In this paper, we report on the transport properties of nanostructured boron doped diamond thin films. The nanostructures made from polycrystalline boron doped diamond show clear evidence of superconductivity with critical temperatures in the Kelvin range and critical field in the Tesla range. Such robust superconducting properties in these superhard materials make them promising candidates for superconducting nanoelectromechanical systems.

1 Introduction

The huge interest seen in the special class of superconducting materials belonging to the covalent metals started with the discovery of superconductivity in MgB2 [1]. Ekimov et al. [2] in 2004 reported the superconducting properties of boron doped diamond for the first time. This led to the study of superhard superconducting materials [3, 4]. Apart from the mechanism of superconductivity in these systems, which remain largely unexplored, the very high Young’s modulus of boron doped diamond makes it a front runner for making nanoelectro mechanical systems (NEMS) of exceptional high quality factor. Before realizing any NEMS made from boron doped diamond it is, however, essential to prove that both the qualities of this system, i.e. superconductivity [5–7] and mechanical properties [8], are preserved when nanocrystalline layers grown on non-diamond substrate are nanopatterned. Earlier studies in this system have concentrated their attention towards the bulk properties of this system. Hence, it is quite essential to investigate the properties of nanostructured boron doped diamond.

In this paper, a comprehensive study of nanostructured superconducting polycrystalline diamond films is presented. It is shown that these nanostructures have superconducting critical parameters like transition temperature and critical field comparable to that of bulk materials.
shown. The wafer was spin coated with 4% polymethyl-
methacrylate (PMMA) to form a 250 nm thick layer which
was prebaked at 180 °C for 5 min. This PMMA layer was
exposed to electron beam with a dose of 360 μC cm⁻² for an
acceleration voltage of 20 kV. For the development of the
exposed wafer, the wafer was dipped in 1:3 solution of
methyl isobutyl ketone (MIBK) and isopropyl alcohol (IPA)
for 1 min. A thin layer of nickel (65 nm) was then deposited
and patterned using a standard electron-gun evaporator and
lift-off technique. This was followed by plasma etching of
diamond structures using electron cyclotron resonance
oxygen plasma [11] and a −27 V dc bias for 8 min. This
leads to an etching rate of ≈40 nm/min. The nickel layer
acted as a mask in this process. The nickel layer was removed
using FeCl₃ solution and ohmic contacts were then obtained
by an evaporation of metals (Ti–Au).

In Fig. 2, a typical microcircuit fabricated by electron
beam lithography technique is shown. The bottom left panel
in the figure shows the picture of the line with the smallest
width ≈80 nm, which is ≈500 nm long and ≈300 nm thick.
Note that in this case the aspect ratio is as high as ≈1:3, the
anisotropy of the plasma etching allowing to pattern one
single grain. The top and bottom right panels are blow up of
200 × 200 μm² and 50×50 μm² area in the centre of the
microcircuit, respectively.

3 Results and discussion In Fig. 3, the resistance of
an unpatterned thin film as a function of temperature is
shown. Four silver paste electrical contacts were deposited
on the surface of as-grown layer for this measurement. A
standard ac lock-in technique with a very low current
injection of 1 μA was used for this measurement. The data
clearly show a superconducting transition with a zero
resistance at ≈3.5 K. The width of the transition is quite
large, typically 0.7 K with a 10–90% of the onset resistance
criterion. In the inset of the same figure a scanning electron
microscope (SEM) picture of the sample with grains of
typical size ≈250 nm, for a film thickness of ≈300 nm is
shown. The large width of transition seen in the resistivity
data is attributed to the distribution of the grain sizes across
the sample.

In Fig. 4a and b, the current–voltage characteristic (V–I
curve) and the calculated differential resistance for a 350 nm
wide and 500 nm long wire is shown. The inset in the panel
(a) shows the critical temperature of a few representative
wires of varying widths. These measurements were
performed in both a 3He and a 3He/4He dilution refrigerators.
The current through the sample for these measurements were
100 nA for the 500 nm wide wire and 10 nA for the rest of the

Figure 1 (online colour at: www.pss-a.com) Schematics of the
nanofabrication process.

Figure 2 The SEM images of a typical microcircuit fabricated by
electron beam lithography.

Figure 3 (online colour at: www.pss-a.com) Superconducting
resistive transition in a boron-doped diamond thin film. The inset
is a SEM image of the surface of the sample, consisting of grains of
typically ≈250 nm.
wires. For the measurement of the $V$–$I$ curves, we have used a standard four probe dc technique. No significant difference was observed from the critical temperature measured on the ‘bulk’ sample (≈2.5 K for this wafer) except for the case of the narrowest wire (below 100 nm wide, $T_c ≈ 1.7$ K). This was seen for all our superconducting diamond thin films: the critical temperature of the wires were those of the bulk material except for the cases where the wire width is less than 100 nm. The $V$–$I$ curves in panel (a) are hysteretic due to thermal effect (Joule heating). The inset shows the $R$ versus $T$ curves for four representative wires. (b) Differential resistance extracted from the $V$–$I$ curves. The resistance goes to zero when the wire is in its superconducting state.

