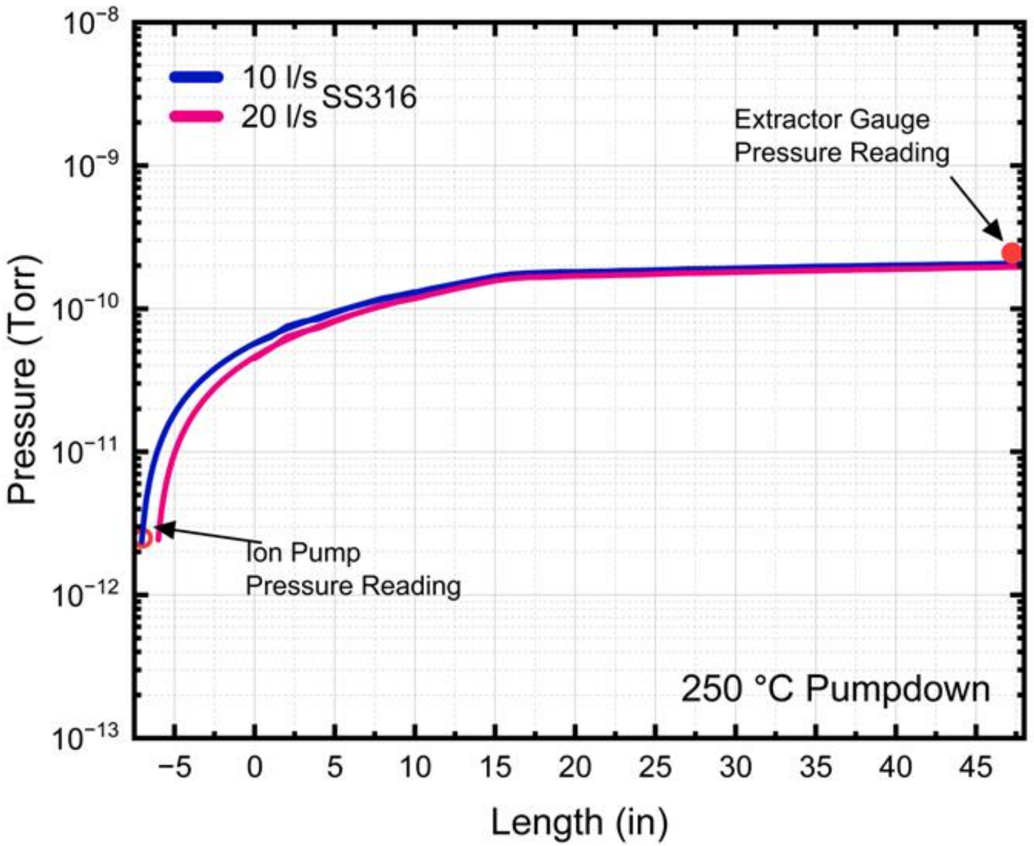
- **Software**: MolFlow+ - a Monte Carlo simulation tool for vacuum systems
- **What do people use MolFlow for?**
  - Predict pressure distributions in complex vacuum systems.
  - Estimate effective pump speed at different locations.
  - Calculate effective conductance of tubes, chambers, and components.
  - Model sticking coefficients and outgassing rates for different materials.
  - Analyze gas flow paths and pressure drops in vacuum systems.
- **Input Parameters:**
  - Outgassing rates: Based on material properties (e.g., hydrogen outgassing rates of SS316 and AISI 1020).
  - Pump speeds: Input as known values or estimated from conductance.
  - Geometry: Tubes, valves, and chamber dimensions.
  - Surface properties: Sticking coefficients, adsorption/desorption rates.

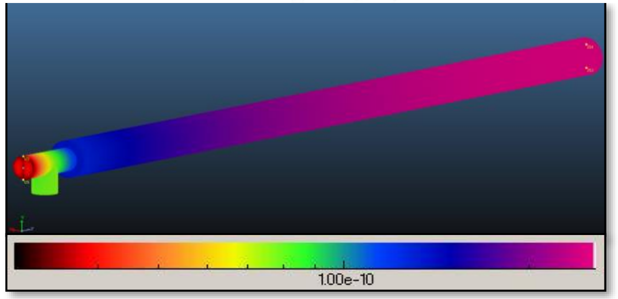


MolFlow+ Simulations

Pump Speed Estimation (SS316)

- **Objective 1**: Use MolFlow to calculate the pump speed at the location of the gauge, based on stainless steel characteristics which are considered to be well known
- **Process**:
  - **SS316 outgassing rate** ($2x10^{-13}$ to $5x10^{-13}$ Torr L/s/cm²) was input into MolFlow+.
  - We assume the pressure measurements were accurate
  - We use the simulation to calculate the **effective pump speed** at the extractor gauge location (far end of the tube).
  - The estimated pump speed was between **10 and 20 L/s**.
  - Very close to the **conductance calculation** of **27 L/s using textbook conductance equations**
- **Outcome**: Now that we know a realistic range of pump speed at the gauge…

