



## Letter

## Stabilizing diamond surface conductivity by phenol-formaldehyde and acrylate resins

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## ABSTRACT

H-terminated undoped nano-crystalline diamond films of 200 nm thickness are deposited by microwave plasma chemical vapor deposition on fused silica substrates seeded by a diamond powder. The films exhibit surface conductivity  $10^{-7} (\Omega/\square)^{-1}$ . Phenol-formaldehyde and acrylate resins are spin-coated on the diamond films in the thickness of 0.2–1.7  $\mu\text{m}$ . After the coating, the surface conductivity changes by  $-12\%$  to  $+52\%$  compared to a bare diamond surface. It also exhibits significantly higher temporal stability. These effects are attributed to an encapsulation of the surface conductive channel from the ambient and to an electrostatic field of molecular dipoles in the resins.

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## 1. Introduction

Diamond exhibits unique properties that can be exploited in electrochemical and electronic applications including bioelectronics [1–4]. The best conductivity can be achieved in monocrystalline diamonds [5]. Yet nanocrystalline diamond (NCD) films may have good enough quality for many applications and the films can be deposited on diverse substrates and on large areas. On both types of diamond, hydrogen-terminated surface can exhibit a two-dimensional surface conductivity [6] which is practical for device fabrication as it can be easily patterned by oxidation through masks. However, unlike bulk doping, the surface conductivity is strongly affected by molecular gas species presented in ambient air and is changing significantly over time [6–8]. Therefore, stabilization of the surface conductivity is crucial for device applications. Diverse organic and inorganic compounds were tested to generate and/or passivate surface conductivity, such as fullerenes [9] or calcium fluoride [10].

Here we report on achieving stabilization of NCD surface conductivity by phenol-formaldehyde and acrylate resin coatings. These resins are commonly available, inexpensive, can be applied by spin-coating, and can be easily micro-patterned by UV or e-beam lithography. These features make them suitable and compatible with common device fabrication technologies. We also show that the resin

coating can increase the surface conductivity compared to the bare surfaces in ambient air.

## 2. Experimental details

Diamond films were deposited in nanocrystalline form on fused silica substrates ( $10 \times 10$  mm). The substrates were mechanically seeded in ultrasonic bath with a diamond powder (nominal size 5 nm) for 40 min [11].

The depositions were performed by microwave plasma chemical vapor deposition process with the following parameters: hydrogen gas flow 300 sccm, methane gas flow 3 sccm, total vacuum pressure 30 mbar, microwave power 800–1400 W. The deposition temperature was in the range 420–800 °C. The resulting layer thickness was 115–240 nm as detected by optical measurements [12].

After the deposition, the films were boiled in acid solution ( $\text{H}_2\text{SO}_4 + \text{KNO}_3$ ) at 200 °C for 30 min. Then the films were re-hydrogenated in pure hydrogen plasma (600 °C, 30 mbar, 1200 W, 10 min), after which the microwave power was switch off, allowing the samples to cool down to room temperature in the hydrogen atmosphere. The re-hydrogenation temperature was the same for all samples, irrespective of their deposition temperature.

For characterizing NCD surface conductivity, conductive channels were defined on H-terminated diamond films by a photolithographic mask. The electrical insulation between the surface channels was achieved by exposing the masked samples to radio-frequency oxygen plasma (300 W, 3 min). After the plasma oxidation, gold contacts (size

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150 × 200 μm, thickness 100 nm) were thermally evaporated on the H-terminated channels using a lithographic mask and lift-off technique. The sheet conductivity of all films was in the order of  $10^{-7} (\Omega/\square)^{-1}$ .

On such in-plane devices, organic phenol-formaldehyde and acrylate resins were spin-coated from liquid solution and dried (hardened) at 100 °C for 45 min in air. The resins are commercially available from MicroChem (ma-P1215, 950PMMA) and Dynachem (Dynalith OFPR 800). Thickness of the resins was varied by adjusting spin-coating rotational speed (1000–5000 rpm) and by diluting the resin using a corresponding solvent (see below). The resin thickness was measured by a profilometer. Before the resin coating, the samples were rinsed in acetone, deionized water, and dried at 100 °C for 30 min in air. Phenol-formaldehyde resins (MA15, OFPR) were composed of phenol-formaldehyde of low molecular weight (5 K) dissolved in 2-methoxy-1-methylethylacetate and diazonaphthoquinone (DNQ) as a photoactive compound. Poly-methylmethacrylate (PMMA) with high molecular weight of 950 K was used as the acrylate resin. The resins are highly electrically insulating and can be used also as gate dielectrics in field-effect transistor structures. Further details about the resin properties can be found at the manufacturers and in the literature [13,14].

Effect of the resins on the NCD surface conductivity was characterized by current–voltage measurements using co-planar Au electrodes, which facilitate Ohmic contact to the H-terminated surface. The current–voltage characteristics were always linear. Their slope was changing with channel length as well as with applied resin coatings. That confirms that the current is controlled by the diamond and not by the ohmic contact resistance. Stability of the conductivity was detected by monitoring electrical current  $I(t)$  at a fixed voltage (typically 1 V) as a function of time (on a time scale of minutes and hours). Measurements were performed in the following sequence: i) in ambient air (humidity 25%), ii) after coating the sample surface by the resin, iii) after removing the resin by rinsing the sample in acetone, deionized water, and drying the sample in air at 100 °C for 30 min. This cycle was repeated with all the resins for a particular sample. The effect of the resins was investigated on four different samples and on five different microscopic channels on each sample. Hence the effect of each resin was recorded on 20 channels. From this procedure a statistical significance was obtained.