Figure 4 (online colour at: www.pss-a.com) (a) Voltage–current ($V$–$I$) characteristic of a 350 nm wide wire at different temperatures. The $V$–$I$ curves are hysteretic due to thermal effect (Joule heating). The inset shows the $R$ versus $T$ curves for four representative wires. (b) Differential resistance extracted from the $V$–$I$ curves. The resistance goes to zero when the wire is in its superconducting state.

wires. The critical field of this sample was also measured by applying magnetic field in and out of plane of the sample. The field is applied perpendicular to the plane of the sample. The behaviour is hysteretic until the applied field reaches 20 mT. (b) Differential resistance extracted from the $V$–$I$ curves. The resistance goes to zero when the applied field is below 100 mT.

Figure 5 (online colour at: www.pss-a.com) (a) Voltage–current ($V$–$I$) characteristic of a 350 nm wide wire at 50 mK under different magnetic fields. The field is applied perpendicular to the plane of the sample. The behaviour is hysteretic until the applied field reaches 20 mT. (b) Differential resistance extracted from the $V$–$I$ curves. The resistance goes to zero when the applied field is below 100 mT.
in Fig. 5b. One can see that when the applied field is smaller than 100 mT a clear zero is observed around \( I = 0 \). Now, when the field is increased further, a clear dip near the zero current even for applied fields as high as 2 T is seen. This shows that there are still pockets of superconductivity in the nanowires at such high fields. The most likely explanation would be that some grains in the wires remain superconducting even at such high magnetic field.

In Fig. 6a and b, the \( V-I \) characteristics and the differential resistance of the 350 nm wide wire when the applied field is in the plane of the sample and perpendicular to the current through the sample is shown. In this case, the hysteresis in the sample was completely removed only when the field was above 80 mT. So this means, that for the same applied field, the critical current is higher for the case when the applied field is in the plane of the sample. We do not have any definitive explanation to this effect but it is quite possible that the introduction of vortices plays a key role in the determination of the critical field. In this case, the difference in aspect ratio of the devices when applying the field perpendicular to the structures or in the plane of the structures may account for the observed behaviour. The differential resistance calculated for this configuration (shown in Fig. 6b) shows that there is distinct zero in the resistance even at fields as high as 200 mT.

In Fig. 7a and b, the \( V-I \) characteristics and the calculated differential resistance, respectively, are shown for a 250 nm wide and 500 nm long wire. The wire shows hysteresis due to
thermal effects until the temperature reaches 600 mK. The retrapping current in this case is of the order of 70 nA while for 350 nm wire it was 83 nA. If one defines the retrapping power as $I_r^2 R_r$, where $I_r$ is the retrapping current and $R_r$ is the retrapping resistance, then for a given sample this quantity should be constant. To check this fact with our experimental data, we compare the retrapping powers of two wires of different widths. Since in the nanowires presented in this paper the lengths and the thicknesses are constant one can say that the ratio of the widths of any two wires should be equal to the ratio of the squares of their respective retrapping currents.

When one compares these ratio for the 350 and 250 nm wire, it is seen that the ratio of the widths is 1.4 which is similar to the ratio of the squares of the retrapping current for the corresponding wires. Hence, we can conclude that the retrapping power is an intrinsic property of the sample.

In Fig. 8a and b, the field dependence of critical current and differential resistance at a fixed temperature of 50 mK for the 250 nm wide wire is shown. One can see that the curves are hysteretic but the hysteresis disappears on applying a magnetic field of 30 mT. The differential resistance plotted in panel (b) of the same figure reveals the presence of few grains (as seen for 350 nm wide wire as well) having critical field above 2 T.

In Fig. 9a and b, the $V–I$ characteristics and differential resistance respectively, of a 90 nm wide and 500 nm long wire as a function of temperature is shown. Here the
characteristic is not hysteretic. This can be explained as follows. The retrapping power for this sample is \( \sim 4.5 \, \text{pW} \). For 90 nm wide wire, the power dissipated at the critical current at 50 mK is equal to 0.65 pW, which is much less than the retrapping power and hence the hysteretic behaviour is not seen. The differential resistance plotted in panel (b) shows that the sample is completely superconducting below 400 mK and above this temperature, the wire remains in the superconducting transition region until it reaches a temperature of \( \sim 1.7 \, \text{K} \) (inset of Fig. 4).

4 Conclusion In conclusion, nanostructures from boron-doped nanocrystalline superconducting diamond has been successfully fabricated. Using electron beam lithography, devices of characteristic size less that 100 nm and aspect ratio as high as 1:3 have been prepared. These structures have critical temperatures in the Kelvin range, similar to what is observed in ‘bulk’; only a slight decrease of \( T_c \) is observed for wires thinner than 100 nm. Critical fields close to 100 mT were measured and traces of superconductivity were observed even under magnetic fields as strong as 2 T. The experiments also revealed that there is stronger suppression of critical properties when the applied magnetic field is perpendicular to the sample plane. 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 nanoelectro-mechanical systems.

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