# A New System for $MgB_2$ Coating R&D at LANL

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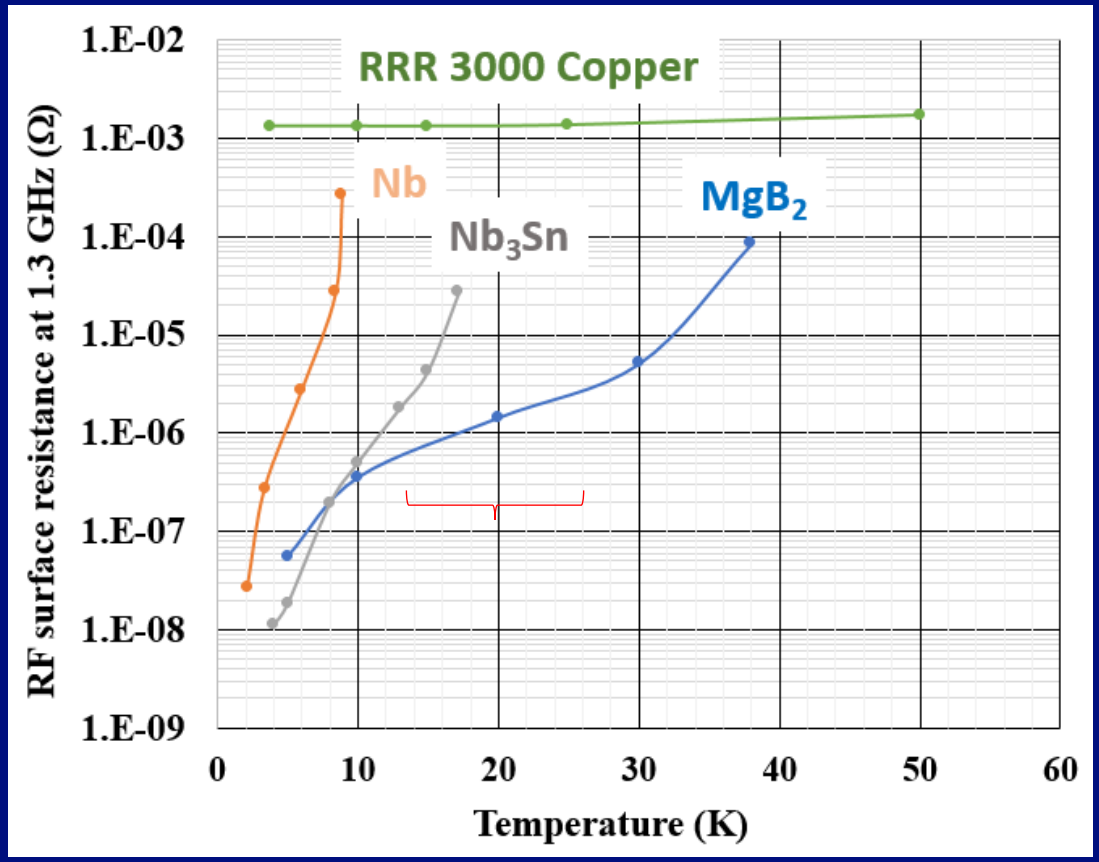
# Background

- $MgB_2$ could be useful if we can run the cavity at ~20 K with cryocoolers



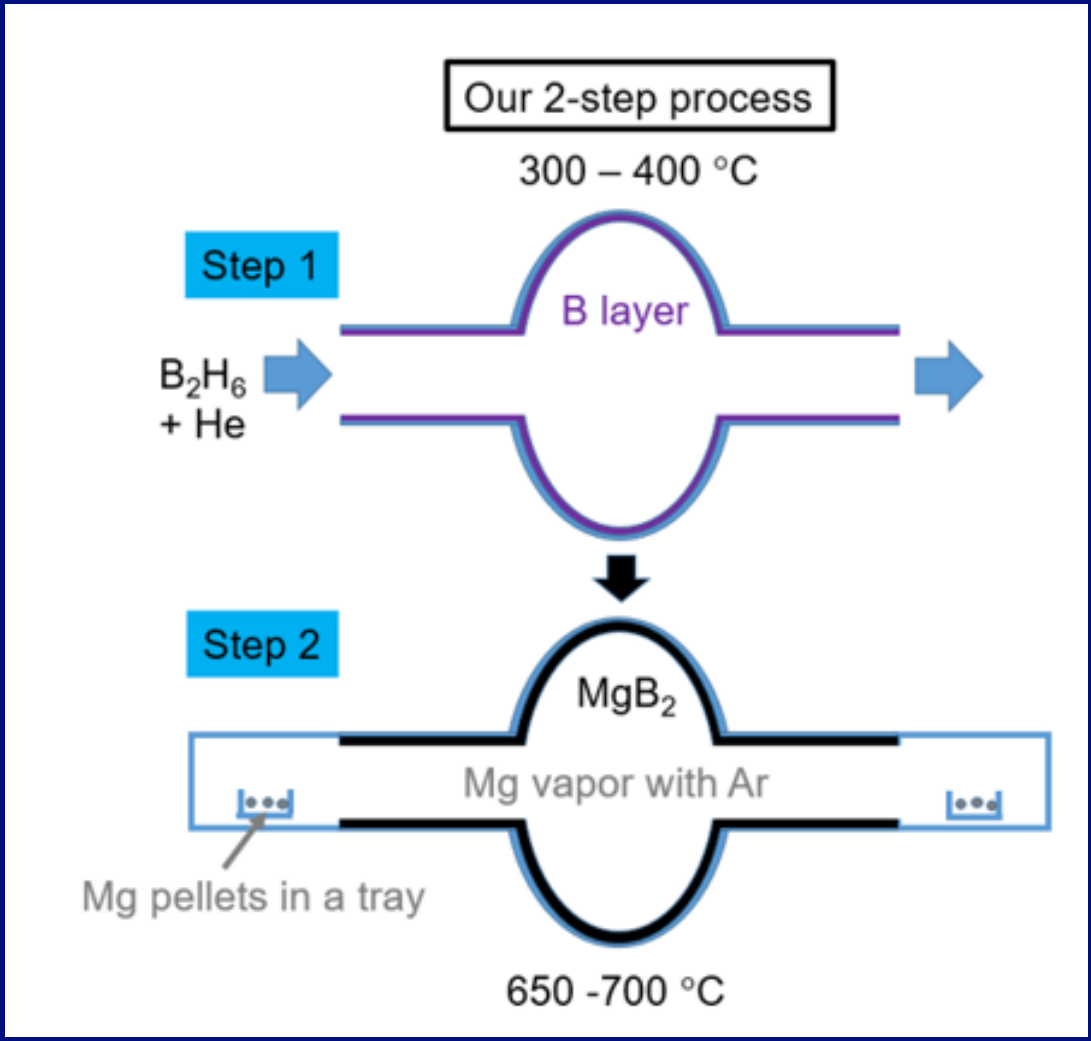

[T. Tajima, Teikon Kogaku 57 (2022) 23, DOI: 10.2221/jcsj.57.23]

