High-field superconductivity in alloyed MgB$_2$ thin films

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We investigated the effect of alloying on the upper critical field $H_{c2}$ in 12 MgB$_2$ films, in which disorder was introduced by growth, carbon doping or He-ion irradiation, finding a significant $H_{c2}$ enhancement in C-alloyed films, and an anomalous upward curvature of $H_{c2}(T)$. Record high values of $H_{c2} \perp (4.2) \approx 35$T and $H_{c2} \parallel (4.2) \approx 51$T were observed perpendicular and parallel to the $ab$ plane, respectively. The temperature dependence of $H_{c2}(T)$ is described well by a theory of dirty two-gap superconductivity. Extrapolation of the experimental data to T=0 suggests that $H_{c2} \parallel (0)$ approaches the paramagnetic limit of $\sim 70$T.
Discovery of superconductivity in MgB$_2$ with the critical temperature $T_c = 39\text{K}$ renewed intense interest in the novel effects in two-gap superconductors. \textit{Ab initio} calculations\textsuperscript{1,2} showed that MgB$_2$ has two weakly coupled gaps $\Delta_{\sigma}(0) \approx 7.2\text{meV}$ and $\Delta_{\pi}(0) \approx 2.3\text{meV}$ residing on disconnected sheets of the Fermi surface formed by in-plane $p_{\text{xy}}$ boron orbitals ($\sigma$ band) and out-of-plane $p_z$ boron orbitals ($\pi$ band). The two-gap Eliashberg theory\textsuperscript{2,3} has explained many anomalies in tunneling, heat capacity and electrodynamics of clean MgB$_2$ single crystals.\textsuperscript{4} However, the physics of two-gap MgB$_2$ alloys determined by the multiple impurity scattering channels, and by the complex substitutional chemistry of MgB$_2$\textsuperscript{5} are still poorly understood. The behavior of disordered MgB$_2$ is particularly interesting because it exhibits enormous enhancement of $H_{c2}$ by nonmagnetic impurities,\textsuperscript{6-8} well above the estimate $H_{c2}(0)=0.69T_c H_{c2}'(T_c)$ of one-gap theory\textsuperscript{9}, and anomalous temperature-dependent $H_{c2}$ anisotropy.\textsuperscript{4} Some of these features have been recently explained by generalized two-gap Usadel equations\textsuperscript{10,11} in which impurity scattering is accounted for by the intraband electron diffusivities $D_{\sigma}$ and $D_{\pi}$, and interband scattering rates $\Gamma_{\sigma\pi}$ and $\Gamma_{\pi\sigma}$. In this Letter we present high-field transport measurements of $H_{c2}(T)$ for 12 MgB$_2$ films made by 6 different groups using very different ways of introducing disorder. We show that $H_{c2}$ is radically increased in dirty films, and $H_{c2 \parallel}(0)$ extrapolates to $H_p \approx 70\text{T}$ for a C-alloyed film, comparable to the paramagnetic limit ($H_p=1.84T_c = 64\text{T}$ for $T_c = 35\text{K}$).

Our films were made by different deposition techniques including pulsed laser deposition (PLD),\textsuperscript{12,13} molecular beam epitaxy (MBE),\textsuperscript{14} hybrid physical-chemical vapor deposition (HPCVD),\textsuperscript{15} sputtering,\textsuperscript{16,17}, and reactive evaporation.\textsuperscript{18} Growth was performed by \textit{in-situ} techniques\textsuperscript{14,15,17,18}, as well as \textit{ex-situ} methods with post-annealing in Mg vapor\textsuperscript{12,13}. C-doped films were produced by HPCVD\textsuperscript{15} with the addition of 75 sccm of ($\text{C}_5\text{H}_5$)$_2\text{Mg}$ to the $\text{H}_2$ carrier gas. Some films were damaged with $10^{16}$ cm$^{-2}$, 2 MeV $\alpha$-particles to controllably alter the scattering by irradiation point defects.\textsuperscript{19} Elemental compositions were determined by wavelength dispersive spectroscopy (WDS) and Rutherford Backscattering Spectroscopy (RBS), and film orientation and lattice parameters with a four-circle X-ray diffractometer. Film parameters are summarized in Table I. In some samples RBS detected through-thickness composition variations, likely due to surface reactions

Measurements of $H_{c2}(T)$ on samples A, B, E, F, H, I, L were performed in a 33T resistive magnet at the NHMFL in Tallahassee. Film resistances $R(H)$ were measured in parallel and perpendicular fields at a sweep
rate of 1T/min while temperature was stabilized to ~10mK. The measuring current density J was varied between 10 and 100A/cm². Detailed study of film A did not show any significant change in R(H) for 4 < J < 4000 A/cm². Samples G, M and N were measured in the 300 msec 60 T pulsed facility at the LNCMP in Toulouse, at a lock-in frequency of 40 KHz and J varying from 50 to 200 A/cm² with no change in R(H). In all cases $H_{c2}$ was defined as $R(H_{c2}) = 0.9R(T_c)$.

