Final Technical Report

1. **DE-SC0004410**
   Temple University

2. **Project Title: Magnesium diboride thin films for superconducting RF cavities.**
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3. **Reporting Period: 6-15-10 to 6-14-13**

4. **Accomplishments:**

- *Large-Area MgB_2 Films Fabricated by Scaled-Up HPCVD Setup.*

For SRF applications, there is a need for developing large-area MgB_2 films for characterization at RF frequencies. In this project, we used a resistive heater in a scaled-up stainless-steel HPCVD reactor to grow large area MgB_2 films with 2" diameter. The film deposited using this technique are uniform and show T_c, J_c and surface morphology similar to those in small-size clean MgB_2 samples deposited by the original HPCVD technique.

A schematic of the HPCVD setup used for the 2-inch film deposition in this work is shown in Fig. 1. In an HPCVD system for small films an inductive heater can be used to heat the susceptor and the Mg bulk pieces on it inside a quartz tube reactor. When the susceptor becomes larger, the inductive heating becomes less uniform, limiting the size of the films that can be grown. In this work, we employed a flat resistive heater coil made of Thermocoax™ heating element to heat a molybdenum susceptor. The heater and the susceptor are housed in a 6-inch diameter stainless steel reactor and a quartz liner tube. The Mo susceptor is 3" in diameter, large enough to accommodate a 2" substrate and magnesium pellets placed in a groove around the substrate. To ensure sufficient Mg vapor pressure across the 2" substrate, it is essential that the center of the susceptor is slightly cooler than the edge. This temperature gradient drives the Mg vapor from the edge of the susceptor, where it is generated, to the center of the substrate. The temperature gradient is achieved by adjusting the radial density of the heating element in the heater coil. Figure 1 presents a typical resistivity-temperature curve of 2" MgB_2 films.

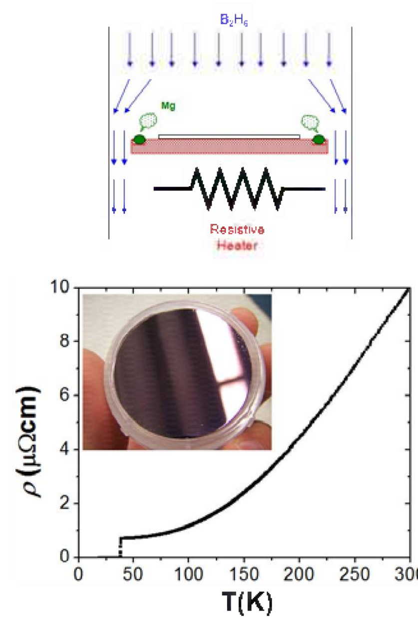

Fig. 1 Top: Schematic diagram of the scaled-up HPCVD reactor. Bottom: Resistivity versus temperature curve for a 350 nm-thick 2" MgB_2 film on *c*-sapphire substrate. Inset shows a picture of the film.

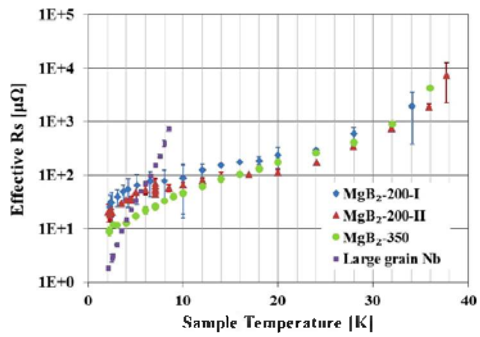

Fig. 2 Effective surface resistance versus temperature of 2" diameter MgB_2 films on sapphire and a large grain Nb sample.

It shows a T_c of 39.1 K, a residual resistivity at 42 K of 0.70 μΩcm, and a residual-resistivity ratio (*RRR*) of 14, indicating excellent film properties. The thickness of the film varies by less than 6% across the 2" substrate. The variation of the film properties are 0.5% for T_c (around 39 K), 14% for *RRR* (around 14), and 16% for J_c(4.2 K) (between 1.5 ~ 1.8 × 10^7 A/cm^2). The results indicate that the uniformity of the sample is good.