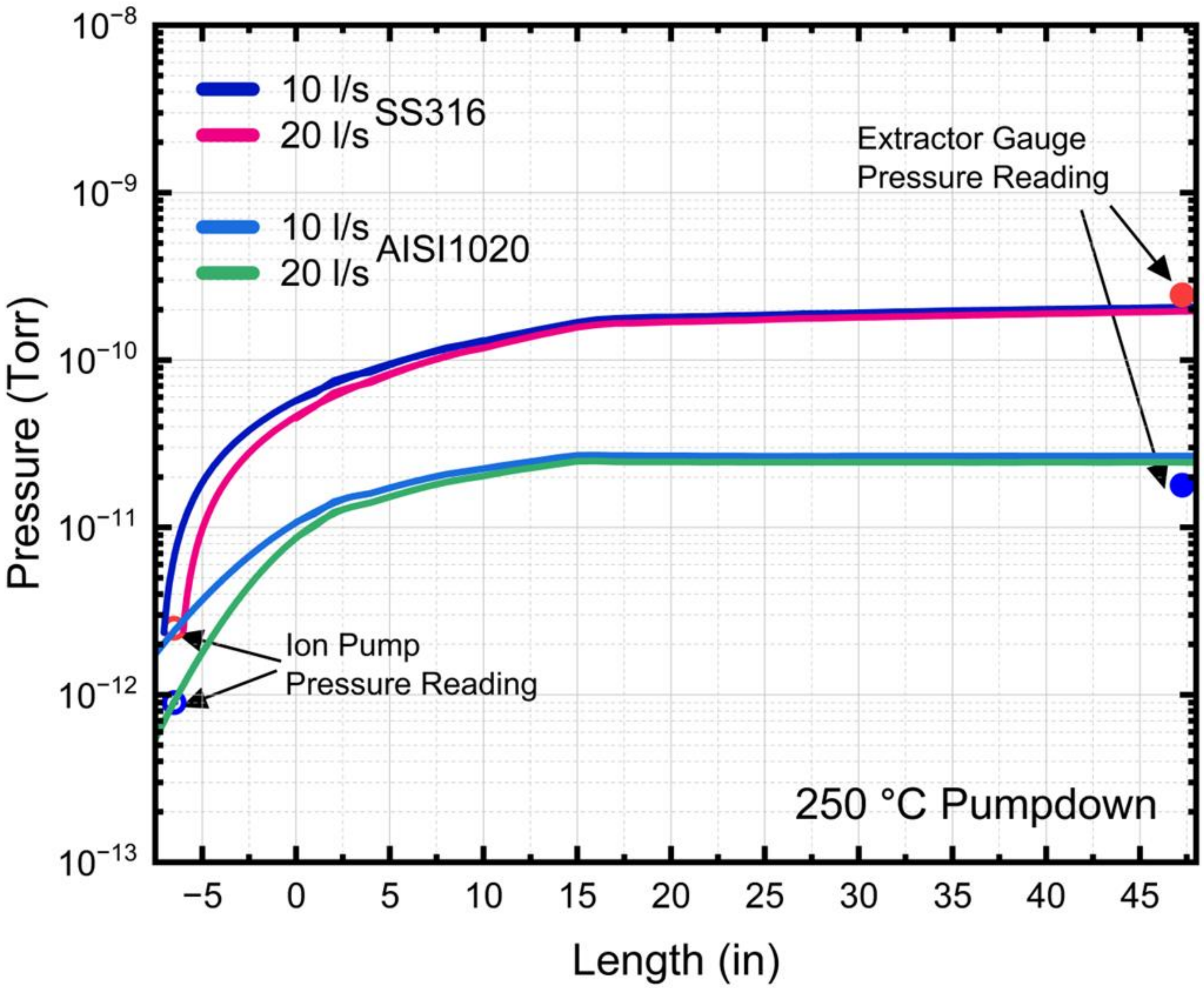




MolFlow+ Simulations

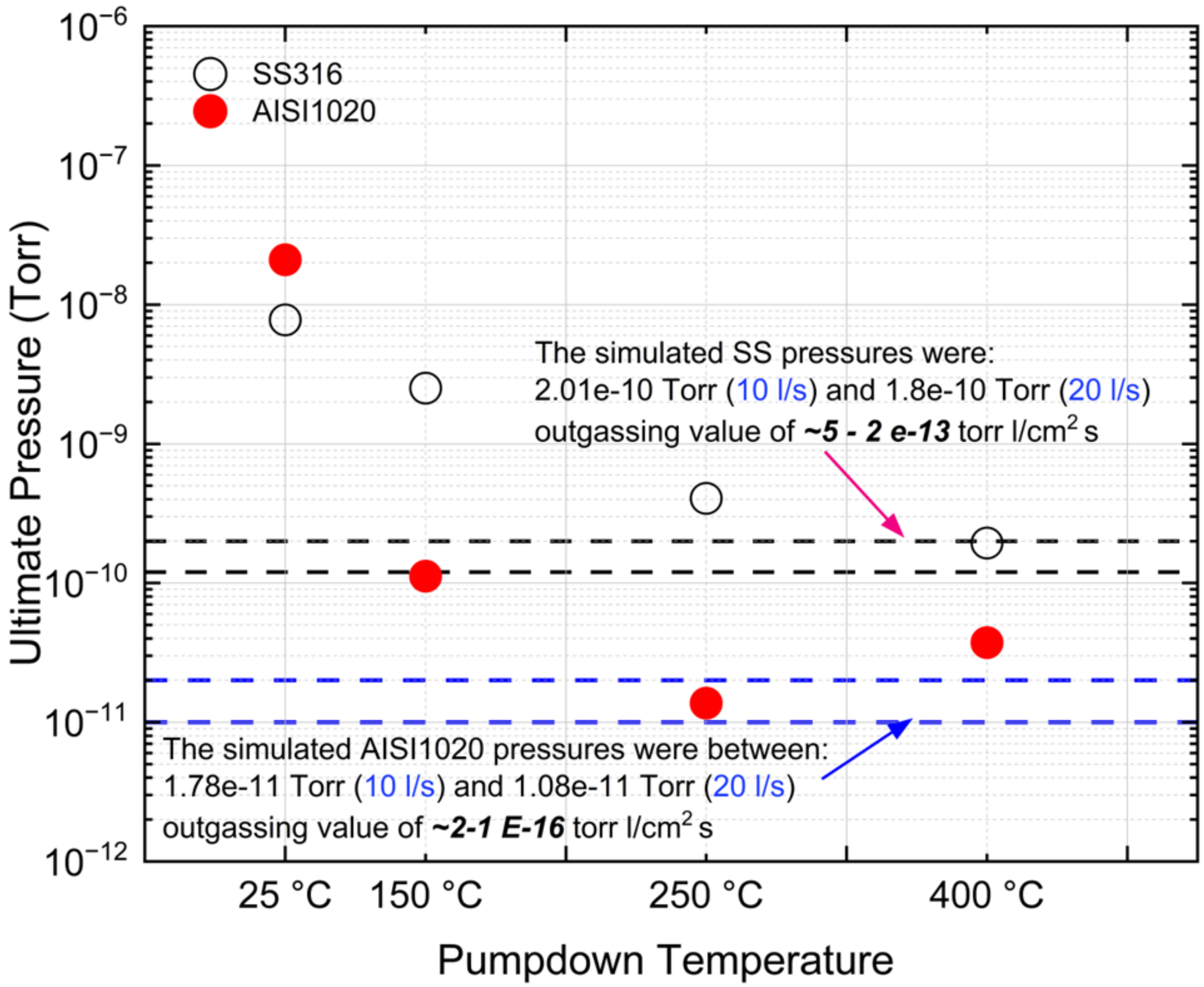
Pressure prediction for low carbon steel tube

