Superconducting magnesium diboride coatings for radio frequency cavities fabricated by hybrid physical-chemical vapor deposition

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We have investigated the coating of an inner surface of superconducting radio frequency cavities with a magnesium diboride thin film by hybrid physical-chemical vapor deposition (HPCVD). To simulate a 6 GHz rf cavity, a straight stainless steel tube of 1.5-inch inner diameter and a dummy stainless steel cavity were employed, on which small sapphire and metal substrates were mounted at different locations. The MgB₂ films on these substrates showed uniformly good superconducting properties including T_c of 37–40 K, residual resistivity ratio of up to 14, and root-mean-square roughness R_q of 20–30 nm. This work demonstrates the feasibility of coating the interior of cylindrical and curved objects with MgB₂ by the HPCVD technique, an important step towards superconducting rf cavities with MgB₂ coating.

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I. INTRODUCTION

Superconducting radio frequency (SRF) cavities are widely used in modern particle accelerators. The lower surface impedance of the superconductor leads to a higher quality factor Q of SRF cavities than normal metal rf cavities [1]. At present, niobium is the most commonly used material for SRF cavities [2]. The rf magnetic field in the Nb cavities has approached the thermodynamic critical field H_c of Nb (~ 200 mT at 0 K [3]), which is the limiting field of the accelerating gradient (E_{acc}). It is therefore desirable to explore new materials that would exceed this limit and allow for higher E_{acc} , leading to shorter and more powerful particle accelerators at significantly lower operational costs.

One such material is the binary intermetallic compound magnesium diboride. The superconductivity in MgB₂ was discovered in 2001 [4] and has attracted great interest for SRF applications [5]. The superconducting transition temperature of MgB₂ is around 39 K, significantly higher than Nb (9.2 K). Its lower residual resistivity [6] and larger thermodynamic critical field [7–9] make it a promising candidate for SRF cavity coatings. According to Collings *et al.* [5], MgB₂-coated cavities can potentially exhibit a higher superheating critical field H_{sh} than that of Nb, and as a result, a much higher E_{acc} would be imaginable [10]. In addition, MgB₂ exhibits a low surface resistance (R_s) [11–14] and is free from the weak link behavior that is encountered in high T_c superconductors [15–20], implying

better power dependence of R_s [21]. While the issue has been raised that H_{c1} could be a limiting factor for MgB₂ since its bulk H_{c1} value is lower than that of Nb, studies suggest that it can be enhanced through the employment of multilayer structures containing superconductive films thinner than the London penetration depth, placed between insulating layers [22]. It is also currently being debated that single thin films would have a similar impact like multilayer structures on the performance of SRF cavities [23–25], and recent results obtained for rf cavities coated with Nb₃Sn indicate that H_{c1} might not be a fundamental limitation for the operation of novel SRF cavities [26].

Among the successful MgB₂ growth techniques, hybrid physical-chemical vapor deposition (HPCVD) [27] provides MgB₂ thin films with the highest T_c , the lowest residual resistivity, and low root-mean-square (rms) surface roughness (R_q) [6]. In particular, it is compatible to coating curved surfaces, such as the interior of an rf cavity. In this paper, we describe the approach and results of using HPCVD to deposit MgB₂ thin film on a 6 GHz rf cavity. In order to achieve this goal, we first deposited films on substrates of the same dimensions mounted on a straight stainless steel tube or a dummy cavity, facing the interior of the tube or cavity. Excellent superconducting properties were observed for the films fabricated in this fashion and the results represent the important first step towards the coating of actual cavities with MgB₂ using HPCVD.

II. EXPERIMENTAL DETAILS

The principle of the HPCVD process has been described in detail by Zeng *et al.* [27]. In order to grow MgB₂ thin films, the Mg vapor pressure has to be high enough so that MgB₂ is the thermodynamically stable phase. For clean MgB₂ films with low residual resistivity, the environment of the deposition should be free from oxygen and carbon. In the original HPCVD setup, the high Mg vapor pressure was

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accomplished through the evaporation of Mg bulk pieces placed next to the substrate on a heated circular susceptor. Diborane (H_2B_6) gas was used as the boron source, which decomposes near the heated susceptor and reacts with the Mg vapor to form MgB_2 on the substrate. To ensure a reducing environment, hydrogen gas was used as the carrier gas during deposition. The high purity Mg and boron sources and the reducing environment lead to very clean high quality MgB_2 films. Because the Mg bulk pieces and the substrate are placed together on the susceptor, the evaporation of Mg and heating of the substrate were linked.