# Background (Cont.)

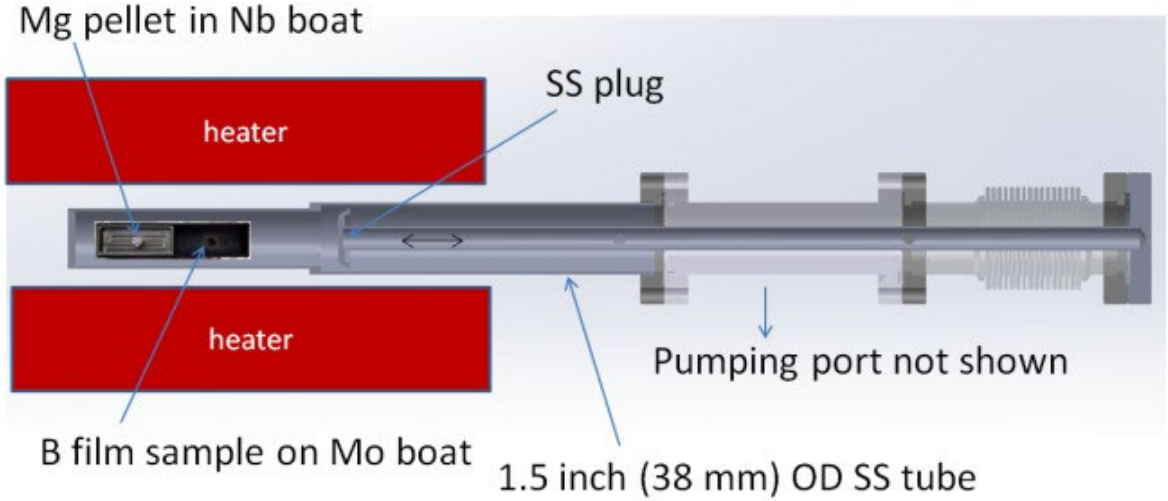
- After evaluating $MgB_2$ film samples prepared with different techniques since ~2005, we concluded that it is worth trying to coat a full-size cavity.
- Although we think HPCVD and Reactive Co-evaporation are the best techniques, we decided to start with a 2-step technique due to its simplicity.
- The 2-step technique consists of coating B layer on the cavity inner surfaces in the first step, then react it with Mg vapor in the second step. See the next slide.
- We started with the second step first using B films we obtained in the previous project that ended in 2015.
- Since we lost a large furnace in 2015, we used a small tube heater and tested the reaction of B with Mg vapor starting in 2015 with promising results [Tajima et al. SRF2015 p. 700]
- The $MgB_2$ project funding at LANL ended in 2015, but we got a new funding from DOE through the US-Japan Cooperation Project. (H. Sakai is Japan PI) from 2018.
- We continued the B-Mg reaction tests with good results of $T_c$ up to ~38 K [Pizzol et al. SRF2021]
- DOE funding was not large enough to build a new system, but the good results led to a larger LANL internal funding to be able to build a new system that will be shown in this workshop with the first B-Mg reaction test conducted recently.

# Concept of our 2-step coating process

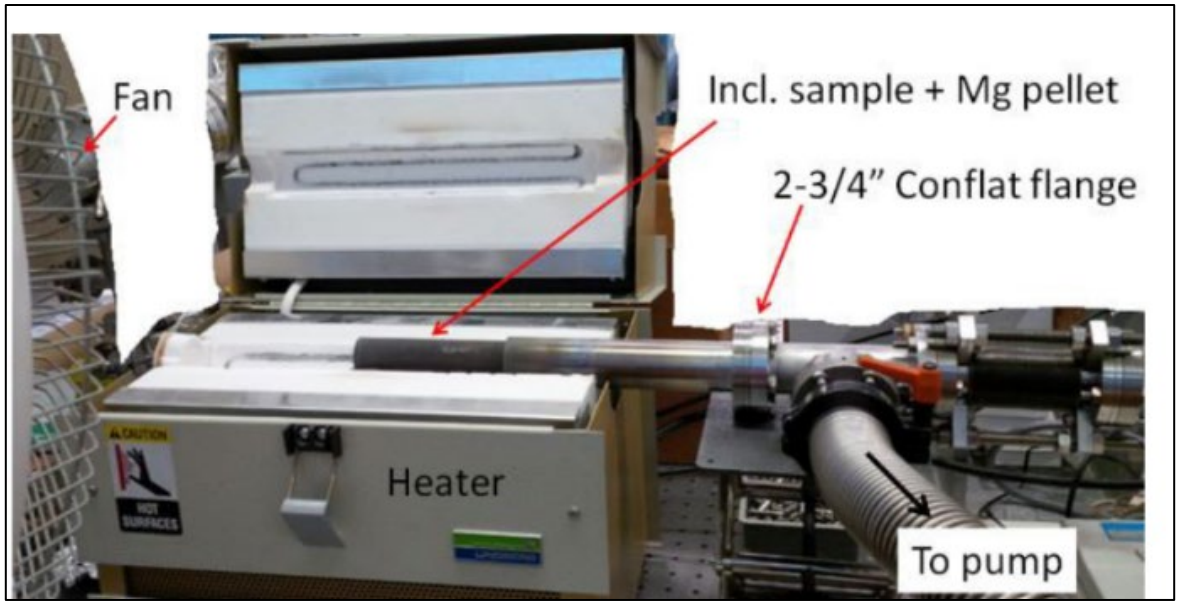
[2]



# B-Mg reaction test set up with a small tube heater [Tajima et al., SRF2015, p. 700]




6mm x 6 mm samples



# B-Mg reaction tests using the small tube furnace since November 2019. The B films we have been using are those taken from the 1.3-GHz cavity used during the previous project that ended in 2015.

| Sample ID | Run# (date) | Reaction temperature (°C) | $T_c$ (K) | Notes |
|---|---|---|---|---|
| 3-A3 | 16 (27NOV2019) | 750 | 33.3 | |
| 3-A4 | 16 (27NOV2019) | 750 | 36.8 | |
| 1-E3 | 17 (20JAN2020) | 700 | 35.3 | |
| 1-E5 | 17 (20JAN2020) | 700 | 38.0 | |
| 1-E7 | 18 (15-16MAR2020) | 650 | 35.5 | Held for ~21 h |
| 1-E8 | 18 (15-16MAR2020) | 650 | 37.0 | Held for ~21 h |
| 1-F1 | 19 (23OCT2020) | 700 | 37.9 | Slow cooling |
| 1-F3 | 19 (23OCT2020) | 700 | 35.6 | Slow cooling |

**Due to COVID-19, no tests were performed between March and September 2020.**

The nominal holding time is 1.5 h.
All but run #19 were fast cooling, i.e., <20 min to <40 °C.

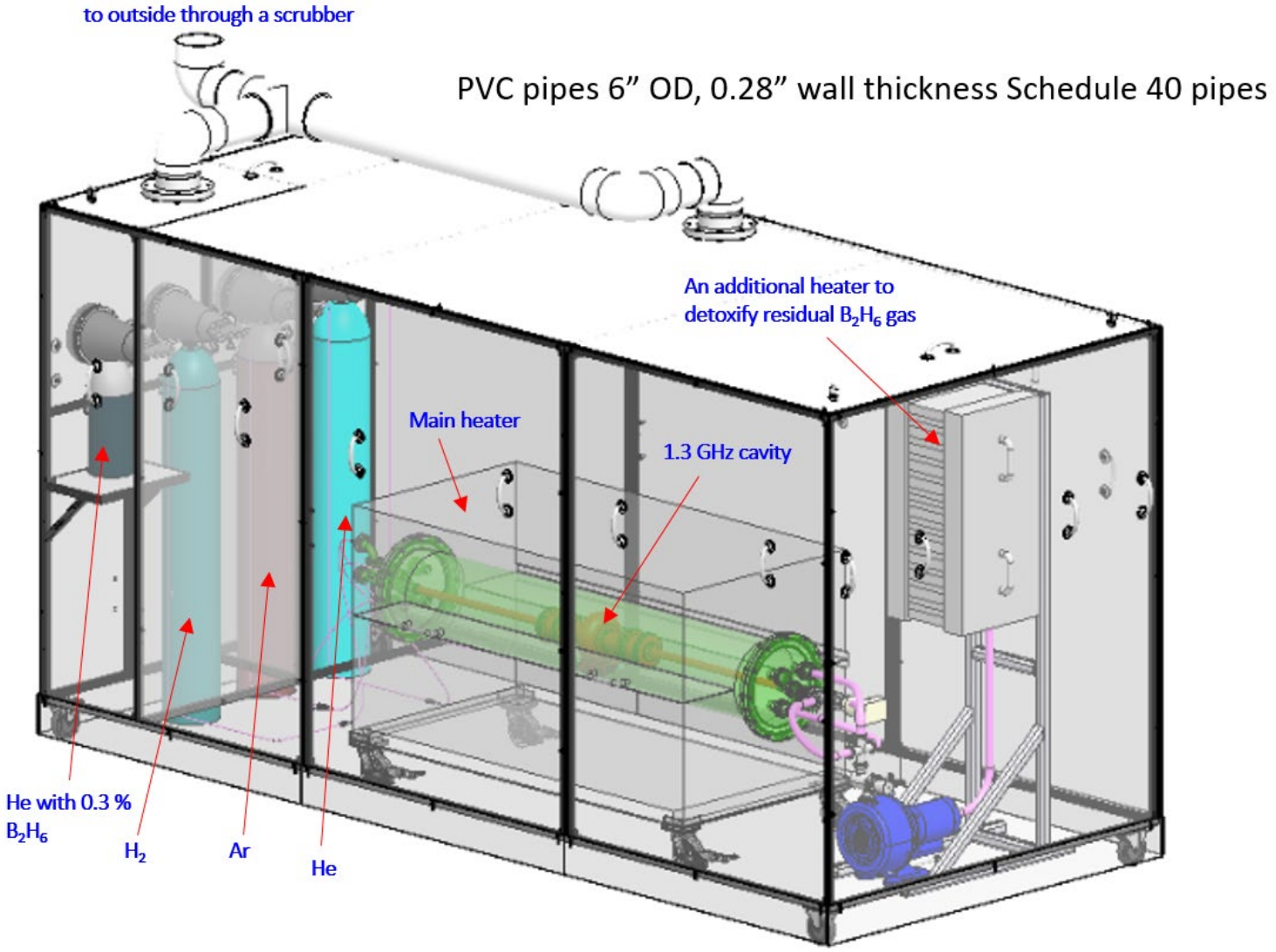
So far, 700 °C coating seems to be the best.

