

**Superconducting anisotropy and evidence for intrinsic pinning in single crystalline MgB<sub>2</sub>**

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(Received 28 February 2002; published 20 June 2002)

We examine the superconducting anisotropy  $\gamma_c = (m_c/m_{ab})^{1/2}$  of a metallic high- $T_c$  superconductor MgB<sub>2</sub> by measuring the magnetic torque of a single crystal. The anisotropy  $\gamma_c$  does not depend sensitively on the applied magnetic field at 10 K. We obtain the anisotropy parameter  $\gamma_c = 4.31 \pm 0.14$ . The torque curve shows the sharp hysteresis peak when the field is applied parallel to the boron layers. This comes from the intrinsic pinning and is experimental evidence for the occurrence of superconductivity in the boron layers.

DOI: 10.1103/PhysRevB.66.012501

PACS number(s): 74.25.Ha, 74.60.-w

**I. INTRODUCTION**

Since the discovery of superconductivity in MgB<sub>2</sub> (Ref. 1) considerable progress has been made in determining the physical properties of this material. The new materials are metallic and hence promising when considering the applications in the various fields. The extremely high  $T_c$  (39 K) sheds doubt on whether or not the superconductivity can be explained within a conventional BCS framework. Tunneling studies show that the material is reasonably isotropic and has a well-developed  $s$ -wave energy gap.<sup>2,3</sup> The anisotropy parameter  $\gamma_c = (m_c/m_{ab})^{1/2}$  of MgB<sub>2</sub> in the literature ranges from 1.2 to 9 in polycrystalline samples.<sup>4</sup> Therefore, it is highly desirable to investigate the fundamental properties of MgB<sub>2</sub> by using a single crystal. Recently, Xu *et al.*<sup>5</sup> succeeded in synthesizing the single crystals by the vapor transport method and reported the superconducting properties of MgB<sub>2</sub>.

The first-principles calculation by Kortus *et al.*,<sup>6</sup> by Choi *et al.*,<sup>7</sup> and by Yildirim *et al.*<sup>8</sup> suggested that the boron layers govern the superconductivity. The boron isotope effect on  $T_c$  supports this idea.<sup>9</sup> However, the experimental confirmation of the superconductivity in the boron layers is not thoroughly convincing so far.

Torque is a sensitive tool for probing the various kinds of anisotropy, and has been successfully applied to investigate the highly anisotropic high- $T_c$  cuprates. The electronic anisotropy of high- $T_c$  cuprates was investigated by Farrell *et al.*<sup>10</sup> and Ishida *et al.*<sup>11</sup> The high- $T_c$  cuprates are characterized by the extreme electronic anisotropy  $\gamma_c = \sqrt{m_c/m_{ab}} = 7-200$  as well as the layered structure of superconductivity. The superconductivity is governed by the CuO<sub>2</sub> layers or by the CuO<sub>2</sub> bilayers. An alternative stacking of the CuO<sub>2</sub> layer and the blocking layer is a key concept both in crystal structure and in the occurrence of the superconductivity. This is also the origin of the intrinsic pinning for vortices in high- $T_c$  superconductors. The MgB<sub>2</sub> structure consists of an alternative stacking of the boron layer and the magnesium layer, too. It is of special interest to investigate the similarity and

dissimilarity between this material MgB<sub>2</sub> and the cuprate superconductors.

It is crucial to discriminate the intrinsic pinning from various other pinning sources. The magnetic torque has an advantage to see the vortex pinning as a function of angle with respect to a crystalline axis. Since the intrinsic pinning works effectively when the field is almost in parallel to the CuO<sub>2</sub> planes, it manifests as a hysteresis peak in torque.<sup>12,13</sup> Therefore, a torque is a sensitive probe for sensing an intrinsic (directional) pinning.

In this paper, we report the electronic anisotropy of MgB<sub>2</sub> by means of the magnetic torque. We also describe evidence for the intrinsic pinning in MgB<sub>2</sub> when the field is applied parallel to the boron layers. This ensures that the superconductivity occurs in the boron layers.

