Anisotropy of the Upper Critical Field and Critical Current in Single Crystal MgB$_2$

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We report on specific heat, high magnetic field transport and ac–susceptibility measurements on magnesium diboride single crystals. The upper critical field $H_{c2}$ for magnetic fields perpendicular and parallel to the Mg and B planes is presented for the first time in the entire temperature range. A very different temperature dependence has been observed in the two directions which yields to a temperature dependent anisotropy with $\Gamma \sim 5$ at low temperatures and about 2 near $T_c$. A peak effect is observed in the susceptibility measurements for $\mu_0 H \sim 2$ T parallel to the $c$–axis and the critical current density presents a sharp maximum for $H$ parallel to the $ab$–plane.

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I. INTRODUCTION

Since the discovery of superconductivity in magnesium diboride at 39 K in January 2001, an enormous amount of work has been done which helped to elucidate many of its physical properties. Among others the multiband electronic structure proposed theoretically with the multiple superconducting energy gaps has been approved. The two main superconducting energy gaps have already been experimentally evidenced by different techniques like for instance specific heat measurements or Andreev reflection. A larger gap is attributed to two-dimensional $p_{x-y}$ orbitals and a smaller gap to three-dimensional $p_z$ bonding and antibonding orbitals. Such a picture indicates a significant anisotropy of the superconducting state. Data reported so far on the anisotropy factor $\Gamma = H_{c2||ab}/H_{c2\perp ab}$ scatter from 1.1 to 13 depending on the form of material (polycrystals, thin films, single crys-
tals) and method of evaluation. The problem remains to be clarified on high quality single crystals. The paper is organized as follows. The experimental details are given in section II. In section III, we show a consistent way of extracting the upper critical field $H_{c2}$ for magnetic fields parallel and perpendicular to the basal $ab$ planes. $H_{c2}$ has been determined in the entire temperature range by high field magnetotransport, and those values are compared to the ones deduced from ac-susceptibility below 5 T and specific heat measurements below 7 T. The perpendicular critical field reveals a conventional character with a linear temperature dependence near $T_c$ and $\mu_0 H_{c2,\perp ab}(0) \approx 3.5$ T while $H_{c2,\parallel ab}$ shows a positive curvature at temperatures above 20 K with $\mu_0 H_{c2,\parallel ab}(0) \approx 17$ T. As a consequence the anisotropy factor $\Gamma$ is temperature dependent with $\Gamma(0 \text{ K}) \approx 5$ and $\Gamma$(near $T_c$) about 2. The angular dependence of the upper critical field measured at 5.4 K and 26 K show an elliptic form as predicted by a one-band Ginzburg-Landau theory, but obviously this theory can not account for the temperature dependent $\Gamma$ parameter. Finally, the magnetic field and angular dependence of the critical current deduced from ac—susceptibility measurements is discussed in section IV.

II. SAMPLE PREPARATION AND EXPERIMENT

Experiments have been performed on high quality MgB$_2$ single crystals coming from one batch. The crystals show clear hexagonal facets with flat and shiny surfaces. The typical dimensions are 50x50x10 $\mu$m$^3$. The resistivity has been measured at 13 Hz as a function of magnetic field up to 28 T for different temperatures and angles ($\theta$) between $H$ and the $ab$-planes of the crystal. The magnetic field was always orthogonal to the measuring current. Gold electrodes have been evaporated as stripes overlapping the top plane of the sample with contact resistance of about 1 $\Omega$ in the four-probe configuration. The ac—susceptibility was deduced from the local transmittivity measured with a miniature Hall probe. The ac excitation field ($h_{ac} \sim 3G$, $\omega \sim 23$Hz) was perpendicular to the $ab$—planes and to the Hall probe plane and superimposed to a $dc$ field which was making an angle $0 < \theta < 90$ with the planes. The specific heat was measured by an $ac$ technique allowing us to measure small samples (here $\sim 100$ nano-grammes). Heat was supplied to the sample at a frequency $\omega$ of the order of 70 Hz by a light emitting diode via an optical fiber. The induced temperature oscillations were measured by a 12$\mu$m-diam chromel-constantan thermocouple that was calibrated in situ.