- **Objective2**: Now use MolFlow to predict the outgassing rate of the low carbon steel tube
- **Process**:
  - **Again, we assume the pressure measurement was accurate**
  - We input into MolFlow a range of pump speeds between **10 and 20 L/s**.
- **Outcome**: MolFlow+ predicts low carbon steel outgassing we measured
  - MolFlow+ predicted the outgassing rates and corresponding ultimate pressure for the low carbon steel tubes (AISI 1020) that were measured experimentally
  - The simulations matched the experimentally observed outgassing rate of 2.35×10^{-16} Torr L/s/cm² after the 400°C bake and ultimate pressure values



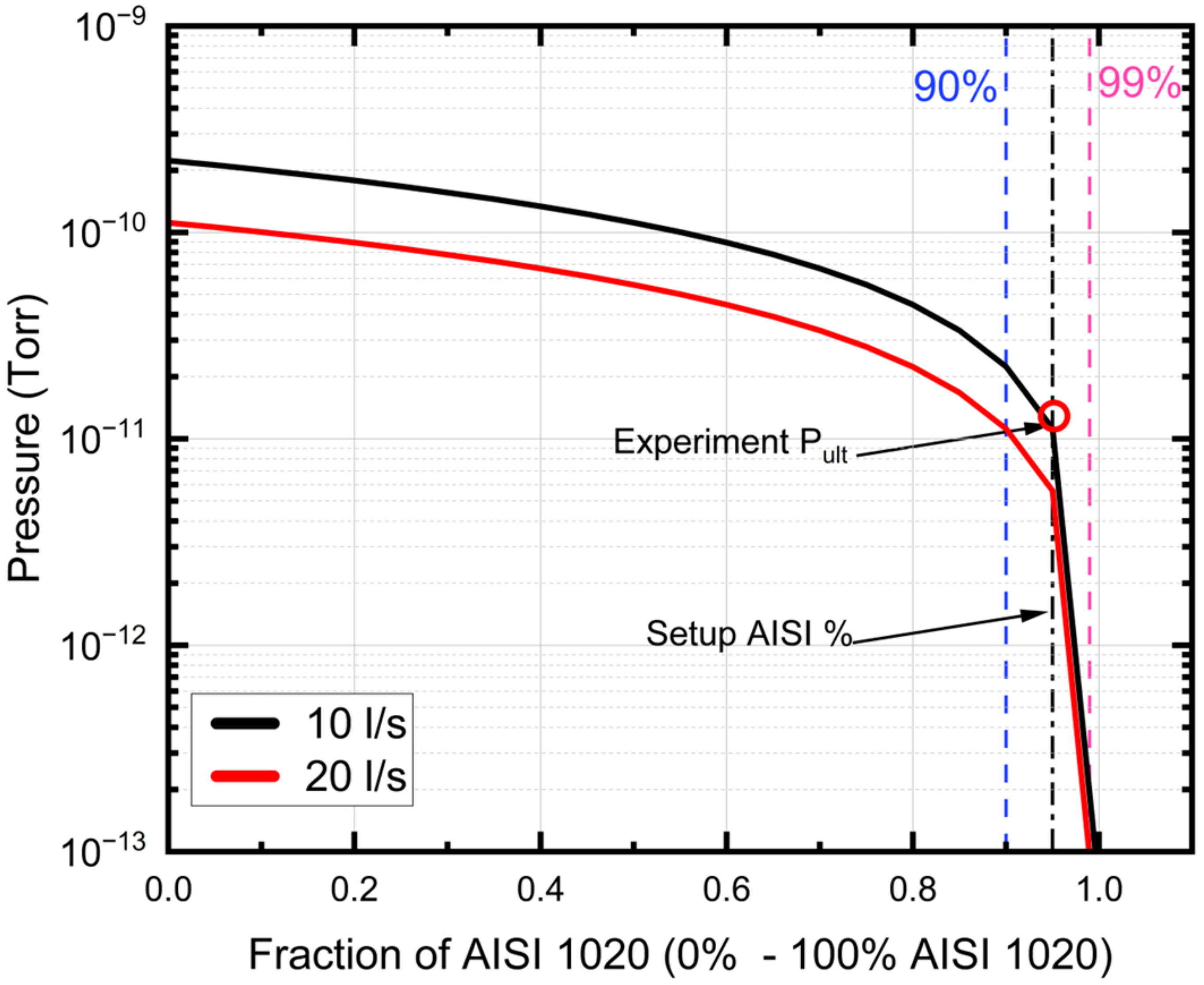
MolFlow+ Simulations

- **Stainless steel components** (flanges, valves, right-angle joints) contributed **measurable outgassing** to both the SS316 and AISI 1020 systems.
- Despite **AISI 1020's significantly lower outgassing rate**, the presence of untreated stainless steel parts **diluted the advantage** by adding additional outgassing sources.
- **Pressure Comparison**:
  - **AISI 1020 (150°C bake)**: Ultimate pressure of ~**1.6x10^{-11} Torr**.
  - **SS316 (150°C bake)**: Ultimate pressure of ~**2.5x10^{-10} Torr**.
- **Conclusion**:
  - The presence of stainless steel parts prevented AISI 1020 from achieving its **theoretical vacuum performance**, highlighting the need for careful material selection and possible treatment of all components in UHV systems.



