Metal-to-insulator transition and superconductivity in boron-doped diamond

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The experimental discovery of superconductivity in boron-doped diamond came as a major surprise to both the diamond and the superconducting materials communities. The main experimental results obtained since then on single-crystal diamond epilayers are reviewed and applied to calculations, and some open questions are identified. The critical doping of the metal-to-insulator transition (MIT) was found to coincide with that necessary for superconductivity to occur. Some of the critical exponents of the MIT were determined and superconducting diamond was found to follow a conventional type II behaviour in the dirty limit, with relatively high critical temperature values quite close to the doping-induced insulator-to-metal transition. This could indicate that on the metallic side both the electron–phonon coupling and the screening parameter depend on the boron concentration. In our view, doped diamond is a potential model system for the study of electronic phase transitions and a stimulating example for other semiconductors such as germanium and silicon.

Keywords: diamond; boron doping; superconductivity; metal–insulator transition

1. Introduction

In 2004, bridging the gap separating the superhardness and the superconducting communities, Ekimov et al. (2004a) discovered the superconducting behaviour of a diamond sample resulting from annealing graphite with B4C at 2500–2800 K under 8–9 GPa for 5 s. They also proposed a mechanism for the transformation of graphite into diamond at high pressure and high temperature (HPHT) and made a thorough characterization of the diamond polycrystal (Ekimov et al. 2004b). Similar results were reported shortly after for polycrystalline (Takano et al. 2004; Winzer et al. 2005) and (100)-oriented single-crystal (Bustarret et al. 2004) diamond films grown by microwave plasma-assisted chemical vapour deposition (MPCVD), showing that (i) ‘zero’ resistivity could be observed up to the boiling temperature of helium (4.2 K), (ii) doping-induced superconductivity appeared above approximately 6×10²⁰ B cm⁻³, and (iii) $T_c$ increased with the boron concentration.

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Other methods such as chemical transport reaction (Sidorov 2005) and hot filament-assisted (Wang 2006) chemical vapour deposition, as well as heavily boron-doped nanocrystalline diamond deposited on seeded glass substrates (Nesládek et al. 2006), were also shown to yield superconducting layers under appropriate conditions, confirming the robustness of Ekimov’s seminal observation. Up to now, $T_c$ has been found to reach the 8–10 K range in a few cases (Winzer et al. 2005; Wang 2006; Mukuda 2007), the exact values depending not only on the preparation method and conditions but also on the experimental characterization and data analysis procedures applied to the transition.

A few years before this finding, the stunning success of graphite-like MgB$_2$, with a superconducting transition temperature $T_c$ of 39 K (Nagamatsu et al. 2001), and the striking properties of many graphite intercalation compounds had shown such light atom-based layered structures to be favourable to superconductivity, as for example in CaC$_6$ where $T_c$ reaches 11.5 K (Emery et al. 2005). Shortly after Ekimov’s discovery, calculations of the band structure of B-doped diamond (Blase et al. 2004; Boeri et al. 2004; Lee & Pickett 2004; Xiang et al. 2004; Ma et al. 2005), combined with the assumption of a BCS-type pairing mechanism (Bardeen et al. 1957), suggested that superconductivity arose from the coupling ($V_c$ potential) of phonons with holes at the top of the $\sigma$-bonding (valence) bands, in a way similar to MgB$_2$. However, the three-dimensional nature of the sp$^3$-hybridized carbon network in diamond leads to a density of states at the Fermi level $g_F$ much weaker than in the quasi-two-dimensional MgB$_2$ compound where B is sp$^2$ hybridized. This results in a lower (calculated) coupling constant $\lambda = g_F V_c$ of the order of 0.4 in diamond doped with 5% holes instead of up to 1 in MgB$_2$.