III. $H – T$ PHASE DIAGRAM

Fig. 1 displays the temperature dependence of the specific heat at various magnetic fields up to 7 T for $H||ab$-planes and up to 2 T for $H$ perpendicular to those planes. In zero field, the specific heat discontinuity at $T_c$, $\Delta C(T_c)$, represents only about 3 % of the total signal, i.e. 5 to 6 times less than in the best polycrystalline samples. However, our samples are extremely small and the addenda contribution are thus very large although impossible to estimate accurately. The data are therefore given in arbitrary units and the temperature range is limited to $T > 12$ K. No significant increase of the transition width, $\Delta T \sim 2$ K, is observed for $\mu_0 H|c < 2$ T and $\mu_0 H|ab < 5$ T. Despite a clear broadening above these chareristic fields, the specific heat anomaly remains well defined in the entire explored temperature range. These results imply that the broadening of the transition width observed in polycrystalline samples is due to randomly oriented anisotropic grains rather than to thermodynamic fluctuations. The curves are remarkably identical for both directions once the field is renormalized by the temperature dependent anisotropy coefficient (see inset of Fig. 1 for instance). It is also worth noticing that the amplitude of the specific heat jump $\Delta C$ decreases much too rapidly with $H$ implying a rapid increase of the Sommerfeld coefficient $\gamma$ and therefore of the density of states. An entropy conservation construction shows that $\gamma \sim 0.9 \gamma_N$ (where $\gamma_N$ is related to the normal state density of states) for $H \sim 0.5 H_{c2}$ in good agreement with recent low temperature measurements by Bouquet et al.

![FIG. 1. Temperature dependence of the specific heat at different magnetic fields for $H||ab$-planes (closed circles) and $H_{\perp ab}$ (open squares). In the inset : specific heat transition at $\mu_0 H_{\perp ab} = 1T$ and $\mu_0 H_{|| ab} = \Gamma, \mu_0 H_{|| ab} \approx 3.5$ T showing that the same curve is obtained in both direction when the field is renormalized by $\Gamma$.](image)

The critical temperature has been determined using the classical entropy conservation construction. The cor-
responding $H_c2$ values as well as the one deduced from susceptibility (onset of the diamagnetic response) and transport measurements have been reported in Fig. 2. In the latter case, the critical field $H_c2(T)$ or temperature $T_c(H)$ values have been defined at the onset of finite resistivity (see Fig. 3).

Some of us have previously shown that the onset of finite resistivity coincides with the location of the specific heat anomaly for $H \perp ab$. Here we show that the same holds for parallel fields $H \parallel ab$. Note that the resistivity reaches the normal state value at a magnetic field $H_{RN}$ higher than the as-deduced upper critical field (see Fig. 3). Below we argue that $H_{RN}$ is due to the surface effects.

![Fig. 2. $H - T$ phase diagram of the MgB$_2$ single crystal. Circles - $H_c2$ from transport measurements. Squares - $H_c2$ from ac-susceptibility. Triangles - $H_c2$ from specific heat. In the insert: temperature dependence of the anisotropy.](image)

One can see that all three presented methods reveal the same results in the common range of applied fields ($\mu_0H \leq 5$ T). The upper critical field perpendicular to the basal plane has a typical temperature dependence for a type-II superconductor with a linear shape near the zero-field transition temperature and a saturation at the lowest temperatures with $\mu_0H_{c2,ab} \approx 3.5$ T. On the other hand the parallel upper critical field has a different strength and shape: close to $T_c$, $H_{c2,ab}$ reveals a positive curvature which changes to negative below 20 K and saturates to about 17 Tesla at the lowest temperatures. A direct consequence of the different forms of the temperature dependencies of these two critical fields is a temperature dependent anisotropy factor $\Gamma$. Then, $\Gamma \sim 5$ is found at low temperatures but it is about 2 near $T_c$ (see inset of Fig. 2).