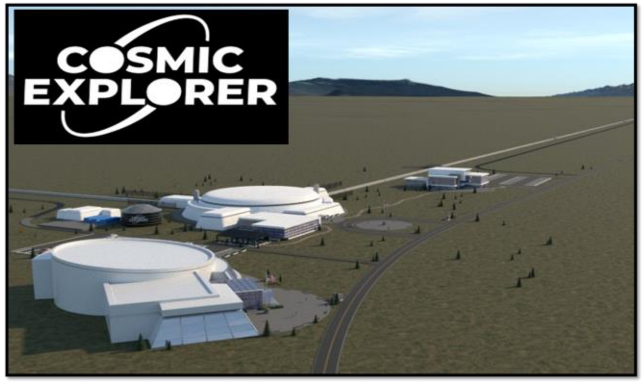
Conclusions and Future Directions

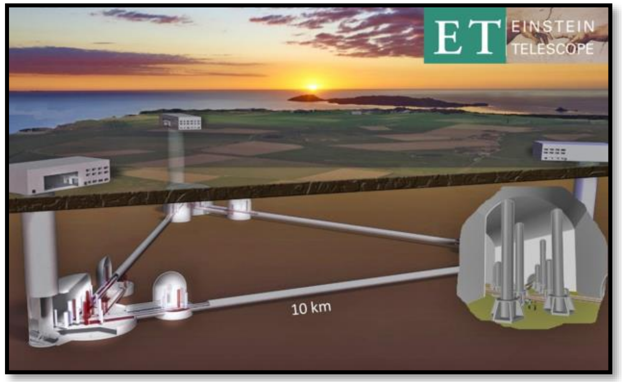
- **AISI 1020** demonstrated significantly lower hydrogen outgassing rates compared to **SS316**, especially after the **150°C** and **250°C** bakes.
- **Increased outgassing after the 400°C bake** was likely due to **degradation of the vacuum gauge**, rather than solely surface degradation of AISI 1020.
- The presence of **stainless steel components** limited the full potential of AISI 1020 in achieving ultra-high vacuum.
- **Simulations using MolFlow+** closely matched experimental results, validating the outgassing behavior and ultimate pressure.
- **Future Considerations**:
  - A chamber made **exclusively from low carbon steel** could have potentially reached **extreme high vacuum (XHV)** conditions, assuming proper gauge protection and material selection.

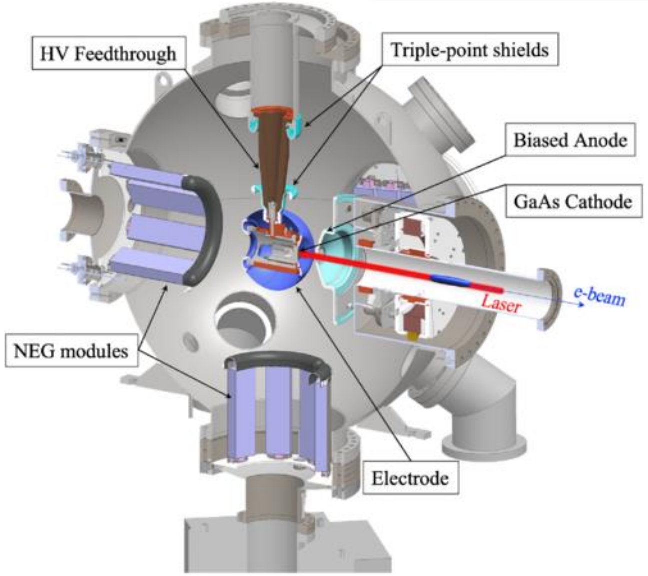


Aiman H. Al-Allaq
aalal005@odu.edu/aimanhq@jlab.org

Questions?



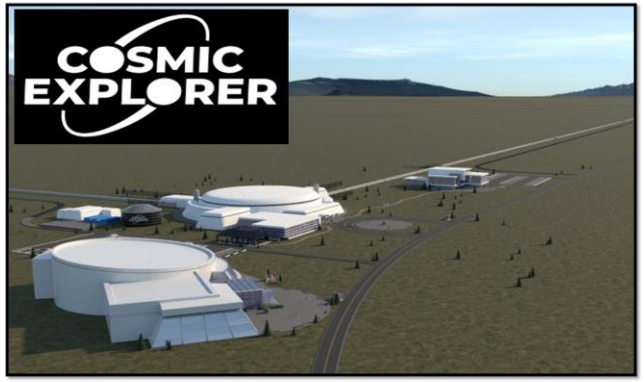


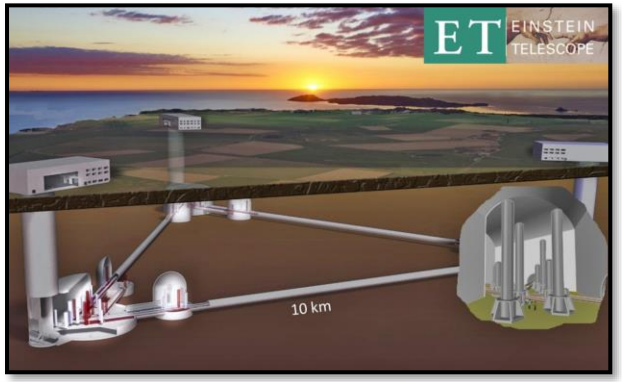


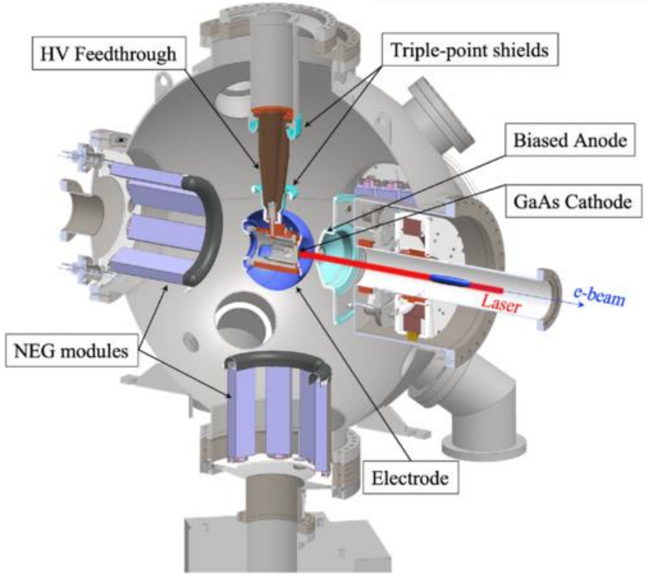
Topics Covered

Aiman H. Al-Allaq
aalal005@odu.edu/aimanhq@jlab.org

- Importance of UHV in Photoguns
- Hydrogen Outgassing Comparisons (AISI 1020 vs SS316)
- Ultimate Pressure Measurements
- MolFlow+ Simulation ResultsI
- Impact of Stainless Steel Components

