A review of recent outgassing studies on mild and stainless steels

(mostly mild steel)

CERN, JLAB, NIST, WM

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Beampipes for Gravitational Wave Telescopes 2023
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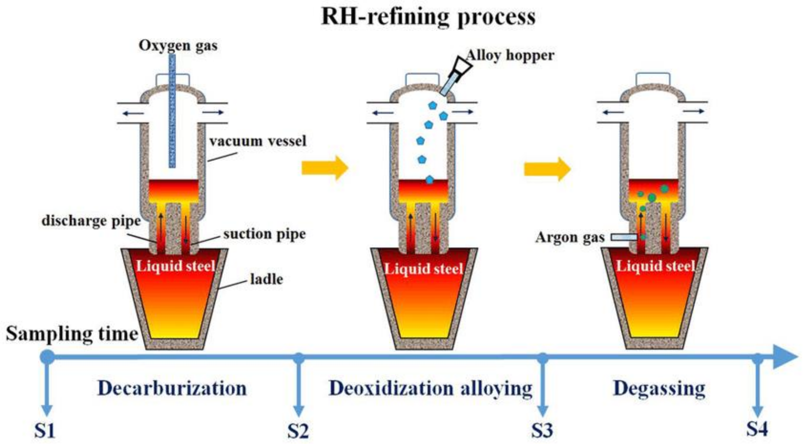
Introduction: Why Mild Steel for Vacuum Systems?

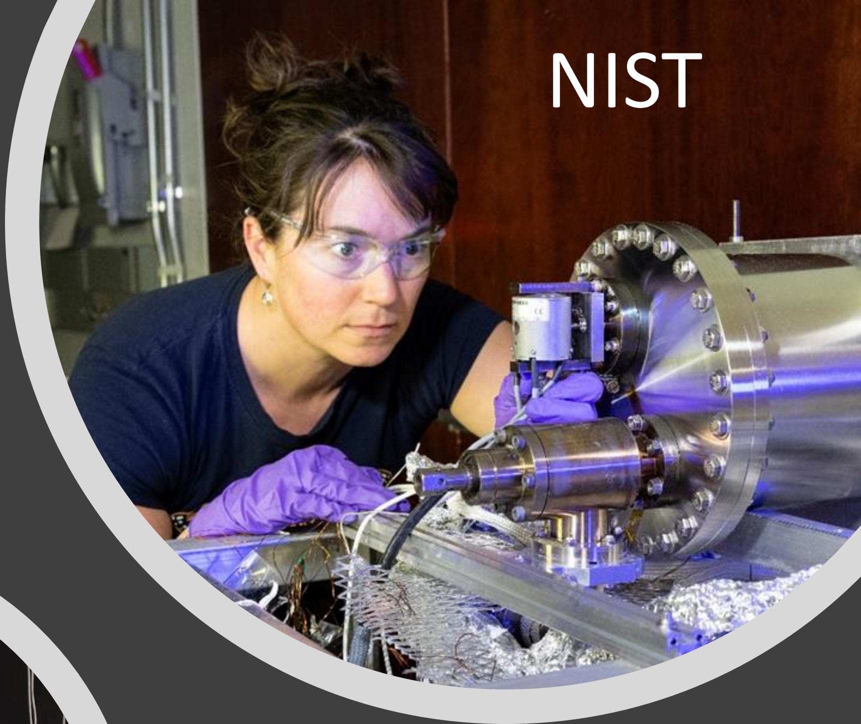
- Commonly used as structural steel or for pipes
- Historically, not considered for UHV systems
- Modern secondary refining processes may reduce H_2
- Possible low-cost alternative to stainless steel
    - Gravity Wave Detectors
    - Large vacuum systems
- Park et al (2016) obtained excellent outgassing results for 3 Korean mild steels
- CERN, JLAB, NIST & WM have confirmed Park et al's H_2 results
- Pump-down (water outgassing) of mild-steel still a subject of research

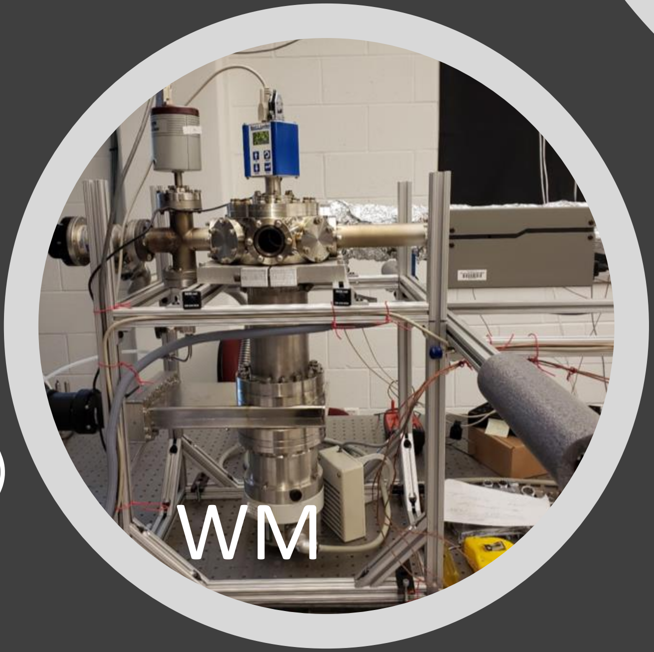


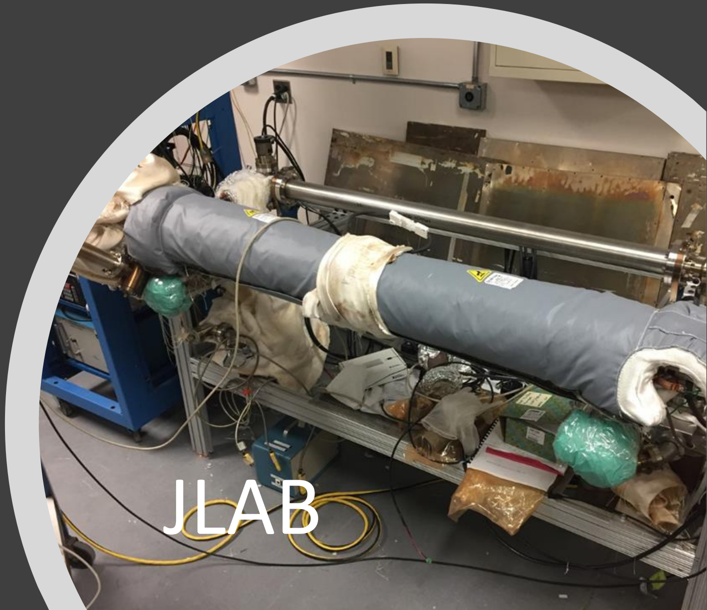
Outline

<u>Recent mild-steel results from NIST, JLAB, CERN, W&M:</u>

- Brief History of mild-steel outgassing
- Brief description of apparatus
    - National Institute for Standards and Technology (NIST)
    - Jefferson National Laboratory (JLAB)
    - William & Mary (WM)
    - CERN described in more detail by Ivo and Carlo
- Compare H_2 outgassing results (after 150 °C bake)
- Compare water outgassing results (pumpdown)







Why Should Mild Steel have low outgassing?

To my knowledge, since 1950s, **mild steel** production **often** uses the **Ruhrstahl-Heraeus** process (RH) or other degassing process for **hydrogen reduction** and decarburization.