**II. EXPERIMENT****A. Sample**

The details of the single-crystal preparation of MgB<sub>2</sub> are reported by Xu *et al.*<sup>5</sup> They reported that the onset temperature of superconductivity is 38.6 K. Because of the severe volatility of Mg and the high melting point of B, MgB<sub>2</sub> single crystals were grown in a closed system. The starting material of Mg was 99.99% pure and B was 99.9% pure. The starting materials, with a molar ratio of 1:1.9 were sealed inside a molybdenum crucible of internal diameter 10 mm, and length 60 mm by the electron-beam welding. The molybdenum crucible was used in a high-frequency induction furnace. The crucible was first heated to 1400 °C at a rate of 200 °C/h and kept for 2 h, then slowly cooled to 1000 °C at a rate of 5 °C/h, and finally cooled to room temperature by switching off the power. A platelike single crystal of MgB<sub>2</sub> ( $\sim 0.11 \text{ mm}^2 \times 14 \text{ }\mu\text{m}$ ) was used for the torque measurements. The  $c$  axis was perpendicular to the plate. The sample weight was too tiny to measure by our electronic balance of resolution 0.01 mg.

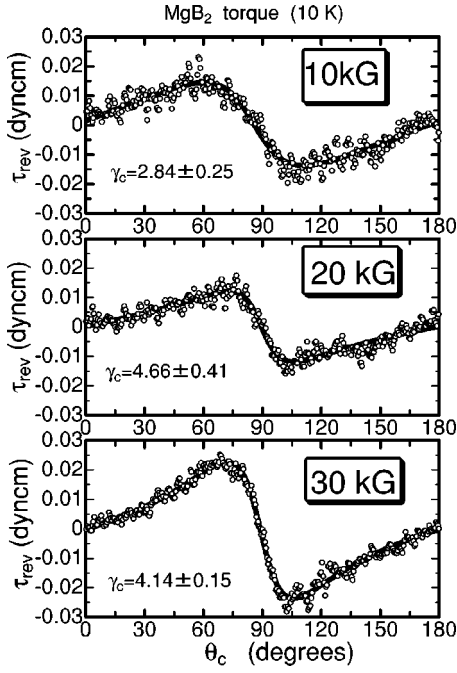


FIG. 1. The reversible torque  $\tau_{rev}$  of  $\text{MgB}_2$  as a function of angle  $\theta_c$  at 10 K (10 kG, 20 kG, 30 kG). The reversible torque is determined as  $\tau_{rev}(\theta) = [\tau_{inc}(\theta) + \tau_{dec}(\theta)]/2$  where  $\tau_{inc}(\theta)$  and  $\tau_{dec}(\theta)$  are the torques as a function of increasing and decreasing angle. The torque curves are analyzed the Kogan model by fixing  $\eta H_{c2} = 60$  kG (see  $\gamma_c$  of the figures).

### B. Torque

A split-type superconducting magnet changed a magnetic field continuously from  $-60$  kG to  $+60$  kG, and had a variable temperature insert from 4 K to 300 K. A torque detection system was attached on the top of the insert. A phosphor bronze string hung a balance arm, a feedback coil, and a quartz sample rod in a helium-gas atmosphere from the main flange. The feedback coil (300 turns of a 0.075-mm Cu wire) was located in a special hollow cylindrical NdFeB permanent magnet (Magnetic Solutions) of a transverse field of 5000 G to 8000 G. A sample torque could be canceled by a controlled torque given by a torque detection mechanism at the top of the insert. We used an optical position sensor to maintain the sample direction. The sample could be rotated by a stepper motor (a resolution of  $0.0036^\circ$ ). The torque dynamic range was from  $-10^3$  dyn cm to  $+10^3$  dy ncm with a sensitivity of  $10^{-3}$  dyn cm.

### III. RESULTS AND DISCUSSIONS

We measured the torque of single crystalline  $\text{MgB}_2$  as a function of  $\theta_c$ . The angular step was chosen as  $0.5^\circ$ . The irreversible torque was extracted by  $\tau_{irr}(\theta) = [\tau_{dec}(\theta) - \tau_{inc}(\theta)]/2$  and the reversible torque was obtained as  $\tau_{rev}(\theta) = [\tau_{inc}(\theta) + \tau_{dec}(\theta)]/2$  where  $\tau_{inc}(\theta)$  and  $\tau_{dec}(\theta)$  are the torques as a function of increasing and decreasing angle, respectively.