Figure 1 shows $H_{c2}^\perp(T)$ (a) and $H_{c2}^\parallel(T)$ (b) for the lower $H_{c2}$ samples A, B, C, E, H, I, L, M, N, for which a wide variety of properties can be developed. R(H) curves for film A are shown in inset. The $H_{c2}^\perp(T)$ data in Fig. 1a fall into two groups, one having $T_c \approx 32-37$K, with relatively low $dH_{c2}/dT$ and $H_{c2}(0) \approx 10.5-15$T, while the lower $T_c$ group (24–32K) has $\approx 50\%$ larger $dH_{c2}/dT$ and $H_{c2}(0)\approx 17–22$T. $H_{c2}(0)\parallel$ data in Fig. 1b range more continuously from 18-40T, with only samples B and L standing out. Film B, with the lowest $T_c \approx 24$K and $H_{c2}(0)$ with $\rho_n \approx 85\mu\Omega\text{cm}$, has almost identical $H_{c2}^\parallel(T)$ and $H_{c2}^\perp(T)$, while non-textured sample L with $\rho_n \approx 9.9\mu\Omega\text{cm}$ also has a low $H_{c2}(0)\approx 22$T in spite of its higher $T_c = 39.4$K. Film E, with highest $T_c = 41.5$ K and $\rho_n \approx 0.4\mu\Omega\text{cm}$ as made (1.6$\mu\Omega\text{cm}$ when measured at the NHMFL) represents MgB² in the clean limit, making it unsuitable for fitting using dirty-limit theory. Although film E has the lowest $\rho_n$, it exhibits the highest $H_{c2}^\parallel(T)$, even though Fig. 1b also includes films with $\rho_n>500\mu\Omega\text{cm}$ but with lower $H_{c2}^\parallel$ (film I). Thus, there is no simple correlation between $\rho_n$ and $H_{c2}$, because the global resistivity may be limited by poor intergrain connectivity while $H_{c2}$ is controlled by intragrain impurity scattering. The anisotropy parameter $\gamma(T) = H_{c2}^\parallel/H_{c2}^\perp$ ranges from $\approx 3$ for the lowest $\rho_n$ film (E, $T_c = 41.5$K) to $\approx 1$ for the lowest $T_c$ textured film B ($T_c = 24$K). For most films, $\gamma(T)$ tends to decrease as $T$ decreases, consistent with the behavior predicted for two-gap MgB² with dirtier $\pi$ band.

Figure 2 shows $H_{c2}^\perp(T)$ and $H_{c2}^\parallel(T)$ curves for the highest $H_{c2}$ films D, F and G, while the insets show the parallel-field R(H) traces. By increasing the nominal carbon content in the HPCVD films, resistivity rises from $\sim 1.6$ (E) to 564 (F) and 250 $\mu\Omega\text{cm}$ (G), while $T_c$ only decreases to 35K. However, $H_{c2}^\parallel(0)$ increases from 12T (E) to 28T (G) and $\approx 40$T (F). Furthermore, $H_{c2}^\parallel(0)$ rises from $\approx 35$T (E) to 51T (G) and more than 70T in sample F, while the anisotropy parameter $\gamma(T) = H_{c2}^\parallel(T)/H_{c2}^\perp(T)$ decreases as $\rho_n$ increases. Figure 2c presents $H_{c2}(T)$ for sample D, which has high nominal O (17at.%) and C (14at.%) content. Unlike the two in situ films made by HPCVD, film D was made ex situ by PLD. This film has $H_{c2}^\parallel(0)\approx 33$T and $H_{c2}^\parallel(0)\approx 48$T.
The $H_{c2}(T)$ curves in Fig. 2 have an upward curvature inconsistent with the dirty limit one-band theory. For two-gap pairing, intraband scattering does not affect $T_c$, but $T_c$ decreases as the pair-breaking interband scattering parameter $g = (\Gamma_{\sigma\sigma} + \Gamma_{\pi\pi})/2\pi k_B T_{c0}$ increases, where $T_{c0} = T_c(g=0)$. Due to the orthogonality of the $\sigma$ and $\pi$ orbitals in MgB$_2$, $g$ is usually small, and $T_c$ does not change much, even if $\rho_n$ is significantly increased. The insensitivity of $T_c$ to scattering makes it possible to increase $H_{c2}$ in MgB$_2$ to a much greater extent than in one-gap superconductors by optimizing the diffusivity ratio $D_{\pi}/D_{\sigma}$. The equation for $H_{c2}$ and $T_c$ in a dirty two-gap superconductor has the form

