Relationship between surface superconductivity, bulk superconductivity and the peak effect in MgB$_2$ single crystals

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Abstract. Utilizing a systematic study of transport measurements, we constructed a detailed $H$–$T$ phase diagram of an MgB$_2$ single crystal. There was no tricritical point of the surface superconductivity, the bulk superconductivity and the peak effect, in contrast to the existence of this point in Nb single crystals, as obtained from the magnetic susceptibility. We found that the surface effect was still strong up to the zero-field superconducting transition temperature and that the peak effect did not disappear on the $H_{c2}$ line because a disordered flux flow was present just below $H_{c2}$. The disappearance of the peak effect is closely related to thermal fluctuations.

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1. Introduction

The recently discovered MgB$_2$ [1] has attracted much attention for its various interesting properties, such as two-gap superconductivity [2]–[7], surface superconductivity [8]–[11] and peak effect [8]–[10], [12, 13]. Many efforts have focused on the two-gap nature, but systematic studies of a possible interplay between the surface superconductivity, the bulk superconductivity and the peak effect are relatively scarce. Recently, a detailed study of Bragg-glass (BG) melting, the peak effect, and the surface superconductivity in a Nb single crystal by using ac magnetic susceptibility and small-angle neutron scattering measurements was reported by Park et al [14]. They identified a tricritical point at which the surface superconductivity and the peak effect met at a single point on the bulk superconductivity of $H_{c2}$. The participation of surface superconductivity at the tricritical point has been a matter of conjecture and still remains an open question.

The surface superconductivity ($H_{c3}$) predicted theoretically by Saint-James and de Gennes [15] was confirmed experimentally in a Nb–Ta film by Hempstead and Kim [16]. When the magnetic field was applied parallel to the plane of the film, residual superconductivity was found above $H_{c2}$ and below $H_{c3}$. There have been several reports of surface superconductivity in bulk systems [14], [16]–[18], including MgB$_2$ [8]–[11]. The peak effect occurs due to a transition from a long-range ordered vortex phase to a short-range disordered phase [19]–[23] and has been especially observed in clean single-crystalline superconductors with weak pinning centers. When the temperature or the field is increased, the peak effect reveals itself as a sudden increase in the critical current just below the superconducting transition temperature or the upper critical field [24, 25]. This peak effect is also characterized by a sudden drop in the magnetoresistance, followed by a reentrance of superconductivity. A number of experimental results regarding the peak effect have been reported in MgB$_2$ based on the ac susceptibility [12], torque magnetometry [13] and electrical transport [8]–[10], [26, 27].

Because the unusual phenomena of surface superconductivity and the peak effect have been observed in MgB$_2$ single crystals, this material is ideal for studying the existence of tricriticality. Up to now, most efforts have concentrated either on the peak effect itself or on the surface superconductivity, but not on both of them at once. The vortex dynamics related to the peak effect in MgB$_2$ is also very interesting because thermal fluctuations in MgB$_2$ are no longer small compared to the pinning strength or the current-driven force. Because, compared to magnetic susceptibility and torque measurements, transport measurements can pick up signals from the surface, the bulk, and moving vortices much more sensitively at the same time, our main interest, the possible interplay among $H_{c3}$, $H_{c2}$ and the peak effect in MgB$_2$, can be studied quite accurately by using this method.

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In this paper, utilizing the electrical transport, we address the issues of tricriticality by mapping out a detailed $H-T$ phase diagram. We found that the surface superconductivity and the peak effect did not merge at a tricritical point and that surface superconductivity did not contribute to the peak effect.

2. Experimental details

In order to investigate the surface superconductivity as well as the peak effect in one sample, high-quality single crystals are needed because a superconducting sheath can be formed only on a clean, flat surface and because the peak effect can appear only in weakly pinned single crystals when a magnetic field is applied. The high-quality MgB$_2$ single crystals used in this study were grown under high pressure, as described earlier [5]. At 3.5 GPa, the sample was heated at a temperature of $\sim$1500 $^\circ$C for 60 min inside a 14 mm cubic multi-anvil-type press. After the heat treatment, the sample was slowly cooled to 900 $^\circ$C for 5 h, followed by a fast cool to room temperature.