The possibility of a superconducting state arising in a degenerate semiconductor or a semimetal was theoretically explored in the late 1950s and early 1960s (Pines 1958; Cohen 1964a, b). Experimentally, after many unsuccessful attempts, type II superconducting transitions were observed between 50 and 500 mK in self-doped GeTe, SnTe and reduced SrTiO$_3$. In all three cases, $H_c(T_c)$ phase diagrams were obtained, while specific heat measurements established the bulk character of the transition. Finally, tunnel spectroscopy performed on GeTe (Stiles et al. 1966) measured the superconducting gap width $2\Delta$ and showed that a $2\Delta/k_B T_c$ ratio of 4.3 could be estimated. These results were considered at the time to further validate the BCS model (Bardeen et al. 1957) for superconductivity (Cohen 1964a, b). Despite this apparent success, the ‘superconducting semiconductors’ angle had not raised much interest during several decades until Ekimov’s results (Ekimov et al. 2004a) triggered a renewed excitement, which will probably last for some time now that superconductivity has also been induced by heavy boron doping in the parent silicon crystal (Bustarret et al. 2006). We shall stress here the features specific to diamond that could lead this crystal to become a model system for this class of materials.

2. Boron incorporation at high concentrations

In diamond, contrary to nitrogen, boron is readily incorporated on substitutional bonding sites of the lattice and behaves as an electronic acceptor centre with an ionization energy of approximately 370 meV. In the $10^{20} - 10^{21}$ cm$^{-3}$ boron concentration range of interest here, polycrystalline p$^{++}$ films grown by
MPCVD on Si substrates as well as nanocrystalline layers (which have industrial applications as chemically inert electrodes for advanced electrochemical sensing or processing devices) have been found to have electrical properties similar (at room temperature) to those of their single-crystal counterparts. We shall therefore focus here mostly on single-crystal epilayers grown by MPCVD, which are generally better suited for quantitative studies.

As a result of the larger covalent radius of boron ($r_B = 0.88 \text{ Å}$) compared with that of carbon ($r_C = 0.77 \text{ Å}$), the substitutional introduction of boron into diamond leads to an expansion of the lattice parameter. This has been found to follow the linear interpolation attributed to Vegard as long as the boron content was lower than 0.2 at.% in MPCVD epilayers (Brunet et al. 1998; Bustarret et al. 2003). Above those threshold concentrations, the expansion is less pronounced (Bustarret et al. 2003; Brazhkin et al. 2006). This lower expansion rate has been attributed to (i) the contribution of free holes (Bustarret et al. 2003), (ii) the negative deformation potentials at the valence band maximum (Brunet et al. 1998), and (iii) the occurrence of substitutional boron pairs (Brazhkin et al. 2006) that are stabilized by p-type doping (Bourgeois et al. 2006). In homoepitaxial films, this expansion is severely limited in the film plane by pseudomorphic growth under compressive biaxial stress, but the boron-induced positive strain along the growth direction is easily detected in the double crystal X-ray diffraction curves (Kačmarčík et al. 2005). A typical dataset obtained at room temperature on both a (100)- and a (111)-oriented homoepitaxial film is represented by intensity contours in figure 1a,b and shows that the downshifted \{400\} and \{111\} diffraction peaks originating from the epilayer have a lineshape very similar to that of the type Ib diamond substrate. In particular, the width of the rocking curve along the vertical axis ($\omega$ scan) shows that the mosaicity of the HPHT substrate was maintained in the epilayer for both orientations, whereas the strain distribution represented by the lineshape along the horizontal axis (2$\theta$ – $\omega$ scan) became somewhat broader in the epilayer.

Finally, the possibility of non-substitutional boron incorporation at high doping levels has been investigated by photoelectron intensity angular distribution circular dichroism (Kato et al. 2006) and by nuclear magnetic resonance (NMR) on the $^{11}$B isotope (Mukuda 2007). Both studies confirmed that the main incorporation site for boron was substitutional. However, $^{11}$B NMR studies detected a second boron-bonding site of lower symmetry, most abundant in relatively thin (100)-oriented epilayers, and which was proposed to be a local B–H complex (Mukuda 2007). Hydrogen is known to be present in MPCVD diamond layers and to passivate effectively boron acceptors as well as other defects (Uzan-Saguy et al. 2001; Goss et al. 2003). Interstitial boron as well as boron-vacancy or boron-self-interstitial pairs are also mentioned in the literature, but the stability of these incorporation sites has been questioned.

