RESEARCH ON MgB_2 AT LANL FOR THE APPLICATION TO SRF STRUCTURES*

T. Tajima#, L. Civale, R.K. Schulze, LANL, Los Alamos, NM 87545 USA

Abstract

This paper is focused on the development of MgB_2 coating technique at LANL. Using boron film samples obtained at a large furnace system, we succeeded in obtaining superconducting MgB_2 films (T_c of up to 37 K so far) by reacting them with Mg vapor. The major improvements were 1) confinement of the Mg vapor in a hot zone to mitigate the insufficient Mg pressure due to condensation on low temperature surfaces of the connected vacuum pipes and 2) reduction of cooldown time, i.e., ~13 minutes instead of ~1 day with the large system to prevent MgB_2 from decomposing.

INTRODUCTION

We have constructed and used a large coating system in a building at Technical Area 35 (TA-35) in LANL. This system has a furnace capable of coating up to a 9-cell 1.3 GHz elliptical cavity [1]. We have been unsuccessful, however, in obtaining superconducting MgB_2 films stably. The identified causes include 1) insufficient Mg vapour pressure due to the condensation on cooler surfaces on the pipes and 2) a long cooling time (~1 day). Since it was difficult to modify the current large system to address these issues, we used a small system in the SRF lab at TA-53 to verify these.

EXPERIMENTAL SETUP

While the large coating system described in Ref. [1] consisted of a hood with air venting to outside and the pressure in the room was kept slightly negative relative to outside as well as with gas monitors for hydrogen and diborane (B_2H_6 toxic) gases for safety, the small setup described here is very simple because of little concern on safety, i.e., reacting Mg vapour with the boron films obtained using the large coating system.

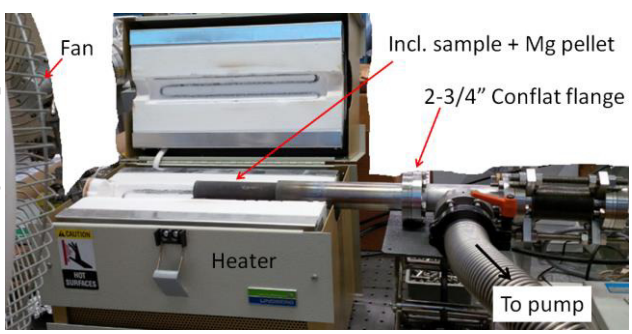

Figure 1: Experimental setup

The system consists of a tube heater (Lindberg type 55035 max. temp. 1100 °C), a turbo pump, a, diaphragm pump and vacuum gauges. Figure 1 shows a photo of the setup.

Figure 2 shows a schematic showing the inside of the stainless steel tube. A plug to confine the zone with a sample and a Mg pellet was used to keep sufficient Mg vapor pressure.

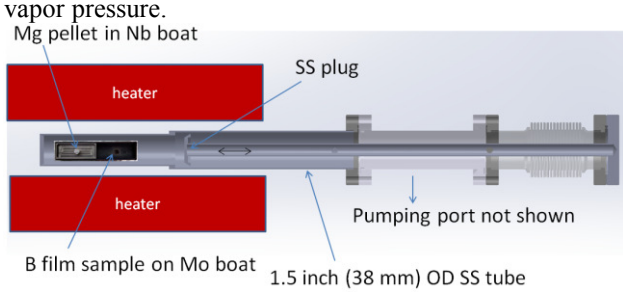

Figure 2: Inside the SS tube. The Mg pellet is on a Nb boat in a Mo boat and the B sample is in the same Mo boat. This Mo boat is confined in a smaller diameter tube after the plug closes this tube.

PROCEDURE TO GET MgB_2

At first, we tried the reaction without Ar gas, i.e., pumping down the pipe and plugged the hot zone and raised temperature, but we did not get any stoichiometric MgB_2, only got B rich films. We might not have had a good sealing of the plug, but we decided to use Ar gas to mitigate the issue of leaking of the Mg vapor since Hanna et al. were successful in obtaining clean MgB_2 films by annealing CVD-deposited B films in Mg vapor in Ar atmosphere [2].

While we need more parameter optimization to get better films, the following procedure was used to get MgB_2 films successfully.

