Physica C 469 (2009) 1055–1058

Contents lists available at ScienceDirect

Physica C

journal homepage: www.elsevier.com/locate/physc

Observation of magnetization step at order–disorder transition in MgB_2 single crystals

T. Nojima [a,*], K. Takahashi [a], M. Chotoku [a], A. Ochiai [b], H. Aoki [b], H.-G. Lee [c], S.-I. Lee [c]

[a] Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan
[b] Department of Physics, Graduate School of Science, Tohoku University, Sendai 980-8578, Japan
[c] Department of Physics, Sogang University, Seoul 121-712, Republic of Korea

ARTICLE INFO

Article history:
Available online 30 May 2009

PACS:
74.25.Dw
74.25.Ha
74.25.Op
74.25.Qt
74.70.Ad

Keywords:
Vortex matter physics
Order–disorder transition
Single crystal
MgB_2

ABSTRACT

We have studied the order–disorder transition in high quality MgB_2 single crystals, using a torque magnetometry combined with a 'vortex shaking' technique. In the wide range of temperature T, field H and the H direction, we succeed in obtaining reversible magnetization curves $M_{rev}(T, H)$ by shaking the pinned vortices. Especially at low temperatures below 25 K and high fields, where the irreversible magnetization curve exhibits the peak effect due to the order–disorder transition, it is found that the peak is transformed into the clear step in $M_{rev}(H)$. Similar step-like behavior is also observed in the temperature dependence of magnetization $M_{rev}(T)$. These results give direct evidence that the order–disorder transition, which is hidden by the large hysteresis of magnetization, has the nature of first-order transition.

1. Introduction

Soon after the discovery of cuprate superconductors, it has been established that there is a rich vortex matter phase diagram in the magnetic field vs temperature (H–T) plane of these compounds [1]. Recently the interests in this subject have extended to various type-II superconductors, containing low-T_c conventional superconductors, and the H–T phase diagrams of them have been re-examined carefully [2–5]. In addition to its famous nature of the two-gap superconductivity [6–9], MgB_2 is a typical type-II superconductor with the relatively strong anisotropy $\gamma = 3 - 6$ [10] and GL parameter $\kappa = 5 - 35$ [11], which are intermediate between high-T_c cuprates and low-T_c conventional superconductors, as well as the relatively high critical temperature $T_c \sim 39$ K [12]. Since the combination of these parameters is considered to be the key to characterize the various vortex phases and the corresponding phase transitions, the examination of the H–T phase diagram in MgB_2 can be useful for a deep understanding of the vortex matter physics.

The peak effect of the magnetization hysteresis or the critical current is one of the common features observed in a wide range of superconductors. Especially, when the sample is a clean single crystal with weak pinning, the peak effect is considered to occur through a transformation of the ordered (or weakly pinned) vortex state at low fields into the disordered (or strongly pinned) state at high fields. In high-T_c cuprates such as $YBa_2Cu_3O_{7-\delta}$ and $Bi_2Sr_2CaCu_2O_8$, it has been recognized that the sharp peak effect is related to the first-order transition form a quasi-ordered Bragg glass phase into a highly disordered vortex glass phase, which results in an abrupt enhancement of vortex pinning [13,14]. In case of MgB_2, there have been some reports on the peak effect in the magnetization M or torque τ curves near the upper critical fields H_{c2} just after the discovery of the superconductivity. The possible existence of the order–disorder transition with a first-order nature has been discussed using the history effect of the magnetization curves around peak region [15,16]. However, the underlying nature of the phase transition or the disordered phase is not still clear.

In order to clarify the above question, it is necessary to measure the physical quantities in thermal equilibrium using clean samples. In this study, we have examined the reversible magnetization M_{rev} in MgB_2 single crystals using highly sensitive torque magnetometry, combined with a vortex shaking method [17], in the H–T region where the peak effect emerges. A step in M_{rev} as a function of H and

* Corresponding author. Address: Low Temperature Materials Science Lab. Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan. Tel.: +81 22 215 2167; fax: +81 22 215 2168.
E-mail address: nojima@imr.tohoku.ac.jp (T. Nojima).

0921-4534/$ - see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.physc.2009.05.165

T, which can be ascribed to the first-order transition, was observed successfully.

2. Experimental

MgB_2 single crystals were prepared by a high pressure synthesis method with 3 GPa [18]. We selected a shiny crystal with almost hexagonal shape and with the dimensions of $\sim 100 \times 100 \times 50\ \mu m^3$. This sample shows the superconducting transition temperature $T_c = 37$ K at $H = 10$ mT and the upper critical field $H_{c2}(\theta = 10°) = 3.0$ T at 4.2 K. Here, θ is the angle between the crystal c axis and H direction. The small value of H_{c2} implies that the sample is clean and suitable for the study of the phase transition of vortices.

The magnetization measurements were performed utilizing a piezo-resistive cantilever commercially developed for the AFM. In this method, which was first introduced by Ohmichi and Osada [19], the sample was glued on the sensitive cantilever and the fine strain occurring on it due to the magnetic torque $\tau = |\boldsymbol{M} \times \boldsymbol{H}|$ of the sample was detected by the piezo resistance, which was also deposited on the cantilever. In order to obtain reversible magnetization curves, a vortex shaking technique [17] was combined to the torque measurements. We set the sample with the cantilever at the center of the epoxy sample holder surrounded by the Cu coil. By applying a small AC transverse magnetic field ΔH_{AC} with amplitude of 20–40 Oe, which shakes the pinned vortex lines, in addition to the longitudinal DC field, we attempted to relax the magnetization to the thermal equilibrium state. The direction of ΔH_{AC} was always kept perpendicular to both the c axis and the DC field. In the measurements of H dependence, the seep rate of the DC field was fixed at 0.1 T/min.