The presented data extend the ones previously obtained by some of us for $\mu_0H < 7$ T on crystals from the same batch. It has been suggested that the positive curvature observed here for $H \parallel ab$ is a consequence of the two-gap structure. However, it is worth mentioning that a very similar behavior has also been observed in conventional superconductors including NbSe$_2$ borocarbides and was believed to be a universal character of layered compounds. This is even more "general" as it has also been observed in the isotropic (K,Ba)BiO$_3$ system. The origin of this effect thus still has to be clarified.

![Fig. 3. Magnetic field dependence of the MgB$_2$ resistance at $T = 5.4$ K for various angles between the field and the $ab$-plane. The critical fields $H_c2$ and $H_c3$ are marked by the arrows for two principal field orientations. For these fields also the effect of current density is presented (dashed lines from right to left: $j = 17, 50, 170$ A/cm$^2$, solid lines $j = 500$ A/cm$^2$).](image)

Fig. 3 represents the resistive transitions from the superconducting to normal state taken at 5.4 K for different angles between $H$ and the $ab$-planes of the crystal. The measurements have been done at the current density 500 A/cm$^2$ (solid lines). The current dependence of the transition for perpendicular fields ($H \perp ab$) - which is also highly non-ohmic - has been attributed to surface conductivity in a previous paper by some of us. We have found similar, albeit a bit weaker effects for parallel fields ($H \parallel ab$) down to the lowest temperatures. In Fig. 3 for both principal field orientations $H \parallel ab$ and $H \perp ab$ an effect of the measuring current is presented. It shows that the transitions are indeed current dependent. For current densities equal to 17, 50, 170 A/cm$^2$ transitions are smooth and for $H \perp ab$ the onset of the finite resistivity is even shifted towards higher fields for decreasing current densities. For $j \geq 500$ A/cm$^2$ the onset of the transition is not changed anymore (measured up to 2000 A/cm$^2$) and shows up as an abrupt jump at $H = H_{c2}$ (for $H \parallel ab$ already at 170 A/cm$^2$) followed by a smooth transition towards the normal state resistance $R_N$. The ratio between the fields at the onset of finite resistivity at 500 A/cm$^2$ ($H_{R=0} = H_{c2}$) and the one at the end of the transition at the lowest current $H_{RN} = H_{c3}$ is about 1.8 for $H \perp ab$ and 1.4 for $H \parallel ab$. This ratio remains constant for all temperatures apart those very close to $T_c$, where it is affected by the intrinsic width of the transition $\Delta T_c$. Our measurements can be compared to the ones obtained

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in a pioneering work on surface effects in type-II superconductors by Hempstead and Kim. They performed measurements on Nb$_{0.5}$Ta$_{0.5}$ and Pb$_{0.83}$In$_{0.17}$ sheets. For fields parallel to the surface of the sheets (that is a favorable configuration for appearance of surface superconductivity) they obtained results strikingly similar to ours. For sufficiently low current densities, the onset of a finite resistance is sensitive to the excitation current since the surface sheath could bear a current keeping the zero resistance even above $H_{c2}$. However, above some threshold current, this surface superconductivity is destroyed and the onset field does not change anymore; it has thus been identified with the upper critical field $H_{c2}$. As observed in our data, for $H = H_{c2}$ the resistance of the sheets increased sharply to a fraction of $R_N$ which was bigger for higher current and then, the resistance finally went smoothly to the normal state $R_N$. $H_{c2}$ was best defined when measured at the lowest possible current density, but the experimental $H_{c2}/H_{c1}$ ratio scattered between 1.6 and 1.96 and dropped down to 1.14 for copper coated Pb-In sheets. Indeed, the configuration of our transport experiment with $H\parallel ab$ is very favorable for surface effects which must then be taken into account. On the other hand, surface superconductivity effects should be negligible for $H\perp ab$. However, in our experimental set-up the current and voltage electrodes overlapping the sample go over the vertical side planes which thus inevitably contribute to surface superconductivity as well.