Electrical transport measurements were performed on a rectangularly shaped, clean, flat MgB$_2$ single crystal, as shown in the inset of figure 1 (dimensions: 220 $\times$ 40 $\times$ 10 $\mu$m$^3$). The gold pads for the four-probe measurements were prepared on a flat surface of the sample by using photolithography, which gave a low contact resistance of around 1 $\Omega$.

During the measurements, magnetic fields were applied with various angles tilted from the sample’s $ab$-plane. From the angular dependent experiments, we observed that the surface superconductivity exists at all angles from the $ab$-plane to $c$-axis. This means that even though the field is tilted, the supercurrent flows along the side-face of the crystal, because a vertical component of the field is still alive. However, the peak effect has an angular dependence.

![Figure 1](http://www.njp.org/)
Therefore, to maximize the peak effect, we oriented the applied magnetic field 30° from the \(ab\)-plane while keeping it normal to the current flow. The field-dependent magnetoresistance was measured at different temperatures for various driving currents. A number of ac current \((f = 17\text{ Hz})\) levels between 0.5 and 7\(\text{mA}\) were used to investigate the Lorentz-force-driven flux motions.

3. Results and discussion

Typical magnetoresistance traces are shown in figure 1. Data at two different current levels, 1\(\text{mA}\) (open dots) and 7\(\text{mA}\) (closed dots), taken at various temperatures are displayed selectively. At one fixed temperature, an onset of a superconducting transition from a normal state appears at the same field with various values of the current, which was defined as the surface superconductivity \(H_{c3}\) by Hempstead and Kim [16]. The \(H_{c3}\) is denoted by vertical arrows in figure 1. After the surface superconducting transition, a current-independent sharp drop occurs in the magnetoresistance curves, which was interpreted as the bulk superconductivity \(H_{c2}\) by Hempstead and Kim [16]. The \(H_{c2}\) is indicated by tilted arrows in figure 1. The magnetization and the heat capacitance measurements carried out on our MgB\(_2\) single crystals by Welp \textit{et al} [9] and Rydh \textit{et al} [10] confirmed that the sharp drop in the magnetoresistance actually corresponds to the onset of bulk superconductivity. In addition, several results obtained from our MgB\(_2\) single crystals, even those picked from different batches, presented evidence of surface superconductivity, such as a highly non-Ohmic current dependence of the resistive transition as in [8]–[10], a ratio of \(H_{c3}/H_{c2}\) of around 1.7 as in [8]–[10], and locally enhanced superconductivity in magnetic fields aligned parallel to surfaces sculptured by using a focused ion beam as in [10].

A finite magnetoresistance occurs inside the bulk superconducting region at temperatures above 10\(\text{K}\) when a current of 7\(\text{mA}\) is applied. The finite magnetoresistance inside the superconducting state originates from the dissipation of moving vortices when the driving force is large enough. The driving force is produced from the product of the applied current and the field \((F = |J \times B|)\). Therefore, the onset of the finite magnetoresistance and its size are sensitive to the magnitude of the applied current.