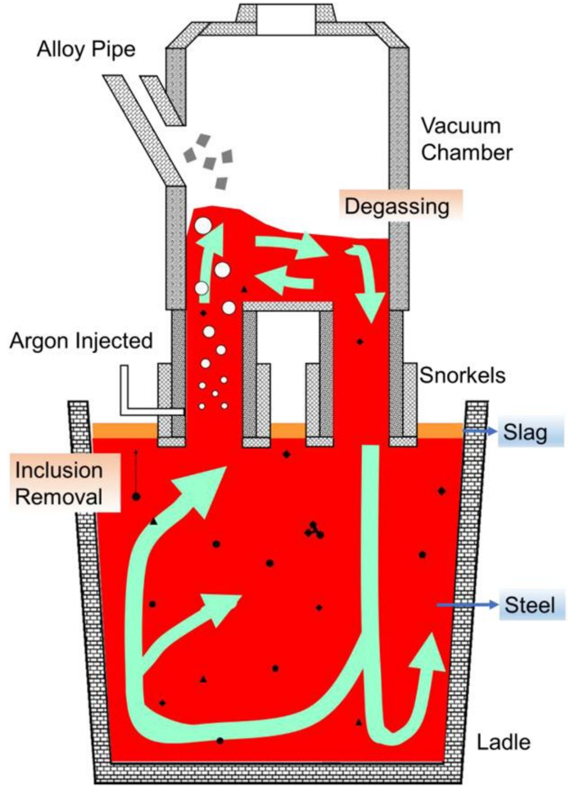
- RH is a secondary metallurgy process in which liquid steel is subjected to a vacuum treatment for decarburization and degassing H_2 and O_2

Stainless steel is typically produced from recycled steel in an electric arc furnace

- As produced, **most stainless steels contain significant dissolved H_2**
- High temperature **heat-treatment** (vacuum-firing) is required to

We sent out 4 samples for hydrogen concentration testing

Sample Identification:	Hydrogen ppm
A36 Steel # 1	0.5
A36 Steel # 2	0.5
304L Blank	3.6
316L Blank	1.9



RH process

Older Literature Values on Mild Steel Outgassing

Table 4. Metals

Material	K_1 torr l. s^{-1} cm^{-2} × 10^{10}	α_1	K_{10} torr l. s^{-1} cm^{-2} × 10^{10}	α_{10}	Ref
Aluminium (fresh)	63	1.0	6.0	1.0	9
Aluminium (degassed 24 h)	41.4	3.2	3.06	0.9	9
Aluminium (3 h in air)	66.5	1.9	4.75	0.9	9
Aluminium (fresh)	62	1.0	3.25	0.9	9
Aluminium (anodised-2 μm pores)	2760	0.9	322	0.9	9
Aluminium (bright rolled)	—	—	75	1	13
Duralumin	1700	0.75	350	0.75	13
Brass (wave-guide)	4000	2.0	100	1.2	13
Copper (fresh)	400	1.0	41.5	1.0	9
Copper (mech. polished)	35	1.0	3.56	1.0	9
OFHC copper (fresh)	188	1.3	12.6	1.3	9
OFHC copper (mech. polished)	19	1.1	1.63	1.1	9
Gold (wire fresh)	1580	2.1	5.1	1	9
Mild steel	5400	1	500	1	13
Mild steel (slightly rusty)	6000	3.1	130	1	13
Mild steel (chromium plated polished)	100	1	9.0	—	13
Mild steel (aluminium spray coated)	600	0.75	100	0.75	13
Steel (chromium plated fresh)	70.5	1	5.8	1	9
Steel (chromium plated polished)	91	1	8.0	1	9
Steel (nickel plated fresh)	42.4	0.9	4.94	0.9	9
Steel (nickel plated)	27.6	1.1	2.33	1.1	9
Steel (chemically nickel plated fresh)	83	1	7.05	1	9
Steel (chemically nickel plated polished)	52.2	1	4.6	1	9
Steel (descaled)	3070	0.6	2950	0.7	9
Molybdenum	52	1.0	3.67	1	9
Stainless Steel EN58B	—	—	14	1.6	13
Stainless Steel 18/9/1 (electro polished)	—	—	2	—	15
(vapour degreased)	—	—	1	—	15
(diversey cleaned)	—	—	3	—	15
Stainless steel	1750	1.1	210	0.75	13
Stainless steel	900	0.7	200	0.75	13
Stainless steel ICN 472 (fresh)	135	0.9	14.7	0.9	9
Stainless steel ICN 472 (sanded)	82.8	1.2	10.4	0.8	9
Stainless Steel NS22S (mech. polished)	17.1	0.5	4.6	0.7	9
Stainless Steel NS22S (electro polished)	42.8	1.0	4.28	1.0	9
Stainless Steel NS22S	144	1.3	13.5	1.9	9
Zinc	2210	1.4	322	0.8	9
Titanium	113	0.6	18.4	1.1	9
Titanium	40	1.0	3.68	1	9

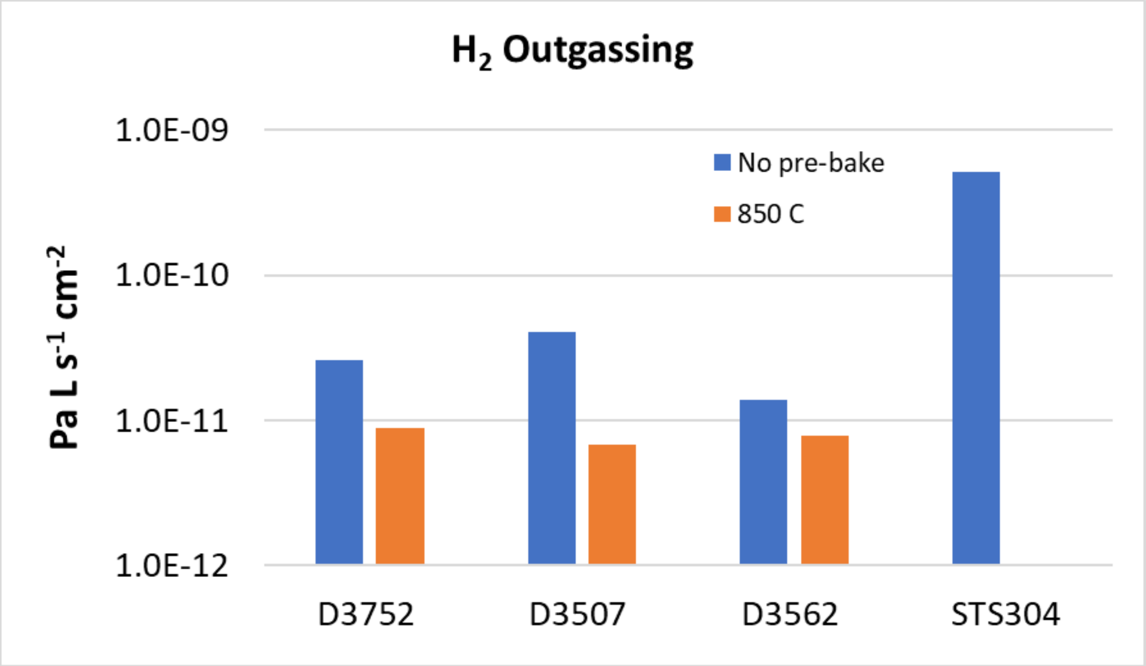
Elsey, R. J. (1975). Outgassing of vacuum materials-II. Vacuum, 25(8), 347–361.
https://doi.org/10.1016/0042-207X(75)91653-X

- Elsey (1975) and Ishimori et al (1971), for e.g., show mild steel outgassing higher than stainless steel
- Back in the 1990's, Dylla and Blanchard questioned the values of outgassing for mild steel relative to SS
- According to Park et al. (2016), vacuum degassing was developed in the 1950's, but only 10% of Japanese plants used the process by the 1970's

