Nanostructures made from superconducting boron-doped diamond

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Abstract
We report on the transport properties of nanostructures made from boron-doped superconducting diamond. Starting from nanocrystalline superconducting boron-doped diamond thin films, grown by chemical vapour deposition, we pattern by electron-beam lithography devices with dimensions in the nanometer range. We show that even for such small devices, the superconducting properties of the material are well preserved: for wires of width less than 100 nm, we measure critical temperatures in the kelvin range and critical fields in the tesla range.

(Some figures in this article are in colour only in the electronic version)

1. Introduction
The discovery of superconductivity in MgB2 [1] has generated a lot of interest for a special class of superconducting materials belonging to the covalent metals. In this context, the observation of superconductivity in highly doped boron diamond [2] paved the way to the study of superhard superconducting materials [3, 4]. Apart from the fundamental point of understanding the physical mechanisms leading to the superconductivity in these systems, their interest lies in a very high Young’s modulus, which makes them promising candidates for the fabrication of superconducting nano-electromechanical systems of exceptional quality factor. Evidence that both the superconductivity [5–7] and the mechanical properties [8] are essentially preserved in nanocrystalline layers grown on non-diamond substrates such as silicon has to be checked in order to bring further credit to this approach.

Most of the studies, however, have so far focused on the properties of bulk superconducting diamond, and it still remains to be shown what happens when the material is patterned into nanostructures. In this paper, we present a comprehensive study of nanostructured superconducting polycrystalline diamond films. Our measurements show that the critical temperature of the nanostructures is \( \approx 2.5 \) K while for the bulk it is \( \approx 3.5 \) K. The critical field for these structures is approximately 500 mT at 50 mK.

2. Experimental details
The nanocrystalline boron-doped diamond was obtained by chemical vapour deposition on a silicon oxide wafer. Prior to growth, wafers were cleaned with NH3OH:H2O2:H2O (1:1:5) solution at 75 °C for 10 min, and rinsed in pure DI water in an ultrasonic bath. In order to enhance nucleation, wafers were then seeded with diamond nano-particles from an aqueous colloid of mono-disperse diamond particles known to have sizes less than 7 nm in solution as confirmed by dynamic light scattering [9, 10]. Atomic force microscope (AFM) measurements have shown this technique to result in uniform nucleation densities far in excess of \( 10^{11} \) cm\(^{-2}\). Diamond growth was then performed by microwave plasma enhanced chemical vapour deposition (MPCVD) from 4% CH4 diluted in H2 with additional boron from a trimethylboron gas source. The microwave power was 3000 W at 60 mbar, the substrate...
3. Results and discussion

A SEM picture (inset of figure 2) of the sample shows grains of typical size \( \approx 150 \) nm for a film thickness of \( \approx 250 \) nm, consistent with the nucleation density. Four silver paste electrical contacts, about 5 mm apart from each other, were deposited at the surface for the characterization of the as-grown layer. The four-point resistance was measured as a function of temperature using these contacts and a standard ac lock-in technique under a very low current injection of 1 \( \mu \)A. Typical data are presented in figure 2: a clear superconducting transition is observed with a zero resistance at \( \approx 3 \) K. The width of the transition is quite large, typically 0.7 K with 10%–90% of the onset resistance criterion; we attribute this width to the distribution of the sizes of the grains in the material.

Electrical characterization of the devices was performed in both \(^3\)He and \(^3\)He/\(^4\)He dilution refrigerators. The bottom panel of figure 3 shows the critical temperature \( T_c \) of wires of various widths, measured with very low current (typically 100 nA); no significant difference was observed from the critical temperature measured on the ‘bulk’ sample (\( \approx 2 \) K for this wafer) except for the case of the narrowest wire (below 100 nm wide, \( T_c \approx 1.7 \) K). This is a generic observation for all our samples: the critical temperature of our wires is that of the bulk material except for wires thinner than typically...
Figure 3. Voltage–current (V–I) characteristics of a 500 nm wide wire at different temperatures are shown in the top panel. The V–I curves are hysteretic due to the thermal effect (Joule heating). The bottom panel shows the R versus T curves for four representative wires.

The asymmetry seen in the V I characteristics mainly arises due to an offset in our experimental set-up; however, there is also a residual asymmetry which is currently not understood.

≈100 nm; in this case, Tc it is slightly reduced. Figure 3 shows the voltage–current (V–I) characteristics of a 500 nm wide wire measured at different temperatures\(^\text{5}\). The V–I curves are hysteretic due to the thermal effect: when the critical current is reached, the Joule effect heats up the wire and the critical current measured when subsequently decreasing the current is thus much lower than the critical current measured when increasing the current \([12, 13]\); moreover, this ‘retrapping’ current is independent of the temperature of the refrigerator, as the actual temperature of the sample is then fixed by the dc current through the wire.

Critical field measurements were performed by applying a magnetic field perpendicular to the structure. V–I characteristics for a 500 nm wide wire under different magnetic fields and at 50 mK are presented in the top panel of figure 4. As expected, the critical current decreases when applying a magnetic field. It should be stressed that for magnetic fields larger than 30 mT, the V–I characteristic is no more hysteretic: in this case, the Joule heating becomes negligible as the critical current is strongly lowered, and both ‘increasing’ and ‘decreasing’ critical currents become similar. In order to get more insight into the superconductivity of our devices under magnetic field, we have numerically calculated the derivatives of the V–I characteristics: the obtained \(\frac{dV}{dI}(I)\) curves are displayed in the bottom panel of figure 4. For magnetic fields smaller than 500 mT, a clear zero is observed around \(I = 0\), as expected for a superconductor. More interestingly, we observe a dip at zero current in the \(\frac{dV}{dI}(I)\) characteristic for magnetic fields above 3 T. This demonstrates that one can still observe traces of superconductivity above 3 T in our nanometric wires, the most probable mechanism being that some grains remain superconducting under very high magnetic fields. More local studies, such as scanning tunnelling spectroscopy measurements \([14, 15]\), are certainly desirable to fully understand the robustness of the superconductivity observed in our system.

4. Conclusion

In conclusion, we have successfully fabricated nanostructures from boron-doped nanocrystalline superconducting diamond. Using electron-beam lithography, we have fabricated devices of characteristic size less than 100 nm and aspect ratio as high as 1:3. These structures have critical temperatures in the kelvin range, similar to what is observed in ‘bulk’ films; typical bulk \(T_c\) being 3.5 K while for nanostructures it is 2.5 K except for 90 nm wire where it is \(\approx 1.7\) K. We measure critical fields close to 500 mT and traces of superconductivity are observed above 3 T. This study proves that superconductivity in boron-doped diamond is a very robust phenomenon which makes it a promising candidate for future applications in the field of superconducting nano-electro-mechanical systems.
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