At low fields, the magnetoresistance remains zero. With further increase in the applied field, the magnetoresistance turns around, moves toward zero, and meets a transition of \(H_{c2}\). At the lower fields, the vortices form a static BG phase [28], which is a quasi-long-range-ordered phase without topological defects. When the applied magnetic field is sufficient to overcome the critical driving force, the BG starts to depin and coherently flows. Therefore, the voltage response associated with the motion of the flux line lattice picks up and grows as the field increases. Doussal and Giamarchi [29, 30] interpreted that a BG phase flows along highly correlated static channels above a threshold critical force; this phase is called the moving BG phase. For a sufficiently large pinning strength, the BG flow begins to pin, so the resistance falls to zero and remains at zero until the mostly disordered vortex phase depins just below \(H_{c2}\), at which point the resistance increases steeply. The reentrance of superconductivity is a demonstration of the pinning–depinning mechanism associated with the peak effect. The onset of the peak effect, originally referring to the field or the temperature at which the critical current starts to increase, is identified as the peak in the magnetoresistance in figure 1. The strength of the peak effect, which is essentially the field interval between the positions of the peak and the dip in the magnetoresistance, progressively decreases as the temperature is increased. Eventually, the peak effect becomes faint around \(T^* = 28\text{K}\), as marked by the star in figure 1.
Figure 2. Magnetoresistance curves around 28 K taken at current levels of 1, 2, 3, 5 and 7 mA counting from the bottom. The peak effect due to pinning of the coherent motion of a BG gradually diminishes as the temperature is raised and finally vanishes near 28 K. However, the surface superconductivity persists to near $T_{c0} = 35$ K.

The results of a detailed study of the relation between $H_{c3}$ and $H_{c2}$ near $T_{c0}$, where $T_{c0}$ is the zero-field superconducting transition temperature, and the dynamics of depinned vortices near $T^*$ are shown in figure 2. The data displayed were taken at current levels of 1, 2, 3, 5 and 7 mA, counting from the bottom. Coherent BG motion requires a critical driving force, as mentioned above. Thus, the BG motion, which is manifested by a sudden rise in the magnetoresistance, should be initiated at a lower field for a higher current, as shown in detail in figure 2. In Nb, $H_{c3}$ merges to $H_{c2}$ below $T_{c0} \approx 9$ K [14]. However, in MgB$_2$, $H_{c3}$ converges to $H_{c2}$ near $T_{c0}$, which is clearly demonstrated by the dotted lines shown in figure 2. In addition, it is worth noting that even though the peak effect has vanished at $T^*$, the kink at the intersection of the two resistance slopes below $H_{c2}$, which is indicated by a horizontal arrow in figure 2, continues up to 32 K. This means that the disordered flow region just below $H_{c2}$ still exists near $T_{c0} \approx 35$ K.

All the data discussed so far can be summed into $H$–$T$ phase diagrams of moving vortices, as shown in figures 3(b)–(d). The phase diagrams show the details of progressive changes that occur when the applied current is varied from 1 to 7 mA. To explain the criteria used to build the phase diagrams, we display differently patterned areas in the magnetoresistance curve, especially for 7 mA, in figure 3(a). In figure 3(d), following the upward dashed line from the bottom, the phases separate into the BG region, the BG flow region (hatched area), the peak-effect region (dark-gray area) indicating the pinning or the reentrance of superconductivity, the disordered flow region (light-gray area), the upper critical field line ($H_{c2}$), and finally the surface superconductivity region between the $H_{c2}$ and the $H_{c3}$ lines. The phase boundary between the BG and the BG flow was determined at a fixed temperature by using the criterion of the magnetic
field at which the resistance reached 5 \( \mu \Omega \). The boundary line between the BG flow and the peak-effect regions represents the onset of the peak effect \((H_{\text{onset}})\). The phase boundary between the peak effect and the disordered flow was obtained from the magnetic field at the resistance of 5 \( \mu \Omega \) when the resistance remains zero or at the minimum point when the resistance is falling. The criteria for the \( H_{c2} \) and the \( H_{c3} \) lines were mentioned before. None of the phase boundaries depended very much on the criteria used to determine them.