The difference in $T_c$ in the same test seems to be due to the difference in the substrate.

# The new system

- The design was performed with US-Japan Cooperation Project, but there was no funding to build it.
- We got an internal funding in FY21 and started purchasing components.
- The major component was a large main furnace, and it was ordered to a U.K. company through a U.S. company.

# A 3D model of the new $MgB_2$ coating system at LANL. A ventilation system to handle toxic $B_2H_6$ gas has been installed.



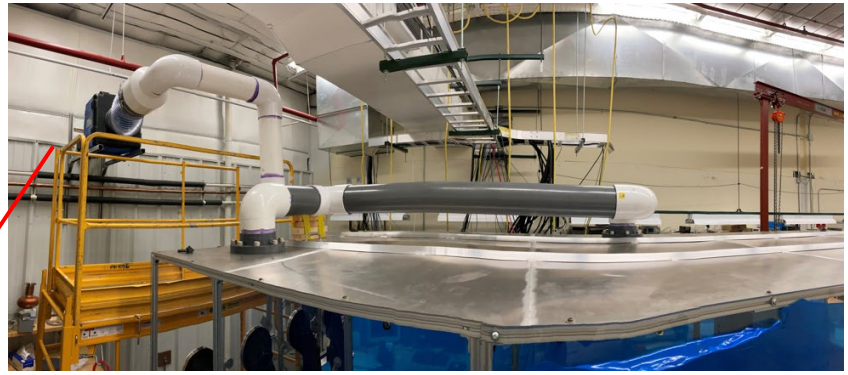
# The main furnace was delivered in September 2021 but found damaged in transit.


The initial scheduled delivery date was June 2021, but it was delayed due to COVID-19.

This furnace was returned to the company in U.K. and repaired and returned to LANL in late January 2022.

# Some photos of the new ventilation piping. The new ventilation system cleared intentionally created smoke inside the booth in 3 min.

**A panoramic view of the PVC piping**



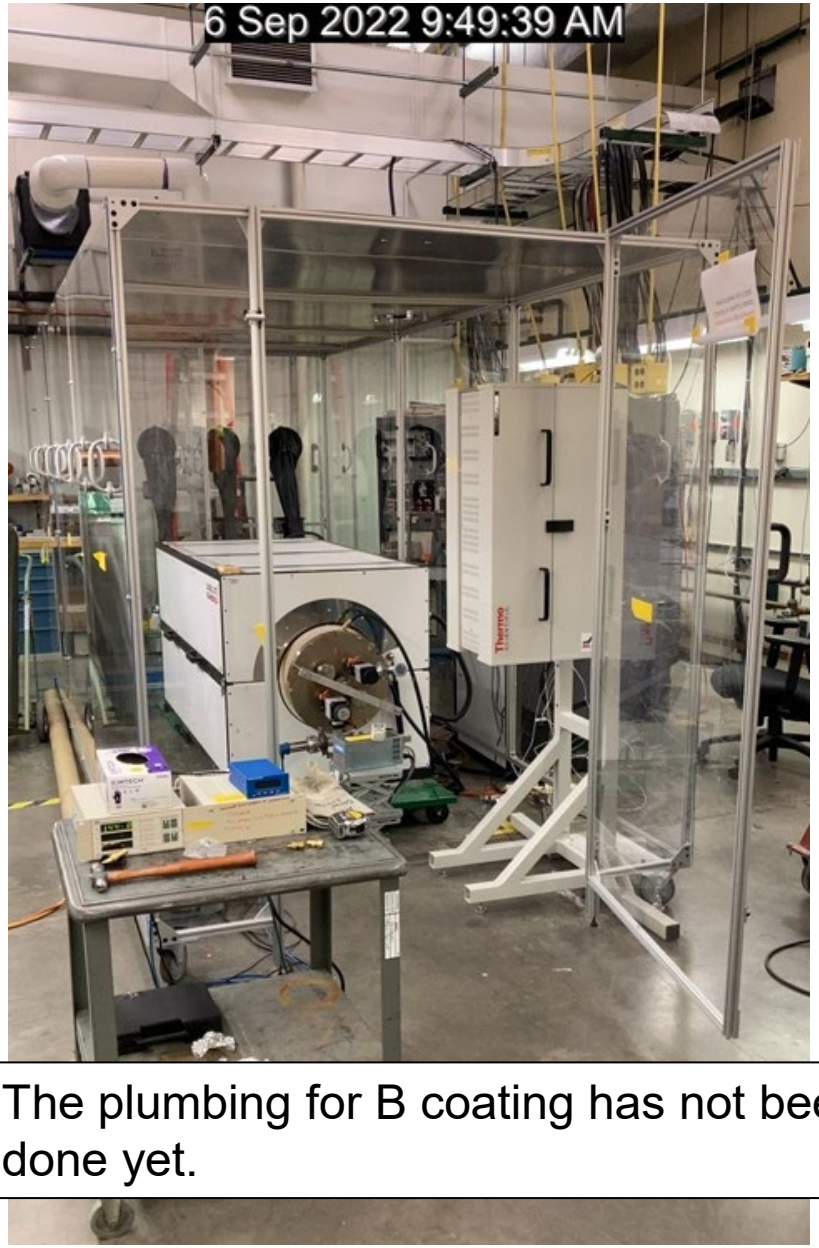
**outside**



**Scrubber**

# Some recent photos


Dr. Sakai measuring some dimensions

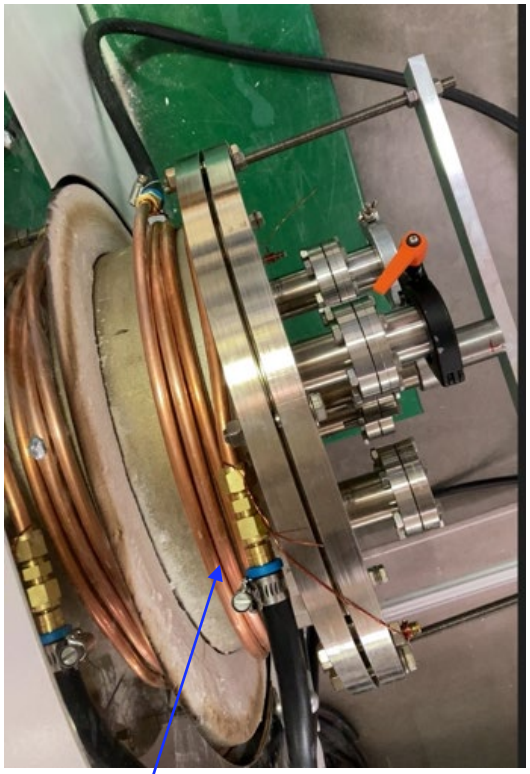



The plumbing for B coating has not been done yet.