3. Doping-induced metal-to-insulator transition

As shown in figure 2, when the boron concentration $n_B$ was increased from 4 to approximately $5 \times 10^{20} \text{ cm}^{-3}$, the low-temperature transport properties of (100)-oriented $p^{++}$ layers changed drastically from an insulating behaviour (i.e. the resistivity diverges when $T \to 0$) to a metallic (and even superconducting)
dependence which extrapolated to a finite normal-state resistivity value at zero temperature. Given the 10% uncertainty of secondary ion mass spectrometry (SIMS) absolute calibrations, these experimental results are in excellent agreement with the zero-temperature model calculations of the vanishing ionization energy or of the chemical potential for both the insulating and the 

Figure 1. (a) Iso-intensity contours around the \{400\} diffraction peak of a (100)-oriented heavily B-doped diamond epilayer. The horizontal axis corresponds to the $2\theta - \omega$ scanning direction while the vertical axis corresponds to the omega ($\omega$) offset angle (rocking curve). (b) Iso-intensity contours around the \{111\} diffraction peak of a miscut (111)-oriented heavily B-doped diamond epilayer. The horizontal axis corresponds to the $2\theta - \omega$ scanning direction while the vertical axis corresponds to the $\omega$ offset angle (rocking curve).

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metallic phase (Kaćmarčik et al. 2005), which yield a critical boron concentration $n_c$ of approximately $5.2\times10^{20}$ cm$^{-3}$. A generalized Drude approach, taking into account the influence of temperature but neither the hopping transport nor weak localization effects, has also been applied to this system (Klein et al. 2007) and yielded a similar critical concentration value lying between 4 and $5\times10^{20}$ cm$^{-3}$.

Following Mott, we may assume that this transition results from boron-related hydrogenic states with a Bohr radius $a_H = \epsilon a_0/m^*$, which overlap when $n_B$ reaches $n_c$ with $a_H n_c^{1/3} = 0.26$ as observed in numerous materials. In this case, taking $\epsilon = 5.7$ for diamond and $m^* = 0.74$ for the holes, the Bohr radius $a_H$ is estimated at 0.35 nm in fair agreement with values based on the acceptor excited states. The present experimental and theoretical estimates for $n_c$ agree with experimental data recently obtained on free-standing polycrystalline p$^+$ layers where $n_c < 4.5\times10^{20}$ cm$^{-3}$ (Takano et al. 2005) and $3.4\times10^{20}$ cm$^{-3} < n_c < 5.5\times10^{20}$ cm$^{-3}$ (Winzer et al. 2005). However, they are one order of magnitude lower than those measured in ion-implanted diamond (Tshepe et al. 1999) where the doping efficiency may be strongly reduced by the non-substitutional incorporation of boron discussed above.

Below the critical boron concentration, the resistivity increases as $\rho = \rho_0 \exp(T_0/T)^m$, where the value of the exponent $m$ depends on the hopping mechanism: $m = 1$ for hopping to the nearest accessible site; $m = 1/4$ for variable range hopping (VRH) assuming (Mott 1968) a nearly constant density of states

![Figure 2. Temperature dependence of the AC resistivity (four point) of various (100)-oriented diamond epilayers with different boron contents $n_B$ measured by SIMS. The doping-induced metal-to-insulator transition (MIT) is found to occur between 4.0 and $4.8\times10^{20}$ cm$^{-3}$.](image-url)
at the Fermi level \( g_F \); and \( m=1/2 \) when \( g_F \) is reduced by a Coulomb gap (ES regime; Efros & Shklovskii 1975; Efros 1976). The \( T_0 \) parameter is related to the localization length \( \xi_{\text{loc}} \) which is of the order of the Bohr radius far from the metal–nonmetal transition, in a concentration range where the formation of both the impurity band and the valence band tail are expected. Typical values of \( 10^6 \) K and crossovers from the VRH to the ES regime and then to a \( m=1 \) variation at even lower temperature have been reported for type Ib single crystals with \( n_B = 2 \times 10^{19} \text{ cm}^{-3} \) (Sato et al. 2000). Closer to the transition, \( \xi_{\text{loc}} \) is expected to increase, leading to lower \( T_0 \) values, so that the VRH regime extends to lower temperatures, as is the case in figure 2 where \( T_0 \) is of the order of a few \( 10^3 \) K for \( n_B = 2.4 \times 10^{20} \text{ cm}^{-3} \) and several \( 10^2 \) K for \( n_B = 4 \times 10^{20} \text{ cm}^{-3} \) with a VRH regime extending down to 10 K (Klein et al. 2007).