Figure 2 shows the effective surface resistance of 2" diameter MgB_2 samples at 7.4 GHz, measured at temperatures between 2.1 and 40 K, with peak fields between 0.6 and 3.7 mT. The surface resistance of a large grain buffered chemical polished Nb sample measured using the same apparatus is plotted as a reference. At 4 K, the MgB_2-350 sample has a comparable surface resistance to Nb.

- *MgB_2 Thin Films on Metal Substrates*

In this project, we have investigated the growth of MgB_2 films on different metal substrates including Nb, Mo, Ta, and stainless steel. All the films were polycrystalline, as indicated by x-ray diffraction and scanning electron microscopy, and showed T_c ~ 39 K, determined by resistance versus temperature, magnetic susceptibility, and dielectric resonator measurements. MgB_2 films deposited on Nb substrates polished to various degrees of smoothness exhibit similar T_c.

Because the metallic substrates are conducting, it is often difficult to obtain the resistivity of the MgB_2 films by the transport measurement even though T_c can be determined by a drop of resistance at the superconducting transition. In Fig. 3, the magnetic susceptibility vs. temperature curves for four MgB_2 films on Nb, Mo, Ta, and stainless steel foils, respectively, are shown. All the films show sharp superconducting transitions with T_c around 39 K. These T_c values are in agreement with those obtained by the transport measurement, and indicate good quality in the MgB_2 films on these metal

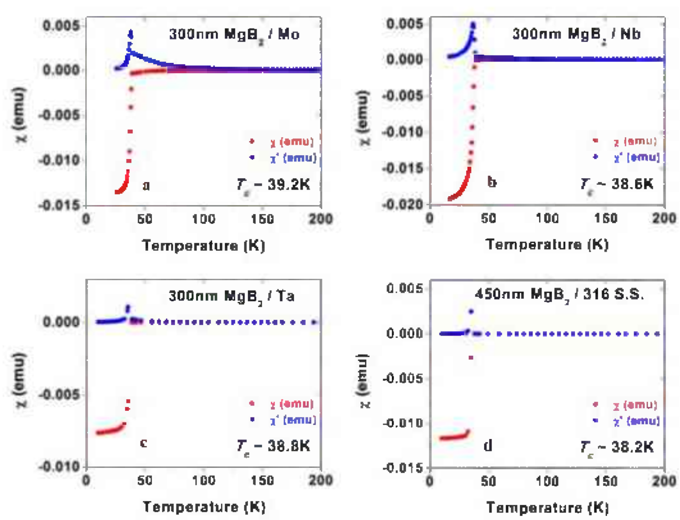

Fig. 3 AC susceptibility measurement of T_c for MgB_2 films on Mo, Nb, Ta, and stainless steel.

substrates. We have also measured the upper critical field H_{c2} by transport measurement under different applied field. On Ta, Mo, and Nb foils, the films show $H_{c2}(0)$ of about 7 T. This low value is similar to that in the clean MgB_2 films on SiC or sapphire single-crystal substrates, an indication of weak scattering in the films. From the *M–H* hysteresis loop measurements using the Bean model, we obtained the critical current density $J_c(H)$ values for MgB_2 films on Ta, Nb, Mo, and stainless steel substrates. The self-field J_c values for all the substrates are around or above 10^7 A/cm^2 even at 20 K, indicating high quality in these films. The J_c values are suppressed rapidly by the applied magnetic field, another indication of the cleanness of the films such that they lack vortex pinning centers.

- *H_{c1} Measurement of Epitaxial and Polycrystalline MgB_2 Thin Films*

It has been suggested that the vortex dissipation loss, which occurs for magnetic fields higher than H_{c1}, is limiting the RF breakdown field rather than H_c. Gurevich proposed a multilayer coating of alternating insulator and high-H_c superconductor layers with a thickness less than its London penetration depth λ to delay the vortex penetration into the cavity bulk. In this project, we have investigated the thickness dependence of H_{c1} in two groups of MgB_2 thin films: epitaxial films on (0001) SiC and polycrystalline films on a room temperature sputtered MgO buffer layer on (0001) SiC. Although H_{c1} enhancement in thin epitaxial films has already been shown, MgB_2 coatings on cavity walls and the top MgB_2 layers in S-I-S multilayer structures are inevitably polycrystalline. Therefore, measurement of H_{c1} in both types of MgB_2 thin films is necessary. To determine H_{c1}, we measured the *m-H* curves of zero-field cooled films. When the applied field is increased to above H_{c1}, vortices enter the superconductor and *m* deviates from the linear *H* dependence.