Park et al 2016 H_2 Outgassing

3 Korean Mild Steels Tested: D3752, D307, D3562

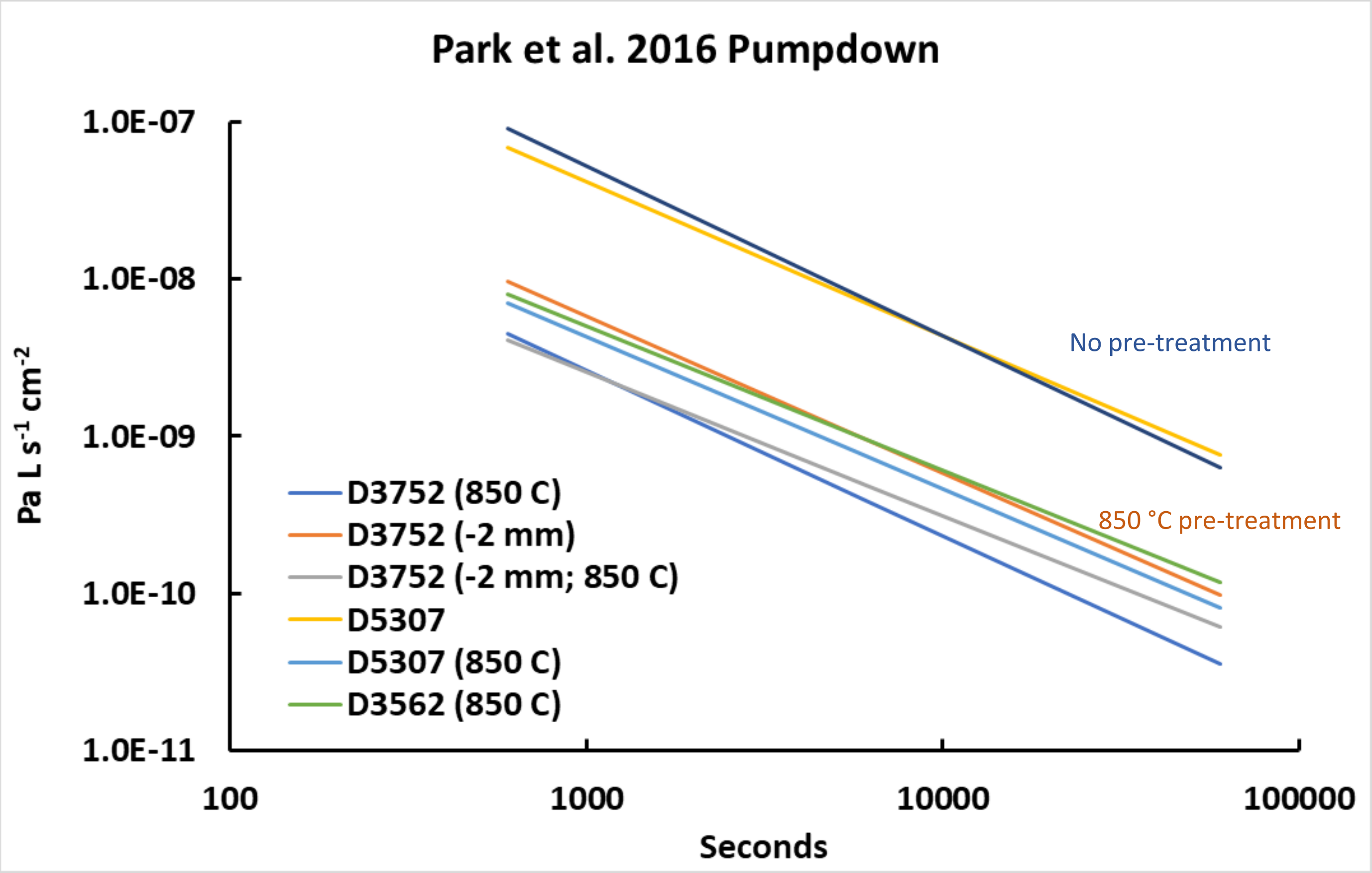
- Rate-of Rise (RoR) with spinning rotor gauge (SRG)
- 304 measurements similar to NIST and other benchmarks
- Mild steel better by more than 10X
- 850 °C shows modest improvement
  - (this will not be true for H_2O outgassing)
- QUESTION: was there a background subtraction for non-mild-steel components?
  - Very important for low outgassing RoR measurements



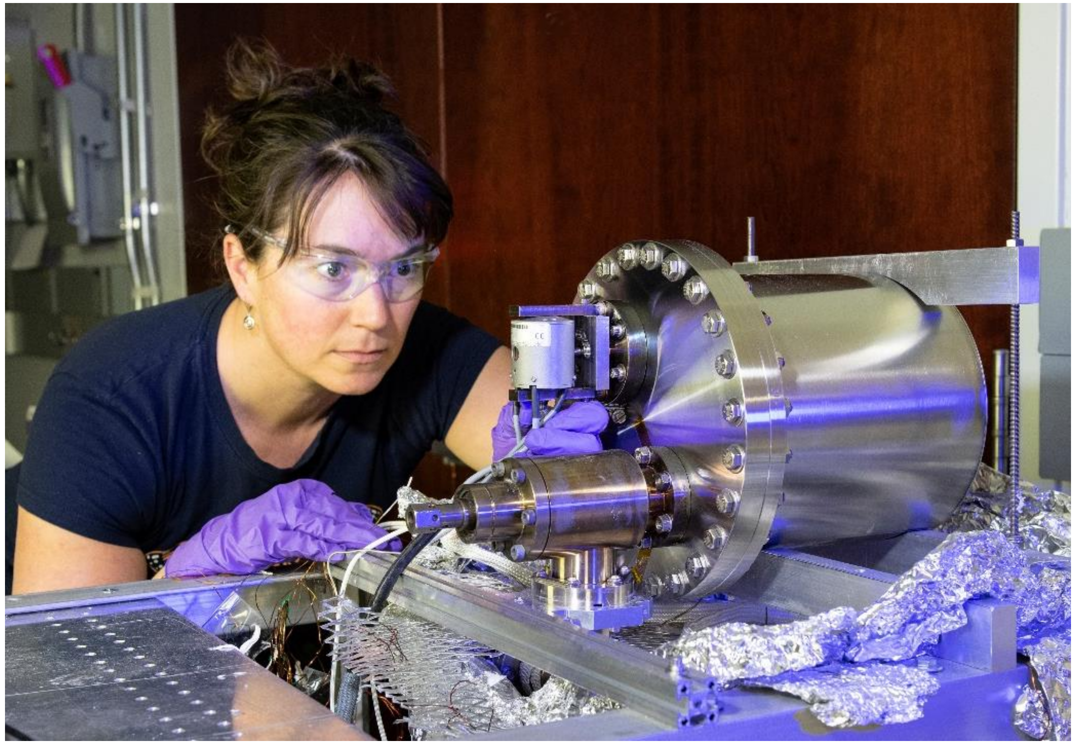
sample chambers were baked at 150 °C for 48 h

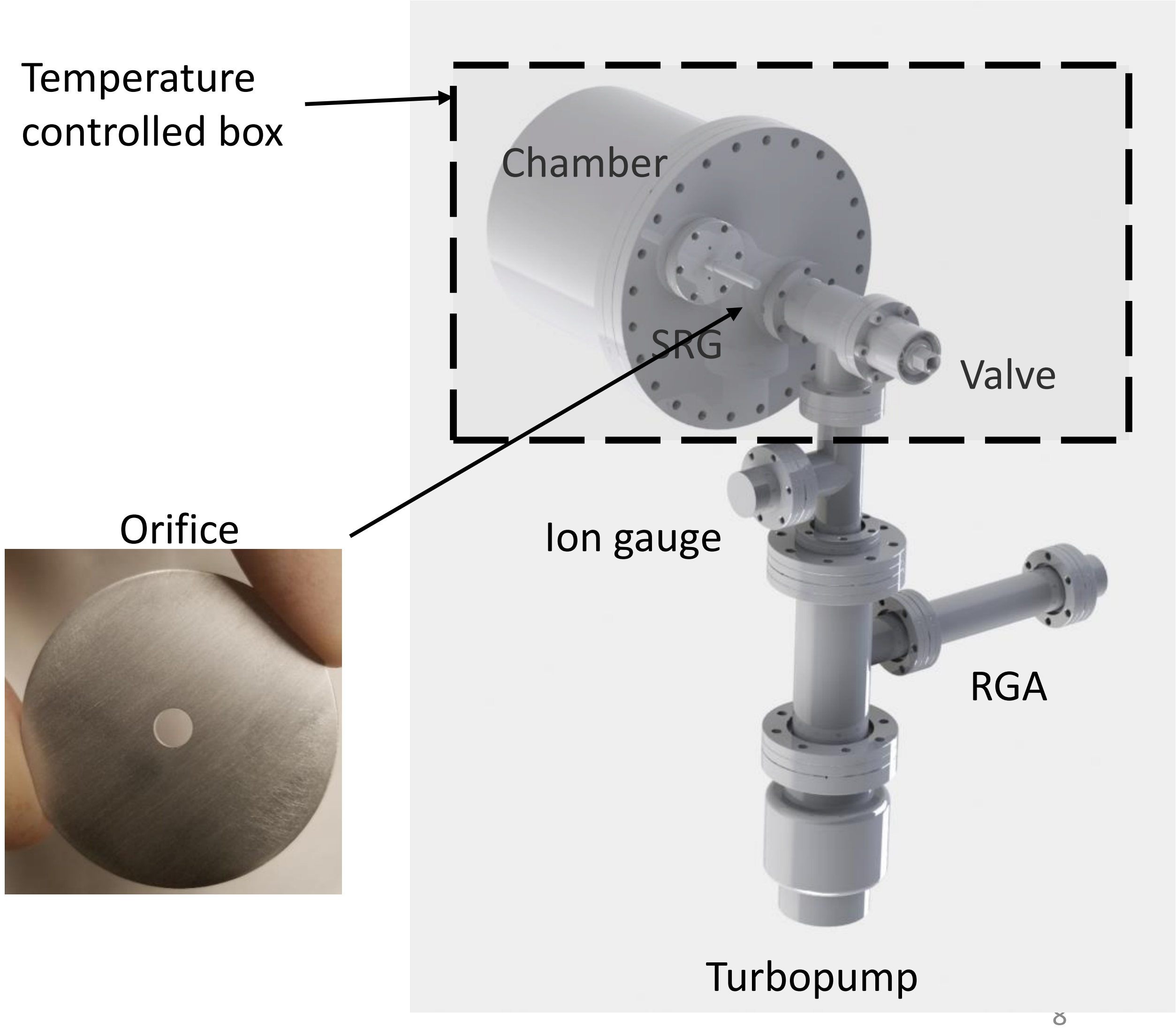
Park et al 2016 Pumpdown Curves

- Outgassing was measured at 24 °C after 48-h in situ bakeout followed by a 5-h N_2 exposure
- Not an air exposure?
- 2 to 3 orders of magnitude lower than stainless steel exposed to air
  - Same slope
- Baking at 850 °C lowed H_2O outgassing