Above the critical concentration, the normal-state conductivity can be extrapolated to a finite value \( \sigma_0 \) as \( T \rightarrow 0 \) K, as deduced from the resistivity variations shown in figure 2. As a matter of fact, as shown in the insert of figure 3a, for \( n_B \) greater than \( 6 \times 10^{20} \text{ cm}^{-3} \) (Klein et al. 2007), and below a certain temperature at which the inelastic mean free path becomes of the order of the elastic one, the resistivity increases slowly when the temperature is reduced. In this regime, where weak localization effects arising from electron–phonon scattering as well as other electronic correlations are expected, the experimental temperature dependence of the conductivity was found (Klein et al. 2007) to follow between 3 and 30 K an expression of the type: \( \sigma = \sigma_0 + A T^{1/2} + B_{\text{e-ph}} T \). Pronounced ‘weak localization’ effects have also been detected in heavily boron-doped polycrystalline (Winzer et al. 2005) and nanocrystalline (Mareš et al. 2006) diamond, while a \( \sigma = \sigma_0 + A T^{1/3} \) variation has been observed in ion-implanted samples (Tshepe et al. 1999).

The critical regime of a second-order phase transition is generally described by two characteristic exponents, \( \nu \) and \( \eta \) (McMillan 1981). \( \nu \) relates the correlation length (here \( \xi_{\text{loc}} \)) to the external parameter driving the transition (here \( n_B \)) through \( \xi_{\text{loc}} \sim 1/|n_B - n_c|^\nu \), whereas \( \eta \) relates the energy and length scales of the system (\( E \sim 1/L^\eta \)). It has been suggested (McMillan 1981) that \( \eta \) ranges from 1 to 3 depending on the relative importance of one-electron localization, many-body correlations and screening effects, and there are some preliminary indications (Winzer et al. 2005; Klein et al. 2007) that for \( p^+ \) diamond \( \eta \approx 3 \) is in agreement with the results obtained on doped silicon. As \( \sigma \) is expected to vary as \( 1/\xi_{\text{loc}} \), one expects (McMillan 1981) that \( \sigma_0 = 0.1(e^2/h)/\xi_{\text{loc}} \) with \( a_H/\xi_{\text{loc}} = (n_B/n_c - 1)^\nu \). As shown in figure 3a (solid line), \( \sigma_0 \) follows closely the prediction of the scaling theory with \( \nu = 1 \) and without any other adjustable parameter (Klein et al. 2007). In contrast to \( \eta \), a unique \( \nu \) value of the order of 1 has been obtained numerically in all systems, independent of the relative importance of the one-electron and many-body effects. The \( \nu = 1.7 \) value reported previously (Tshepe et al. 1999) for implanted diamond thus remains to be explained.

4. Doping-induced normal-to-superconducting transition

Although an increase of \( T_c \) with either the boron concentration \( n_B \) or the free carrier density has been expected on the basis of VCA calculations (Barnard et al. 2003; Boeri et al. 2004; Lee & Pickett 2004; Ma et al. 2005) and experimentally observed in some of the early reports (Bustarret et al. 2004; Kačmarčík et al. 2005; Winzer et al. 2005),...
Figure 3. (a) Conductivity extrapolated to zero temperature as a function of the boron content $n_B$ deduced from SIMS measurements on boron-doped diamond (100)-oriented epilayers. The solid line corresponds to the prediction of the scaling theory of the MIT, taking $\nu=1$. Insert: temperature dependence of the electrical resistance (for two samples also characterized in figure 2), rescaled to their resistance at 100 K, illustrating the weak localization regime (more pronounced closer to the MIT) and the definition of $T_c$ for the superconducting transition. (b) Critical temperature $T_c$ deduced from resistivity curves (at 90% of the normal-state resistance) as a function of the boron content $n_B$ deduced from SIMS measurements. The open circle has been taken from Ekimov et al. (2004a). The solid line corresponds to $T_c \sim (n_B/n_c - 1)^{0.5}$ with $n_c = 4.5 \times 10^{20}$ cm$^{-3}$. 