- Bake the system at ~150 °C under vacuum for ~20 min
- Fill the system with UHP Ar gas up to 1/3 psi
- Plug the hot zone with a Mg pellet and a B film
- Raise the temperature to a planned value
- Hold it for 50 min
- Quench it to ~40 °C in ~13 min

CHARACTERIZATION OF THE FILMS

The elemental composition and their depth profile were obtained using XPS and Auger electron spectroscopy. The superconductivity transition temperature T_c was determined with magnetization measurements using a SQUID magnetometer. Figure 3 shows a summary of 3 successful samples that showed superconductivity. Figures 4 through 6 show the depth profile of B (red), Mg (green) and O (purple) for the 3 samples. Although we have not fully optimized the parameters yet, so far, the sample (c) that was annealed at

*Work supported by the US Department of Energy (DOE).
#tajima@lanl.gov

Copyright © 2015 CC-BY-3.0 and by the respective authors

750 °C showed the highest T_c of ~37 K with the sharpest drop of the magnetic moment as shown in Fig. 3. The ideal ratio of B:Mg for MgB_2 is 2:1, i.e., 66% of B and 33% of Mg. As you can see in Figures 4-6, samples (a) and (b) are slightly B rich compared to sample (c). The oxygen content of sample (c), however, is higher than ideal as shown in Fig. 6. This could be due to insufficient pumping before introducing Ar gas. We will try to improve the pumping and lower the O content to see if the T_c goes up. In our experience, the T_c needs to be high (>~35K) to show high vortex penetration field [3].

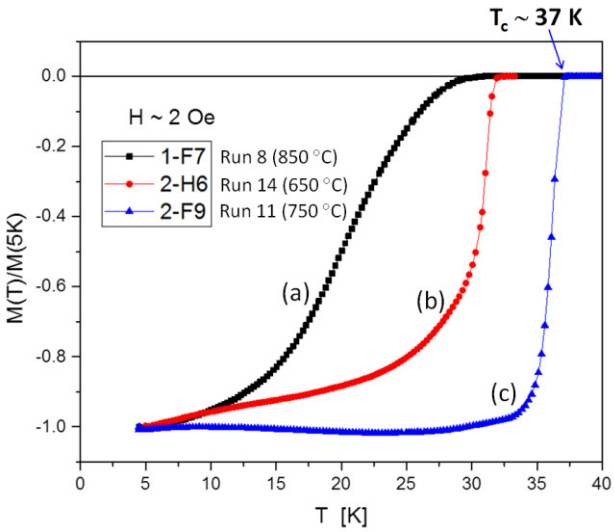

Figure 3: Magnetic moment normalized with the value at 5 K for different reaction temperatures; (a) 850° C, (b) 650 °C and (c) 750 °C.

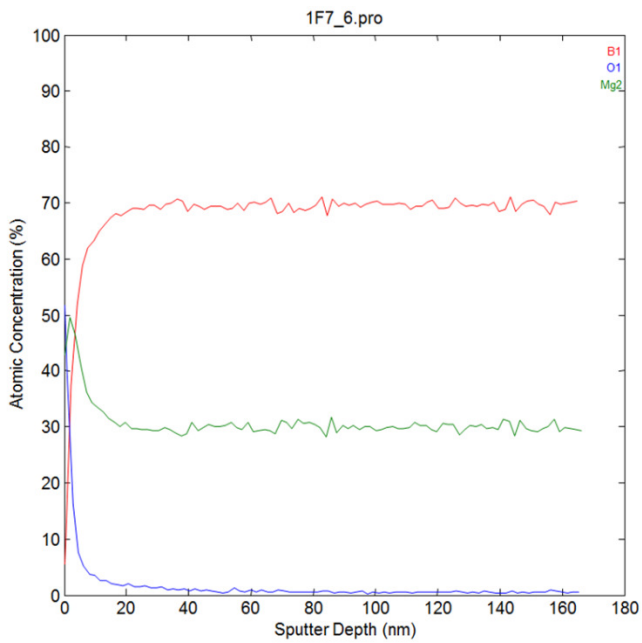

Figure 4: The Auger depth profile for sample (a) in Fig. 3, i.e., reaction at 850 °C for 50 min.

