Comparison of critical properties in MgB$_2$ nanometer films prepared on SiC/Si substrate

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Abstract. Critical superconducting properties in 100nm MgB$_2$ film are investigated and examined in comparison with our previous results in 10nm and 50nm films. MgB$_2$ films were prepared by sequential evaporation of boron and magnesium on SiC-buffered Si substrate followed by in situ annealing. The amount of supplied boron was controlled so as to result in the required MgB$_2$ film thickness with excess Mg top layer. The superconducting transition temperature $T_c$ is estimated to be 33.1K from the onset of AC diamagnetic susceptibility. The upper critical field $H_{c2}$ is also estimated from the AC susceptibility. Their temperature dependences are almost linear with $|dH_{c2}/dT| \approx \pm 5.6\text{kOe/K}$ for the parallel magnetic field and $|dH_{c2}/dT| \approx \pm 3.9\text{kOe/K}$ for the perpendicular field. These values are larger than those in our previous 50nm film ($T_c$ of 34.5K) in spite of a little lower transition temperature. The critical current density $J_c$ and irreversibility field $H_{irr}$ are estimated from DC magnetization hysteresis. Scaling behavior of $H_{irr} \sim [1 - (T/T_c)^2]^n$ is examined in comparison to our previous studies in 10nm and 50nm films with different critical exponents $n$.

1. Introduction

Investigations of the binary metallic MgB$_2$ superconductor in the form of thin films are important both from basic as well as application points of view, since the form of thin film can provide variety of different sample conditions and variety of electronics applications [1].

In our previous article [2], nanometer thickness effects were investigated in MgB$_2$ thin films (10nm thick with the superconducting transition temperature $T_c = 26.0\text{K}$ and 50nm thick with $T_c = 34.5\text{K}$). Under magnetic field perpendicular to the film surface, temperature dependence of the upper critical field $H_{c2}$ was almost linear with $|dH_{c2}/dT| \approx -3.1\text{kOe/K}$ for the 50nm film, while for the 10nm film overall dependence was about $|dH_{c2}/dT| \approx -4.8\text{kOe/K}$ yet with apparent positive curvature. The lowest temperature value of $H_{c2}$ for the 10nm film with short coherence length exceeded the one for the 50nm film in spite of much lower $T_c$. Scaling behavior of the irreversibility field $H_{irr} \sim [1 - (T/T_c)^2]^n$ with different critical exponents $n$ was examined in terms of superconducting character.

In this work, we prepare thicker film of 100nm and comparatively investigate superconducting critical properties along with examinations on $T_c$, $H_{c2}$, $J_c$ and $H_{irr}$. 
2. Experimental

MgB\(_2\) films were prepared by sequential electron beam evaporation of boron and magnesium on SiC-buffered Si substrate followed by in-situ annealing [3]. The precursor Mg-B bilayers were deposited at room temperature substrate, where the amount of supplied boron was controlled so as to result in film thickness of 100nm. As-deposited film was in-situ heated to 280°C for 30 min in an argon atmosphere at a pressure of 0.06 Pa. Subsequently, the pressure was increased up to 16 Pa and the temperature was increased to maximum temperature of 750°C and kept there for 10 min. The sample was then cooled down to room temperature at 10\(^{-3}\) Pa Ar pressure.

AC and DC magnetizations were measured with magnetic fields parallel and perpendicular to the film using PPMS magnetometer (Quantum Design). The transition temperature \(T_c\) was evaluated from the onset of AC diamagnetic susceptibility at each magnetic field \(H\). The critical current density \(J_c\) was estimated from DC magnetization hysteresis with the Bean critical state model: \(J_c = 30\Delta M/r\), where \(\Delta M\) is the height of the magnetization loop and \(r\) is the sample half-width (about 0.16 cm). Irreversibility field \(H_{irr}\) was also estimated from diminishing magnetization hysteresis.

3. Results and discussion

3.1. AC magnetization measurements and upper critical field

Figure 1 shows onsets of AC diamagnetic susceptibility \(\chi'\) under various magnetic fields perpendicular to the surface of the present 100nm MgB\(_2\) film. The measurements were performed with the field-cooling condition. The superconducting transition temperature \(T_c\) is estimated to be 33.1K at minimal magnetic field. This is a little lower than \(T_c\) of 34.5K in our previous 50nm film. Decrease of \(T_c\) suggests some more impurities or defects mingled by chance into the present 100nm film. From onset transition temperatures \(T_c(H)\) as indicated by arrows, we can estimate temperature dependent upper critical fields \(H_{c2}(T)\). Diamagnetism was much weaker with magnetic field parallel to the film surface.

Thus estimated \(H_{c2}(T)\) is indicated in figure 2 as a function of temperature \(T\) for the perpendicular (solid circles) and parallel (open circles) magnetic fields. As seen in the figure, temperature dependence is almost linear except for just below \(T_c\). The temperature coefficient \(\frac{dH_{c2}}{dT}\) is about 5.6kOe/K for the parallel magnetic field and 3.9kOe/K for the perpendicular field. These values are larger than those in our previous 50nm film in spite of a little lower transition temperature for 100nm film. This infers that the coherence length \(\xi\) is shorter in the present film, which is possibly due to more impurities or defects reducing mean-free path along with the dirty limit.
Almost linear temperature dependence in $H_{c2}(T)$ is consistent with the behavior in the previous 50nm film and absence of distinct positive curvature indicates that the present 100nm film (as well as 50nm film) is in the category of three-dimensional superconductors (though anisotropic).

