

Conductive-AFM Study of Emerging Graphitic Channels Implanted in CVD Diamond

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INTRODUCTION

Carbon has different allotropic forms at ambient conditions with radically different structural and electrical properties (most importantly, diamond and graphite): this peculiarity allows the fabrication of all-carbon devices for various technological applications. In this context, the employment of focused MeV ion beams in diamond is a versatile tool to create different structural forms of carbon by the progressive conversion of the sp^3 -bonded diamond lattice to a sp^2 -bonded amorphous/graphitic phase.

The use of variable-thickness masks on the diamond surface allows the tuning of the ion penetration depth in order to realize channels with emerging endpoints, with several applications in radiation detection, bio-sensing and color center emission [1-3].

In the present paper we report about the Conductive Atomic Force Microscopy (C-AFM) characterization of the emerging areas of buried graphitic channels.

ION BEAM IMPLANTATION

A commercial synthetic single-crystal diamond grown by Chemical Vapor Deposition (CVD) from ElementSix (Ascot, UK) was used in this study. The $4.5 \times 4.5 \times 0.5$ mm³ sample was classified as "optical grade", cut along the [100] crystal direction and optically polished on the two opposite large faces.

The sample was implanted across one of the polished surfaces with 1.2 MeV He⁺ ions at the AN2000 accelerator of the INFN National Laboratories of Legnaro (INFN-LNL) with a broad 5×5 mm² - ion beam, in order to deliver an ion fluence of 1×10^{17} cm⁻². Implantation was performed at room temperature using current densities in the order of $2 \mu\text{A cm}^{-2}$ to minimize sample heating. MeV ions induce structural damage in matter: in the initial stages of the ion path, the ion energy is progressively reduced by Coulombian interactions, while at their end of range the cross section for nuclear collisions is strongly enhanced, promoting vacancy formation. Non-uniform depth profile of the damage density was evaluated with Monte Carlo numerical simulations: the code employed in this work is "Stopping and Range of Ions in Matter" (SRIM) [4], in its 2013.00 version.

The high density of damage induced by ion implantation promotes the conversion of the diamond lattice into an amorphous phase within a layer ~ 250 nm thick

located ~ 2 μm below the sample surface.

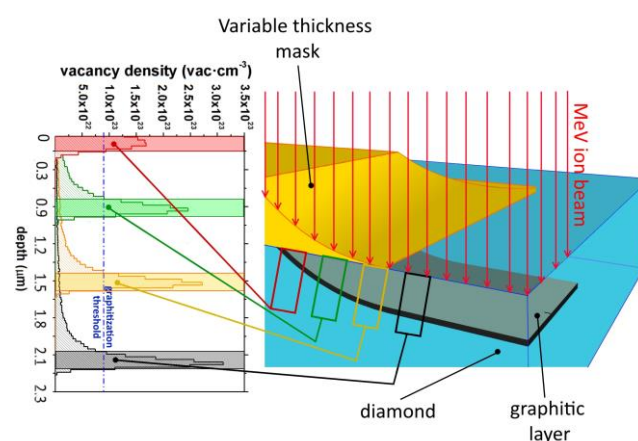


Fig. 1: Schematics of the implantation through a variable-thickness mask (right) and corresponding SRIM evaluation of vacancy density profiles generated in diamond at variable depth by 1.2 MeV He⁺ irradiation after crossing the copper mask

The geometry of the implantation was defined by a novel two-level masking process which guarantees a control of the structure geometry in the three spatial directions [5].

At the first level, a stencil mask defines the shape of the implanted areas; at the second level, a 3D copper mask modulates with high spatial accuracy the penetration depth of the ions from their range in the unmasked material up to the sample surface with increasing thickness of stopping material. Figure 1 shows the implantation process through the variable thickness mask and the vacancy profiles generated by 1.2 MeV He⁺ ions in diamond after crossing increasing thicknesses of copper.

Thermal annealing at 950 °C for 2 hours was performed in order to induce the graphitization of the highly-damaged regions.

CONDUCTIVE-AFM MEASUREMENTS

In C-AFM, during imaging, while a conductive cantilever scans the sample surface in contact mode to produce a topography map, the electrical current between the cantilever and the sample is simultaneously measured. While the topography is acquired using the deflection signal of the cantilever, the electrical conductivity is measured with a current amplifier.

Park Scientific XE-100 AFM and rectangular conductive cantilevers NS18 Ti-Pt (Mikromash, Sofia, Bulgaria) were used for this study. These cantilevers have a spring constant around 2.8 N m^{-1} . The topological and the current intensity signals were simultaneously recorded under C-AFM mode.