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systematic studies have remained rather scarce. Assuming that the mechanism for superconductivity was that proposed by the BCS theory (Bardeen et al. 1957), and further that the semiempirical solution of the Eliashberg equations proposed by McMillan (1968) could be applied to diamond, most theoretical studies have evaluated the critical temperature $T_c$ using the following equation:

$$T_c = \frac{\hbar \omega_{\log}}{1.2k_B \exp[-(1.04(1 + \lambda))/(\lambda - \mu^*(1 + 0.62\lambda))]}$$

(4.1)

where $\omega_{\log}$ is a logarithmic-averaged phonon frequency (approx. 1020 cm$^{-1}$ in diamond) and $\mu^* = g_F U_c(0)$ the strength of the zero-frequency limit of the retarded Coulomb pseudopotential $U_c(0)$. This screening parameter, $\mu^*$, has generally been taken by the authors to range between 0.1 (Boeri et al. 2004), 0.13 (Blase et al. 2004) and 0.15 (Lee & Pickett 2004; Xiang et al. 2004), either because these values lead to good agreement with experiments or because the last value is typical of usual metals where the Fermi energy is about two orders of magnitude greater than the phonon energy. Within a simplified parabolic band description of the valence band maximum and a BCS model for superconductivity, the curvature of the $T_c$ versus $n_B$ curve may then be discussed (Bustarret et al. 2004; Winzer et al. 2005; Cardona 2005, 2006; Klein et al. 2007) as a function of the $\lambda$ and $\mu^*$ parameters governing equation (4.1).

Moreover, a comparison between (111)- and (100)-oriented $p^{++}$ diamond epilayers (Mukuda et al. 2007; Umezawa et al. 2005) confirmed that above a threshold concentration $n_c$, approximately $5 \times 10^{20}$ cm$^{-3}$, $T_c$ increased sub-linearly with $n_B$ in the case of (100)-oriented growth and superlinear for (111)-oriented substrates. The fact that the same boron concentration could lead to quite different $T_c$ values depending on the preparation conditions (Umezawa et al. 2005) has raised the question of the doping efficiency of boron atoms depending on their incorporation site. For optimized growth conditions, the influence of such extrinsic mechanisms should however remain limited, in particular, close to the critical boron concentration $n_c$ (Winzer et al. 2005; Klein et al. 2007). As shown in figure 3b, surprisingly large $T_c$ values (greater than 0.4 K) have been obtained when $n_B$ is only 10% higher than $n_c$ in (100)-oriented epilayers where $T_c$ followed a $(n_B/n_c)^{1/2}$ dependence.

Under the assumption that equation (4.1) still applied despite the fact that close to $n_c$ the Fermi energy becomes smaller than the phonon energy, those relatively high values of $T_c$ could be explained (Klein et al. 2007) by a slow variation of the electron–phonon coupling parameter, $\lambda$ (typically $\lambda \sim (n_B/n_c - 1)^{0.2}$), together with a pronounced but gradual decrease in the screening parameter $\mu^*$ when $n_B \rightarrow n_c$. Such a view, where both $\lambda$ and $\mu^*$ are rescaled by the proximity of the metal-to-insulator transition (MIT), is not so common although the influence of the proximity of the MIT on the superconducting behaviour of disordered metals has been studied extensively in order to explain the enhancement of $T_c$ in the vicinity of the MIT (Osofsky et al. 2001, 2002; Soulen et al. 2003). Such an enhancement has not been reported for diamond so far.

5. Boron-doped diamond: a type II superconductor in the dirty limit

As indicated by the $H_c$ versus $T_c$ phase diagrams and $H_{c2}(0)$ values published in the first experimental studies (Bustarret et al. 2004; Ekimov et al. 2004a; Takano et al. 2004), $p^{++}$ diamond is a type II superconductor. A striking confirmation of
Figure 4. (a) Vortex image (1.5×1.5 μm²) obtained at 100 mK at a magnetic field of 1900 Oe on a (100)-oriented 75 nm thick boron-doped diamond epilayer, superimposed with a topographic scanning tunnelling microscopy map yielding an r.m.s. roughness of 1.8 nm. The vortices appear as darker spots. (b) Fourier transform mapping of the vortex image shown in (a), illustrating the disorder affecting the triangular Abrikosov lattice. The average vortex spacing was 110 nm.