# We started testing the main furnace in July 2022

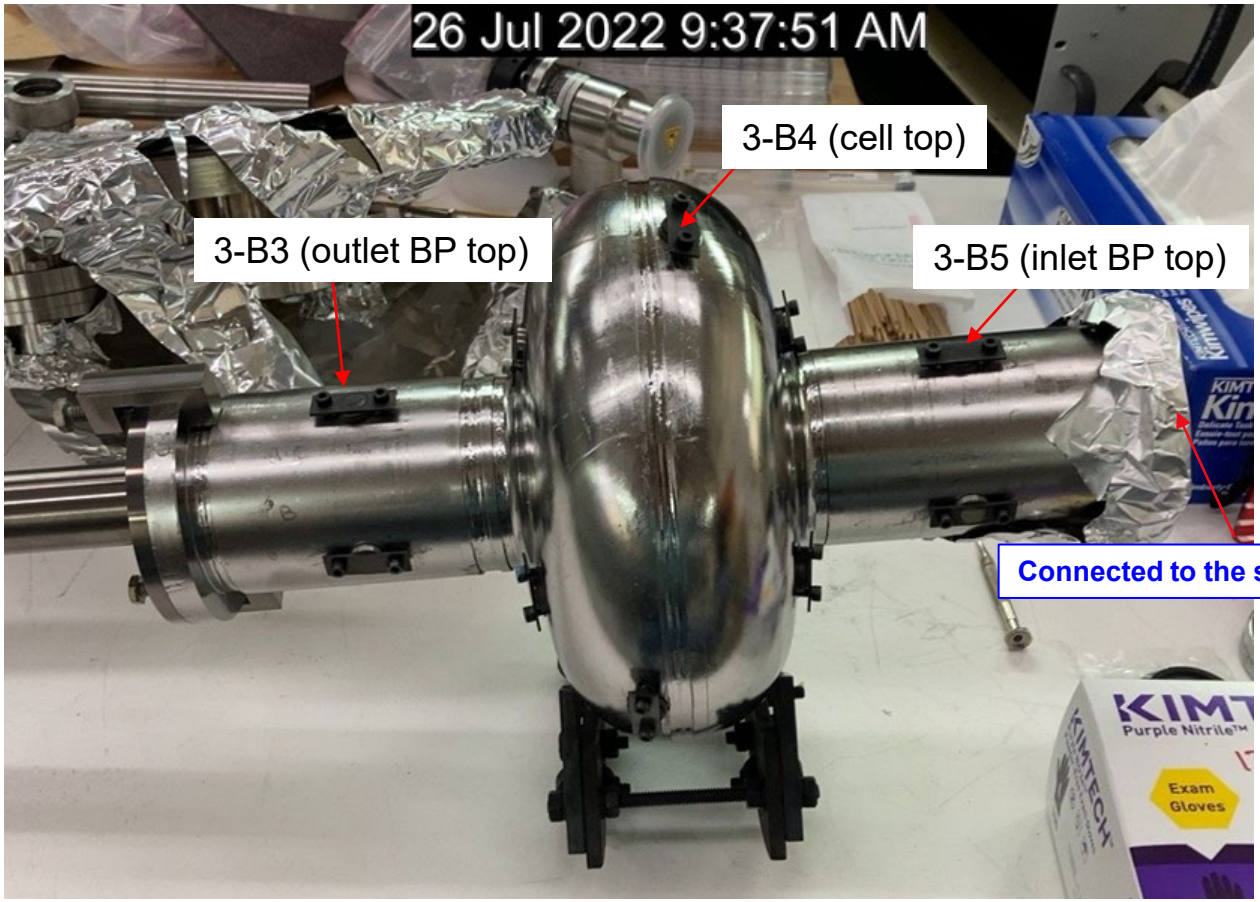
- Raised the furnace temperature up to 800 C and found that the Viton o-ring at the end flanges got damaged due to too high temperature, probably much higher than its limit of ~205 C.
- Added 3/8” OD copper cooling pipes around the beam pipes outside the furnace.
- We tested up to 700 C with the flange cooling and the Viton o-ring was not damaged.


Damaged Viton o-ring

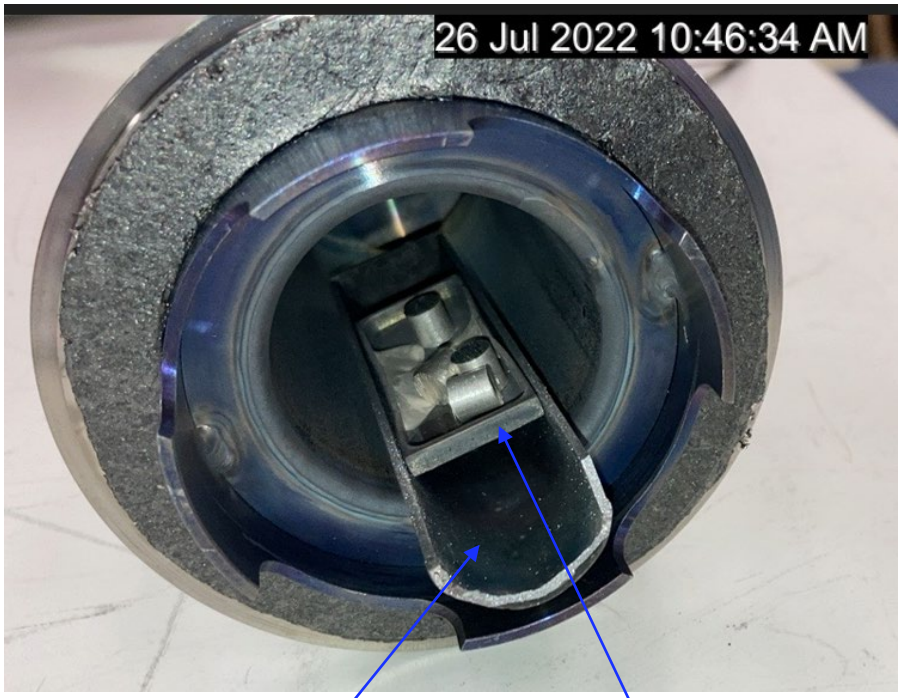


Cooling pipe added

# We performed our first B-Mg reaction test on 28 July 2022. Three B coated Nb samples were attached at 3 locations on a 1.3-GHz 1-cell Nb cavity.