In Fig. 4, the temperature dependence of H_{c1} for MgB_2 films from both groups is shown. Among the films grown on (0001) SiC substrates, the 300 nm thick film has the lowest H_{c1} at all temperatures, about 600 Oe at 5 K, and demonstrated a linear H_{c1}-T dependence. These properties are similar to previous measurements on bulk MgB_2 samples reported in the literature, indicating that we have reached the thick film limit for MgB_2. H_{c1} increases with decreasing MgB_2 film thickness. The highest value of H_{c1} (5K) is 1880 Oe when the MgB_2 film thickness is 100 nm on bare SiC substrates. The H_{c1}-T curves of the MgB_2 films grown on (0001) SiC and sputtered MgO substrates with the same MgB_2 film thickness are comparable, indicating that the top films in the S-I-S structures or a polycrystalline film

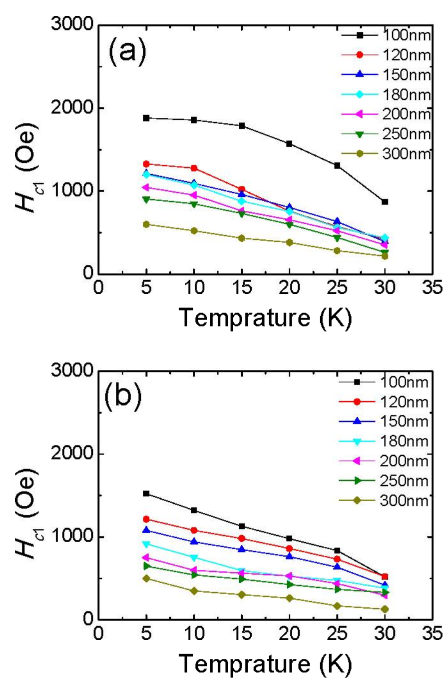

Fig. 4 Temperature dependence of H_{c1} for different film thicknesses for (a) epitaxial films on (0001) SiC substrates, and (b) polycrystalline films on 15 nm sputtered MgO buffer layers on SiC.

grown on metal cavities would have the similar ability to protect the cavity materials from magnetic vortices as clean, epitaxial MgB_2 films.

- *Coating of RF Cavities.*

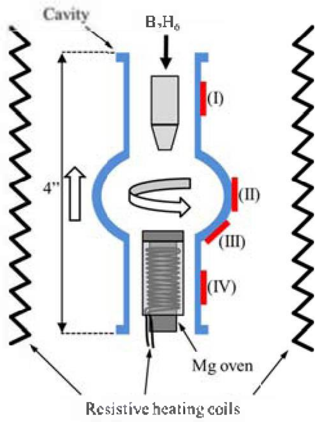

Fig. 5 Schematic of the cavity coating setup.

In this project, we have studied the coating of MgB_2 on the inside wall of the 6 GHz cavity produced by Enzo Palmieri of INFN in Padua, Italy. Figure 5 shows the schematic of the deposition setup. A water-cooled B_2H_6 gas line mounted on the top flange and a Mg oven mounted on the bottom flange of the vacuum chamber come within about an inch from each other coaxially inside the RF cavity. During the growth, the cavity is heated by a clamshell heater and both spins and moves vertically for a uniform coating. Four windows were opened on the top, equator, tilted, and bottom part of the cavity, as shown in the figure, against which sapphire substrates were mounted.

Figure 6 shows the results of 4 MgB_2 films deposited on these substrates through the windows. Of the four films, the bottom one exhibits the lowest residual resistivity of 0.43 μΩcm with a T_c of 40.2 K. The sample positioned at the equator of the cavity has the lowest T_c of 37 K and highest residual resistivity of 30 μΩcm. The result demonstrates the viability of the HPCVD technique for coating the inside wall of SRF cavities.