The original HPCVD system was appropriate for small (5×5 mm) and flat substrates. In order to coat the interior of a cavity, the original configuration was significantly modified. In the new setup (Fig. 1), located in a stainless steel vacuum chamber, the Mg source and the cavity (the substrate) were separated from each other. The Mg source is a tube-shaped molybdenum oven, wrapped with a resistive coaxial heating element and mounted on the bottom flange of the vacuum chamber. Two resistive clam-shell heaters, fabricated using coaxial heating elements, were used to heat the cavity independently from the Mg source during the deposition. By splitting the heater into two parts, the system can be opened for easy access. Diborane gas (diluted to 5% in H_2) was introduced into the chamber through a water-cooled stainless steel supply line from the top. It is necessary to cool the gas inlet during the deposition in order to prevent premature thermal decomposition of B_2H_6 . However, the cooled diborane line can act as a cold spot for Mg condensation, which can negatively affect the deposition process. The balance between the two conflicting requirements needs to be carefully managed.

FIG. 1 (color online). HPCVD setup for coating rf cavities with MgB_2 . (a) Stainless steel tube sample holder capable of rotational and linear movement; (b) water-cooled diborane supply line; (c) clam-shell heater; and (d) Mg oven.

FIG. 2 (color online). (a) 6 GHz Nb cavity provided by Dr. Enzo Palmieri of INFN in Padua, Italy. (b) Stainless steel dummy cavity with similar dimensions to the 6 GHz Nb cavity. Openings at different locations for mounting the substrates are marked.

The distance of the tip of the diborane line and the top of the Mg oven was kept at about 1.5 inches and MgB_2 forms within this space. A stainless steel tube (coaxial with the diborane supply line) was mounted on the top flange of the vacuum chamber. It held the sample holder via a mounting flange and provided rotational and linear movement to the sample during the deposition.

Figure 2(a) shows the 6 GHz Nb cavity that was chosen to coat the interior with MgB_2 . The cavity was provided by Dr. Enzo Palmieri of INFN in Padua, Italy. The cavity is about 4 inches long, its equator about 1.8 inches in diameter, and the end tubes about 0.8 inches in inner diameter. In the search for optimum deposition parameters, we first used a stainless steel tube, 2.5 inches in length and 1.5 inches in diameter. In the next step, a stainless steel dummy cavity of similar dimensions to the actual 6 GHz Nb cavity was fabricated for the deposition experiments [see Fig. 2(b)]. Openings were made at different locations of the stainless steel tube and the dummy cavity so that $10 \text{ mm} \times 10 \text{ mm}$ substrates could be mounted to face the interior of the tube and cavity and MgB_2 films deposited on them. The substrates employed in the experiments were mostly c-cut sapphire, while metal substrates (including stainless steel, molybdenum, and niobium) were also used.

For each deposition, 8 magnesium pellets of 1/4 inch diameter and 1/4 inch length (99.95% purity) were placed in the Mg oven. Before heating up the system, the chamber was pumped down to better than 10^{-5} Torr. Subsequently, it was purged with 400 sccm of hydrogen for 10 min, and the hydrogen pressure was raised to 40 Torr afterwards. The temperature of the clam-shell heaters and the Mg oven was then gradually increased to 780°C and 750°C , respectively. Heating the Mg oven to 750°C guarantees that a high enough Mg vapor pressure is reached for MgB_2 phase stability. The deposition was started by introducing 80 sccm diborane gas mixture through the supply line. The film thickness was controlled by the flow rate of the diborane gas mixture and the deposition time.

The thickness of the films was measured by a Veeco Dektak step profiler after etching a step into the films. The

FIG. 3 (color online). Schematic view of the setup for stainless steel tube with samples mounted at the (I) top, (II) middle, and (III) bottom locations.

structure of the films was measured by x-ray diffraction (XRD) with $\theta - 2\theta$ and Φ -scans using a Bruker D8 Discover system. The surface morphology was analyzed with atomic force microscopy (AFM) using a Veeco Icon system. The temperature-dependent resistivity and the superconducting transition were measured using the four-probe technique according to the Van der Pauw method [28].