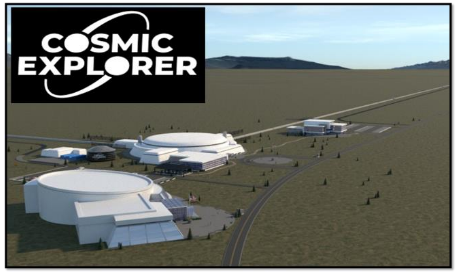


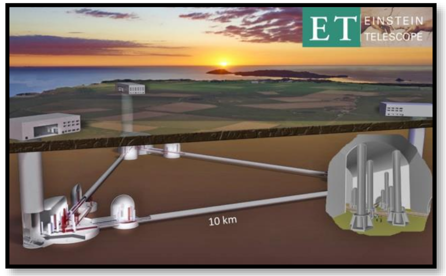


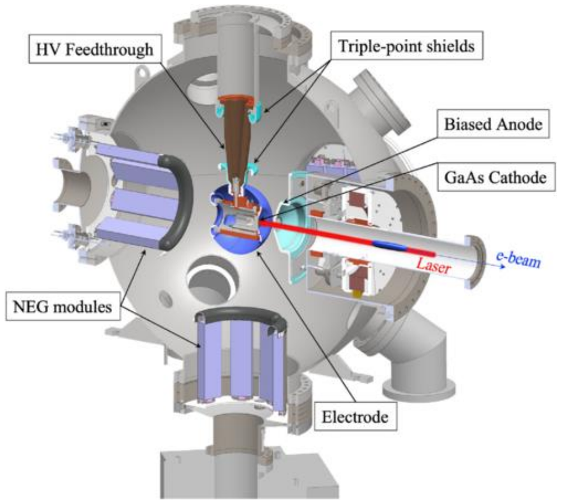


Vacuum – why should we work to improve it?

- This study evaluates the **hydrogen outgassing rates** and **ultimate pressure performance** of **AISI 1020 low carbon steel** and **SS316 stainless steel** tubes, focusing on their potential applications in **ultra-high vacuum (UHV)** systems.
- **Motivation**:
- **Low carbon steel** offers a **cost-effective alternative** to stainless steel for UHV systems due to its lower cost and ability to achieve **significantly lower outgassing rates** after appropriate heat treatment.
    - **Gravitational wave observatories**.
    - **Spin-polarized electron sources**.
- Understanding the surface transformations, especially **magnetite formation**, helps in improving vacuum performance and cost efficiency in these systems.
- **Approach**:
- **Experimental Comparison**:
    - Side-by-side evaluation of AISI 1020 and SS316 tubes.
    - Measurement of **outgassing rates** and **ultimate pressures** after various heat treatments.
- **Surface Science**:
    - Analysis of surface changes using **SEM**, **EDS**, and **AFM** to investigate the impact of **oxidation** and **magnetite layer formation** on vacuum performance.