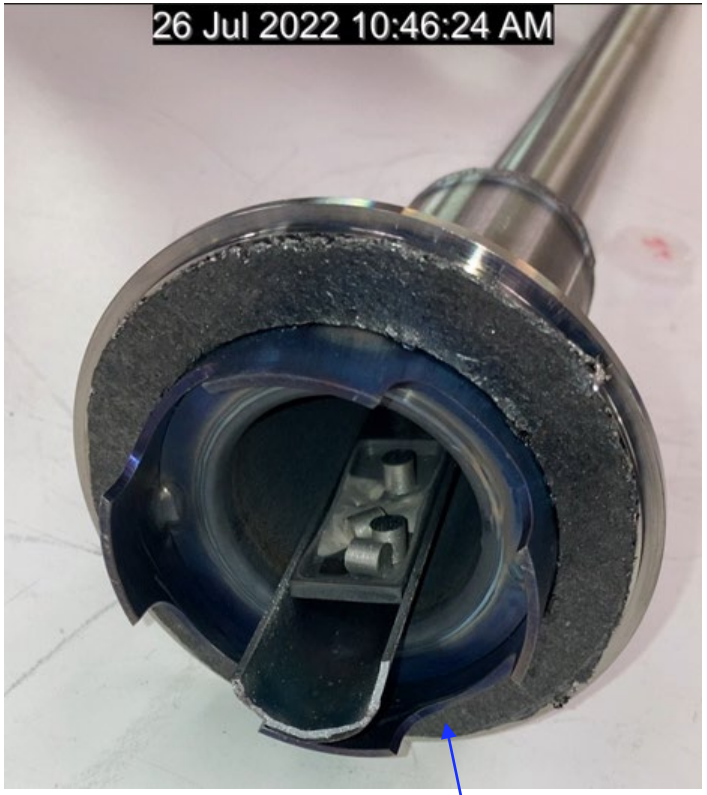


# Four new Mg pellets were placed on a Nb tray on a Mo boat in a section next to the cavity




Mo boat

Nb tray

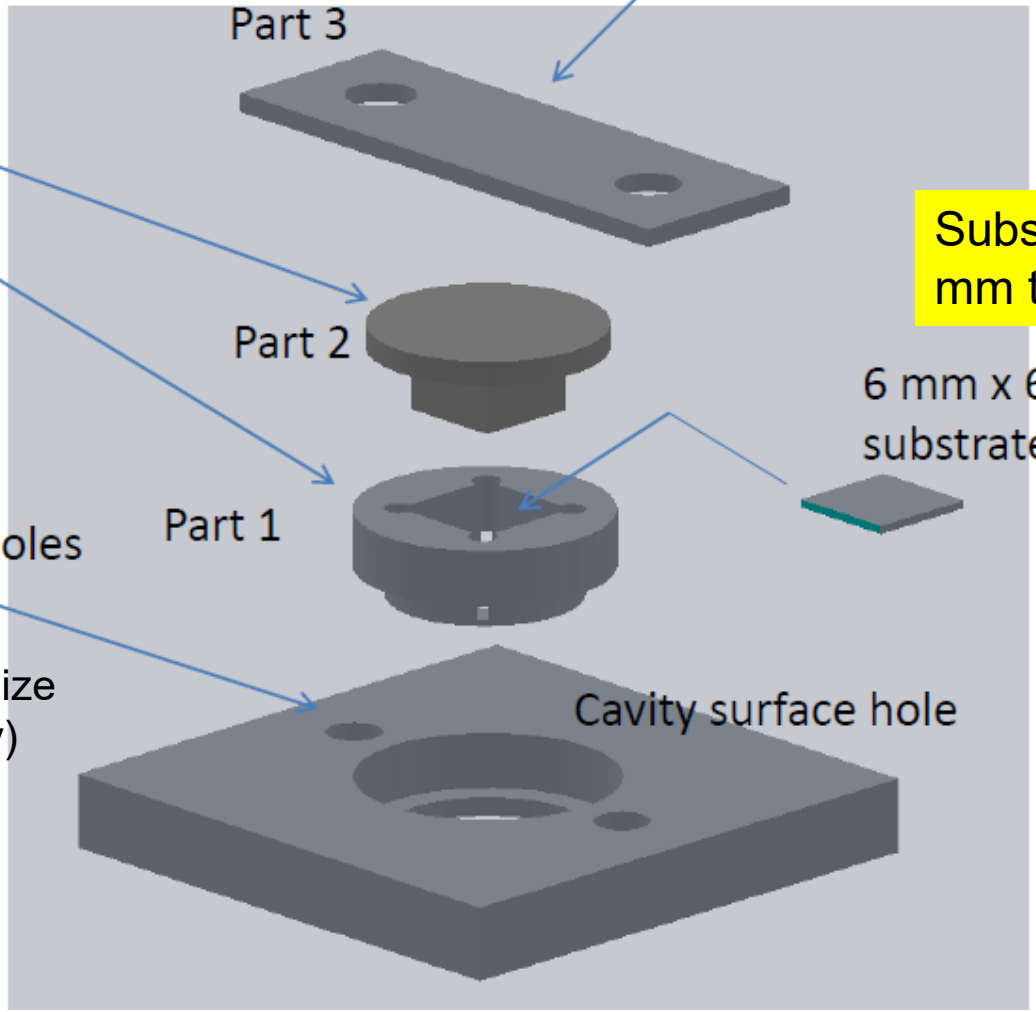



These cutouts are to pump down the cavity

# This time, the sample coupons were Nb coated with B.

A sample looking from outside.

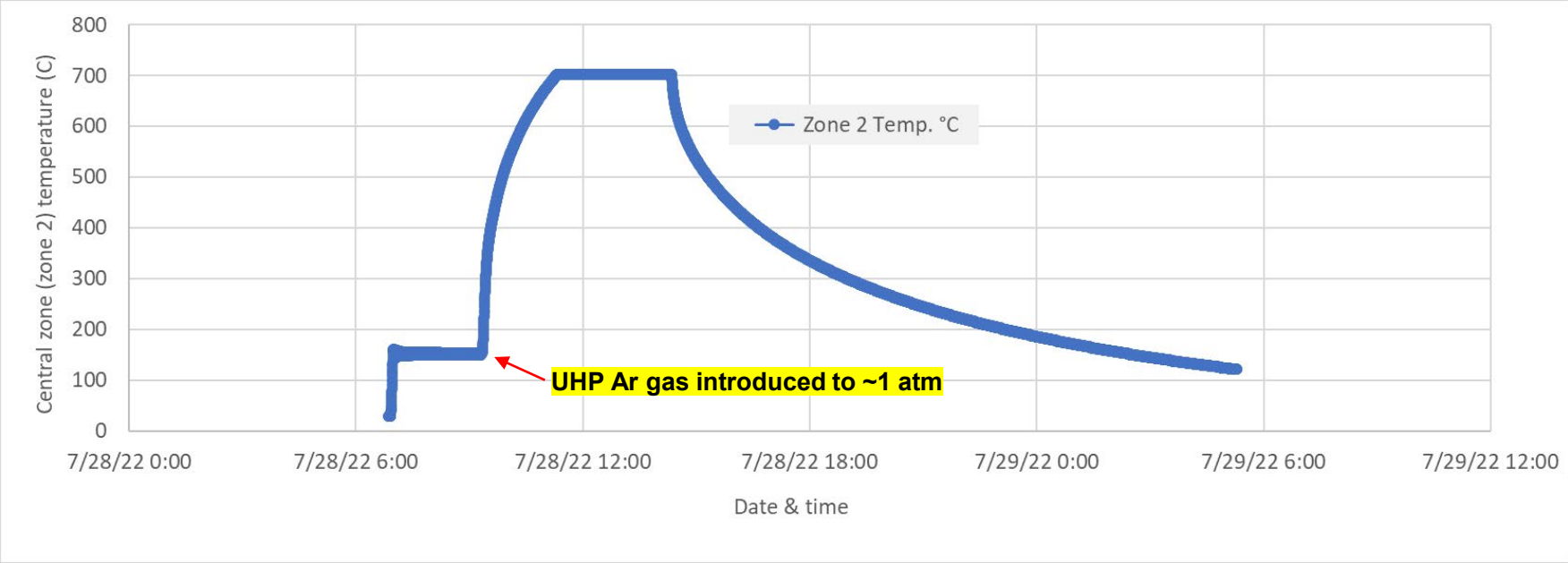

Sample holder parts



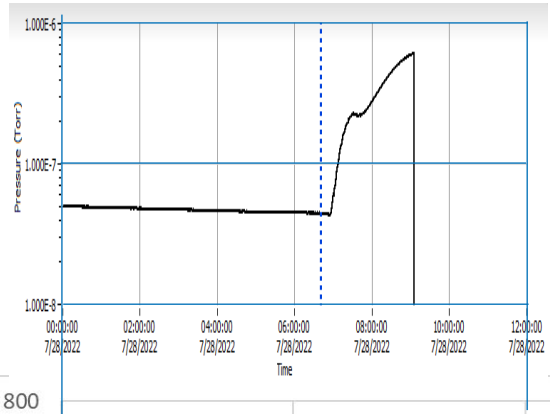

Substrates were ~2 mm thick Nb this time

# Furnace temperature trend during the first B-Mg reaction test

The furnace was turned on with 150 C setting at 06:53:55 at 27 C on 28JUL2022.
It reached ~150 C at 06:59:00, i.e., it took 5 m 5 s. It overshot to 160 C.
The set temperature was raised to 700 C at 09:19:50, i.e., it was kept at 150 C for 2 h 20 m 50 s.
It reached 700 C at 11:18:50, i.e, it took 1 h 59 m 00 s.
The furnace was turned off at 14:20:13, i.e., it was kept at 700 C for 3 h 1 m 23 s.



