

LI Fen, GUO Tao, ZHANG Kai-cheng, CHEN Chin-ping,
FENG Qing-rong

MgB₂ thick films deposited on stainless steel substrate with T_c higher than 39 K

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Abstract Thick MgB₂ (magnesium diborate) films, ~10 μm, with T_c (onset) = 39.4 K and T_c (zero) = 39.2 K have been successfully grown on a stainless steel substrate using a technique called hybrid physical-chemical deposition (HPCVD). The deposition rate is high, ~ 6.7 nm/s. The X-ray diffraction (XRD) indicates that it is highly (101) and *c*-axis oriented. The scanning electron microscope (SEM) images demonstrate that the film grown is in “island-mode”. The uniform superconducting phase in the film is shown by the *M-T* measurement.

Keywords MgB₂ thick films, stainless steel substrate, superconductivity

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1 Introduction

Since the discovery of superconductivity in a simple binary compound MgB₂ [1], a tremendous amount of research activities have come about. The interest lies in two areas: one is dedicated to understanding the basic properties, and the other, to seeking out a mass production process for high quality wires, tapes, etc., towards large scale applications. Application-wise, MgB₂ is a promising substitute for Nb-based conventional superconductors due to its higher transi-

tion temperature at 39 K. The operating temperature can be easily set at around 20 K to 30 K, much higher than the liquid helium temperature. Hence, the required cryogenic environment can be conveniently obtained using a cryocooler without the cumbersome facilities necessary to handle the cryogenic liquid. The benefits are obvious, immediate and immense. For example, one MgB₂ superconducting magnet, without resorting to liquid helium, could reduce the size of a clinical NMR-CT system. The corresponding medical service would, therefore, become much more mobile and convenient, leading to a much easier and wider access in daily life. The impact would be apparent and far-reaching.

Various techniques have been applied, including PIT [2–5], diffusion of Mg vapor into B-fiber [6], etc., in an effort to find out an effective and efficient process to produce the superconducting elements such as wires, tapes etc. Modest successes have been achieved. However, alternative approaches that would provide easier and more efficient processes are still under intensive studies in laboratories. One alternative may rely on the fabrication of thick film as a pre-stage towards the mass production of these superconducting elements.

In this report, we will demonstrate that HPCVD is an effective and efficient approach to the synthesis of thick MgB₂ films on a stainless steel substrate, and the films indicate good superconducting properties.

2 Experiment

Experimentally, the reaction chamber inside which the vapor deposition takes place is very simple, similar to that reported previously [7]. The mixture of 75 % B₂H₆ in H₂ and Mg ingots serves as the active sources. The Mg ingots were placed around the substrate located on an iron sample holder, which can be heated up inductively by an RF-generator to vaporize the Mg. What is different from the past [8] approaches, is

LI Fen, GUO Tao, ZHANG Kai-cheng, CHEN Chin-ping,
FENG Qing-rong (✉)

Department of Physics and State Key Laboratory for Artificial Structure and Mesoscopic Physics, Peking University, Beijing 100871, China
E-mail: qrfeng@pku.edu.cn

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that the iron sample holder is now supported by a columnar graphite with a bigger thermal capacity, so we can control the temperature of the sample holder more easily. Additional pure H_2 also flows in the reaction chamber, thus reducing the oxygen content and suppressing any further oxidation of the sample during the deposition process. Also, the H_2 would cut down the decomposition rate of B_2H_6 . The flow rate of the B_2H_6 mixture gas was about 10 sccm at a pressure of 2 kPa and the background gas, H_2 , was about 100 sccm at 20 kPa. The temperature of the chamber was regulated within the range of $680^\circ C \sim 720^\circ C$. Under this condition, the deposition rate can reach 6.7 nm/s, which is much higher than that reported by the group in Penn State University [9]. The high growth rate can be ascribed to the high concentration of the B_2H_6 , $\sim 75\%$.