In Figs. 1 and 2, we show the reversible torque  $\tau_{rev}$  of  $\text{MgB}_2$  at 10 K (10 kG, 20 kG, 30 kG) and the reversible

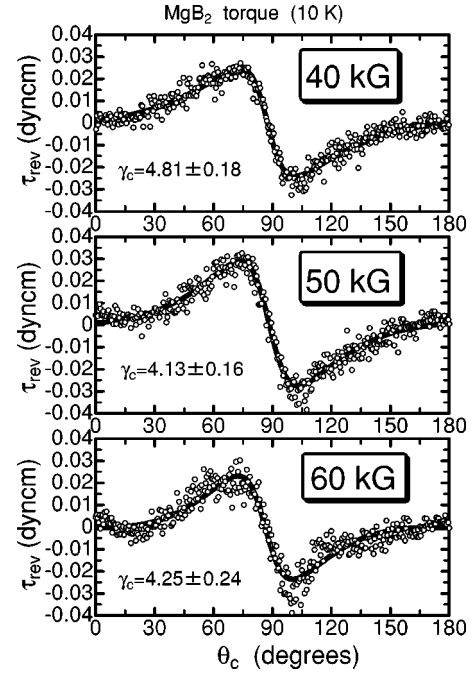


FIG. 2. The reversible torque  $\tau_{rev}$  of  $\text{MgB}_2$  as a function of angle  $\theta_c$  at 10 K (40 kG, 50 kG, 60 kG). The reversible torque is determined as  $\tau_{rev}(\theta) = [\tau_{inc}(\theta) + \tau_{dec}(\theta)]/2$  where  $\tau_{inc}(\theta)$  and  $\tau_{dec}(\theta)$  are the torques as a function of increasing and decreasing angle. The torque curves are analyzed the Kogan model by fixing  $\eta H_{c2} = 60$  kG (see  $\gamma_c$  of the figures).

torque  $\tau_{rev}$  of  $\text{MgB}_2$  at 10 K (40 kG, 50 kG, 60 kG), respectively. The shape of the torque curves in Figs. 1 and 2 is similar to the torque curve of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ .<sup>11</sup> This indicates that the  $\text{MgB}_2$  superconductor is anisotropic with respect to the  $c$  axis. In the three-dimensional anisotropic London model in the mixed state, the angular dependence of the torque is given by Kogan<sup>14</sup> as

$$\tau_{rev}(\theta_c) = \frac{\phi_0 H V}{16\pi\lambda^2} \frac{\gamma_c^2 - 1}{\gamma_c^{1/3}} \frac{\sin 2\theta_c}{\epsilon(\theta_c)} \ln \left[ \frac{\gamma_c \eta H_{c2}^\perp}{H \epsilon(\theta_c)} \right], \quad (1)$$

where  $\epsilon(\theta_c) = (\sin^2\theta_c + \gamma_c^2 \cos^2\theta_c)^{1/2}$ ,  $\theta_c$  is the angle between the applied field and the  $c$  axis,  $\gamma_c = \sqrt{m_c/m_{ab}}$ ,  $H_{c2}^\perp$  is the upper critical field perpendicular to the  $ab$  plane ( $\eta \sim 1$ ), and  $V$  is the sample volume. This equation is relatively simple, and has frequently been employed in the literature to analyze the electronic anisotropy of various high- $T_c$  cuprates.<sup>10,11,15</sup> The computer fitting of  $\tau_{rev}$  to the Kogan model gives the anisotropy parameter  $\gamma_c = 2.8 - 4.8$  where  $\eta H_{c2}$  is fixed to 60 kG.<sup>5</sup>

As shown in Fig. 3, the torque curve measured in 10 kG at 10 K has a hysteresis against the angle scans. The torque as a function of increasing as well as decreasing angle has a sharp peak at  $\theta_c \approx 90^\circ$ . This is well known as an intrinsic pinning peak for the high- $T_c$  cuprates.<sup>12,11</sup> The dashed line is the reversible component (see the fitted line in the top figure of Fig. 1). The peak appearing near  $90^\circ$  represents the manifestation of intrinsic pinning in this superconductor. This is experimental confirmation of the superconductivity in the