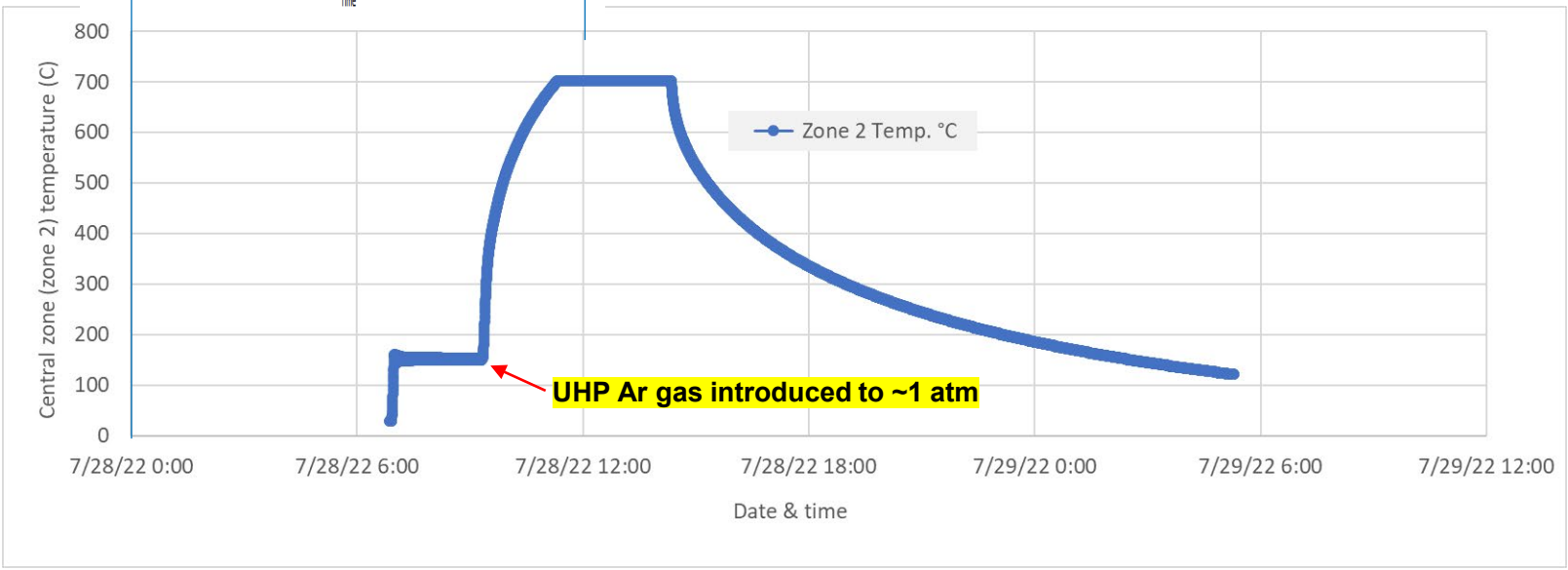
# The degassing at 150 C was probably not long enough. We should have waited until the pressure goes down.




1E-6

1E-7 Pressure (Torr)

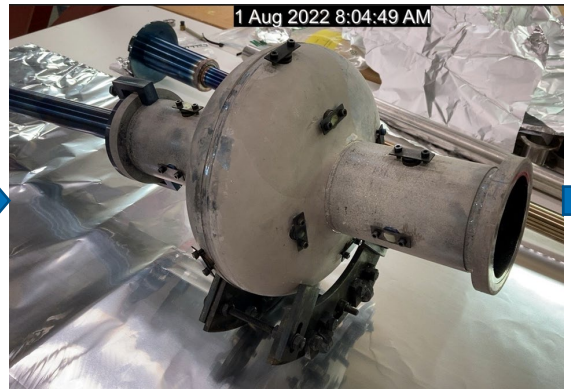
1E-8



# Magnetometer measurements on the 3 samples were performed on 12-13SEP2022 with no superconductivity detected.

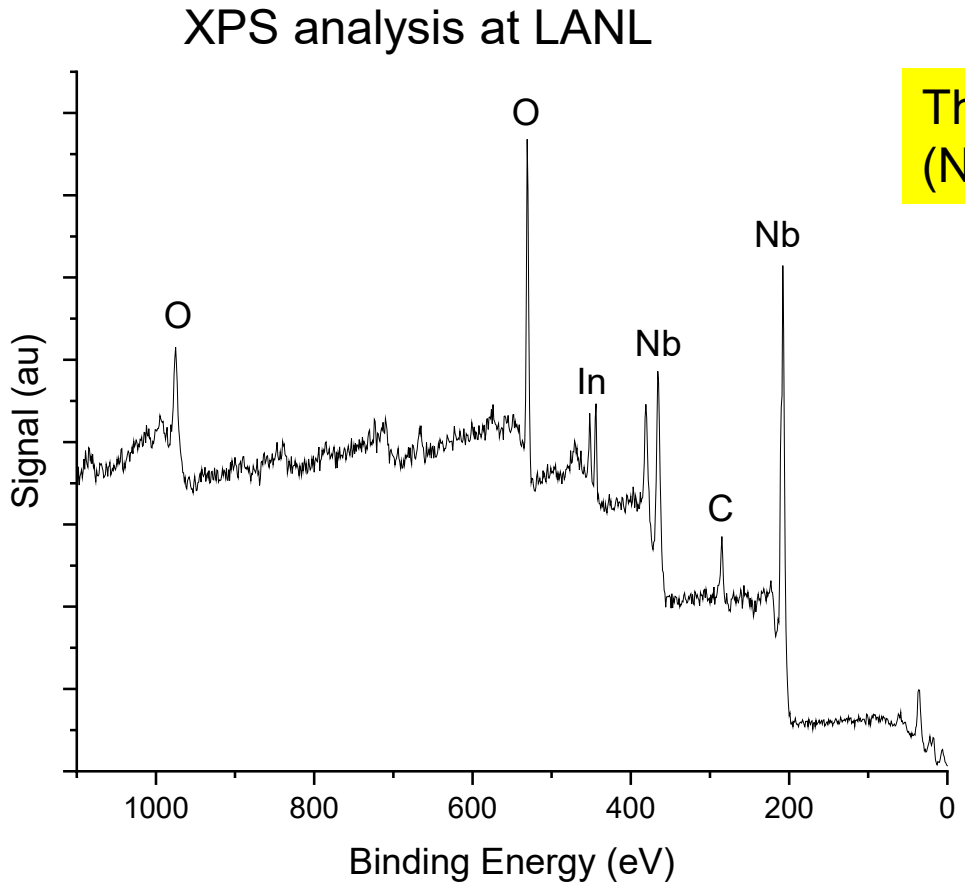
- One thing we noticed was that the cavity outer surface color changed after this reaction, then after heating under vacuum.


**After B-Mg reaction in Ar at 700 C (furnace)**




**After heating at 700 C under vacuum**

# We did not analyze the surface material after the B-Mg reaction test but did analyze the surface after heating the cavity under vacuum.



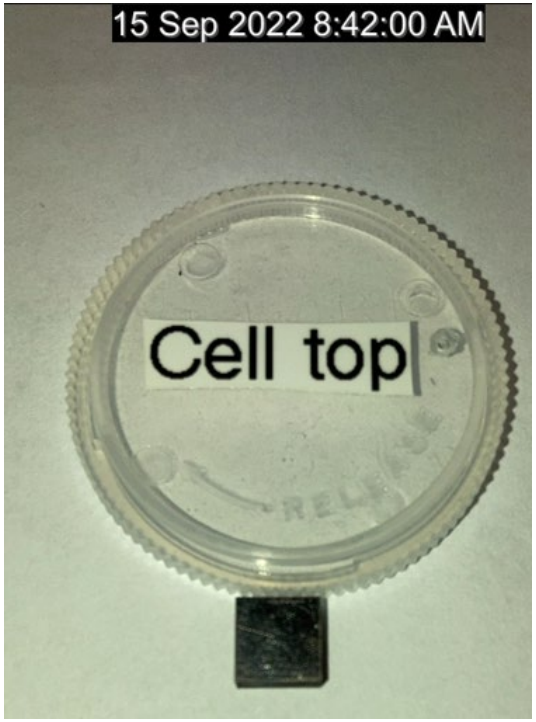

The surface material is Nb oxide ($NbO_2$ and $Nb_2O_5$).