$$2w(\ln t + U_+) + (\lambda_0 + \lambda_i)(\ln t + U_+) + (\lambda_0 - \lambda_i)(\ln t + U_-) = 0,$$

$$\psi\left(\frac{1}{2} + \frac{g}{t_c}\right) - \psi\left(\frac{1}{2}\right) = -\frac{2 \ln t_c}{2w \ln t_c} \left(\lambda_0 + \lambda_i\right),$$

where $t = T/T_{c0}$, $t_c = T_c/T_{c0}$, $T_{c0} = 1.13 T_\psi 10^{-12} s$, $w = \lambda_{\sigma\sigma} \lambda_{\pi\pi} - \lambda_{\sigma\pi} \lambda_{\pi\sigma}$, $\lambda_0 = (\lambda_+ + 4 \lambda_{\sigma\pi} \lambda_{\pi\sigma})^{1/2}$, $\lambda_i = \lambda_{\sigma\pi} + \lambda_{\pi\sigma}$, $\lambda_{\sigma\sigma}$ is $2\times2$ matrix of BCS coupling constants, $\lambda_{\sigma\pi}$ and $\lambda_{\pi\sigma}$ describing intraband pairing, and $\lambda_{\sigma\pi}$ and $\lambda_{\pi\sigma}$ describing interband pairing. Here $\lambda_+ = ((\omega + \Gamma_+) - 2\omega_{\sigma\pi} \Gamma_{\sigma\pi} - 2\lambda_{\sigma\pi} \Gamma_{\pi\sigma})/\Omega_0$, $\Gamma_\pm = \Gamma_{\sigma\sigma} \pm \Gamma_{\pi\pi}$, $\omega_{\pi} = (D_{\pi} \pm D_{\sigma}) \pi H/\phi_0$, $\Omega_0 = (\omega_+^2 + 2\Gamma_+ \omega_+)^{1/2}$, $U_+(H,T) = \psi(1/2 + \hbar \omega_+ / 2\pi k_B T) - \psi(1/2)$, $\Omega_\pm = \omega_+ \pm \Omega_0$, where $\psi(x)$ is the digamma function, and $\phi_0$ is the flux quantum. For $H||ab$, the diffusivities in Eq. (1) should be replaced according to: $D \rightarrow [D^{(ab)} D^{(c)}]^{1/2}$.

The evolution of $H_{c2}(T)$ and $T_c$ with $g$ is shown in Fig. 3. For dirty $\pi$ band ($D_{\pi} << D_{\sigma}$) and $g = 0$, $H_{c2}(T)$ has an upward curvature, because $dH_{c2}/dT$ at $T_c$ is determined by the larger $D_{\sigma}$, while $H_{c2}(0)$ is determined by the smaller $D_{\sigma}$. As $g$ increases, the upward curvature of $H_{c2}(T)$ diminishes, and $T_c$ decreases. In the limit $g >> 1$ of complete interband mixing, $T_c(g)$ approaches $T_{c0} = T_{c0} \exp[-(\lambda_0 + \lambda_i)/2w]$, while $H_{c2}(T)$ for fixed $D_{\pi}$ and $D_{\sigma}$ evolves toward the one-gap $H_{c2}(T)$ curve. The case of a dirtier $\sigma$ band, $D_{\pi} >> D_{\sigma}$ corresponds to a one-gap-like $H_{c2}(T)$ curve broadened near $T_c$ by the band mixing.

We used Eqs. (1)-(2) to describe the observed $H_{c2}(T)$, taking $ab$-initio values $^3 \lambda_{\sigma\pi} = 0.81$, $\lambda_{\pi\pi} = -0.28$, $\lambda_{\sigma\sigma} = 0.115$ and $\lambda_{\pi\pi} = 0.09$ as input parameters. First, $g$ was calculated from Eq. (2) with the observed $T_c$ and $T_{c0} = 39K$. Next, we calculated $D_{\sigma}$ from the observed (or extrapolated) $H_{c2}(0)$, leaving the ratio $D_{\pi}/D_{\sigma}$ as the
only fit parameter determining the shape of $H_{c2}(T)$. This procedure is based on a conventional assumption of the dirty limit theory that impurities only change the scattering rates, but do not affect the coupling constants $\lambda_{mn}$, or the electron density of states. The fits describe well the observed $H_{c2}(T)$ curves in Fig. 2, indicating that $\pi$ scattering is stronger than $\sigma$ scattering in all our high-$H_{c2}$ films. The extrapolated $H_{c2}(0) \approx 55T$ for film G and $>70T$ for film F.  