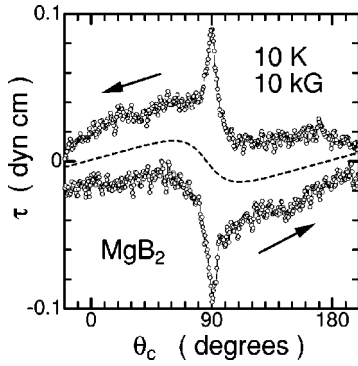


FIG. 3. The torques as a function of increasing as well as decreasing angle are presented in 10 kG at 10 K. The dashed line is the reversible component (see top figure of Fig. 1). The peak appearing near  $90^\circ$  represents the manifestation of intrinsic pinning in this superconductor. This is clear evidence for layered superconductivity in the boron layers.

boron layers.<sup>6-8</sup> The remarkable hysteresis of the torque curve give rise to the uncertainty in the reversible torque obtained by  $\tau_{rev}(\theta) = [\tau_{inc}(\theta) + \tau_{dec}(\theta)]/2$ . Actually,  $\gamma_c$  in 10 kG is appreciably less than those in other fields at 10 K. A tiny intrinsic pinning peak can be seen in the torque curve at 20 kG. Note that the torque curves are almost reversible for 30 kG, 40 kG, 50 kG, and 60 kG.

The  $\gamma_c$  is almost independent of field between 20 kG and 60 kG at 10 K. We omitted  $\gamma_c$  in 10 kG to obtain the averaged  $\langle \gamma_c \rangle = 4.31 \pm 0.14$ . This is somewhat larger than those reported in the literature.<sup>4</sup>

Angst *et al.*<sup>16</sup> also reported the torque measurement of MgB<sub>2</sub>. Their measurement regime is complimentary to ours. They mainly use the torque as a sensitive means of determining  $H_{c2}$  as a function of angle  $\theta_c$ . However, the determination of  $H_{c2}$  is dependent on the criterion of the torque onset, and their criterion contains a target parameter  $\gamma_c$ . The upper critical field  $H_{c2}(\theta_c)$  thus obtained is analyzed by the effective-mass model. They obtained  $\gamma = 6$  at 15 K and  $\gamma = 2.8$  at 35 K. They also used the Kogan model to analyze the torque data and found the field dependence of  $\gamma_c$ . They interpreted it in terms of the double gap structure of MgB<sub>2</sub>. We note that this is not the case in our torque measurements because  $\gamma_c$  is almost independent of field at 10 K.

We attempted to measure the temperature dependence of the torque curve in 60 kG, but the signal-to-noise ratio was not satisfactory enough to analyze the torque data. This is due to the small size of the MgB<sub>2</sub> crystal. Our results were inconclusive regarding the temperature dependence of  $\gamma_c$ . Measurements using a larger crystal are desirable for the detailed studies of the MgB<sub>2</sub> system.

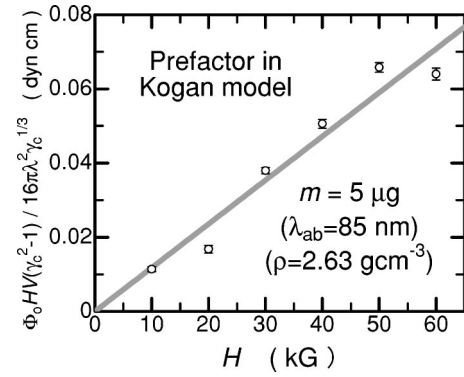


FIG. 4. A prefactor  $\phi_0 HV(\gamma_c^2 - 1)/16\pi\lambda^2\gamma_c^{1/3}$  of the Kogan formula as a function of  $H$ . From the slope, the sample mass is estimated to be approximately  $5 \mu\text{g}$ .

Finally, we estimate a weight of our MgB<sub>2</sub> sample from the torque amplitude. In the Kogan model, we obtain a prefactor of  $\phi_0 HV/16\pi\lambda^2$  by the least-squares fitting where the penetration depth should read as  $\lambda = (\lambda_c \lambda_{ab}^2)^{1/3}$ . In Fig. 4, we plot  $\phi_0 HV/16\pi\lambda^2$  as a function of  $H$ . The data points are approximately fitted by a straight line. From the slope, one can determine the sample volume  $V$ . By assuming  $\lambda_{ab} = 85 \text{ nm}$ ,<sup>4</sup> the density  $\rho = 2.63 \text{ g/cm}^3$ , and  $\gamma_c = 4.31$  we estimate the mass  $m$  of our sample as  $\sim 5 \mu\text{g}$ . We note that the mass calculated from the measured size of the crystal and the density of MgB<sub>2</sub> is approximately  $3.3 \mu\text{g}$ .