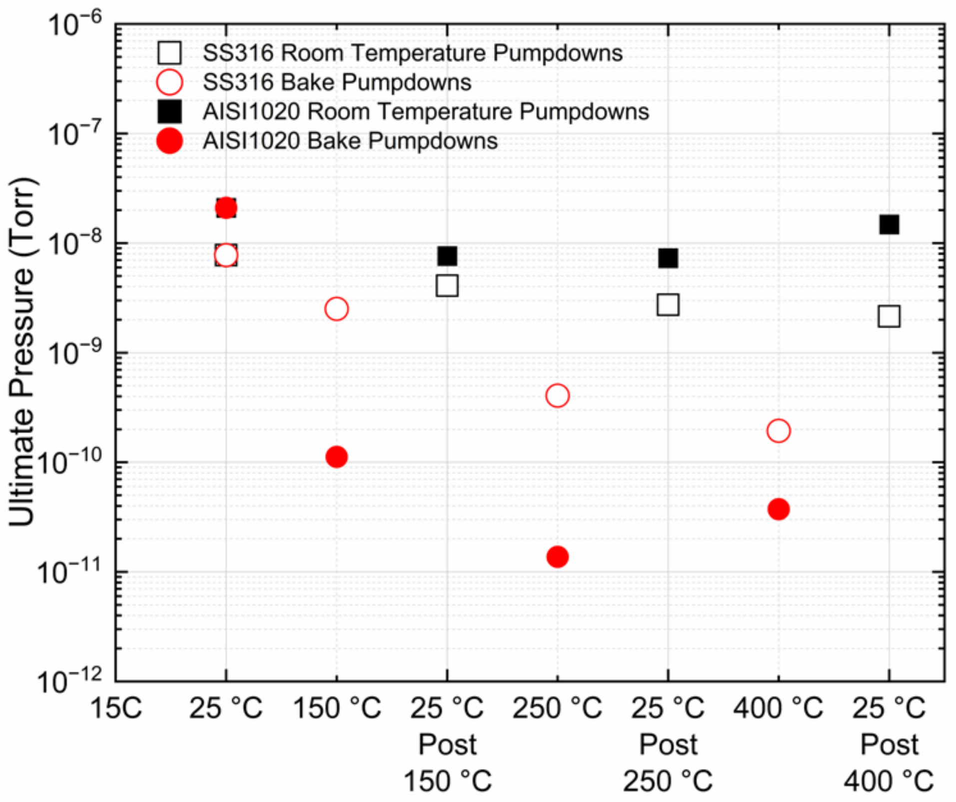




Ultimate Pressure Measurements

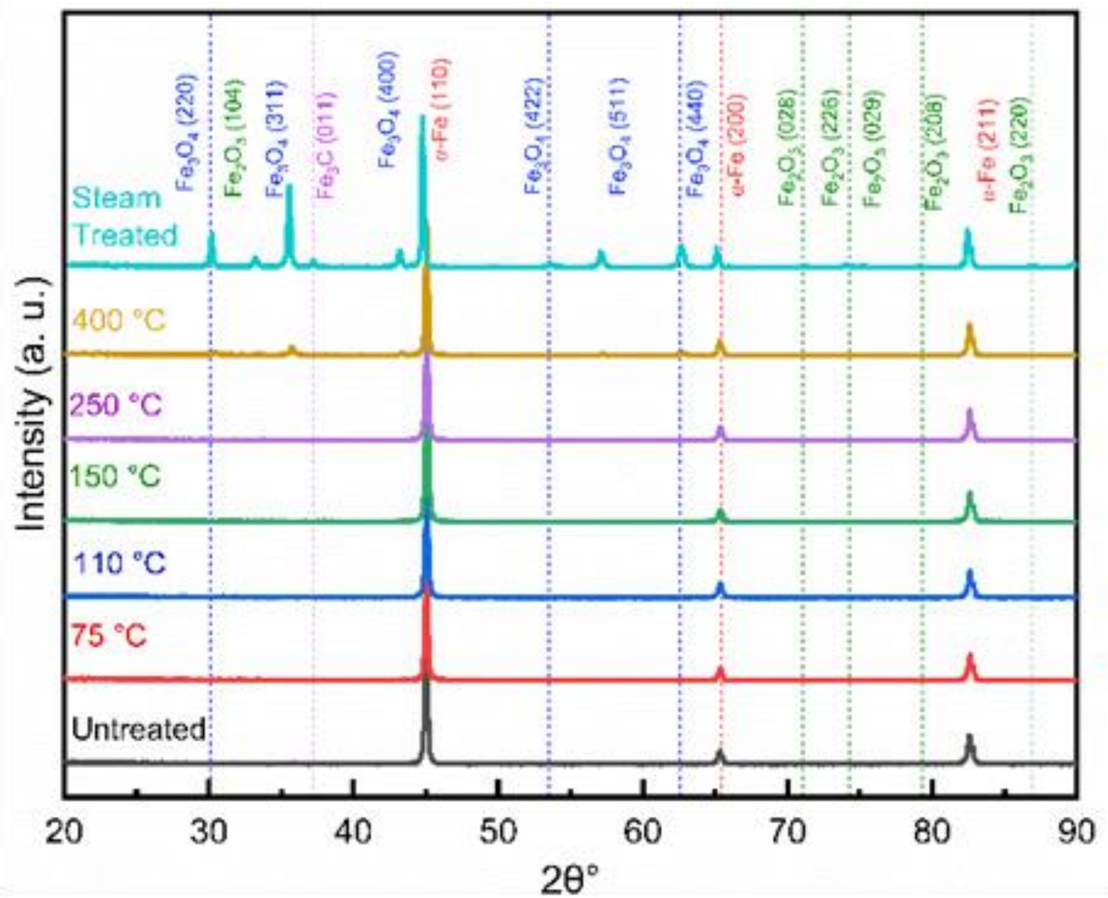
- Magnetite surface layers have garnered significant interest due to indications that they may enable rapid pumpdown at modest temperatures

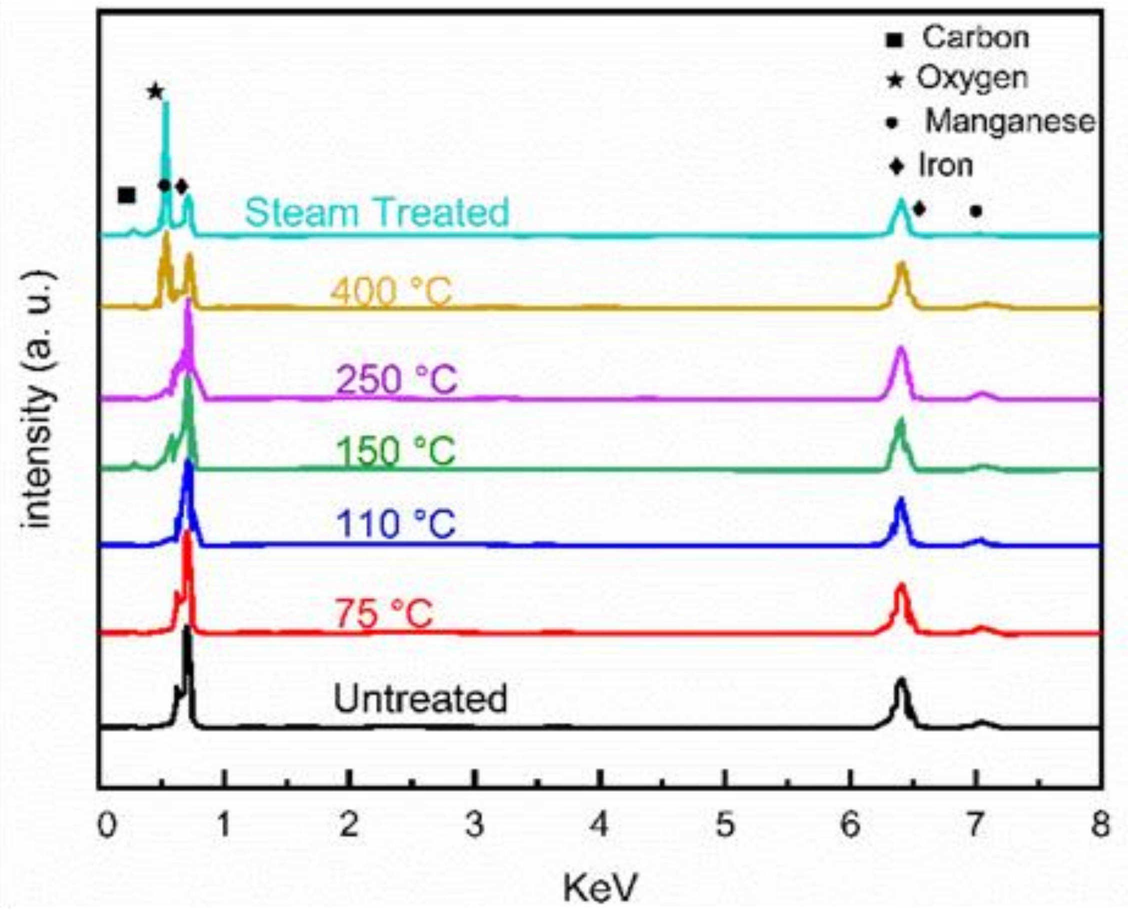
- We indeed created a magnetite coating on the interior surface of the low carbon steel tubes.



