Flux pinning and improved critical current density in superconducting boron doped diamond films

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Abstract
The ability to carry transport current in a magnetic field is the most important aspect of a superconductor. We present a detailed analysis of the upper critical field ($H_c(0)$) and vortex dynamics in superconducting boron doped diamond (BDD) films. $H_c(0)$ measured on the samples of different doping levels revealed a high critical field of up to 7.3 T. Pinning potential $U_0$, estimated using thermally activated flux-flow (TAFF) model shows that $U_0$ is of the order of $10^7$ K. Self-field critical current density ($J_c$) estimated for the superconducting BDD films showed large $J_c \sim 10^7$ A/cm$^2$ due to enhanced flux trapping.

1. Introduction

Owing to some extraordinary physical properties such as high hardness (100 GPa) and high thermal conductivity (2000 Wm$^{-1}$K$^{-1}$), diamond has the potential for a wide range of applications. The possibility of growing diamond and doped diamond in thin film form further enhances its area of applications [1–3]. Of all the features of diamond, properties exhibited as a result of bandgap engineering by virtue of impurity doping are the most significant. This aspect has a particular interest in the field of superconductivity.

Superconductivity in high pressure high temperature (HPHT) grown boron doped diamond (BDD) was first reported by Ekimov et al [4] and then shortly after, superconductivity in chemical vapour deposited (CVD) BDD films was demonstrated by Tokano et al [5, 6]. Discovery of superconductivity in diamond was immediately followed by theoretical works from various groups stressing on the study of the mechanism of superconductivity in BDD. Ab-initio calculations [7, 8] have revealed that beyond the critical concentration $n_c$, an insulator to metal transition (IMT) sets in and the impurity band merges with the valence band driving the Fermi level into the valence band. Thus, it was pointed out that superconductivity in BDD is due to a phonon mediated pairing mechanism. An alternative theory proposed by Baskaran suggests the existence of resonant valence bond (RVB)-type mechanism in the rigid impurity band of superconducting BDD [9, 10].

In addition to the aforementioned theoretical works, many noteworthy experimental works have been carried out. Among these works, mention can be made of the work by Bustarret et al [11], which showed the dependence of $T_c$ on the doping level in high quality single crystalline diamond and Klein et al [12] studied boron doping induced IMT where the dependence of $T_c$ on boron concentration ($n_B$) in granular BDD was demonstrated. The work by Willem et al [13] and Zhang et al [14], showed the microscopically inhomogeneous nature of superconductivity in BDD. Importance of spin ordering and disorder have been highlighted by Zhang et al, revealing ferromagnetism [15] and anomalous resistance peak [16, 17] in superconducting diamond. Furthermore, the spectroscopic studies that showed existence of hole states near the valence band maximum (VBM) using x-ray absorption spectroscopy (XAS) [18] and the angle resolved photoelectron spectroscopy (ARPES) studies of homoepitaxial BDD films by Yokoya et al which demonstrated merging of the impurity band into valence band in the metallic side of IMT [19].
Here, we present details of vortex dynamics in the superconducting BDD films. Despite extensive studies on the evolution of IMT with $n_h$ in BDD and the exhaustive research on the electrical transport properties, work on magnetic properties of BDD in the superconducting regime is scarce. Discussions on the vortex phase diagram in the superconducting regime and existence of the possible effect of spin paramagnetism on pair breaking mechanism needs immediate attention. Critical information such as vortex dynamics and effect of disorder on flux pinning is still not understood. One of the reasons for this is because of the tremendous difficulties faced by the experimentalist in synthesizing samples with high $T_c$. It is, therefore, important for one to understand the flux dynamics in highly inhomogeneous and granular samples. Unlike in the case of high $T_c$ and intermediate $T_c$ superconductors \cite{20}, the issue of intrinsic vortex pinning in BDD has not been probed.

2. Experimental details

Granular intrinsic diamond film and BDD films were deposited on Si substrate using a hot filament chemical vapour deposition (HFCVD) reactor, which is described in detail elsewhere \cite{21}. Deposition of each film was carried out for 3 h. The substrate temperature was 850 °C and the chamber pressure during the deposition was maintained at 7 Torr. The flow rates of H$_2$ and CH$_4$ during the deposition process were maintained at 3000 sccm and 80 sccm, respectively. Boron doping was achieved by inletting B(CH$_3$)$_3$ gas during the depositions. Several depositions were carried out by systematically varying the concentrations of boron in the gaseous phase viz., 7500 ppm (D1), 8750 ppm (D2) and 9500 ppm (D3).