The anisotropy parameter $\gamma$ can be estimated from the temperature coefficient ratio between parallel and perpendicular $H_{c2}$ as $\gamma = \xi_{\parallel} / \xi_{\perp} = |dH_{c2\parallel}/dT| / |dH_{c2\perp}/dT|$. In table 1 we summarize critical superconducting parameters evaluated in our MgB$_2$ films of 10nm, 50nm and 100nm thickness.

| Thickness (nm) | $T_c$ (K) | $|dH_{c2\parallel}/dT|$ (kOe/K) | $|dH_{c2\perp}/dT|$ (kOe/K) | $\gamma$ |
|---------------|-----------|-------------------------------|-------------------------------|--------|
| 10            | 26.0      | 5.6                           | 4.8                           | 1.2    |
| 50            | 34.5      | 4.9                           | 3.1                           | 1.6    |
| 100           | 33.1      | 5.6                           | 3.9                           | 1.4    |

The very low $T_c$ of 26.0K as well as the very small value of $\gamma$ in 10nm film probably reflects anomalous two-dimensional character (granular superconductivity) as discussed previously [2].

A little smaller $\gamma$ in 100nm film than 50nm can be explained by the presence of more defects or impurities as mentioned above. In our previous report [2], we discussed possibility of limitation of coherence length by the crystal grain size. Because the present films are not single crystal, grain sizes would be less than at least 1/10 of the film dimensions and almost the same between 50 and 100 nm films. Thus, the perpendicular coherence length $\xi_{\perp}$ may be limited by the perpendicular grain size of a few nanometer and almost unchanged, while the parallel coherence length $\xi_{\parallel}$ may not be limited by larger grain sizes in the layer direction and then mingled impurities may result in decrease of $\xi_{\parallel}$ as well as $\gamma$ in 100nm film.

### 3.2. Critical current density and irreversibility field

Critical current density $J_c$ in 100nm MgB$_2$ film is given in figure 3 as a function of magnetic field $H$ perpendicular to the film, where $J_c$ was deduced from DC magnetization hysteresis and Bean model.

**Figure 3.** Critical current density $J_c$ in 100nm MgB$_2$ film as a function of perpendicular magnetic field $H$.
Obtained \( J_c \) is rather small compared with maximum values of 0.6MA/cm\(^2\) and 5MA/cm\(^2\) in the previous 10 and 50 nm films, respectively. The reason is not entirely clear at present, though above mentioned defects or impurities may obstruct coupling between crystal grains and reduce \( J_c \) values throughout the whole sample dimension.

We next estimate the irreversibility field \( H_{irr} \) based on vanishing criterion of \( J_c=\text{300A/cm}^2 \) (about 1/20 of the maximum observed \( J_c \)). Figure 4 indicates variation of \( H_{irr} \) in 100nm film under perpendicular magnetic field as a function of \( 1-T^2 \), where \( t=T/T_c \). In the inset of the figure we also represent the former results in 10 and 50 nm films [2]. For high-\( T_c \) superconductors \( H_{irr} \) is often examined in relation to the scaling law as \( H_{irr}(T) = H_{irr}(0)\left[1-(T/T_c)^{1/2}\right]^{4}[4,5] \). The straight lines in figure 4 correspond to the least square fitting to the data based on this scaling equation. From such fitting, the critical exponent \( n \) is deduced to be about 8 for 100nm film in contrast with 3 and 1.5 for 50nm and 10nm films. The \( n \) value seems to increase with increasing thickness of the MgB\(_2\) film.

The difference in \( n \) values reflects difference in the pinning mechanism. Matsushita et al. [6] examined the pinning mechanism in melt-textured Bi-2212 superconductor and discussed that smaller \( n \) value corresponds to flux pinning due to non-superconducting inclusions, while larger \( n \) value corresponds to the pinning due to small defects such as point defects or dislocations. Thus, the large value of 8 for our 100nm film infers that the main pinning centers in this film are defects or impurities, whose presence was repeatedly suggested in the above discussion in §3.1. The irreversibility field at 0K, \( H_{irr}(0) \), can also be derived from the least square fitting to the data, the results being 46kOe, 14kOe and 3.8kOe, respectively, for 100nm, 50nm and 10nm films. The largest value of \( H_{irr}(0) \) in 100nm film is again explained by the presence of many defects in this film.

4. Conclusion

Thick film of 100nm MgB\(_2\) was prepared and comparatively investigated on various critical superconducting properties along with examinations on \( T_c, H_{c2}, J_c \) and \( H_{irr} \). MgB\(_2\) films were prepared by sequential electron beam evaporation of boron and magnesium followed by in-situ annealing.

Comparing our critical parameters in three different MgB\(_2\) films, 100nm thickness resulted in anisotropic three-dimensional superconductor with rather many defects or impurities, showing small \( J_c \) and large \( H_{irr}(0) \). Such impurities may come from rather long evaporation time needed for accumulating the very thick B and Mg layers. On the other hand, the smallest thickness of 10nm resulted in anomalous two-dimensional superconductivity with the largest \( H_{c2} \) but the smallest \( H_{irr} \). After all, as far as we prepared, the 50nm thickness seems to result in the best film, indicating fair values of \( H_{c2} \) and \( H_{irr} \) along with the highest \( T_c \) as well as the largest \( J_c \).

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