Our $H_{c2}(T)$ data are striking because the highest $H_{c2}$ values are attained for films with weak $T_c$ suppressions, and the three highest-$H_{c2}$ films (48 $< H_{c2}(0) < 70T$) greatly exceed the $H_{c2}(0)$ values reported for C-doped MgB$_2$ single crystals ($\sim$35T) \cite{22,23} and C-doped filaments (32T). \cite{24} The fits to $H_{c2}(T)$ for films D, F, and G, which all have significant C content, indicate much stronger $\pi$ than $\sigma$ scattering.

We find that the very broad range of $\rho_n$ ($\sim$1-600$\mu\Omega$cm) does not directly manifest itself in the atomic-scale scattering that actually determines $H_{c2}$, because current-blocking extended defects control the measured resistivity. TEM on a C-doped HPCVD film found significant amorphous C-rich phase and scattered MgC$_2$ precipitates surround many Mg(B$_{1-x}$C$_x$)$_2$ grains, thus showing that much less than the total C content from Table I is dissolved in MgB$_2$. Because $T_c$ is depressed to zero at $x \sim 0.15$ ($\approx$10 at%) and $\rho_n \geq 50\mu\Omega$cm in Mg(B$_{1-x}$C$_x$)$_2$ single crystals \cite{22,23} and filaments \cite{24}, this would indicate that films with $T_c$ of 33-35K have $x \sim$0.03-0.05 within the MgB$_2$ grains. This heterogeneous microstructure results in $T_c$ inhomogeneities, which manifest themselves in the resistive transition broadening in Figs. 2.

To understand the scattering mechanisms better, we observe that, similar to C-alloyed MgB$_2$ single crystals, \cite{28} our high-$H_{c2}$ films F and G have smaller a-axis lattice spacing than the clean limit value of 0.3085nm. However, the c-axis lattice spacing in our high-$H_{c2}$ films is larger than the value of 0.3524nm measured for bulk pure and C-alloyed single crystals and filaments. \cite{24,28} TEM study of film D showed buckling of the $ab$ planes (perhaps due to strains induced by as-grown nanophase precipitates), naturally causing the c-axis lattice expansion. Furthermore, lattice buckling results in strong $\pi$ scattering due to disturbance of the $p_z$ $\pi$ orbitals, and thus dirtier $\pi$ band ($D_\pi << D_\sigma$) necessary to account for the upward curvature of $H_{c2}(T)$ in Figs. 2b and 2c. This scenario may also explain how C (which normally substitutes for B) can result both in the strong in-plane $\sigma$ band scattering and out-of-plane $\pi$ scattering required for the observed $H_{c2}$ enhancement.
In conclusion, we have performed extensive studies of the effect of disorder on $H_{c2}$ of MgB$_2$ and report record high $H_{c2}$ values, which may approach the paramagnetic limit for C-doped films. The upward curvature of $H_{c2}(T)$ and weak $T_c$ suppression are described well by the two-gap theory.

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26. Given the limited field range in our experiments we used Eqs. (1) and (2) without paramagnetic terms to fit the data. Analysis of paramagnetic effects will be given elsewhere.

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Figure captions

Figure 1:
$H_{c2}^\perp(T)$ (a) and $H_{c2}^\parallel(T)$ (b) for samples A, B, C, E, H, I, L, M, and N. The lines are guides for the eye. Insets show $R(H)$ for sample A for: $T = 2.1, 4.2, 8, 10, 15, 20, 22, 25, 30$ K (a), and $T = 2.47, 3.34, 4.2, 6.8, 10, 12, 15, 18.2, 20, 22, 25, 28, 32$ K (b). The $R(H_{c2}) = 0.9R_{n}$ criterion used to determine $H_{c2}$ is shown with a dashed line.

Figure 2:
$H_{c2}^\parallel(T)$ (triangles) and $H_{c2}^\perp(T)$ (squares) for films G (a), F (b) and D (c). Insets show the raw $R(H)$ traces for $H^\parallel ab$. Solid curves are calculated from Eqs. (1) and (2) with fit parameters given in Table I.

