Observation of first-order transitions of the vortex lattice in MgB$_2$ single crystals

T Nojima$^1$, K Takahashi$^1$, M Chotoku$^1$, A Ochiai$^2$, H Aoki$^2$, H-G Lee$^3$ and S-I Lee$^3$

$^1$Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan
$^2$Department of Physics, Graduate school of Science, Tohoku University, Sendai 980-8578, Japan
$^3$Department of Physics, Sogang University, Seoul 121-742, Republic of Korea

E-mail: nojima@imr.tohoku.ac.jp

Abstract. Measurements of reversible magnetization have been performed on high quality MgB$_2$ single crystals using torque magnetometry combined with the 'vortex shaking' technique. At high temperatures above 26 K, a step like decrease in the magnetization curve is observed at $H_m$ with and without shaking. This anomaly can be ascribed to the first order vortex lattice melting transition. At low temperature below 25 K, where the irreversible magnetization curve reveals the peak effect, we succeed in suppressing the hysteresis completely by shaking and observing the clear step in the reversible magnetization curve at the field $H^*$ for the peak effect. The entropy change, estimated from the step of the reversible magnetization at $H_m$ is 2-3 times larger than that at $H^*$.

1. Introduction

In addition to high superconducting transition temperature $T_c \sim 39$ K, one of the peculiar features of MgB$_2$ is that this intermetallic compound has two superconducting gaps [1-3], $\Delta_\sigma$ ($\sim 7$ meV) and $\Delta_\pi$ ($\sim 2-3$ meV), originating from quasi-two dimensional $\sigma$ band and three dimensional $\pi$ band, respectively [4]. A number of experimental quantities, showing the unusual field $H$ and temperature $T$ dependence, are explained based on the two gaps with small mixing of carriers between them [5-7]. Now it is known that the contribution of the $\pi$ band to the superconductivity is strongly suppressed at high fields above 1 T [7], meaning that the superconductivity is governed by the $\sigma$ band at high fields.

Vortex matter physics in MgB$_2$ is another attractive subject from the aspects of both fundamental physics and application. For single crystal samples, there are some reports on the peak effect in the magnetization curves [8, 9]. From the history effect in the peak region, the possible existence of the order-disorder transition with the nature of the first order transition is discussed. On the other hand, there are few reports for the thermally induced vortex lattice melting transition [10]. This may come from its existence very close to the upper critical field $H_{c2}$, leading to difficulty in the observation. At high fields, this material behaves as a typical type II superconductor with the relatively high GL parameter $\kappa = 5$-35 [11] and anisotropy $\gamma = 3$-6 [12] due to the main contribution of the $\sigma$ band superconductivity. Since these parameters take the intermediate values between high-$T_c$ cuprate and low-$T_c$ conventional superconductors, the studies of the phase transitions in vortex system may give
useful information to the general understanding of the vortex matter physics. It is important to find direct evidences for these vortex matter phase transitions and the relation among these in MgB$_2$.

In this work, we have performed the detailed studies on both the irreversible and reversible magnetization $M$ in high quality MgB$_2$ single crystals, using torque magnetometry combined with the ‘vortex shaking’ technique. In the wide range of temperature, we succeed in observing the step in the reversible magnetization curve, which can be ascribed to the thermally induced melting transition or the order-disorder transition, depending on temperature.

2. Experimental

Single crystals of MgB$_2$ were grown by a high pressure synthesis method [13]. For the measurements, we selected the crystals with the typical size of $100 \times 100 \times 50 \, \mu m^3$, showing the superconducting transition with temperature $T_c = 37.0$ K and the upper critical field $H_c^2 = 3.0$ T (parallel to the $c$ axis) at 4.2 K. The small value of $H_c^2$ implies that the sample is very clean and suitable for the measurements of the first order phase transitions.

The magnetization curves were derived through the torque $\tau$ magnetometry with a piezo-resistive cantilever for the AFM [14], where the displacement of the cantilever due to $\tau = |M \times H|$ occurring on the sample was detected by the piezo resistor deposited on it. In order to get the reversible magnetization or torque, a small AC transverse magnetic field $\Delta H_{AC}$ with amplitude of 20-40 Oe and frequency of 20-100 Hz, which shakes the pinned vortex lines and causes the fast relaxation of magnetization, was applied in addition to the main DC longitudinal field.