Thickness of the deposited films were found to be ~1.5 μm, as revealed by the cross-sectional SEM images (FESEM, Quanta 3D, FEI). Temperature dependent resistivity and Hall effect measurements were performed using physical property measurement system (PPMS, Quantum Design). Electrical transport measurements were carried out in a linear four probe configuration. Hall effect measurements were performed by passing a current of 35 μA. Hall concentrations are $n_h = 1.3 \times 10^{21} \text{cm}^{-3}$ (D1), $n_h = 2.0 \times 10^{21} \text{cm}^{-3}$ (D2) and $n_h = 2.2 \times 10^{21} \text{cm}^{-3}$ (D3). Magnetic measurements were carried out using SQUID based magnetic property measurement system (MPMS, Quantum Design). In all the magnetic measurements, the field was applied perpendicular to the plane of the film.

3. Results and discussion

3.1. Upper critical field

In the following discussions, $T_c$ denotes the onset superconducting transition temperature. Figure 1 shows the resistivity versus temperature curves of the BDD films. $T_c$ of the BDD films are 5.3 K (D1), 6.8 K (D2) and 7.2 K (D3). The normal state resistivity of the BDD films show weakly semiconducting nature with the activation energy less than 10 meV.
In order to understand the mechanism of superconductivity in BDD films, it is important to study the upper critical field \( (\mu_0 H_c^2) \). It provides information on the coherence length \( \xi_{GL} \) and reflects the details of electronic interactions with the magnetic field thereby providing insight into the origin of the pairing strength and the pair breaking mechanism.

In type II superconductors, superconductivity is destroyed when magnetic field penetrates through the material in the form of vortices resulting in the reduction of its gap function [22]. As vortices start overlapping, the superconductor is driven to its normal state. Alternatively, for certain class of superconductors [23], existence of spin paramagnetic effects can lead to suppression of the upper critical field. Critical field, solely, due to paramagnetic effects in a superconductor gives rise to the paramagnetic upper critical field, \( \mu_0 H_c^2(0) = 1.86 T_c \) (in Tesla) [24, 25]. Also, according to the single band theory by Werthamer, Helfand and Hohenberg (WHH) [26], upper critical field for a weakly coupled superconductor in the dirty limit (\( \xi_{GL} > l \)) is the mean free path (i.e., \( \mu_0 H_c^2(0) = -0.692 T_c \frac{2H_c^2}{\alpha T_c} \), when Maki parameter [27] \( \alpha \) and the spin-orbit scattering constant \( \lambda_{so} \) are neglected. The relative effect of Pauli paramagnetism, an effect of the competition between the spin Zeeman energy and the superconducting condensation energy on upper critical field is expressed by the Maki parameter, \( \alpha = 1.414 \frac{H_c^2 \mu_0}{H_c^2(0)} \). Suppression of the upper critical field in a superconductor due to \( \alpha \) is given by \( H_c^2 = \left( \frac{H_c^2(0)}{\sqrt{1 + \alpha^2}} \right) \).

For the present samples, to reduce ambiguity due to the surface superconductivity and effects of vortex motion in the determination of upper critical field, we have used the 0.5\( \rho_0 \) criterion, where \( \rho_0 \) is the normal state resistivity just above \( T_c \). Superconducting parameters obtained from the resistive transitions in applied magnetic field are listed in table 1. Figure 2(a) shows dc resistivity of the BDD films as a function of temperature under applied field up to 5 T with an increment of 0.5 T. Clearly, \( \rho \) versus \( T \) broadens in the presence of applied magnetic field and \( T_c \) shifts to lower temperatures with increasing field. Field-induced broadening of superconducting transition width, remains almost constant for the samples and for the entire range of the applied field. \( \mu_0 H_c^2 \) scales with \( T_c \) which in turn is dependent on \( n_e \).

In figure 2(b), we show \( H_c^2(T) \) versus \( T \). Here, \( H_c^2(0) \) is shown by the continuous curve in olive. Evidently, WHH curves fit well to the experimental data and any further attempt to invoke spin paramagnetic effect (i.e. \( \alpha = 0 \)) leads to deviation of the fit from the experimental values as shown by the solid curves in red. Thus, by considering \( H_c^2(0) \) as the best value for upper critical field in the BDD films we have estimated Ginzburg-Landau coherence length for the films using \( \xi_{GL} = \left( \frac{\phi_0}{2\pi H_c^2(0)} \right)^{1/2} \), where \( \phi_0 \) is the flux quantum, this is listed in table 1. Our findings are in complete agreement with earlier reports in BDD, where upper critical fields were estimated using WHH theory [28], although no attempt was made to investigate spin paramagnetic effects in these reports [14, 29, 30]. Note that \( H_c^2(0) \) is larger than \( H_c^2(0) \), this further points to the fact that superconductivity in BDD is of a conventional BCS type. Spin paramagnetic effect is found to be important for superconductors with high critical fields. For instance, in A15 compounds [31] and in 11 Fe based systems [32], critical fields were found to be suppressed significantly due to spin paramagnetic effects and could be explained only when WHH models with \( \alpha = 0 \) is invoked. However, in the case of BDD, given the relatively low \( H_c^2(0) \) values, paramagnetic effects may not be important. In the above analysis, the effect of orbital pair breaking (\( \lambda_{orb} \)) is ignored as contribution from spin-orbit scattering is effective only in the case of large Z elements [33]. A conclusive test of \( \alpha \) and \( \lambda_{orb} \) effects in BDD, can be conveniently verified by similar studies at ultra-low temperatures in the milli-Kelvin temperature range.