Figure 3:
$H_{c2}(T)$ curves calculated from Eq. (1) for $D_n = 0.03D_n$ and $g = 0.01, 0.05, 0.2, 1, 10$ (from top to bottom). Inset shows $T_{c}(g)$ calculated from Eq. (2) with $\lambda_{mn}$ taken from Ref. 3.
| Samples                  | Substrate       | $T_c$ (K) | $\rho_n$(40K) ($\mu\Omega$cm) | $H_{c2}^\parallel$ (T) | $H_{c2}^\perp$ (T) | $g$ | $D_x/D_\sigma$ | $c$ (Å) | $a$ (Å) | Mg at% | B at% | C at% | O at% |
|-------------------------|-----------------|-----------|-------------------------------|------------------------|-------------------|----|----------------|--------|--------|-------|------|------|------|
| A epitaxial             | (0001)Al$_2$O$_3$ | 35        | 9(4)                          | 13.5                   | 33                | 0.045 | 0.12           | 3.516  | 3.047  | 29    | 53   | 10   | 8    |
| B fiber-textured        | (0001)Al$_2$O$_3$ | 23.7      | 86(56)                        | 17                     | 17                | 0.5   | <<1            | -      | -      | 28    | 57   | 7    | 8    |
| C epitaxial*            | (0001)Al$_2$O$_3$ | 34        | 7                             | 20.5                   | 30                | 0.06  | <<1            | 3.52   | 3.08   | -     | -    | -    | -    |
| D fiber-textured*       | (111)SrTiO$_3$  | 31        | 220                           | 33                     | 48                | 0.075 | <<1            | 3.547  | -      | 37    | 32   | 14   | 17   |
| E epitaxial             | SiC             | 41.5      | 1.6(0.4)                      | 12                     | 34.5              | -     | -              | 3.511  | 3.107  | 30    | 57   | 2    | 11   |
| F fiber-textured        | SiC             | 35        | 564                           | >74                    | 0.045             | <<1   | 3.542          | 3.055  | 26     | 46    | 21   | 6    |      |
| G fiber-textured        | SiC             | 35        | 250                           | 28.2                   | 55.5              | 0.045 | 0.065          | 3.536  | 3.057  | 25    | 42   | 26   | 6    |
| H epitaxial*            | SiC             | 38        | 10.5                          | 10.5                   | 30                | 0.025 | 0.06           | 3.519  | 3.107  | 31    | 63   | 4    | 1    |
| I untextured            | (0001)Al$_2$O$_3$ | 32        | 567(290)                      | 21.7                   | 26.8              | 0.09  | 0.08           | -      | -      | -     | -    | -    | -    |
| L no 00l textured       | r-cut Al$_2$O$_3$ | 39.4      | 9.9(2.8)                      | 10.8                   | 21.4              | 0.025 | 0.07           | -      | -      | 32    | 65   | 1    | 1    |
| M epitaxial             | (111)MgO        | 33.5      | 47                            | 14.6                   | 38.1              | 0.095 | 0.1            | 3.533  | 3.036  | 24    | 41   | 28   | 6    |
| N untextured*           | (001)MgO        | 28.6      | 400                           | 15.8                   | 24.3              | 0.155 | <<1            | -      | -      | 33    | 53   | 5    | 9    |

Sample list with substrate, texture and lattice parameters derived from XRD and chemical compositions deduced from WDS. Impurities detected in amounts less than 1 at.% are not listed. $\rho_n$(40K) was obtained during $H_{c2}$ measurements (as-grown values are given in parentheses). $H_{c2}^\parallel$ and $H_{c2}^\perp$ values were extrapolated to 0K, and $g$ and $D_x/D_\sigma$ were deduced from the fit of $H_{c2}(T)$ curves for all films. ($D_x/D_\sigma << 1$ means that the data point scatter does not allow us to distinguish between finite and zero $D_x/D_\sigma$, so the fit was performed for $D_x = 0$).
Figure 1 a

![Graph showing the relationship between magnetic field (T), temperature (K), and voltage (µV).](image-url)
Figure 1 b
Figure 2 b
Figure 2 c
Figure 3

The graph shows the variation of $H_{c2}(T,g)/H_{c2}(0,0.03)$ with temperature $T$ in Kelvin. The inset in the top right corner highlights the temperature $T_g$ as a function of $g$. The graph displays different colored lines representing various values of the parameter $g$. The x-axis represents temperature $T$ in Kelvin, ranging from 0 to 40, and the y-axis represents the normalized magnetic field $H_{c2}(T,g)/H_{c2}(0,0.03)$.