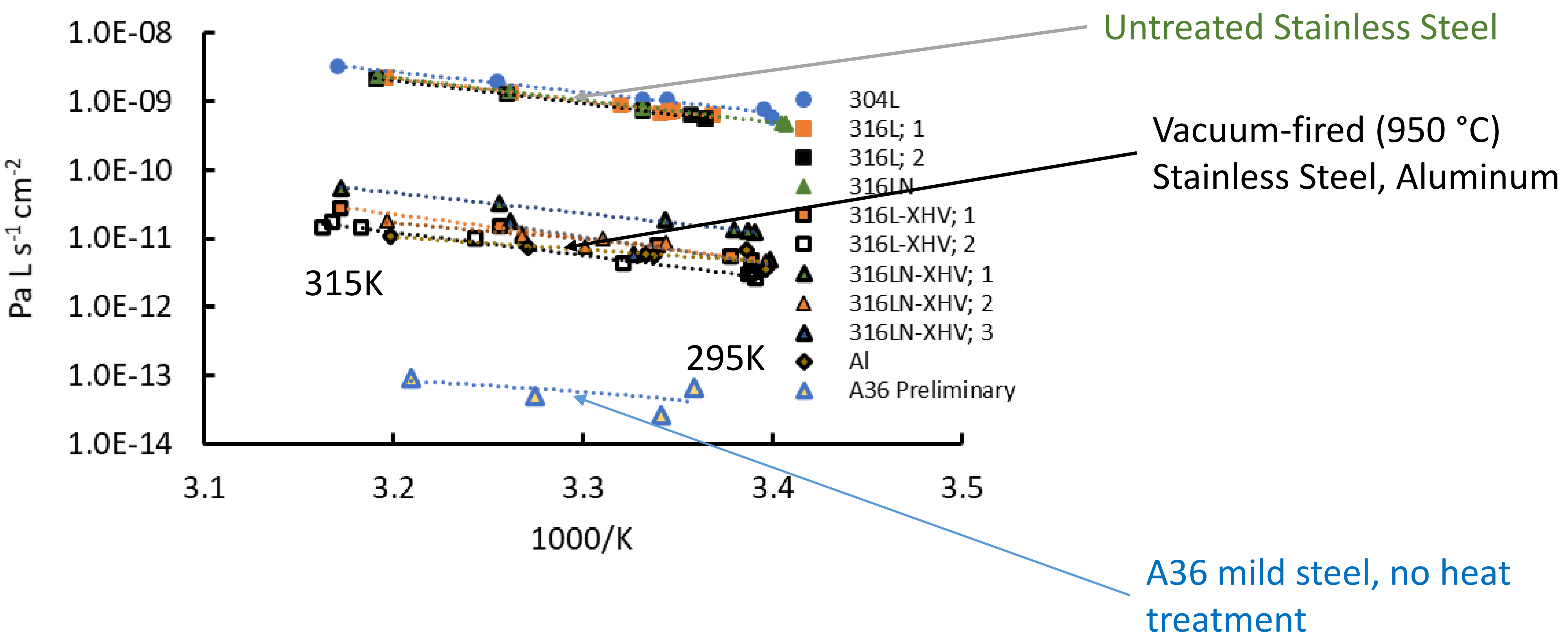
NIST Outgassing Apparatus





- Published outgassing results for 7 geometrically identical chamber– Applied Vacuum Division
    - ≈ 6.4 L
    - ≈ 2000 cm^2
- H_2 Outgassing: rate of rise (RoR)
- Pumpdown: throughput method
- We add A36 mild steel

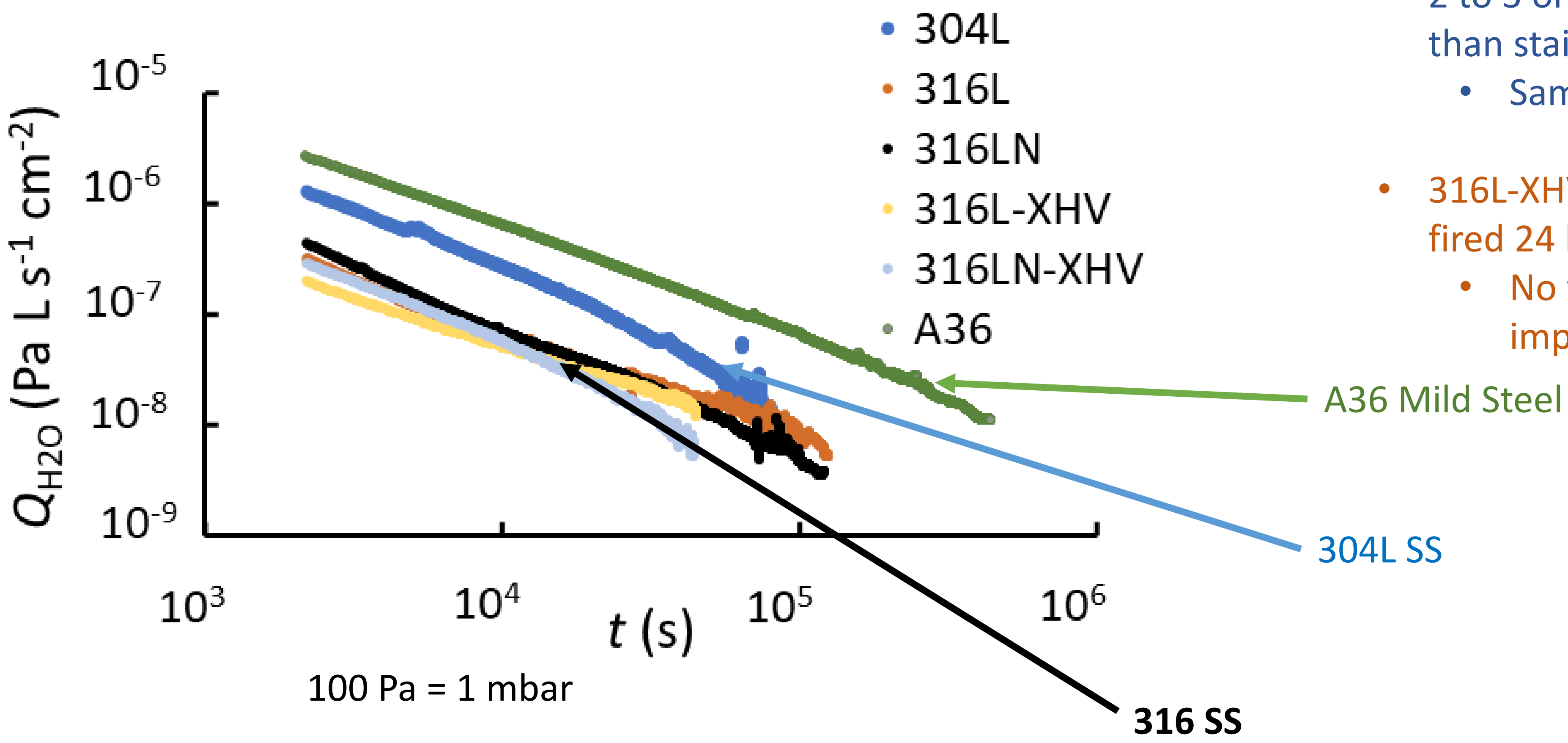
NIST H_2 Outgassing data (A36 PRELIMINARY DATA)



