A New System for MgB₂ 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
B-Mg reaction test set up with a small tube heater [Tajima et al., SRF2015, p. 700]

Mg pellet in Nb boat

SS plug

B film sample on Mo boat

1.5 inch (38 mm) OD SS tube

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.

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

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$_2$H$_6$ gas has been installed.

PVC pipes 6” OD, 0.28” wall thickness Schedule 40 pipes

An additional heater to detoxify residual B$_2$H$_6$ gas

Main heater

1.3 GHz cavity

He with 0.3 % B$_2$H$_6$

H$_2$, Ar, He
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.
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.

These cutouts are to pump down the cavity.
This time, the sample coupons were Nb coated with B.

A sample looking from outside.
Substrates were ~2 mm thick Nb this time
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.
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$_2$O$_5$).

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

Outside BP Top temperature sensor may have been detached. We will check.
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.

Connected to a scroll pump.

70-L/s turbo 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 (B₂H₆) 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.

<table>
<thead>
<tr>
<th>2022</th>
<th>2023</th>
<th>2024</th>
<th>2025</th>
</tr>
</thead>
<tbody>
<tr>
<td>B layer coating optimization</td>
<td>Cavity coating tests, low-power RF tests,</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>improve cavity performance</td>
<td></td>
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</tbody>
</table>

This will be delayed

LANL

Test MgB$_2$ coated cavity, identify issues, give feedback to LANL

KEK

Refine cavity diagnostics system, develop a new T-map sensor for 20 K tests

Design a compact cryomodule based on a MgB$_2$ cavity operating at 20 K
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.

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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.

![Graph showing temperature changes](image-url)