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this fact, featuring vortex images (figure 4a) obtained in the mixed state by ultra-low-temperature scanning tunneling microscopy (Sacépé et al. 2006a,b), has been reported recently: the superconducting gap, its temperature dependence, its relationship to the macroscopic critical temperature as well as the shape of the tunnelling density of states were found to be fully compatible with a conventional weak-coupling mechanism in a thin and homogeneous (100)-oriented epilayer. More precisely, in order to generate an image of the vortices, the scanning tip bias was fixed at the value corresponding to the coherence peak observed in the differential conductance spectra at the gap edge. A contrast on this signal was obtained when this peak disappeared in the locally normal regions present around each vortex core. These vortices formed a partially disordered triangular Abrikosov lattice (figure 4b) and were not pinned by defects or surface morphological features (figure 4a). Spatial variations of the gap width as well as the temperature and orientation dependences of the gap shape (Yokoya et al. 2005) have however been observed, confirming the effect of spatial inhomogeneities on macroscopical signatures of superconductivity such as the marked broadening of the transition under moderate magnetic fields.

The coherence length, $\xi$, can be estimated either from the low $T_c$ part of such phase diagrams or from the $dH/dT$ slope at low field. Typical values for $\xi$ evaluated in one of these ways were 10 nm for polycrystalline HPHT bulk $p^{++}$ diamond (Ekimov et al. 2004a), 10–30 nm for polycrystalline MPCVD films (Winzer et al. 2005) and 15 for the (100)-oriented epilayer leading to the vortex pattern shown in figure 4. An estimate of the mean free path $l_{\text{mfp}}$ for the holes in the normal state has been proposed (Winzer et al. 2005) on the basis that a combination of Hall effect and conductivity measurements at 4.2 K: $l_{\text{mfp}}$ was of the order of 0.5 nm in samples where $\xi=10$ nm. A London penetration length, $\lambda_L$, of 150 nm has moreover been evaluated for the same film. Considering that $l_{\text{mfp}} \ll \xi \ll \lambda_L$, it was concluded that boron-doped superconducting diamond was in the dirty limit. Similar conclusions may be drawn from a free-electron description of the room-temperature macroscopic conductivity values measured on superconductive films (usually in the 200–2000 $\Omega^{-1}$ cm$^{-1}$ range), from momentum distribution curves of angle-resolved photoemission around the Fermi level (which yielded $l_{\text{mfp}}=0.5$ and 0.9 nm in Yokoya et al. (2005)), or from a Drude model analysis of their reflectivity spectrum (microscopic optical conductivity) yielding $l_{\text{mfp}}=2.5$ and 4 nm at room temperature (Bustarret et al. 2001; Mareš et al. 2006). The latter values were obtained on the same epilayers mentioned above for which a phase diagram was published and $\xi$ values of 15 and 20 nm have been proposed (Bustarret et al. 2004): again here, $l_{\text{mfp}} \ll \xi$. Moreover, in the thinnest of these boron-doped layers, both the room-temperature scattering rate $\gamma$ (from Drude analysis; Bustarret et al. 2001) and the superconducting gap $2\Delta(0)$ are known and there is no doubt that the $\gamma/2\Delta$ ratio is of the order of 1700, clearly in the ‘dirty’ limit of superconductivity.

6. Conclusion

In p-type diamond, the critical boron doping of the MIT was found to coincide with that necessary for superconductivity to occur. Some of the critical exponents of the MIT were determined while superconducting diamond was
found to follow a conventional type II behaviour in the dirty limit, with critical temperature values remaining relatively high quite close to the doping-induced insulator-to-metal transition. This could indicate that on the metallic side both the electron–phonon coupling and the screening parameter depend on the boron concentration, in a range where the electronic energy of the system becomes smaller than the phonon energy.

Because diamond has a simple crystallographic structure and does not involve magnetism, it could become an attractive model system for the study of superconductivity in low-dimensional structures of controlled dimensions and where disorder can hopefully be restricted to the chemical randomness of an ideal substitutional alloy.

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References