After heat treatment, the tubes were vented to air and then pumped down again.
Stainless achieved lower pressures following repeated heat treatment
Low carbon steel, not so much, suggesting surface transformation to magnetite did not provide a benefit

Surface Science Evaluation





- Magnetite Layer Formation:
  - Heat treatments caused Magnetite (Fe_3O_4) and Hematite (α-Fe_2O_3) to form on the AISI 1020 surface above 250°C.

Surface Science Evaluation

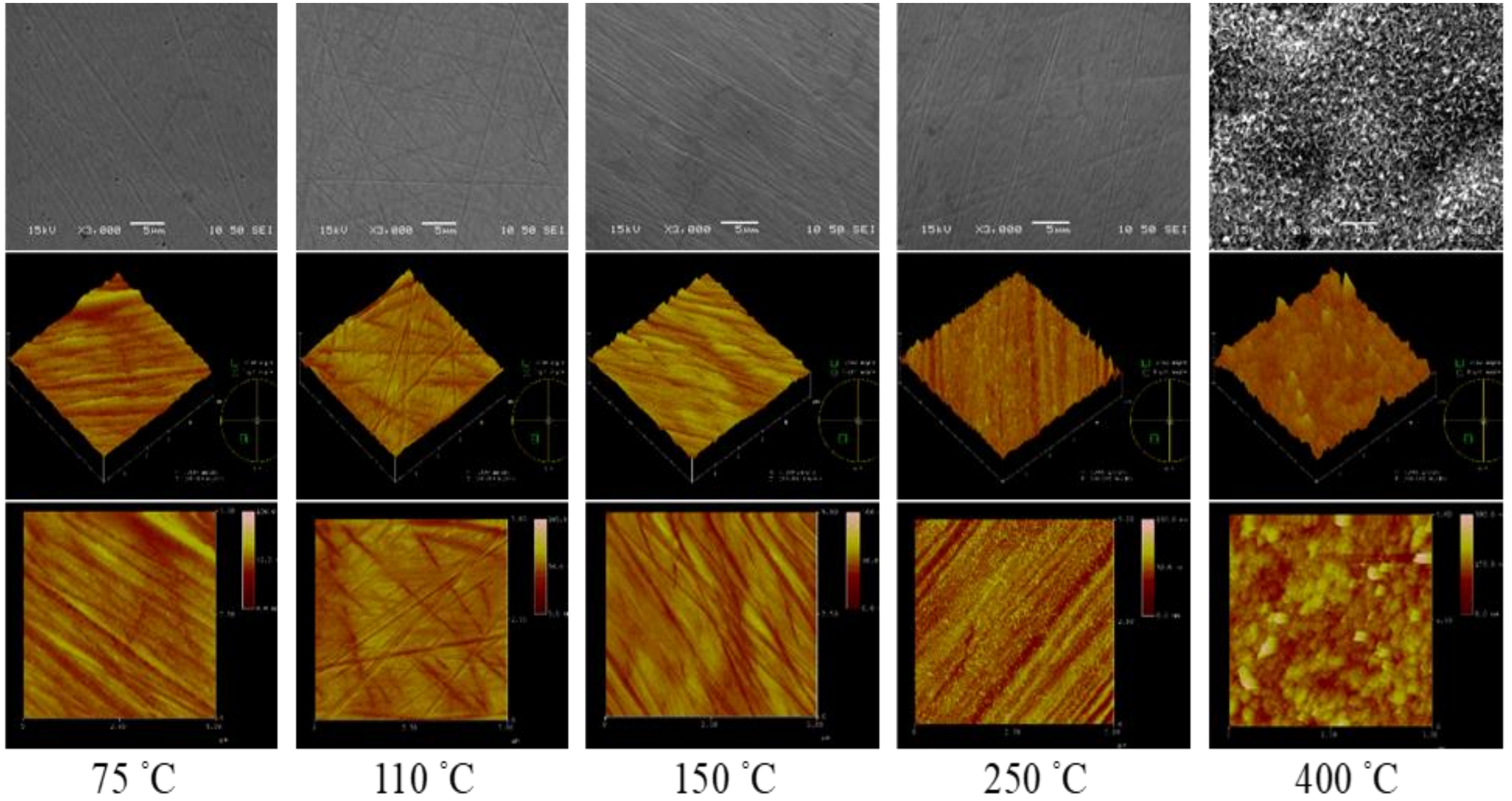
•During the **ultimate pressure experiments**, **venting to air** at various temperatures and **post-400°C degradation** were observed in AISI 1020.
•Surface science analysis was conducted to understand the **microscopic and macroscopic transformations** and their impact on the vacuum performance.

Experimental Setup:
•**AISI 1020 coupons** (5 mm^2 and 10 mm^2 surface area, 2.7 mm thickness) were cleaned and annealed at **25°C, 75°C, 110°C, 150°C, 250°C, and 400°C** to simulate different stages of the ultimate pressure experiment.
•**Analysis Methods**:
•**Energy-Dispersive X-ray Spectroscopy (EDS)**: Quantified **elemental composition**, focusing on **oxygen uptake** as a measure of oxidation.
•**Scanning Electron Microscopy (SEM)**: Investigated **surface morphology** and microscopic changes post-annealing.
•**Atomic Force Microscopy (AFM)**: Provided detailed information on the **surface roughness** and texture changes.

Why It Matters:
•These analyses helped identify the **oxidation behavior** and **surface degradation** of AISI 1020, especially after exposure to air and temperatures beyond **400°C**.
•Understanding surface transformations is crucial for improving the material's performance in **UHV conditions**, especially after high-temperature bakes.