3 Results and discussions

The XRD analysis was performed using a Philips X'Pert diffractometer. Figure 1 shows the poly-crystalline MgB_2 crystal structure. The strong peaks of (101) and (002) for MgB_2 suggests that the film is highly (101) and c -axis textured. Due to the excess Mg vapor during the depositing process, Mg can be present in the deposited films. Furthermore, the presence of MgO may result from the residual O_2 adhering to the chamber inner-wall and the surface of Mg ingots, existing in the background gas, H_2 , etc. Besides, the broad but not strong peak around 23° is due to the poorly crystallized B [10]. The characteristic peaks for the substrate do not appear on the spectrum because the film thickness, $\sim 10 \mu m$, exceeds the typical X-ray penetration length, by a few micrometers.

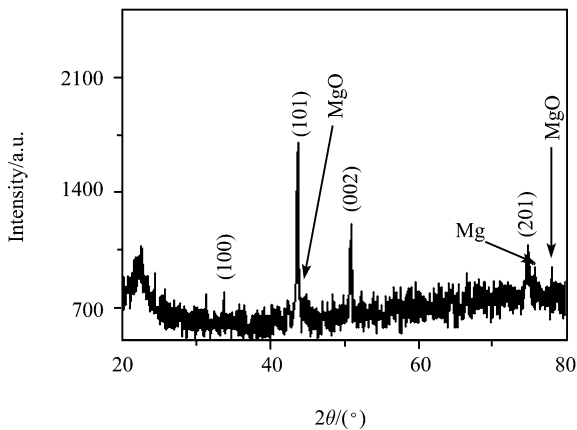


Fig. 1 The X-ray diffraction pattern of MgB_2 film grown on the stainless steel substrate.

The temperature-dependent resistivity relationship (ρ - T curve) was performed with a standard 4-probe measurement

using the Quantum Design PPMS System. Figure 2 clearly shows that the transition temperature, $T_c \sim 39.4$ K, and the 90%–10% transition width, $\Delta T \sim 0.2$ K. This is comparable or sharper than most of the thin film or even bulk samples reported [8, 11, 12]. Based on the resistance at room temperature and the transition temperature, RRR is calculated as 1.9, which is comparable with values reported previously [13]. This is likely due to the small grain sizes and existence of MgO in the film, since precipitates of MgO at the grain boundaries will act as series-connected resistors to the MgB_2 grains.

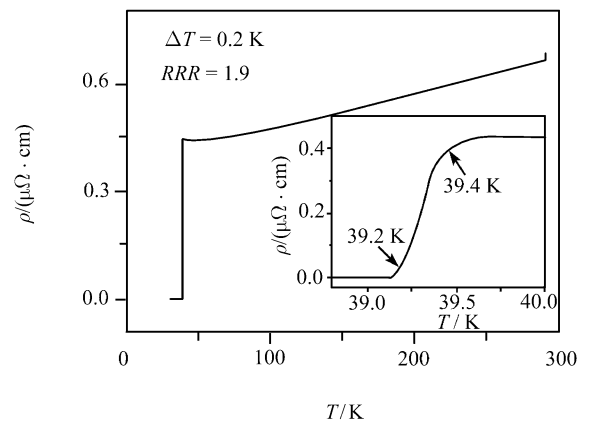
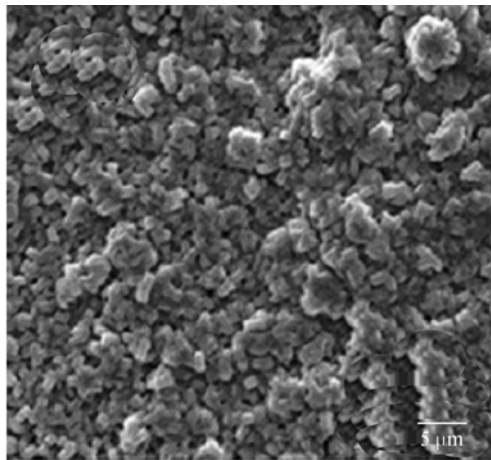