III. RESULTS AND DISCUSSIONS

A. Deposition on stainless steel tube

The setup for depositing MgB_2 thin films on substrates mounted at different locations of the stainless steel tube is schematically shown in Fig. 3. Three holes were opened on the tube, positioned at 0.5, 1, and 1.5 inches from the top, respectively. The substrates were mounted outside the tube with the surface exposed to the interior of the tube where the MgB_2 deposition occurred. During the deposition, the stainless steel tube rotated continuously and travelled for 5 inches along its symmetric axis, evenly exposing the interior to the Mg vapor and diborane gas. A full deposition cycle, in which the Mg oven and diborane line travelled downwards through the tube once, took 5–6 minutes.

$\theta - 2\theta$ XRD spectra of three MgB_2 films on c-cut sapphire substrates mounted at different locations are displayed in Fig. 4(a). The scans from all the three samples show (000 l) reflections of MgB_2 , which indicates a predominant c-axis orientation of the films. There are no other diffraction peaks visible except the substrate related peaks, suggesting phase purity in the films. The diffraction pattern recorded for the bottom sample shows slightly broader MgB_2 peaks, indicating smaller grains in this film as compared to the middle and top samples. The Φ -scans of the MgB_2 film's (10 $\bar{1}$ 1) peak from the top sample and the Al_2O_3 substrate's (10 $\bar{1}$ 4) peak are shown in Fig. 4(b), which

indicates a six-fold symmetry in the MgB_2 film, implying that the hexagonal MgB_2 film is epitaxial and in-plane aligned with the substrate with a 30° rotation. This result clearly demonstrates that epitaxial films were successfully deposited at all locations in the stainless steel tube.

An AFM scan of the top MgB_2 film on Al_2O_3 substrate is shown in Fig. 5. Large interconnected hexagonal grains can be observed, indicating a homogenous film growth. The estimated rms roughness for the $2\text{ }\mu\text{m} \times 2\text{ }\mu\text{m}$ measured area of the 130 nm thick film is 28 nm. The middle film appears to be the thickest among the three and also has the lowest R_q , as can be seen in Table I. The bottom film displays a lower thickness and higher R_q than the middle film. The variation of thickness among different locations is most likely due to the different gas flow patterns at these locations. The diborane gas mixture and Mg vapor were restricted inside the stainless steel tube when the diborane line and Mg oven were located in the center of the tube. The situation was significantly different when the diborane line was still outside of the tube at the top or the Mg oven had exited the tube at the bottom. This is also reflected in the

FIG. 4 (color online). XRD spectra of the MgB_2 films on (0001) sapphire substrates located in the top, middle, and bottom sample pockets on the stainless steel tube. (a) $\theta - 2\theta$ scans. The asterisks represent the peaks originating from the substrate. (b) ϕ scans of the MgB_2 (10 $\bar{1}$ 1) peak and the Al_2O_3 (10 $\bar{1}$ 4) peak for the top sample.

FIG. 5 (color online). AFM image of the MgB_2 film deposited on the Al_2O_3 substrate mounted at the top location of the stainless steel tube. Interconnected hexagonal grains can be clearly seen.

roughness of the films at different locations. We have shown previously that the deposition parameters of HPCVD can have a significant impact on the film roughness and grain size of flat MgB_2 films [29]. The present result on the interior of a round tube is consistent with our previous results.

Figure 6 shows resistivity versus temperature curves for a set of MgB_2 films grown on c-cut sapphire substrates mounted at different locations. Their T_c is in the range from 38.2 to 39.4 K, as displayed in Table I. The residual resistivities are all below $28.2 \mu\Omega\text{cm}$, where the middle film exhibits the lowest residual resistivity. The residual resistivity ratios of the three measured films lie in the range from 3.5 to 7. These values are comparable to properties obtained from samples fabricated using the regular HPCVD setup [6,7,30]. The middle sample consistently shows better properties, including higher T_c , lower resistivity, and smaller surface roughness, suggesting that it is important to restrict the diborane gas mixture and Mg vapor inside the stainless steel tube in order to create a uniform gas flow pattern. For coating a cavity, it is thus necessary to employ extension tubes at both ends to achieve a stable and uniform gas flow pattern throughout the entire cavity.