$H_{c2}$ in MgB$_2$ crystals has been measured by several authors in a limited temperature range. The $H_{c2,ab}$ values of Angst et al. obtained via torque magnetometry are in a good agreement with ours while $H_{c2,ab}$ are slightly higher (implying a slightly higher anisotropy parameter). Similar $H_{c2}$ values have also been obtained by Sologubenko et al. from the magnetic field and temperature dependencies of the thermal conductivity below 6 Tesla. They also found a standard dependence for $H_{c2,ab}(T)$, and a positive curvature for $H_{c2,ab}(T)$ above 30 K. Our data are in very good agreement with those obtained by the measurements of the magnetic moment in MgB$_2$ single crystals by Zehetmayer et al. Finally, Bud’ko and Canfield have arrived to similar conclusions by extracting the superconducting anisotropy from the magnetization measurements on randomly oriented powder samples. It was however of fundamental importance to show that the ”critical fields” obtained from all those measurements coincide with the thermodynamic transition deduced from specific heat measurements. In many novel superconductors fluctuations broaden the transitions from the superconducting to the normal state in magnetic field. One of the most striking phenomenon which has been observed in these systems is the existence of a melting line $T_m(H)$ above which the vortex lattice melts into a liquid of entangled lines. The presence of this liquid phase complicates the determination of the upper critical field $H_{c2}$. For instance, the onset of a finite resistance in those systems is indicating the melting transition (at $H_{R=0}$) while the upper critical field is located at a very end of the resistive transition $R(H_{c2}) = R_N$ (see for instance). However, fluctuations are not expected to play any significant role in MgB$_2$ as confirmed by specific heat measurements: the width $\Delta C$ does not increase significantly with magnetic field and the upper critical field occurs near the onset of finite resistance at $H_{R=0}$. A non correct criterion for the $H_{c2}$ determination partly explains the discrepancy in the values deduced from transport measurements.

Next, we discuss the angular dependence of the critical field. As shown in Fig. 3, the resistivity rapidly drops towards zero at $\mu_0 H \sim 16$ T for $H \parallel ab$ and progressively shifts towards lower fields as the angle increases. The corresponding $H_{c2}(\theta)$ can be well fitted by the simple ellipsoidal form $\left(\frac{H_{c2}(\theta) \sin \theta}{H_{c2,ab}}\right)^2 + \left(\frac{H_{c2}(\theta) \cos \theta}{H_{c2,ab}}\right)^2 = 1$ of Lawrence and Doniach for anisotropic three-dimensional superconductors with $\Gamma = 4.8$ (see Fig.4). A similar dependence has been observed at 26 K (not shown) but with $\Gamma = 3.4$. By triangles we have also plotted the characteristic fields $H_{R_N}$. If surface conductivity becomes important for $H\parallel ab$ and $H\parallel \parallel ab$ ($H_{R_N}$ is then close to $H_{c2}$), this effect is diminished for other field orientations and the $H_{R_N}/H_{c2}$ ratio drops down to $\approx 1.1$. Surface conductivity effects can thus account for the cusp-like behavior of $H_{R_N}$ at small angles which has been first attributed to $H_{c2}$ by other authors.

![FIG. 4. Angular dependence of the upper critical field (open circles) at 5.4 K together with the fits by the Lawrence-Doniach formula. Triangles are "critical fields" taken at the end of the resistive transition $H_{R_N}$ showing the cusp-like behavior at small angles. In the inset: angular dependence of the critical current at $\mu_0 H = 2$ T and $T = 18$ K ($J_0 \sim 5 \times 10^4$ A/cm$^{-3}$).](image)
IV. PEAK EFFECT AND ANGULAR DEPENDENCE OF THE CRITICAL CURRENT

Another interesting phenomenon is the observation of a peak effect in the critical current. This effect shows up in our transport measurements in the same way as in... and is also visible in our susceptibility data in a small magnetic field range $\sim 2-3$ T for $H$ perpendicular to the $ab$-plane. This increase in the critical current appears as a dip in the susceptibility in both temperature and magnetic field sweeps as also reported by Pissas et al. for the same field orientation. Surprisingly, the peak effect is only visible in a narrow magnetic field range. For lower magnetic fields, the peak is replaced by a rapid drop of the susceptibility and it has been suggested by Pissas et al. that this drop could be associated with a first order transition in the vortex lattice in analogy with high $T_c$ oxides. However, in MgB$_2$, the peak effects appear close the $H_{c2}$ line and no sign for the existence of a vortex liquid could be obtained in our experiments.