3. Results and discussion

Fig. 1 shows the typical data of $M(H)$ defined as $\tau(H)/H$ without ΔH_{AC} at $\theta = 60°$. In the low temperature region below 25 K, $M(H)$ curve reveals the peak effect just below H_{c2} determined as the onset field of diamagnetism. In Fig. 1, the magnetization hysteresis curve $\Delta M(H) = M_{down}(H) - M_{up}(H)$ at 24 K is also plotted. Here, M_{up} and M_{down} are M with increasing field and decreasing field, respectively. We can clearly see that the peak in $\Delta M(H)$, which is small at 24 K, is more pronounced with decreasing T. For all the field directions measured ($\theta = 10° - 88°$), similar types of $M(H)$ curves, showing the peak effect, were observed below 25 K. As suggested in the previous reports [15,16], this peak effect can be a change in the pinning strength for the vortices caused by the order–disorder phase transition. We empirically determined the transition field H^* as the inflection point with dM/dH showing a minimum value [16]. It is also noted that the hysteresis in $M(H)$ is very small except for the peak region, which indicates the back-ground pinning force of the present sample is very weak. This feature helps us diminish the hysteresis by the vortex shaking technique and examine the intrinsic nature of phase transition, as described below.

According to the scenario of the order–disorder transition reported for the high-T_c cuprates [13,14,17], we expect a discontinuous step in the magnetization curve in thermal equilibrium state, indicating a first-order transition, around the peak effect. In order to check this, we repeated the torque measurements with shaking fields ΔH_{AC}. In Fig. 2, typical magnetization curves with $|\Delta H_{AC}| = 20$ Oe at $T = 16$ K and $\theta = 60°$ are plotted for different frequencies f of ΔH_{AC}. In this figure, $M_{ave} = (M_{up} + M_{down})/2$ at 3.9 T for each f is subtracted from the $M(H)$ data to reduce the effect of small fluctuation in the background piezo resistance with a long time scale. At high frequencies, as shown in the data at $f = 2$ kHz, a small hysteresis of magnetization curves between the field-up process and the field-down process remains. The hysteresis became smaller with increasing $|\Delta H_{AC}|$ to 34 Oe, but it did not close completely. These results indicate that the AC transverse field, which induces the tilting motion of vortices, does not penetrate enough inside the sample due to the fast oscillation of field in our experimental condition. As well as frequency and amplitude, the effect of the shaking field depends also on the sweep rate of the DC field. It may be possible to cause a larger decrease in the magnetization hysteresis by applying DC field with a much slower sweep rate than 0.1 T/min used in this study. With decreasing frequency, the hysteresis shrinks remarkably around $f = 100$ Hz, so that we can observe the reversible magnetization curves $M_{rev}(H)$ at low f below 100 Hz. We also confirmed that the traces of $M_{rev}(H)$ curves for 100 Hz and 20 Hz did not change practically by the increase in $|\Delta H_{AC}|$ to 30 and 40 Oe, respectively.

Looking at the data in Fig. 2 carefully, we note that a small step-like behavior in $M_{rev}(H)$ emerges at almost the same characteristic field H^* with that derived from the peak effect (the vertical dotted line in Fig. 2). This small step in $M_{rev}(H)$ can be seen more clearly when we replot the field derivative of magnetization dM_{rev}/dH

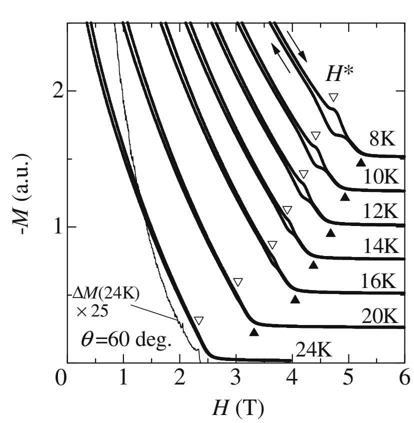

Fig. 1. Typical magnetization curves $M(H)$ defined as $\tau(H)/H$, at low temperatures below 25 K and $\theta = 60°$. Each curve is offset for clarity. Open and solid triangles indicate the positions of the order–disorder transition fields H^* and H_{c2}, respectively. Magnetization hysteresis curve $\Delta M(H)$ at $T = 24$ K (thin line) is also shown.

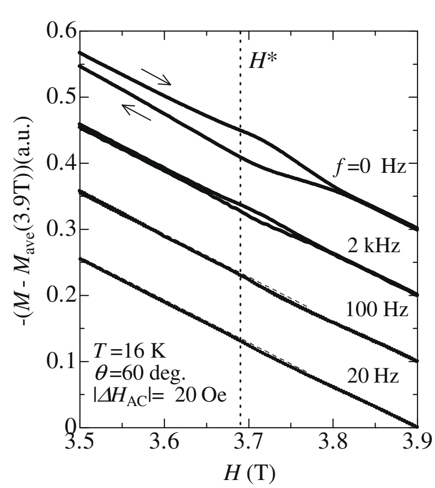

Fig. 2. Magnetization curves $M(H)$ with the shaking field ΔH_{AC} at $T = 16$ K and $\theta = 60°$ for various frequencies f of ΔH_{AC}. The amplitude of the shaking field $|\Delta H_{AC}| = 20$ Oe. For each f, the data are plotted after $M_{ave} = (M_{up} + M_{down})/2$ at 3.9 T is subtracted. Each curve is offset by 0.1 with respect to the previous one for clarity. The vertical dotted line denotes the position of H^*. The broken lines in the data for 100 Hz and 20 Hz are guides to the eye.