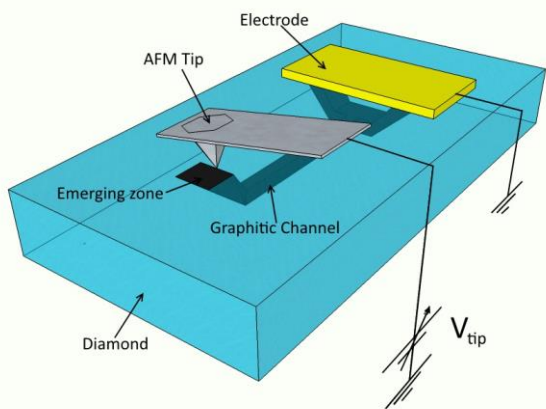


Fig. 2: Schematics of the experimental set-up. The AFM tip is scanned on the emerging end-zone of the graphitic channel. The electrostatic potential on AFM tip is set to a positive value and the current flowing through the tip, the graphitic channel and the electrode is measured for every point of the scan.

The local conductivity of the sample is acquired placing the conductive tip on the sample surface applying a bias of 1 V between tip/cantilever and the sample, thus collecting the current flowing through the tip. Typical scanning area was $45 \times 45 \mu\text{m}^2$ at a scanning rate of 0.75 Hz. All measurements were performed at room temperature.

RESULTS

The topography profile above the buried channel is reported in Figure 3a, which shows a pronounced surface swelling of $\sim 190 \text{ nm}$ localized at the implanted area. The presence of surface swelling in implanted diamonds is a well known effect due to the lower density of the graphitic channel with respect to one of the surrounding diamond crystals [6]. Figure 3b shows the C-AFM map of the same region identifying a well defined conductive area ascribable to the emerging section of the graphitic channel. This map allows to estimate the size of the surface (i.e. $\sim 70 \mu\text{m}^2$) of the graphitic emerging contact with a non-destructive measurement. This is a remarkable feature since these structures will be employed as electrochemical electrodes. This first estimation does not take into account cross-talking effects between the morphology and the current intensity signals. In particular, a contribution in current density variation must be attributed to the influence of the finite contact-area between the tip and the sample surface, that can produce an increasing of current flowing through the tip in correspondence of strong variation in topology.

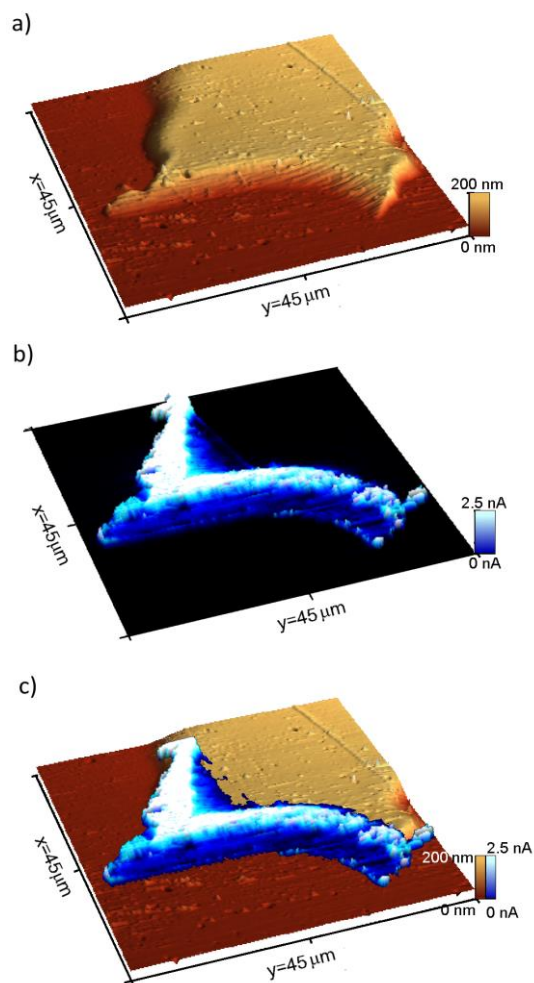


Fig. 3: Conductive-AFM maps collected from surface regions in correspondence of the emerging zone of the graphitic channel (a) Topographic image (b) Image of the logarithm of the current flowing through the tip (c) Overlap of maps (a) and (b)

CONCLUSIONS

These results demonstrate that C-AFM is an effective measurement technique for mapping electrical properties of conductive structures. Future activities will be focused on the characterization of implanted structures having more defined and complex geometries.

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