Broadening of resistivity transitions in applied magnetic field is due to the flux motion, an indication of transition from the vortex lattice or vortex glass state into a vortex liquid state. To understand vortex dynamics in the superconducting BDD films, it is desirable to estimate pinning potential \( U_p \). In this section, we discuss thermally activated flux flow (TAFF) model first proposed by Anderson [34] and Kim [35] that became popular only after the work by Graybeal [36]. This model is employed to calculate pinning potential and to understand the role of thermal fluctuations in vortex dynamics. The necessary condition for applicability of this model is \( U_p > k_B T \). According to this model, even though thermal energy is significantly less than pinning potential energy, the flux bundles have finite probability to overcome a pinning energy barrier. Temperature and

| Sample | \( \frac{dH_c^2}{dT} \) (TK\(^{-1}\)) | \( \mu_0 H_c^2(0) \) (T) | \( \mu_0 H_c^2(0) \) (T) | \( \mu_0 H_c^2(0) \) (T) | \( \alpha \) | \( \xi_{GL} \) (nm) |
|--------|---------------------------------|-----------------|-----------------|-----------------|-------|----------------|
| D1     | 1.87                            | 6.3             | 9.0             | 4.48            | 0.98  | 7.2            |
| D2     | 1.77                            | 7.2             | 10.9            | 5.2             | 0.93  | 6.7            |
| D3     | 1.66                            | 7.3             | 11.9            | 5.38            | 0.86  | 6.7            |
magnetic field dependence of resistivity $\rho(T, H)$ can be described by, $\rho = \rho_{0f} \exp \left( -\frac{U(T, H)}{kT} \right)$, where $\rho_{0f}$ is a parameter and $U(T, H)$ is activation energy required for a vortex bundle to get depinned from a pinning centre of potential $U_0$. We assume that $\rho_{0f}$ is independent of temperature and $U = U_0 \left( 1 - \frac{T}{T_c} \right)$. The linear temperature dependence of activation energy $U$ is valid only for the case in which applied current density for the measurement is sufficiently small. Then we have, $\ln \rho = \ln \rho_0 - \frac{U_0}{T}$ and $\ln \rho = \ln \rho_0 + \frac{U_0}{T}$. We show plots of $\ln \rho$ versus $\frac{1}{T}$ in figure 3, where it can be seen that below $10^{-2} \rho_0$, $\ln \rho$ versus $\frac{1}{T}$ is linear, clearly, representing a thermally activated region. The slopes $U_0$ are represented by the solid lines and its y intercept is $\ln \rho_0$. Each solid lines representing the slopes of the thermally activated regime under various fields, intersects at $T_c$ for all the samples under investigation.

From the slopes of the experimental data, we get, $U_0 = 187$ K, 280 K, and 313 K for D1, D2 and D3, respectively, at the lowest applied field of 0.5 T. The sample with the highest $n_h$ has the largest $U_0$ and decreases with decreasing $n_h$ (or decreasing $T_c$). From the present findings, it is compelling to believe that the rigidity of the vortex lattice at low fields in BDD films is due to the inhomogeneity of boron content in the grains and due to the presence of grain boundaries.

Dependence of $U_0$ on H is expected to be inversely proportional, $U_0 \propto \frac{1}{H^{0.44}}$. In the presence of thermal fluctuations, however, field dependence of $U_0$ gets modified as $U_0 \propto H^{-\alpha}$. This phenomenon has been previously reported in various classes of superconductors [36, 38, 39]. Figure 4 depicts the field dependence of $U_0$. For applied magnetic fields less than 2.5 T, the magnetic field dependency is weak and it varies as, $U_0 \propto H^{-0.44}$ and for fields greater than 2.5 T, there is a strong magnetic field dependency with $U_0 \propto H^{-1.22}$. Such a crossover is due to changeover from a single vortex pinning regime to a regime where interactions between flux lines become significant, a result of weakening of pinning potential as the applied magnetic field is increased. Taking into consideration the field dependencies, field and temperature dependent resistivity can be written as $\rho \sim \rho_{0f} \exp \left( \frac{U_0 \left( 1 - \frac{T}{T_c} \right)}{kT} \right)$, for fields less than 2.5 T and $\rho \sim \rho_{0f} \exp \left( \frac{U_0 \left( 1 - \frac{T}{T_c} \right)}{kT H^{1.22}} \right)$ for fields greater than 2.5 T.
3.2. Magnetization and the critical current density