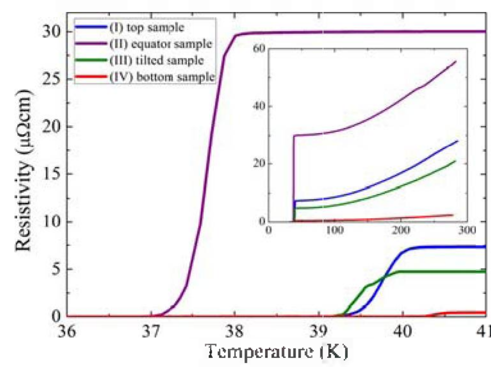

Fig. 6 Superconducting transition for four MgB_2 films on sapphire deposited at four locations on the cavity wall.

In this project, we have also used a two-step approach for coating the cavity. In this approach, the cavity is first coated with a boron layer by flowing diborane through the cavity when the cavity is heated to 500°C. The second step consists of removing the diborane line, closing the top of the cavity off with a stainless steel cap, and exposing the boron layer to Mg vapor. The annealing process takes approximately 30 minutes at 790°C. This procedure successfully produced superconducting films on sapphire substrates mounted on the various locations on the cavity wall with T_c above 37 K. The thickness of the MgB_2 layer on top of the unreacted boron layer can only be estimated. Assuming that resistivity of the MgB_2 layer is the same as the film from the *in-situ* process (29.9 μΩcm), the thicknesses would be approximately 300 nm – 400 nm depending on the location.

5. **List of papers:**

- B. P. Xiao, X. Zhao, J. Spradlin, C. E. Reece, M. J. Kelley, T. Tan, and X. X. Xi, Surface impedance measurements of single crystal MgB_2 films for radiofrequency superconductivity applications, Supercond. Sci. Technol. 25, 095006 (2012). "This work is supported by Jefferson Science Associates, LLC under US DOE Contract No DE-AC05-06OR23177 and by DOE under Contract No DE-SC0004410."
- Tamin Tai, B. G. Ghamsari, T. Tan, C. G. Zhuang, X. X. Xi, and Steven M. Anlage, MgB_2 nonlinear properties investigated under localized high rf magnetic field excitation, Phys. Rev. ST Accel. Beams 15, 122002 (2012). "The work at Temple University is supported by DOE under Grant No. DE-SC0004410."
- Teng Tan, Chenggang Zhuang, Alex Krick, Ke Chen, and X. X. Xi, Large-Area MgB_2 Films Fabricated by Scaled-Up Hybrid Physical–Chemical Vapor Deposition, IEEE Trans. on Appl. Superconductivity 23, 7500304 (2013). "This work was supported in part by the U.S. Department of Energy under Grant DE-SC0004410."
- Chenggang Zhuang, Teng Tan, Alex Krick, Qingyu Lei, Ke Chen, X.X. Xi, MgB_2 Thin Films on Metal Substrates for Superconducting RF Cavity Applications, J. Supercond. Nov. Magn. 26, 1563 (2013). "This work is supported by US Department of Energy under grant No. DE-SC0004410."
- M. A. Wolak, T. Tan, A. Krick, E. Johnson, M. Hambe, Ke Chen, and X. X. Xi, Superconducting magnesium diboride coatings for radio frequency cavities fabricated by hybrid physical-chemical vapor deposition, Phys. Rev. STAB 17, 012001 (2014). "This work was supported by the DOE under Grant No. DE-SC0004410."
- Tamin Tai, Behnood G. Ghamsari, Thomas R. Bieler, Teng Tan, X. X. Xi, and Steven M. Anlage, Near-field microwave magnetic nanoscopy of superconducting radio frequency cavity materials, Appl. Phys. Lett. 104, 232603 (2014). "The work at Temple University was supported by DOE under Grant No. DE-SC0004410."

6. List of people working on the project:

- Teng Tan – graduate student, fully supported by this grant, September 2010 - June 2013.
- Michael Hambe – postdoc, fully supported by this grant, April 2011 – March 2012.
- Matthaeus Wolak - postdoc, fully supported by this grant, November 2012 – June 2013.