The region of BG flow widens as the applied current is increased, which points to the Lorentz force as a key mechanism. It is noteworthy that \( H_{c3} \), \( H_{c2} \) and \( H_{\text{onset}} \) do not meet at a single point in the \( H-T \) phase space, which is in contrast to the previous proposal of tricriticality by Park \textit{et al} [14] based on the results for Nb. As the schematic phase diagram for Nb obtained from [14] shows in figure 4, \( H_{c3} \) and \( H_{\text{onset}} \) merge at one point along the \( H_{c2} \) line below \( T_{c0} \). Park \textit{et al} [14] used \( H_{\text{peak}} \) in the phase diagram of Nb because they obtained the peak position of the peak effect, not the onset position (\( H_{\text{onset}} \)). However, the difference between \( H_{\text{peak}} \) and \( H_{\text{onset}} \) for Nb is very small so that the tendency does not change. Therefore, we used the same notation of \( H_{\text{onset}} \) in the \( H-T \) diagram for Nb. However, for MgB\(_2\), surface superconductivity still appears above the \( H_{c2} \) line up to \( T_{c0} \) due to a stronger surface effect. The peak effect vanishes below \( T_{c0} \)

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**Figure 3.** Detailed \( H-T \) phase diagrams of moving vortices for magnetic fields 30\(^\circ\) away from the \( ab \)-plane. The phase diagrams are constructed for applied ac currents of (b) 1 mA, (c) 4 mA and (d) 7 mA. In (b)–(d), the open circle indicates the disappearance point of the peak effect, which is designated by \( T^* \). The BG flow, the peak effect, and the disordered flow regions are presented by hatched, dark and light-gray areas, respectively. In the magnetoresistance curve of (a), the criteria used to separate the abovementioned phases are displayed with patterned areas, especially for 7 mA.
Figure 4. Schematic $H$–$T$ phase diagram of the Nb single crystal inferred from the result by Park et al [14]. There is a tricritical point of the $H_{c3}$, $H_{c2}$ and $H_{\text{peak}}$ lines at a temperature below $T_c$.

and especially below the $H_{c2}$ line, not along the $H_{c2}$ line, due to the existence of disordered flow just below $H_{c2}$. This disordered flow region was not observed in the static vortex phase obtained from the magnetic susceptibility measurement [14].

The $T^*$ presented by the open circle in figures 3(b)–(d) does not depend on the magnitudes of the applied current and field, which means that the $T^*$ is not affected by the Lorentz force. It is valuable to compare the ratio of the vanishing temperature of the peak effect to the superconducting transition temperature, $T^*/T_{c0}$, in superconductors with various $T_{c0}$. $T^*/T_{c0}$ is about 1 for NbSe$_2$ ($T_c \approx 7.2$ K), 0.9 for Nb ($T_c \approx 9.2$ K) [14], 0.7–0.8 for MgB$_2$ ($T_c \approx 35–38$ K) [12, 13] and 0.4 for Bi$_2$Sr$_2$CaCu$_2$O$_8$ ($T_c \approx 90$ K) [31]. The value of $T^*/T_{c0}$ becomes smaller with increasing $T_{c0}$, that is, the peak effect becomes faint at temperatures well below $T_{c0}$ as $T_{c0}$ increases. The thermal fluctuation effect is stronger near $T_{c0}$ when $T_{c0}$ is high. In addition, according to Larkin–Ovchinnikov’s model, the peak effect becomes more pronounced in proportion to the pinning force and the elasticity of the flux line lattice [32]. The pinning strength and the elasticity are weakened by high thermal fluctuations. Therefore, the vanishing of the peak effect is strongly related to thermal fluctuations.

4. Conclusion

We performed a systematic study of magnetoresistance measurements, which are sensitive to the superconducting transition and to vortex dynamics. A detailed $H$–$T$ phase diagram of moving vortices in an MgB$_2$ single crystal indicates that surface superconductivity and the peak effect do not meet at a tricritical point on the $H_{c2}$ line, which is in contrast to the case of single-crystalline
Nb. Remarkably, a disordered flux flow was found to exist just below $H_{c2}$ so that the peak effect did not vanish on $H_{c2}$.

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