Figure 5 shows magnetization versus temperature curves obtained using an applied field of 10 Oe. Samples cooled below \( T_{\text{c,zero}} \) without applied field and then turning on the field during heating through \( T_{\text{c,zero}} \), results in the low temperature zero-field-cooled (ZFC) flux exclusion curves. Whereas, cooling through \( T_{\text{c,zero}} \) in the field results in the low-temperature field-cooled (FC) flux expulsion. Diamagnetic response starts to drop at 3.6 K for D1, 5.5 K for D2 and at 5.8 K for D3. Clearly, there are inconsistencies in \( T_{\text{c,zero}} \) obtained from magnetization and resistivity measurements. This is due to the fact that the superconducting network for channelling of Cooper pairs in the resistivity measurement is developed much earlier compared to the establishment of bulk

![Figure 3](image3.png)

**Figure 3.** Arrhenius plots of BDD films derived from the resistivity versus temperature curves under applied field in the superconducting region. Each solid line represents the slope in thermally activated regime in the region of \( \rho < 10^{-2} \rho_n \). The point of intersection of the slopes is \( T_c \).

![Figure 4](image4.png)

**Figure 4.** Field dependence of the pinning potential \( U_0 \) for the superconducting boron doped diamond films D1, D2 and D3.
superconductivity, as required for the measurable diamagnetic response from the films. Difference between the temperature at which the onset of superconducting diamagnetic response is observed and the irreversible temperature at which ZFC and FC curves bifurcate during the temperature cycle of the measurement is 0.5 K. Shielding fraction as evidenced from ZFC diamagnetic susceptibility is 74% and 80% for D2 and D3 respectively. Certainly, an overestimation of the superconducting volume fraction as the presence of normal precipitates in the form of non-homogeneity and grain boundaries is appreciable in granular BDD films. Since D1 has a lower $T_c$, zero, it was not possible to obtain saturation in the diamagnetic response up to 2 K.

To further understand the magnetic response of the superconducting films, field dependence of magnetization measurements were carried out. We show the isothermal magnetic hysteresis loops (MHLs) of the BDD films in figure 6(a). MHLs were obtained with a perpendicular field up to 500 Oe at 2 K, 3.5 K, and 5 K for D2 and D3, and for D1, it was obtained at 2 K. Irreversible magnetization decreases monotonously with an increase in the applied field and the MHLs are nearly symmetric about $M = 0$ axis. Critical current density, a technologically important parameter is estimated from the MHLs using critical state Bean model [40]. This model assumes that a superconductor is driven to a critical state as soon as magnetic field enters into it and $F_L = F_P$, where $F_L$ and $F_P$ are Lorentz force and pinning force, respectively. Although this model is constructed on simplistic assumptions, results obtained using this model are realistic in estimating the critical current density of superconductors. In the present calculations for critical current density of the BDD films, we have used the expression for $J_c$ as $J_c = \frac{20\Delta M}{a^2d(1-\frac{b}{a})}$, here, $a = 2.5$ mm and $b = 2$ mm are the sides of the film, $d = 1.5$ μm is the thickness of the film, $\Delta M$ is the difference of magnetization between the increasing and decreasing field and represents the degree of hysteresis [41]. As shown in figure 6(b), the measured self-field $J_c$ is of the order of $10^3$ A/cm$^2$ and it decreases monotonously with increasing field for all of the films under investigation. The significantly large self-field $J_c (= 30 \times 10^3$ A/cm$^2$) in the present samples as compared to the self-field $J_c$ ($\sim 10^4$ A/cm$^2$) of polycrystalline BDD samples [14, 42] can be attributed to (1) relatively high $T_c$s in the present samples, (2) larger grain boundary content and the smaller grain sizes (average grain size 300 nm) that can enhance effective flux pinning and (3) the orientation of the film with respect to the applied magnetic field, as the effective number of vortices are expected to be maximum for the field applied perpendicular to the plane of the film.

4. Conclusions

In conclusion, we have synthesized boron doped diamond (BDD) films by systematically varying the boron concentrations and established their bulk superconductivity by electrical transport and magnetization
measurements. Investigation of the upper critical field revealed that the effect of Pauli paramagnetism is not important for the BDD films. The film with the highest boron concentration ($n_B = 2.2 \times 10^{21} \text{cm}^{-3}$ (D3)) showed a $T_c$ of 7.2 K and the upper critical field, $H_{c2}(0) = 7.3$ T. Pinning potential of BDD films were obtained using TAFF model and was found to be of the order of $10^2$ K. We estimated the critical current using the critical state Bean model and found that the self-field $J_c \sim 10^7 \text{A/cm}^2$.

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