![Diagram](image_url)

FIG. 5. Magnetic field dependence of the critical current at $H_{ab}$ at $T$(K) = 8.5, 13, 17, 21.5 and 26 (from top to bottom) showing a peak effect at low temperatures ($J_0 \approx 5 \times 10^5$ A/cm$^2$). In the inset : temperature dependence of the $ac$-susceptibility at $\mu_0 H = 2$ T for different angles between $H$ and the $ab$-plane.

The peak is here reflecting the fact that the shear energy of the flux lattice drops towards zero more rapidly than the pinning energy in the vicinity of the normal state allowing to the vortex matter to accomodate more efficiently to the disorder close to the transition. Moreover, as shown in Fig. 5, the value of the peak current rapidly decreases for increasing temperature (i.e. for decreasing magnetic field) but probably still exists at high temperature. Similarly, as shown in the inset of Fig. 5, the dip in the susceptibility (that is the peak in the current density) also rapidly disappears as the magnetic fields is turned away from the $c-$axis. However, the peak effect is still visible in transport measurements for $H||ab$ again suggesting that the peak has shifted towards lower $J$ values when the magnetic field was turned and thus disappeared from our experimental window. The $J$ values presented in Fig.5 have been deduced from the magnetic field dependence of the $ac$-susceptibility. Indeed, in the non linear regime, the $ac$ response is related to the current density $J$ through : $\chi(h_{ac}, \omega, T) = F(h_{ac}/J(\omega, T))$ where $F(x)$ depends on the pinning mechanism, the sample geometry and the flux dynamics. $F$ has been determined experimentally following the procedure introduced by Pasquini et al. and is in very good agreement with the numerical calculations performed by Brandt for bulk pinning in cylinders (with the appropriate thickness/radius ratio). Note that our critical current values are much smaller than the one previously obtained in polycrystal but, as shown below, $J$ sharply increases when the field is applied parallel to the $ab-$ planes.

As shown in the inset of Fig.5, at low temperature, the susceptibility (and thus the critical current) only weakly depends on $\theta$ down to $\theta \sim 10$ degrees and the rapid drop of the susceptibility close to $H_{c2}$ is progressively shifted towards higher temperature for decreasing $\theta$ values. Finally close to the $ab$-plane the susceptibility rapidly decreases showing that the pinning of the vortices is much more efficient for $H||ab$. The corresponding $J/J_0$ values at 18 K are displayed in the inset of Fig. 4 showing that $J$ increases by a factor of about 4 for $H$ parallel to the $ab-$planes. A similar behavior has been observed in the critical current deduced from transport measurements in $c-$axis oriented thin films and Elltsev et al. recently reported on a rapid drop of the dissipation when the magnetic field is applied parallel to the $ab-$planes in single crystals. A similar cusp in the critical current has been observed in high $T_c$ cuprates in which the vortices are strongly pinning by the weakly superconducting layers between the CuO$_2$ planes when the field is applied parallel to those planes (so called intrinsic pinning). However, the origin of the strong pinning of the $ab-$ planes in diborides still has to be clarified given the rather small anisotropy of this material.

V. CONCLUSION

In summary, the upper critical field has been deduced from specific heat, high magnetic field transport and $ac$-susceptibility measurments for $H$ parallel and perpendicular to the $ab-$planes. $H_{c2}$ perpendicular to the planes reveals a conventional temperature dependence with $\mu_0 H_{c2||ab}(0) \approx 3.5$ Tesla but the parallel critical field with $\mu_0 H_{c2||ab}(0) \approx 17$ Tesla has a positive curvature at temperatures above 20 K. Consequently, the
anisotropy factor \( \Gamma = \frac{H_{c2}|_{ab}}{H_{c2}^||_{ab}} \) is temperature dependent decreasing from \( \sim 5 \) at low temperature towards \( \sim 2 \) close to \( T_c \). The critical current deduced from the susceptibility measurements present a sharp peak effect for \( \mu_0 H \perp ab \sim 2 − 3 \) T. At low temperatures, \( J \) only depends weakly on the angle between \( H \) and the \( ab \)−plane for \( \theta > 10 \) degrees but rapidly increases for \( H \perp ab \).

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