We have also deposited MgB_2 on metal substrates including niobium, molybdenum, and stainless steel. Figure 7 shows the critical temperature versus sample location for different substrates. The critical temperature was measured by employing the four-probe technique,

TABLE I. Comparison of the characteristics obtained for the three samples mounted on the stainless steel tube.

Sample	Top	Middle	Bottom
Thickness (nm)	130	440	290
Roughness R_q (nm)	28	10	25
Critical temperature T_c (K)	38.2	39.4	39.2
Residual resistivity ($\mu\Omega\text{cm}$)	24.1	5.3	28.2
Residual resistivity ratio	3.5	14	7

FIG. 6 (color online). (a) Resistivity versus temperature for a set of MgB_2 films grown on c-cut sapphire substrates mounted at different locations of the stainless steel tube. (b) Close-up of the transition edge.

where a sudden and significant drop in resistance was taken as a transition of the overlaying MgB_2 layer into the superconducting phase. The T_c value varies between 37.2 and 39.1 K among different substrates and locations, with stainless steel being the worst substrate showing a T_c 2 K below that of the MgB_2 films on Al_2O_3 . Again, the result is consistent with previous reports of MgB_2 films deposited on flat metallic substrates by HPCVD [31–33].

B. Deposition on 6 GHz dummy cavity

The next step of the investigation was to deposit MgB_2 thin films on substrates mounted on the 6 GHz dummy

FIG. 7 (color online). Critical temperatures for different substrates at different locations in the stainless steel tube.

FIG. 8 (color online). Schematic view of the setup used for the dummy cavity with the (IV) top, (V) equator, (VI) curved, and (VII) bottom sample locations.

cavity. The schematic of the HPCVD setup is shown in Fig. 8. Four separate openings were drilled on the dummy cavity to mount substrates at the top and bottom tubes, as well as the equator and the curved face below the equator [see Fig. 2(b)].

Figure 9 shows the temperature dependence of resistivity for a set of MgB_2 films deposited on c-cut sapphire substrates. The bottom film exhibits the lowest residual resistivity of $4.3 \mu\Omega\text{cm}$ with a T_c of 40.2 K, which is a sign of a clean MgB_2 thin film [6]. The samples from the top and the curved face have equal T_c , yet varying residual resistivities, which are shown in Table II. The sample positioned at the equator has the lowest transition temperature and its residual resistivity is significantly higher than the other films. The thicknesses of the four films also vary and are displayed in Table II for comparison. The top film is the thickest of all four films, while the thinnest is the film on the equator. The films at the curved face and bottom tube exhibit similar thicknesses. Among all the films, the film at the equator has the smallest thickness, the lowest T_c , and the highest residual resistivity.

The decrease in film thickness at the equator can partly be explained by the increase of the cross-sectional radius. In the current experiment, the Mg oven and diborane line traveled at a constant speed through the top and bottom tubes, and were slowed down at the equator. When the cross-sectional radius, thus the surface area to be covered, increased significantly at the equator level, the deposition rate still decreased considerably, although the vertical movement was already slowed down. This could be compensated by redirecting the flow of the diborane gas mixture towards the equator. Because of the small size of the dummy cavity, this option has not been explored yet.

It has been shown previously that the sample thickness can directly affect the transition temperature and resistivity of an HPCVD MgB_2 film [29,34]. Films grown in the

FIG. 9 (color online). (a) Resistivity versus temperature for a set of MgB_2 films at different locations of the dummy cavity. (b) Close-up of the transition edge.

Volmer-Weber mode tend to exhibit higher tensile strain with increasing film thickness [35,36], which results in higher T_c . Since the equator film is the thinnest, a lower T_c could be expected. However, the inferior properties of the film at the equator can also be a result of the nonoptimized gas flow pattern and Mg vapor distribution. Unfortunately, the small size of the 6 GHz cavity limits the options in which this can be improved. As shown in Fig. 8, the diameter of the 6 GHz cavity (0.8 inches) is close to that of the water-cooled B_2H_6 line and the Mg oven (~ 0.5 inches), leaving very little space in between. As a

TABLE. II. The characteristics of the MgB_2 films deposited on the four samples mounted on the 6 GHz dummy cavity.