FUTURE PLANS

When funds become available, we will restart optimizing the parameter. In parallel, we will design and build a new coating system that will realize these optimized conditions. We will then fine tune the parameters and coat cavities for RF testing. The new system will be large enough to coat at least 1.3 GHz multi-cell elliptical cavities.

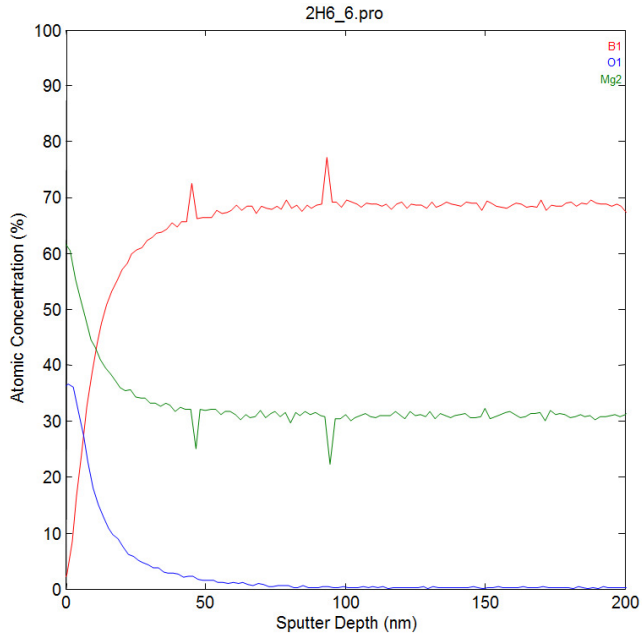

Figure 5: The Auger depth profile for sample (b) in Fig. 3, i.e., reaction at 650 °C for 50 min.

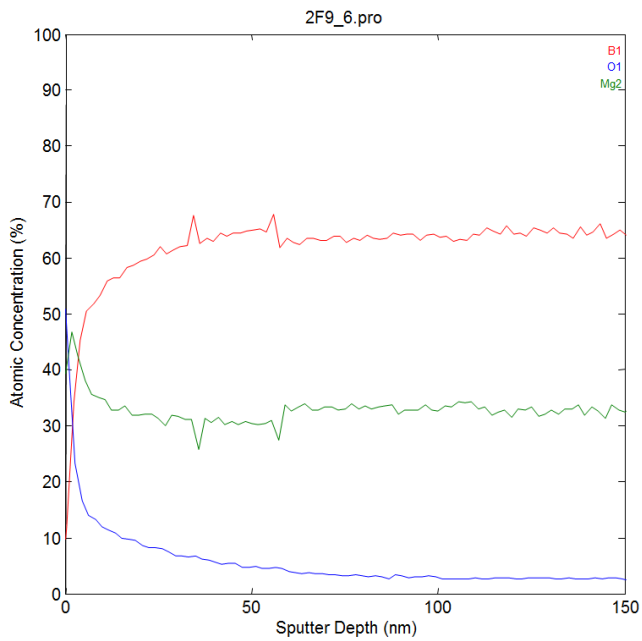

Figure 6: The Auger depth profile for sample (b) in Fig. 3, i.e., reaction at 750 °C for 50 min.

Copyright © 2015 CC-BY-3.0 and by the respective authors

ISBN 978-3-95450-178-6

ACKNOWLEDGMENT

This work was supported by the U.S. Department of Energy (DOE) Office of Science Office of Nuclear Physics Early Career Research Program. We would like to thank Mike Borden for careful review of the paper.

REFERENCES

[1] T. Tajima et al., "Status of MgB_2 Coating Studies for SRF Applications," SRF2013, Paris, France, September 2013, p. 777 (2013); http://www.JACoW.org

[2] M. Hanna et al., "Clean epitaxial MgB_2 films fabricated by the *ex situ* annealing of chemical vapour deposition-grown B films in Mg vapor," Supercond. Sci. Technol. 21 (2008) 045005.

[3] T. Tajima et al., "Studies on Superconducting Thin Films for SRF Applications," LINAC2010, Tsukuba, Japan, September 2010, p. 854 (2010); http://www.JACoW.org

Copyright © 2015 CC-BY-3.0 and by the respective authors