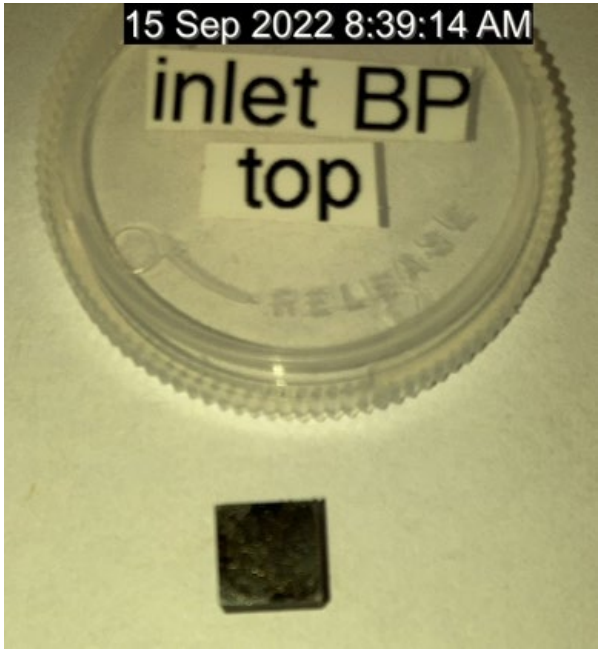
Indium is seen since the sample was placed into indium foil.

# We also measured temperatures at various points in the Inconel pipe when the furnace temperature went up to 700 C


A K-type thermocouple is being attached at the cell equator

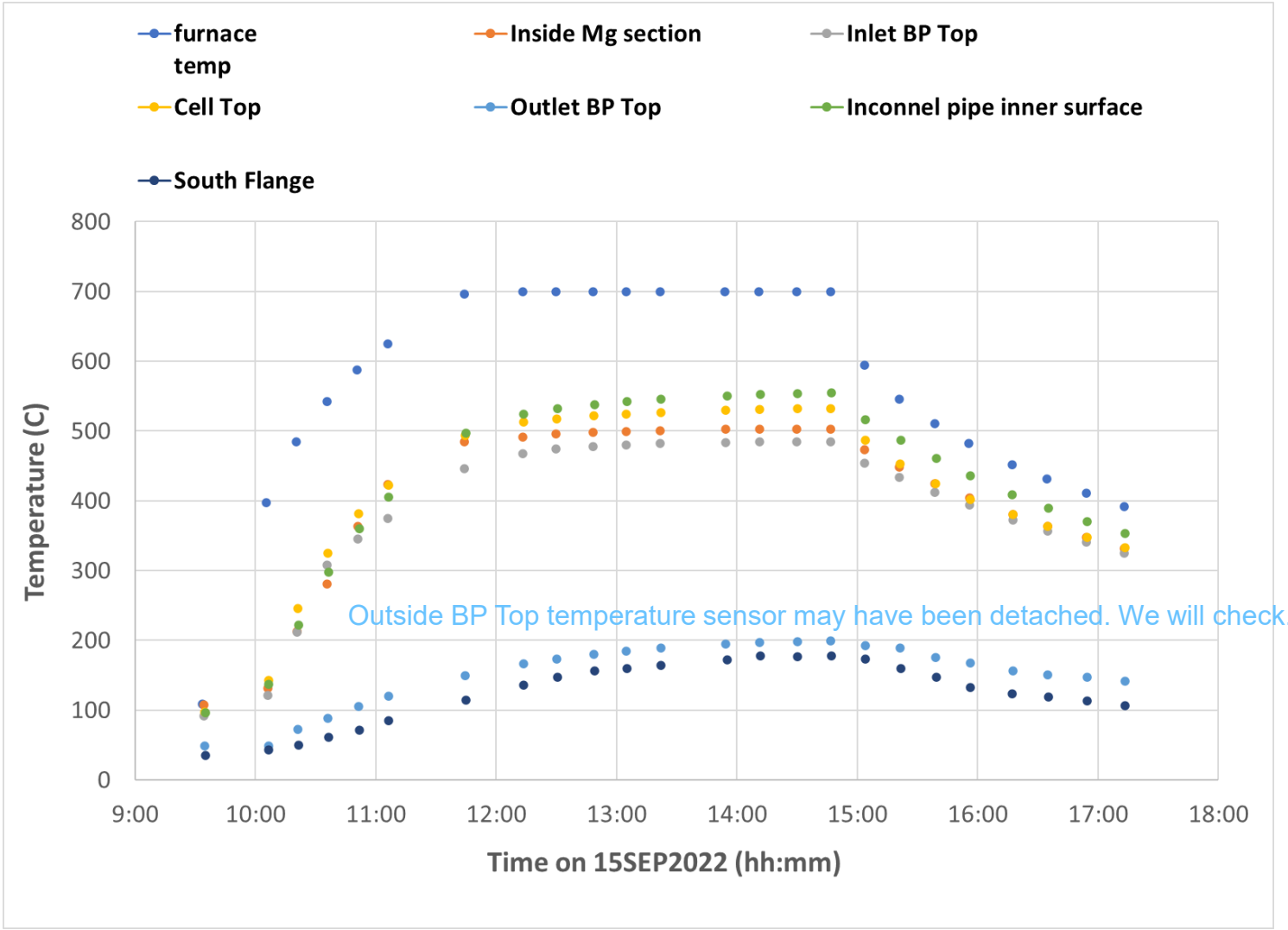
# Photos of the samples after the B-Mg reaction test. No pics were taken before. XPS surface analysis will be done soon.

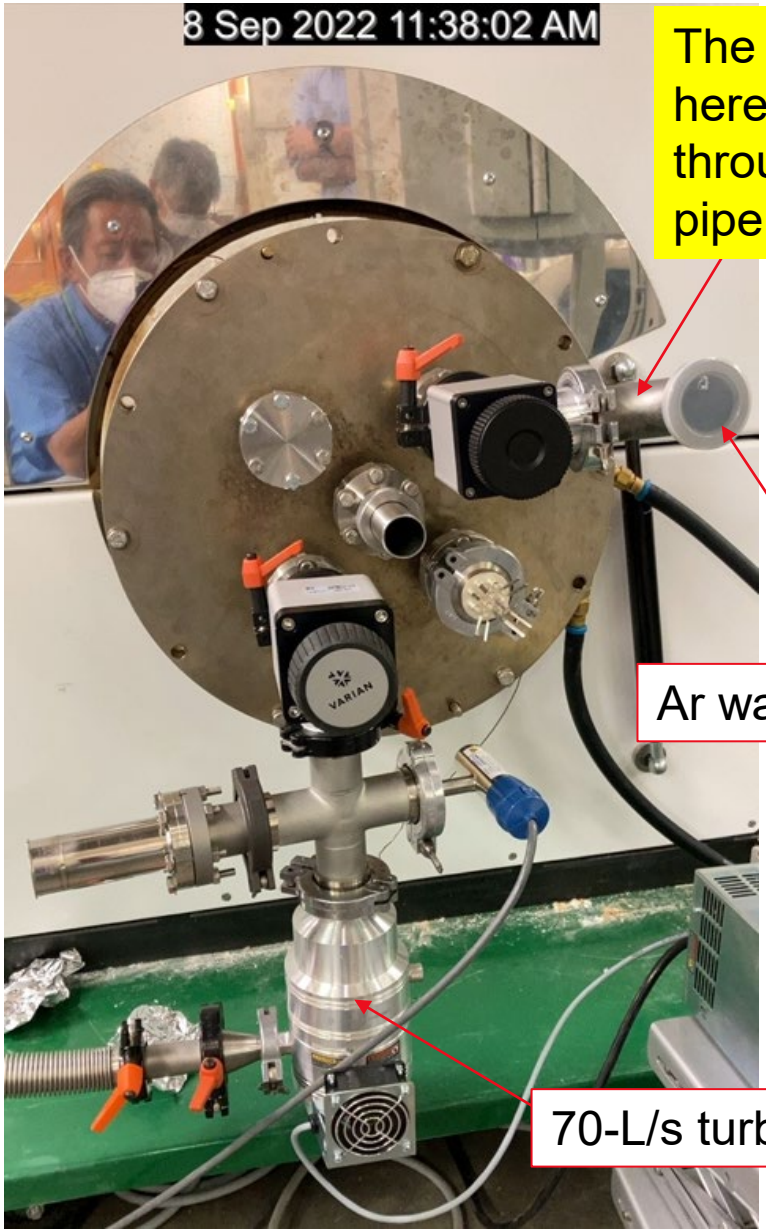



# It was found that the Mg section, cell and beam pipe temperatures are considerably lower than the furnace temperature (<532 C vs. 700 C)