Sample	Top	Equator	Curved	Bottom
Thickness (nm)	350	43	140	120
Roughness R_q (nm)	8	18	15	13
Critical temperature T_c (K)	39.1	36.9	39.1	40.2
Residual resistivity ($\mu\Omega\text{cm}$)	73.3	299	47.5	4.3
Residual resistivity ratio	3.7	1.8	5.4	5.5

FIG. 10 (color online). Schematic views of the two-step process: (a) boron film deposition and (b) annealing of the boron films in Mg vapor.

result, we have not been able to coat the dummy cavity with a uniform MgB_2 film with this setup.

C. Two-step deposition

An alternative approach to the coating of the rf cavity, i.e. a two-step deposition process, was investigated. The process is schematically shown in Fig. 10. In this approach, the dummy cavity was first exposed to the diborane gas mixture at a clam-shell heater temperature of 500°C to

FIG. 11 (color online). (a) Resistivity versus temperature for the top three samples from the two-step process. (b) Close-up of the transition edge.

TABLE. III. A comparison between the MgB_2 films as deposited by the two-step deposition on the top three samples mounted on the 6 GHz dummy cavity.

Sample	Top	Equator	Curved
Estimated thickness (nm)	400	330	380
Critical temperature T_c (K)	37.1	37.6	37.7
Residual resistance (Ω)	0.085	0.220	0.143
Residual resistance ratio	2.1	1.9	2

deposit a boron film on the substrates, as shown in Fig. 10(a). Subsequently, the top of the dummy cavity was closed off with a stainless steel cap and the dummy cavity was exposed to Mg vapor from the Mg oven, as shown in Fig. 10(b). The clam-shell heaters were heated to 790°C during the second step, while the Mg oven was kept at 760°C . Most of the Mg vapor was contained inside the dummy cavity by the stainless steel cap, and the interior was exposed to this vapor for 30 min. At the end of the annealing step, both the clam-shell heaters and the Mg oven were switched off. As the cap at the top had a lower temperature than the dummy cavity because it was subjected to the hydrogen gas flow from the diborane line, the Mg vapor condensed on the inside of the cap rather than on the cavity wall, leaving the MgB_2 films free from the Mg condensate on the surface. The process of annealing boron film in Mg vapor at elevated temperatures to produce MgB_2 film has been reported previously for flat substrates [37,38].

Since only a thin layer at the surface of the boron film was converted to MgB_2 by annealing in Mg vapor, it is difficult to evaluate the resistivity of the MgB_2 films. Thus, the temperature dependence of resistance for the resulting films is instead presented in Fig. 11. Table III shows a comparison of the main characteristics of the three samples at different locations. The films have very similar critical temperatures and the values are slightly lower than the ones obtained for films fabricated with the two-step process as reported in the literature [37,38]. This could be attributed to Mg vapor loss during the annealing step as no tight enclosure was used at the bottom of the dummy cavity, the lower annealing temperature, and the shorter annealing time.

Although the thickness of the films is difficult to define, we can estimate it assuming that the resistivity of the film was comparable to the equator film from the *in situ* process ($300 \mu\Omega\text{cm}$), which had a similar T_c . From the measured resistance values, we estimate the thicknesses of the three films to be above 300 nm, as shown in Table III.

IV. CONCLUSIONS

We have studied coating of MgB_2 on the inside wall of a 6 GHz Nb cavity by HPCVD. In the modified HPCVD 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 came within about 1.5 inches from each other coaxially inside the rf cavity. During the growth, the cavity was heated by clam-shell heaters and spun and moved vertically for a uniform coating. MgB_2 films deposited on substrates mounted at different locations of the cavity show good superconducting properties. In addition, a two-step process was investigated as well, which also produced MgB_2 films with good superconducting properties. We have not been able to coat an actual 6 GHz cavity for rf testing. The main difficulty is the small size of the 6 GHz cavity, which significantly affected the gas flow pattern inside the cavity, leading to different qualities of the films deposited at different locations inside the cavity. This experience suggests that rf cavities of around 3 GHz or lower can provide more space between its wall and the B_2H_6 line and Mg oven, making the gas flow more diffused and the manipulation of gas flow and Mg vapor distribution easier to achieve. The results presented here have established the technical feasibility of coating the inside wall of an rf cavity with MgB_2 using HPCVD, an important first step towards MgB_2 SRF cavity technology.

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