Surface Science Evaluation

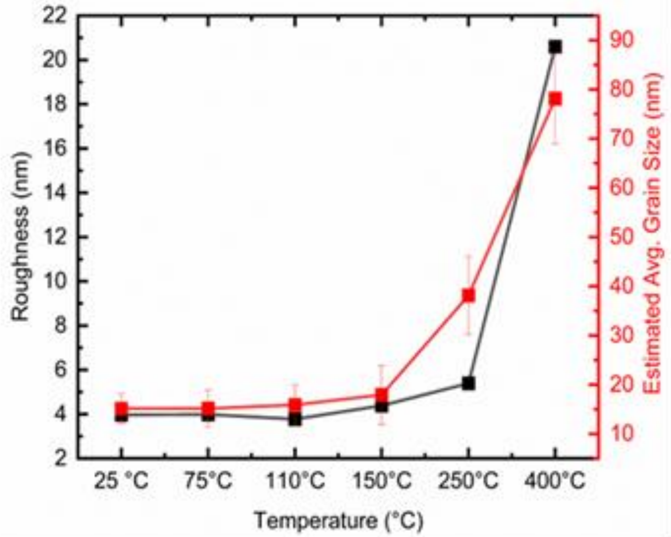
- **SEM and AFM results** showed increased surface roughness and grain growth:
  - Roughness increased from **3.988 nm (75°C)** to **20.598 nm (400°C)**.
  - Grain size increased from **15.15 nm (75°C)** to **78.14 nm (400°C)**.

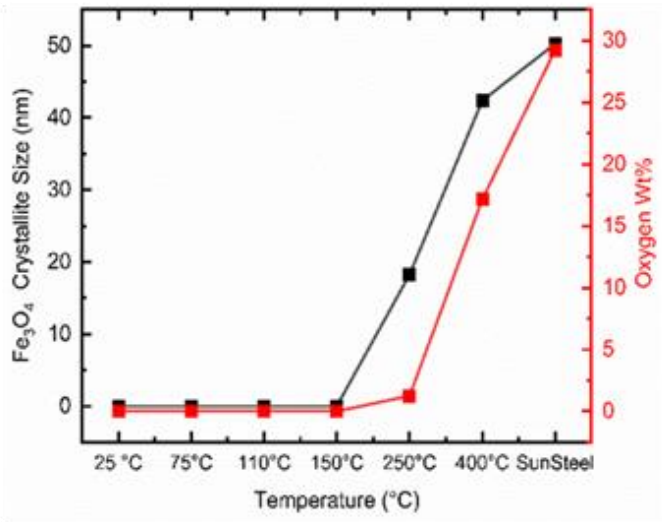


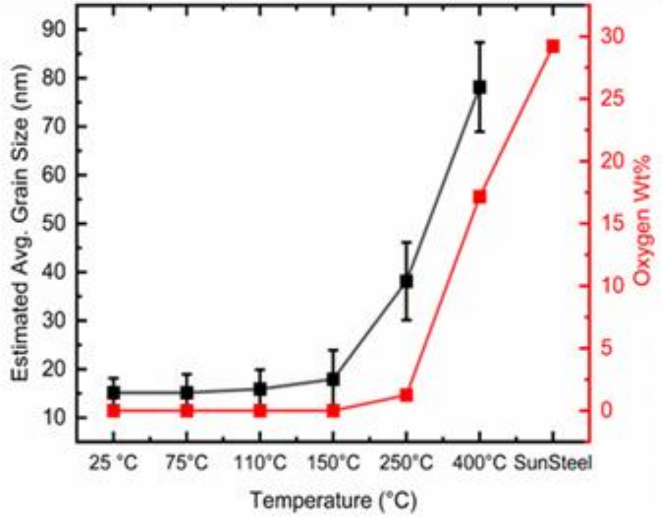
Surface Science Evaluation

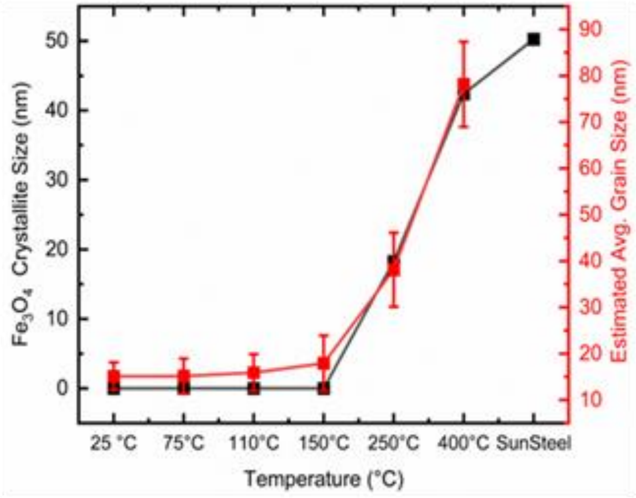
Oxidation Kinetics and Implications

- SEM and AFM results showed increased surface roughness and grain growth:
  - Roughness increased from 3.988 nm (75°C) to 20.598 nm (400°C).
  - Grain size increased from 15.15 nm (75°C) to 78.14 nm (400°C).
- Oxygen Uptake: Oxygen content increased significantly:
  - 1.73% (150°C) to 30.64% (400°C) for 5 mm² samples.
  - 0% (150°C) to 17.16% (400°C) for 10 mm² samples.









Surface Science Evaluation

- **AISI 1020** achieved significantly lower **hydrogen outgassing rates** and ultimate pressure compared to **SS316** after high-temperature bakes.
- The lowest ultimate pressure (~$\mathbf{1x10^{-11}}$ **Torr**) was achieved after a **250°C bake**, while higher temperatures (400°C) resulted in surface degradation and increased outgassing.
- **Surface oxidation**, particularly the formation of a **magnetite layer** (Fe_3O_4), was observed after venting to air at high temperatures. This magnetite layer played a critical role in surface transformations and altered outgassing behavior.

- **Implications for Polarized Gun Sources**:
- The **lower hydrogen outgassing rates** of AISI 1020, especially after moderate baking at **250°C**, offer promising benefits for **polarized electron gun sources**, where maintaining ultra-high vacuum is critical to preserving **quantum efficiency (QE)** of **GaAs photocathodes**.
- The **magnetite layer**, being a dark-colored coating, can contribute to **light absorption**, potentially reducing **stray reflections** and improving system stability in sensitive optical systems such as polarized electron sources.
- The ability of **AISI 1020** to achieve **XHV conditions** with minimal pre-treatment compared to stainless steel suggests it could be a **cost-effective alternative** for vacuum chambers in polarized gun sources, reducing contamination and improving the longevity of electron sources.