#### IV. CONCLUSIONS

The torque curves at 10 K are almost reversible in fields larger than 20 kG. The electronic anisotropy of MgB<sub>2</sub> is determined by the torque as  $\gamma_c = 4.31 \pm 0.14$ . The new superconductor exhibits an intrinsic pinning at 10 K in 10 kG when the field is applied parallel to the boron layers. There is a modulation of the order parameter in MgB<sub>2</sub> along the  $c$  axis. This indicates that the boron layers, as suggested from the first-principles calculations,<sup>7</sup> govern the superconductivity. The MgB<sub>2</sub> superconductor is rather similar to the high- $T_c$  cuprates while the anisotropy of MgB<sub>2</sub> is moderate compared to the cuprates.

#### ACKNOWLEDGMENTS

This work was partially supported by a Grant-in-Aid for Scientific Research (Project Nos. 12554012, and 12874042) granted by the Ministry of Education, Science, and Culture of Japan.

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<sup>1</sup>J. Nagamatsu, N. Nakagawa, T. Muranaka, Y. Zenitani, and J. Akimitsu, *Nature (London)* **410**, 63 (2001).

<sup>2</sup>H. Schmidt, J.F. Zasadzinski, K.E. Gray, and D.G. Hinks, *Phys. Rev. B* **63**, 220504 (2001).

<sup>3</sup>A. Sharoni, I. Felner, and O. Millo, *Phys. Rev. B* **63**, 220508 (2001).

<sup>4</sup>C. Buzea and T. Yamashita, *Supercond. Sci. Technol.* **14**, R115 (2001).

<sup>5</sup>M. Xu, H. Kitazawa, Y. Takano, J. Ye, K. Nishida, H. Abe, A. Matsushita, N. Tsujii, and G. Kido, *Appl. Phys. Lett.* **79**, 2779 (2001). Figure 4 indicates  $H_{c2} = 60 \text{ kG}$  at 10 K. We assume this value for  $\eta H_{c2}$  in the analyses of the Kogan model.

<sup>6</sup>J. Kortus, I.I. Mazin, K.D. Belashchenko, V.P. Antropov, and L.L.

- Boyer, Phys. Rev. Lett. **86**, 4656 (2001).
- <sup>7</sup>H.J. Choi, D. Roundy, H. Sun, M.L. Cohen, and S.G. Louie, cond-mat/0111182 (unpublished).
- <sup>8</sup>T. Yildirim, O. Gülseren, J.W. Lynn, C.M. Brown, T.J. Udovic, Q. Huang, N. Rogado, K.A. Regan, M.A. Hayward, J.S. Slusky, T. He, M.K. Haas, P. Khalifah, K. Inumaru, and R.J. Cava, Phys. Rev. Lett. **87**, 037001 (2001).
- <sup>9</sup>S.L. Bud'ko, G. Lapertot, C. Petrovic, C.E. Cunningham, N. Anderson, and P.C. Canfield, Phys. Rev. Lett. **86**, 1877 (2001).
- <sup>10</sup>D.E. Farrell, J.P. Rice, D.M. Ginsberg, and J.Z. Liu, Phys. Rev. Lett. **64**, 1573 (1990).
- <sup>11</sup>T. Ishida, K. Okuda, H. Asaoka, Y. Kazumata, K. Noda, and H. Takei, Phys. Rev. B **56**, 11 897 (1997).
- <sup>12</sup>M. Tachiki and S. Takahashi, Solid State Commun. **70**, 291 (1989); **72**, 1083 (1989).
- <sup>13</sup>T. Ishida, K. Kitamura, K. Okuda, and H. Asaoka, J. Phys. Soc. Jpn. **70**, 2110 (2001).
- <sup>14</sup>V.G. Kogan, Phys. Rev. B **38**, 7049 (1988).
- <sup>15</sup>T. Ishida, K. Okuda, A.I. Rykov, S. Tajima, and I. Terasaki, Phys. Rev. B **58**, 5222 (1998).
- <sup>16</sup>M. Angst, R. Puzniak, A. Wisniwski, J. Jun, S.M. Kazakov, J. Karpinski, J. Roos, and H. Keller, Phys. Rev. Lett. **88**, 167004 (2002)