100 Pa = 1 mbar

Chambers baked 125-150 °C for ≥ 3 days

NIST Water Outgassing data
(A36 PRELIMINARY DATA)

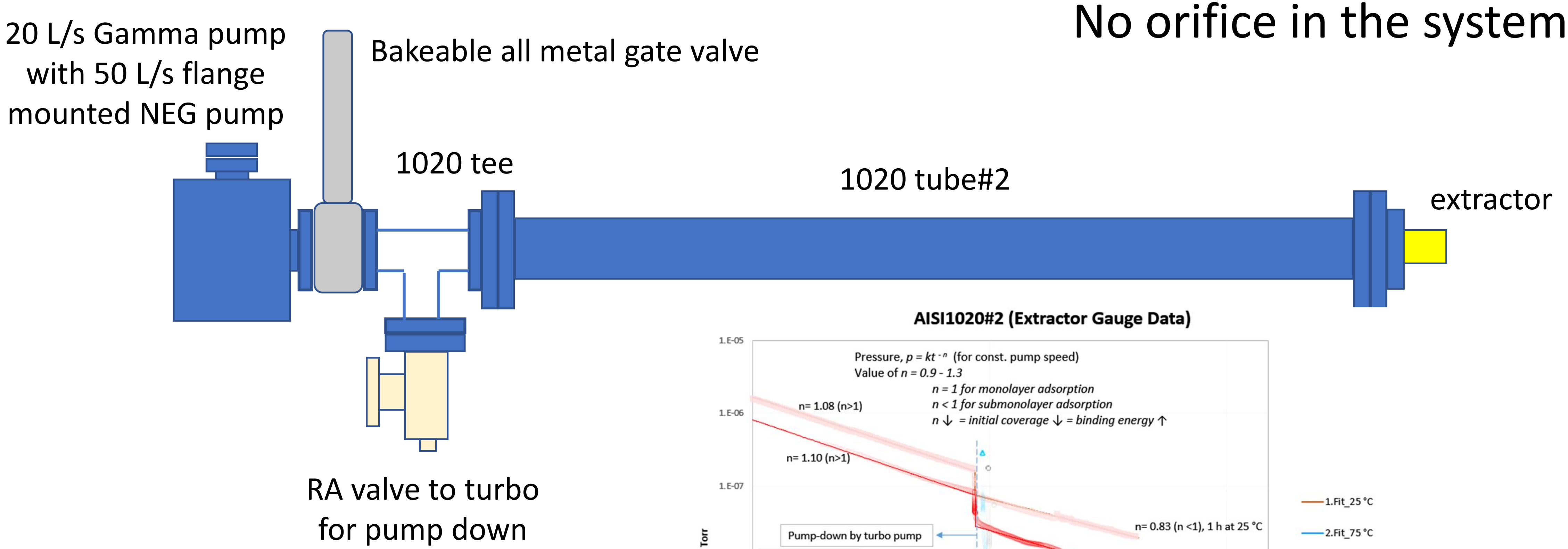


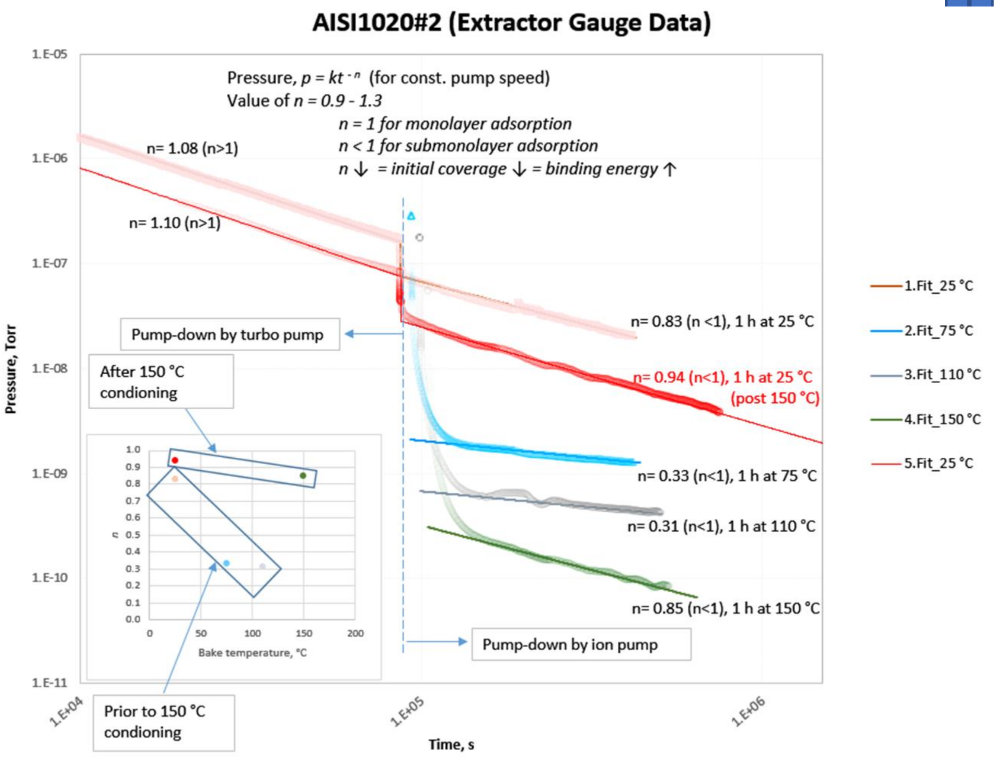
100 Pa = 1 mbar

- Exposed to Air
- 2 to 3 orders of magnitude lower than stainless steel exposed to air
  - Same slope
- 316L-XHV and 316LN-XHV vacuum fired 24 h at 950 C.
  - No water outgassing improvement

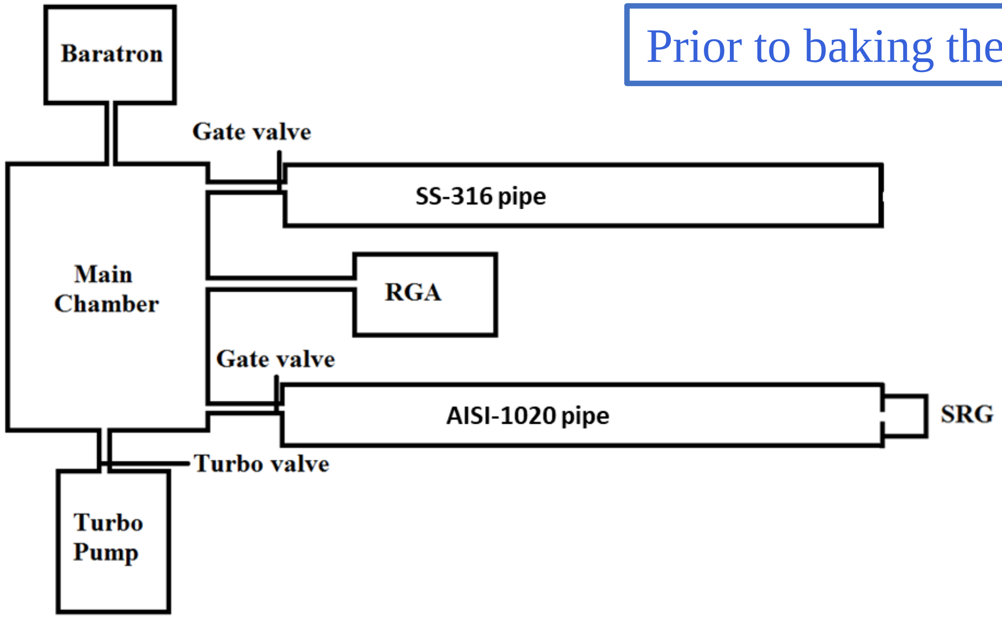
Pump Down setup

No orifice in the system





Jefferson Lab



Prior to baking the pipe

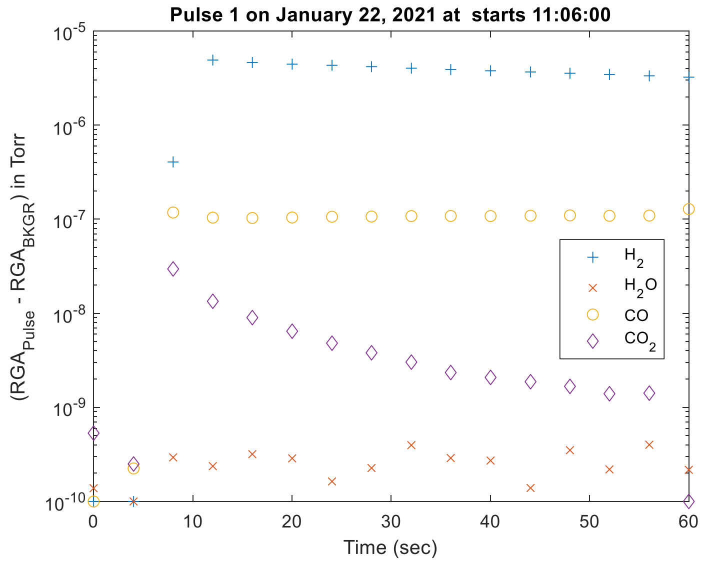
To determine the gas composition, we use a pulse gas release method:

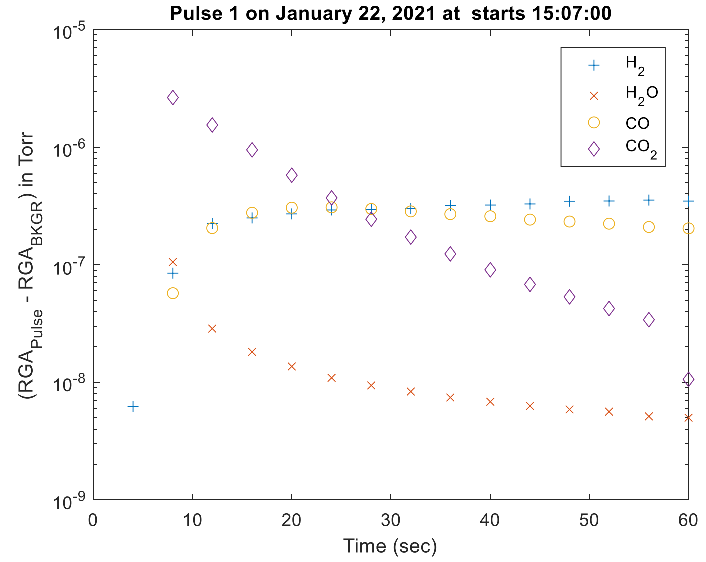
1. Begin by closing Turbo (4-5 seconds) to monitor outgassing of main chamber
2. Pulse pipe (for 1-3 seconds) to equilibrate pressures. The pipe and the RGA chamber have nearly equal volumes.
3. Observe the **time dependent** signals at selected *amu*. With *amu* = [2 14 15 16 18 28 44] scan time is reduced to 2 or 4 seconds.

H_2 dominates by two orders of magnitude at 24C

The CO_2::H_2 ratio increases at temperatures above 60C

CO_2 is 10 times larger than H_2 at 70C.

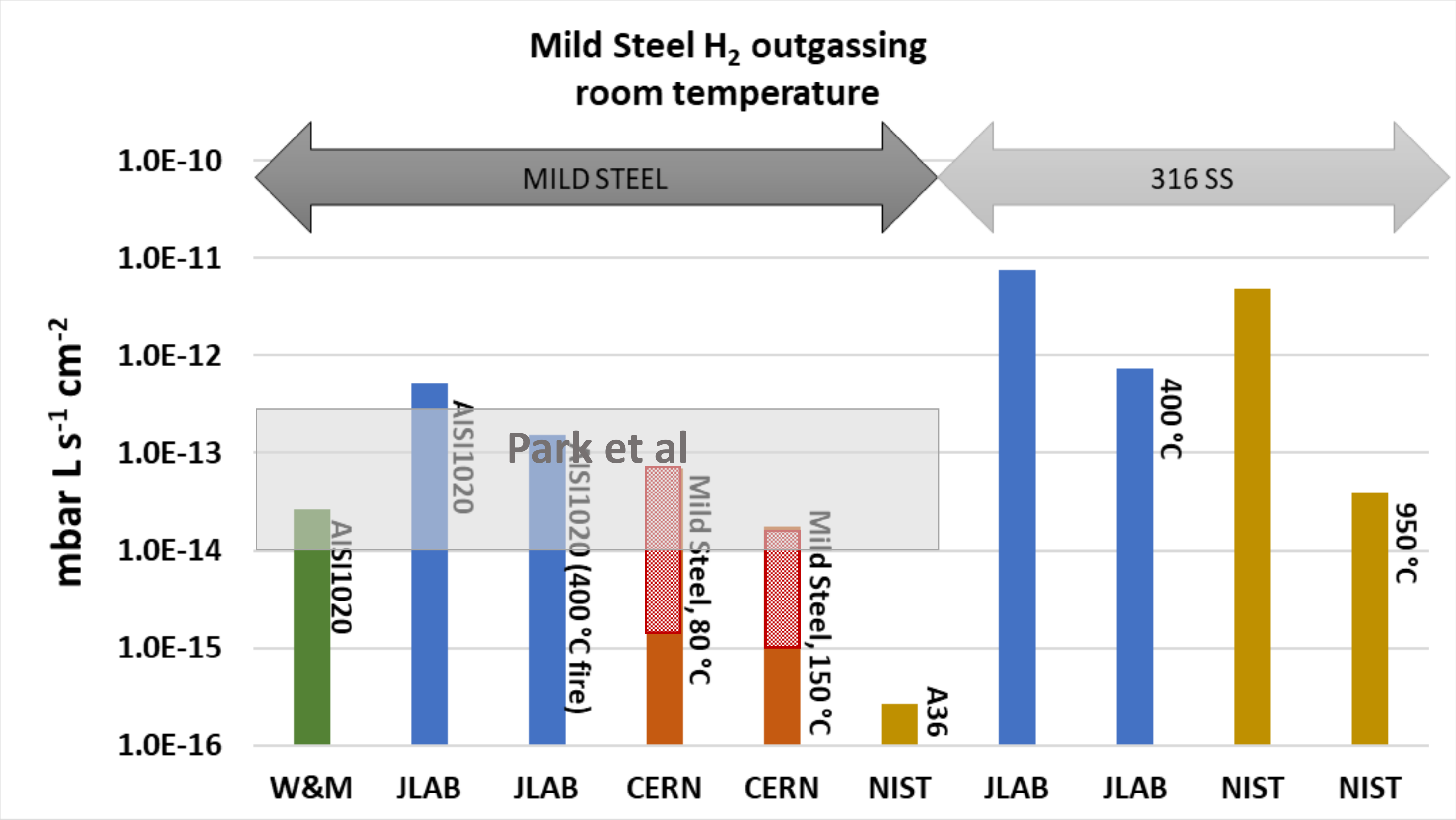




Mild Steel H_2 outgassing

Lab	Mild Steel	mbar L s-1 cm-1
W&M	AISI1020, 150 °C, 48 h bake	2.6E-14
JLAB	AISI1020, 150 °C, 69 h bake	5.2E-13
JLAB	AISI1020 (pre-bake 400 °C), 150 °C, 48 h bake	1.5E-13
CERN	Mild Steel, 80 °C, 48 h	1.5E-15
CERN	Mild Steel, 80 °C, 48 h	7.0E-14
CERN	Mild Steel, 150 °C, 48 h	1.0E-15
CERN	Mild Steel, 150 °C, 48 h	1.7E-14
NIST	A36, 299 K, 100-150 C bake, 5 days	2.6E-16

Lab	Stainless Steel	mbar L s-1 cm-1
JLAB	SS316, 150 °C, 48 h bake	7.6E-12
JLAB	SS316 (pre-bake 400 °C) 150 °C, 49 h	7.3E-13
NIST	316L	4.9E-12
NIST	316L-XHV (950 °C Fire)	3.9E-14



- 150 °C, minimum 48 hour bakes (except where noted)
- All exposed to air before bake
- Other constituents (H_2O, CO CO_2) likely present