3. Results and discussion

The inset of figure 1 shows the typical data of $\tau(H)$ without $\Delta H_{AC}$ at $\theta = 60^\circ$ above 26 K with $\theta$ the angle between the field direction and the $c$ axis. At high temperatures, $\tau(H)$ curves are almost reversible, in the wide range of field below $H_c^2$, even without the shaking field, implying that the pinning force in the present sample is very weak. Here, $H_c^2$ is determined as the onset of diamagnetism. As shown in the main panel of figure 1, we note that the small but extra decrease in $\tau(H)$ with positive curvature occurs at $H_m$ in the enlarged scale. This anomaly can be clearly seen when the field derivative of magnetization, $dM/dH$ calculated as $d(-\tau/H)/dH$, is plotted using the same data. The peak in $dM/dH$ at $H_m$ is indicative of a step-like change in the magnetization. We observe almost the same behavior in the data with $\Delta H_{AC}$. From the analogy to the case of high-$T_c$ cuprates, this anomaly can be ascribed to the first order vortex lattice (or Bragg glass) melting transition. As shown in figure 1, with increasing $H$, $dM/dH$ decreases rapidly to the normal state after the peak without any plateau. This suggests that in MgB$_2$ the region of vortex liquid phase is extremely narrow or overlaps with the fluctuation region of the order parameter amplitude around $H_c^2$. Similar results are observed in $\tau(H)$

![Figure 1. Field dependence of torque $\tau$ and $dM/dH$ around $H_m$ at $T = 26$ K and $\theta = 60^\circ$. The dotted line is guide to the eye. Solid and open triangles denote $H_c^2$ and $H_m$, respectively. The inset shows the typical $\tau(H)$ curves at high $T$.](image-url)
above 26 K at all the direction of $H$ except for $\theta = 90 \pm 1^\circ$ (The behavior for $H//ab$ will be shown in elsewhere).

In the inset of figure 2, the $\pi(H)$ curves without $\Delta H_{AC}$ at $\theta = 60^\circ$ below 25 K are shown. For almost all the field direction, we observe that the irreversible $\pi(H)$ starts to reveal the peak effect just below $H_{c2}$ around $T = 25$ K, and the peak becomes more pronounced with decreasing temperature. As suggested in the previous reports [8, 9], this can be the phenomenon accompanied by the order-disorder transition from the weakly pinned order phase to the strongly pinned disorder phase, which results in the sudden increase of the hysteresis in $M(H)$ or $\pi(H)$. According to this scenario, we expect discontinuous change in thermal equilibrium quantities around the peak. Indeed, it is found that the peak is transformed into the clear step at $H^*$ in the reversible $M(H)$ after the hysteresis of irreversible $M(H)$ is suppressed almost completely with the shaking field $\Delta H_{AC}$, as shown in figure 2. In figure 3, $dM/dH$ curves at various $T$ derived from the reversible $\pi(H)$ with $\Delta H_{AC}$ are plotted. The peak in $dM/dH$, corresponding to the step-like change in $M(H)$, is observed at $H^*$ in the wide range of $T$ below 25 K. We also note that $H^*$ is very close to the transition field empirically determined as the inflection point of irreversible $M(H)$ around the peak. These results clearly demonstrate that there exist a first order transition, which has the same origin with the peak effect in the irreversible $M(T)$.

We observe two types of first order phase transitions. As discussed above, it is inferred that these transitions have different underlying origins, which are thermally-driven at high temperature above 26 K and disorder-driven below 25 K. In order to check this, we calculated the entropy change $\Delta S$ at the phase transition using the Clausius-Clapeyron equation, $\Delta S = -(dH_0/dT)\Delta M$. Here, $H_0$ is $H_{c1}$ or $H^*$.

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**Figure 2.** Field dependence of -$M$ calculated as -$\pi/H$ around $H^*$ at $T = 12$ K and $\theta = 60^\circ$. The dotted line is guide to the eye. Solid and open triangles denote $H_{c2}$ and $H^*$, respectively. The inset shows the typical $\pi(H)$ curves at low $T$.

**Figure 3.** Plots of $dM/dH$ at $\theta = 60^\circ$ as a function of $H$ at low $T$. $dM/dH$ calculated from the reversible $\pi(H)$ data with the shaking field as $d(-\pi/H)/dH$. 

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depending on temperature, and $\Delta M$ is the magnetization change at the step. Since there is no region with constant $M(H)$ slope, or the $dM/dH$ plateau at high temperatures as shown in figure 1, $\Delta M$ around $H_m$ is estimated as the magnetization increase over the field width of the peak in $dM/dH$. With increasing temperature, $\Delta S$ showing the almost constant value at low temperatures, suddenly increase at 25 K and becomes 2-3 times larger, indicating the change in the nature of phase transition. The rough estimation of $\Delta S$ per vortex per c-axis length at 30 K is $0.1 k_B$ which is the same order of magnitude with the value for high-$T_c$ cuprate (Noted that getting correct $\Delta S$ is difficult in this experiment since the calibration of the cantilever and the calculation of $M$ from $\tau$ must be based on the assumption that anisotropy $\gamma$ is independent of $H$, which may not be a case of MgB$_2$). Considering the solid to solid phase transition at $H^*$, the smaller value of $\Delta S$ at low temperatures than that at high temperatures may be natural.

4. Conclusion
Using torque magnetometry combined with vortex shaking technique, vortex matter phase transition occurring in the MgB$_2$ single crystals has been examined in detail. We succeed in obtaining the direct evidence for the first order transition of vortex lattice (or Bragg glass) which changes the nature from the thermally-driven melting transition to order-disorder one with decreasing temperature. It is clearly confirmed that the later transition has the same origin with the peak effect.

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