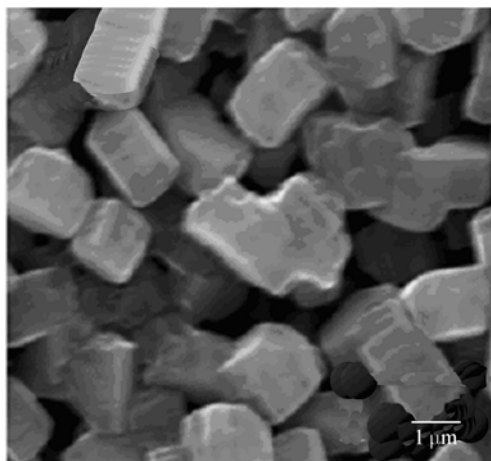
Fig. 2 The ρ - T measurement of the MgB_2 thick film. The inset shows a magnified view near the transition region.

The SEM observations were carried out by a QUANTA 200 FEG scanning electron microscope. The characteristic surface morphology of the MgB_2 thick film shown in Figs. 3 (a) and (b) are the film images with the magnification of 8000 and 60 000 respectively. From Fig. 3 (a), we can see that although the film is not very smooth, which is related to the high concentration of B_2H_6 and the deposition rate in the film synthesizing process, it is quite dense. Seeing Fig. 3 (b), we can learn that the film consists of crystallites, ~ 800 nm long and 400 nm wide. And these crystallites are made up of many smaller grains, ~ 130 nm. This phenomena demonstrates that the film is grown in an “island-model”. The gaps between the big grains, thus reducing the connectivity of the crystallites, may be another reason for the low RRR , ~ 1.9 .

At the same time, the M - T measurement is being carried out with the applied magnetic field H parallel to the film surface using a Quantum Design SQUID magnetometer. The result is plotted in Fig. 4, from which we can learn that the superconducting transition temperature is 38.1 K. In the transition region, the M - T curve is sharp without the “shoulder” structure, indicating that the superconducting phase in the film is uniform. In the lower temperature region, especially 11 \sim 31 K, the presence of flux jumping can be observed. This relates to the very fine disorder structure and the relatively small thermal diffusion in the films at low temperature [14].



(a)



(b)

Fig. 3 SEM images of the fabricated thick MgB_2 film on stainless steel substrate. **(a)** The characteristic surface morphology of the MgB_2 thick film under 8000 magnification. **(b)** The image with higher magnification of 60 000.

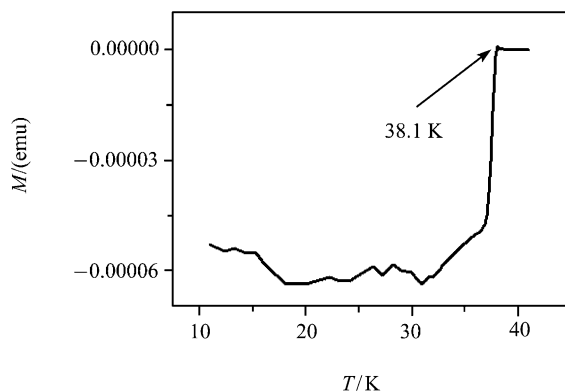


Fig. 4 The M - T measurement carried out with the applied magnetic field H of 10 Oe parallel to the film surface.

4 Conclusion

In conclusion, the present experiment demonstrates that thick MgB_2 films of about $10\ \mu\text{m}$, with T_c (onset) $>39.4\ \text{K}$ and sharp transition $0.2\ \text{K}$, have been grown on a stainless steel substrate by using the HPCVD technique. The deposition rate is high, $\sim 6.7\ \text{nm/s}$, and the film is (101) and c -axis textured. The sharp but no “shoulder” transition in the M - T curve indicates a uniform superconducting phase in the film. These results indicate that the HPCVD technology is fit for synthesizing thick MgB_2 films.

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