With 1 atm Ar inside



**The oxidation may have been due to the air trapped at the port for introducing Ar gas.**




The air trapped here was pumped through the Inconel pipe

Ar was connected here

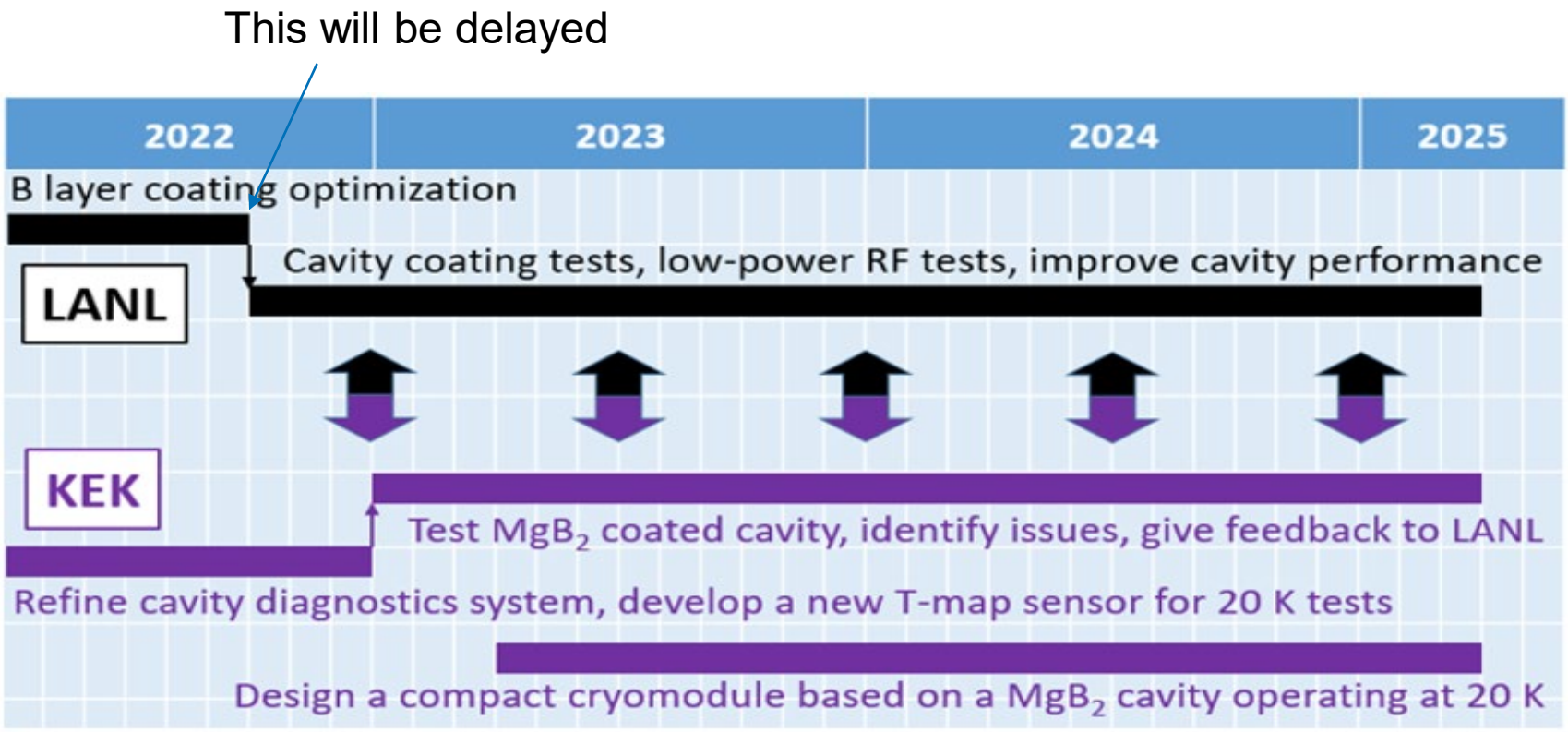
70-L/s turbo pump

Connected to a scroll pump

# Conclusion and near-term next steps

- We were able to build a new system for the full-size 1.3-GHz cavity coating R&D in the SRF lab at LANL and started B-Mg reaction tests.
- The first B-Mg test was unsuccessful with no superconductivity detected on all the 3 samples.
- We measured the temperatures on the cavity, Mg section, etc. and found that the temperatures were <532 C when the furnace temperature was 700 C, which is likely to be the cause of failed test.
- Next steps
  - Add RGA sensor at the pump port (ongoing)
  - Analyze the surfaces of recent samples (XPS analysis ongoing)
  - Raise the temperature of the Mg section and cavity to ~700 C. We need to improve the cooling of flanges so the flange temperature will not exceed ~200 C (Viton limit).
  - Add a pumping at the port for introducing Ar gas
  - Continue preparing for the B layer coating. Since diborane ($B_2H_6$) gas is toxic, we will need to be very careful to ensure safety.

# US-Japan Cooperation Project Schedule (in the official proposal). Mg-B reaction tests continue first.



# Acknowledgments


- We would like to thank Stephen Milton, the previous division leader at the Accelerator Operations and Technologies Division at LANL, for his enthusiastic support that led to the LANL internal funding.
- This R&D have been supported by the US-Japan Science and Technology Cooperation Program in High Energy Physics and LANL LDRD project 20210720ER.

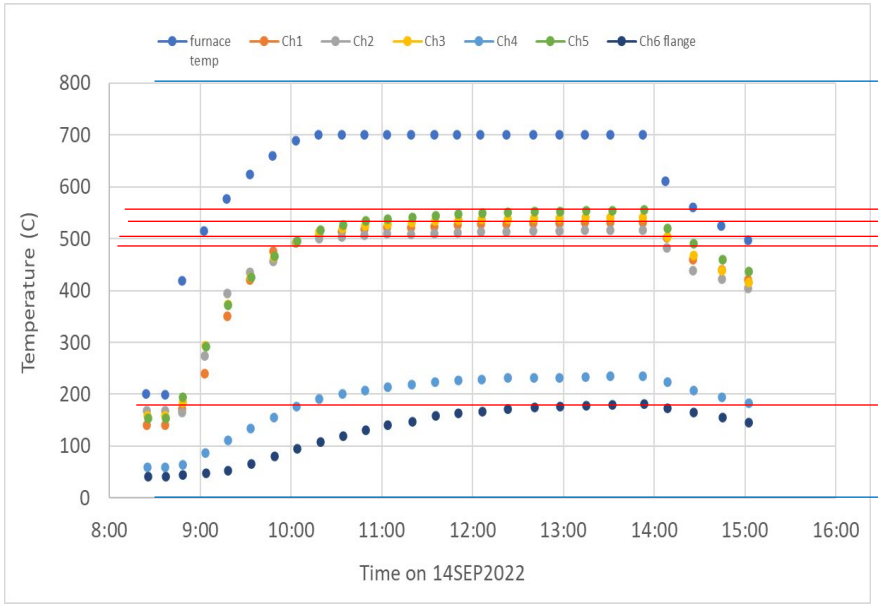
# Backup slides

# Temperatures of cavity, Mg section, etc. when the furnace temperature went up to 700 C. Comparison between vacuum and 1 atm Ar inside.

- The Mg section temperature (Ch1, orange) got lower with 1 atm Ar from 531 C to 503 C
- The Inlet BP Top temperature (Ch2, gray) got lower with 1 atm Ar from 516 C to 485 C.
- The flange temperature (Ch6, dark blue) did not change much.
- Ch4 is probably not showing correct temperature.

| |
|---|
| Ch1 (Orange) : Inside Mg section |
| Ch2 (Gray): Inlet BP Top |
| Ch3 (Yellow): Cell Top |
| Ch4 (Light blue): Outlet BP top |
| Ch5 (Green): Inconel pipe inner surface |
| Ch6 (Dark blue): South flange |

vacuum




With 1 atm Ar inside