### 3. Results and discussion

Fig. 1 illustrates that the surface conductivity of H-terminated NCD films deposited at 600 °C is generally preserved after rinsing as well as after application of resin coatings. The resin coatings also exhibit an

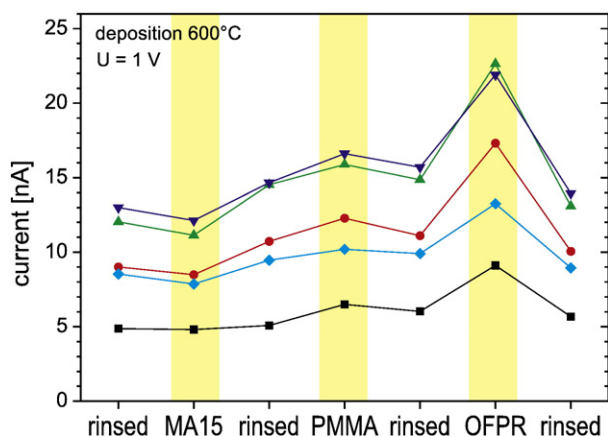


Fig. 1. Variations of current through five different microscopic channels on the diamond samples deposited at 600 °C caused by application of PMMA (thickness 0.2 μm), OFPR (thickness 0.9 μm), and MA15 (thickness 1.5 μm) resins as well as by rinsing procedures.

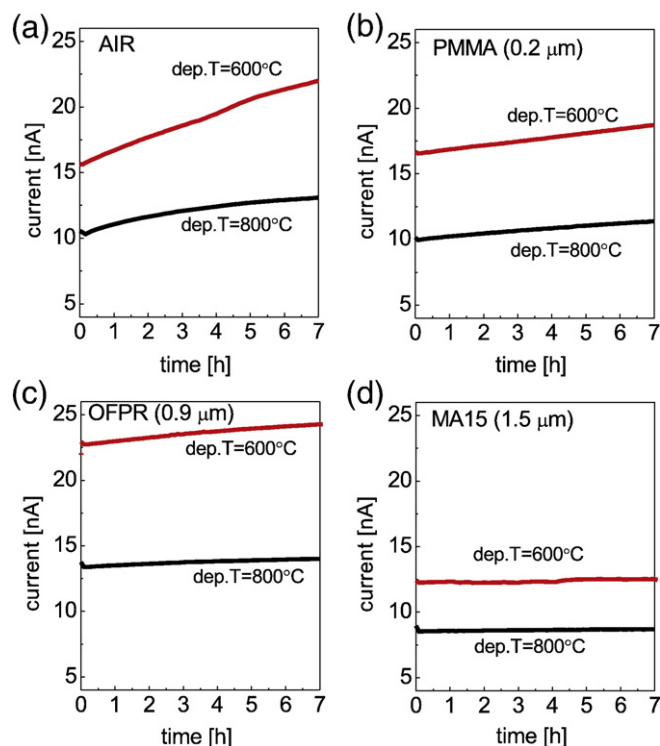


Fig. 2. Time dependence of NCD surface conductivity (a) in air and after coating by (b) PMMA (thickness 0.2 μm), (c) OFPR (thickness 0.9 μm), and (d) MA15 (thickness 1.5 μm) resins.

additional effect of slight decrease or enhancement of the surface conductivity. By repeating the experiments on four different samples and on five different microscopic channels on each sample we obtained the statistical information about these effects. The MA15 decreases the surface conductivity in 75% of cases, PMMA increases the conductivity in 40% of cases (decreases in 60% of cases), and OFPR increases the conductivity in 100% of cases compared to bare surfaces after the rinsing procedure. The above data were obtained within 30 min after particular processing steps (rinsing or coating). We also treated each channel as an independent sample to calculate average percentual change and standard deviation out of 20 channels. From there we established a confidence interval around the average percentual change by using a confidence level of 95%. The change of surface conductivity due to the coating by the particular resin is then following: MA15 =  $(-7 \pm 4)\%$ , PMMA =  $(-12 \pm 11)\%$ , OFPR =  $(+52 \pm 4)\%$ . Based on these numbers we conclude that the effects of resins on the surface conductivity are statistically significant. Nevertheless, in the case of PMMA the confidence range is very close to zero. This indicates that the effect of PMMA may be very close to random one.

The long-term effect of the resins on the surface conductivity is shown in Fig. 2. It is illustrated for two different samples, deposited at 600 °C and 800 °C. The NCD films deposited at 600 °C show higher surface conductivity compared to films deposited at 800 °C due to lower amount of sp<sup>2</sup> phase on the surface [15]. Surface conductivity of bare diamond films exposed directly to air exhibits an ongoing increase on a time scale of hours with small fluctuations (Fig. 2(a)).

On the NCD surfaces passivated with resins, the current increase is slower and fluctuations become flattened (Fig. 2(b–d)). In case of MA15 resin, the effect is so pronounced that the surface conductivity is almost perfectly stabilized and keeping nearly a constant value from the very beginning.

Note that each resin has also inherently different thickness based on its properties and standard preparation parameters. Fig. 3 shows the stabilization effect as a function of the resin thickness. The data are

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