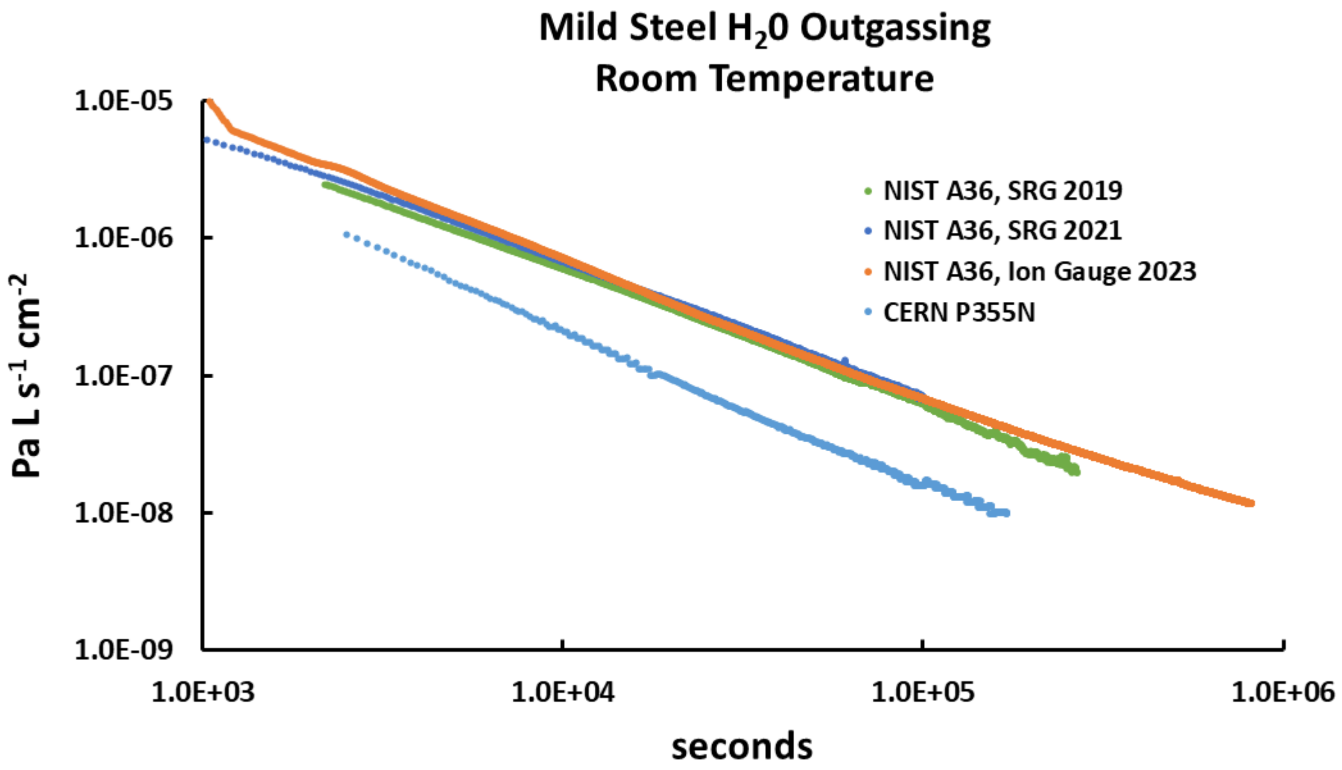
Evidence suggests that for a wide variety of mild steels H_2 outgassing will not be a concern for most applications

Mild Steel H_2O Outgassing Comparison

Throughput Method: NIST & CERN

- NIST has measured A36 3 times over 4 years
  - Two different gauges: SRG & IG
  - Very consistent measurements

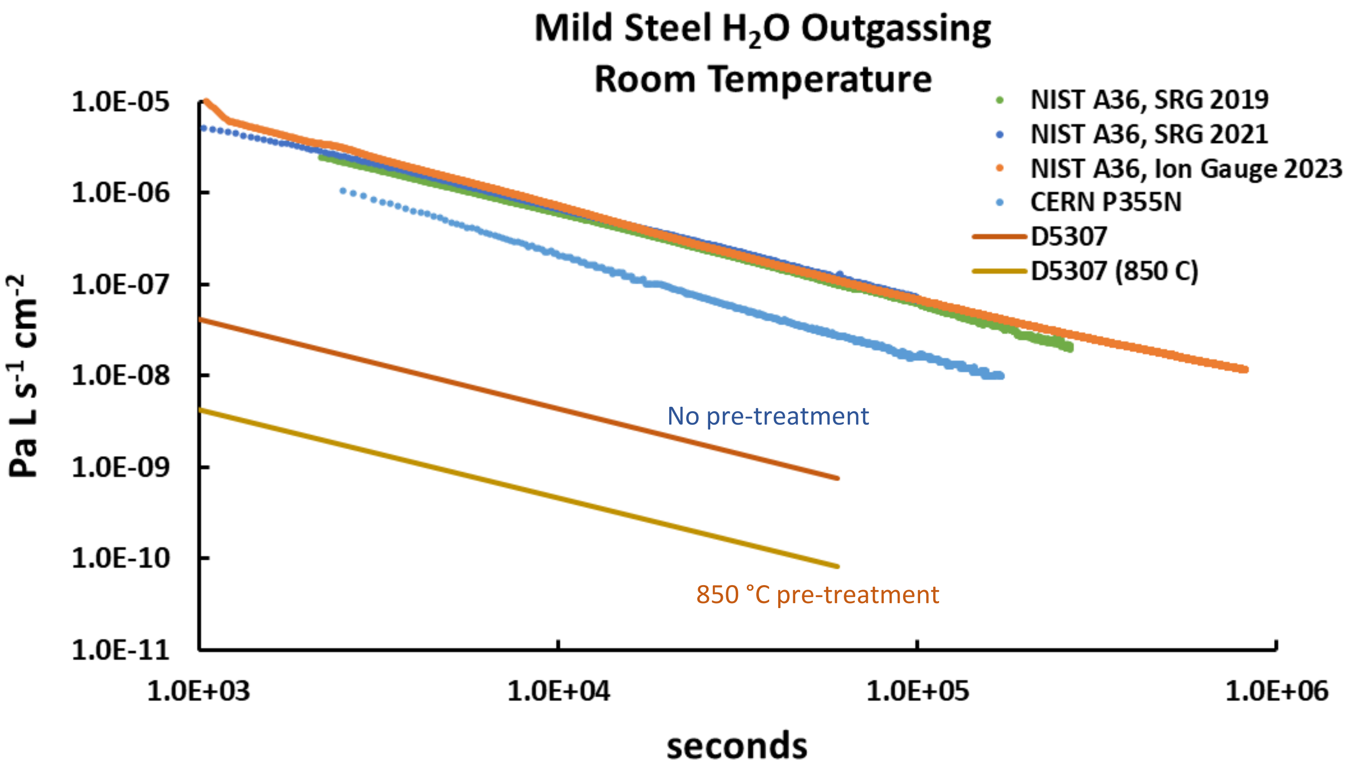
- CERN measured 355N mild steel
  - 355N may be close to order of magnitude lower than A36
  - Similar slope



Mild Steel H_2O Outgassing Comparison

Throughput Method: NIST, CERN & Park

- Park et al 2016 results for D3507 (Korean) are 1 to 3 orders of magnitude lower
  - They probably vented with N_2 not air
- Similar slope to CERN and NIST

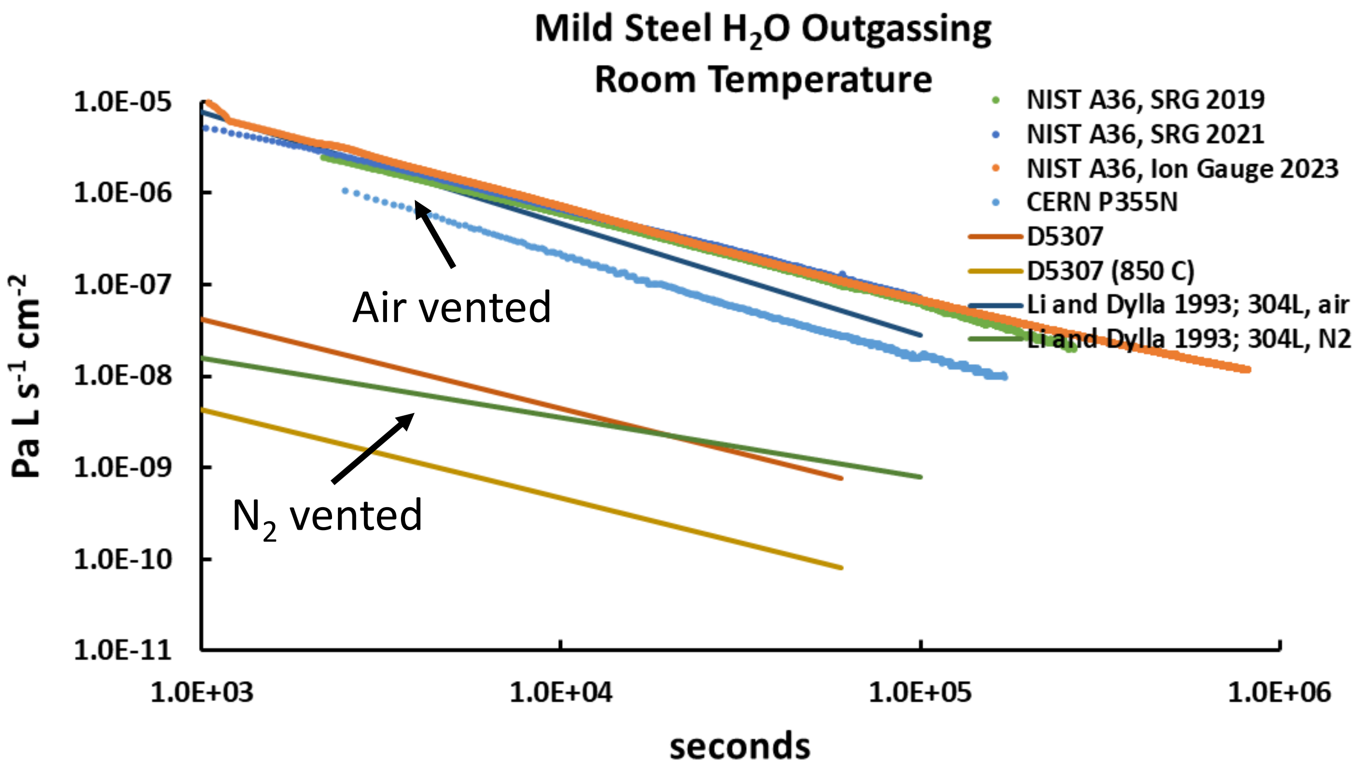


Mild Steel H_2O Outgassing Comparison

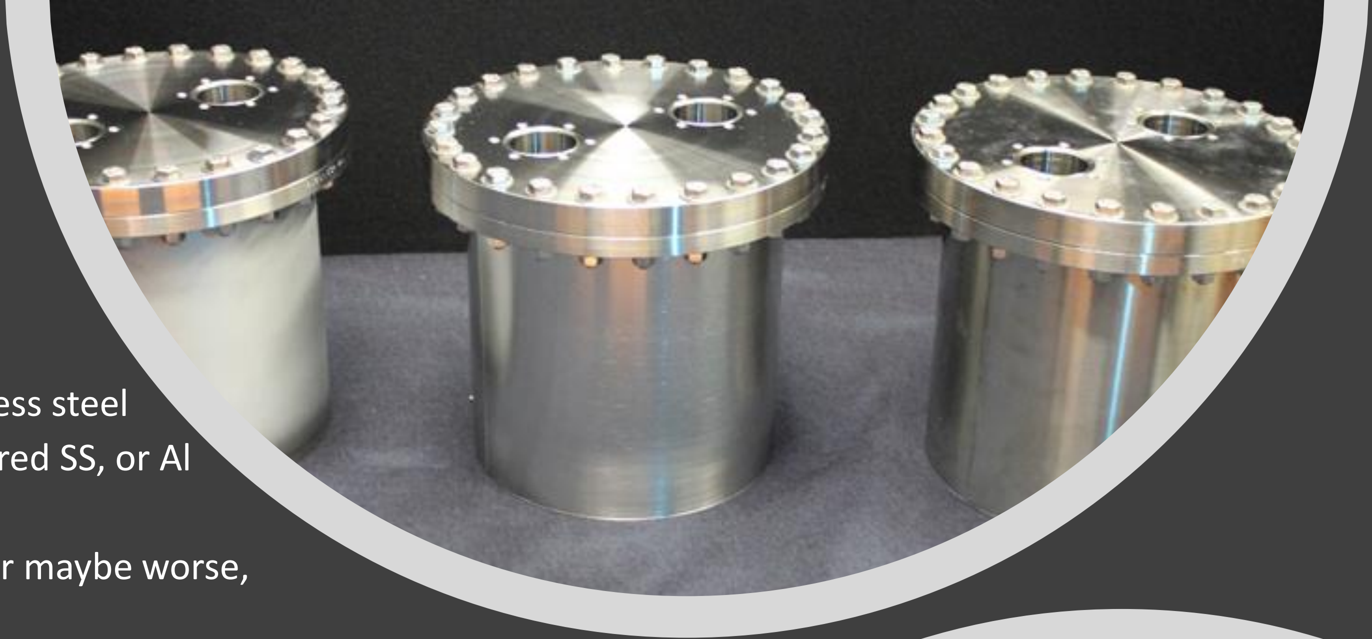
Throughput Method: NIST, CERN, Park, & Dylla

- Li & Dylla 1993 vented with air and dry N_2
- CERN & NIST Data same order of magnitude as air vented 304L SS
- Slope of mild steel may be slightly slower for mild steel
- Park et al 2016 results similar order of magnitude to N_2 vented 304L SS

Pumpdown of mild steel from air may not be any improvement over 304L SS. It may be a little worse.



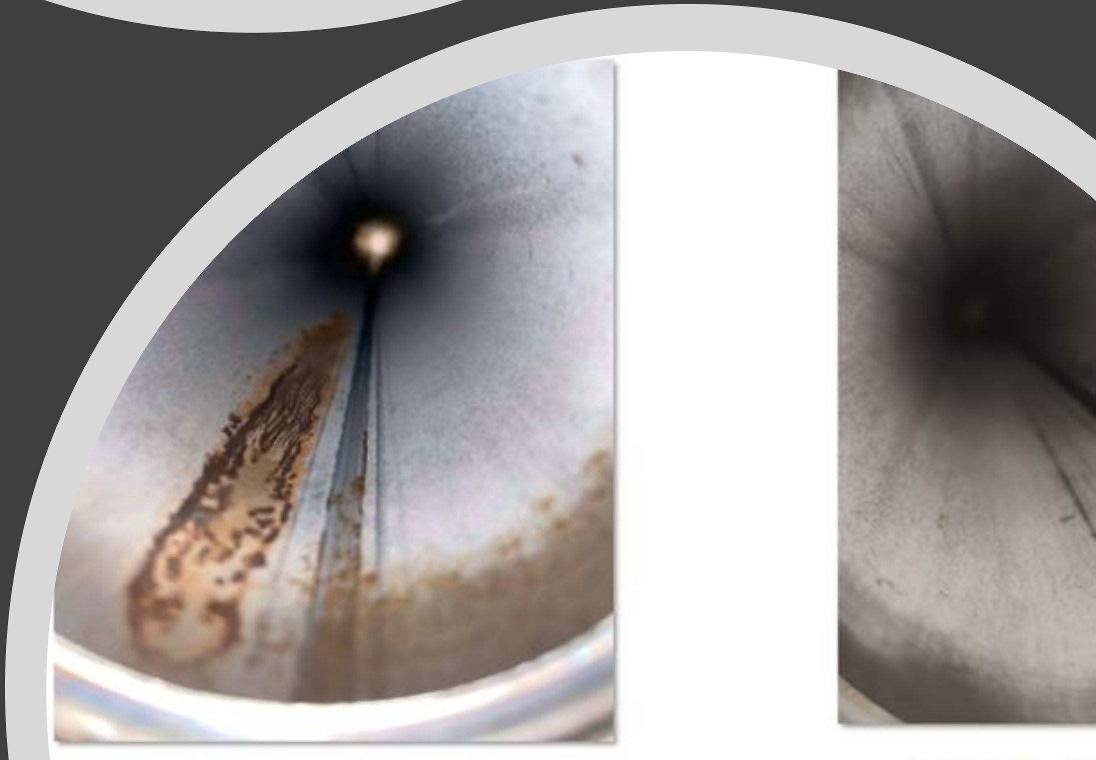
Wrapping up …



- Untreated Mild Steel H_2 outgassing is superior to stainless steel
  - Likely as good as, if not better than, Ti, vacuum-fired SS, or Al
- Water outgassing of untreated mild steel is similar to, or maybe worse, than stainless steel
- There is an indication that heat treatment improves mild steel water outgassing performance
  - Stainless steel water outgassing is not improved by this

<u>Going forward</u>:

- Modifying mild steel surface may improve water outgassing
  - We want to develop a process for magnetite coating
  - Test Magnetite coated mild steel for water outgassing
- Still need full investigation of water outgassing as a function of temperature



Aspect of the internal surface after accumulation measurements

Aspect of the inte... pressure mea...

Thank You!

Many thanks to Emmanuel Newsome, Fred Dylla and the folks at JLAB, WM, & CERN!

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