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Realization of a diamond based high density multi electrode array by means of Deep Ion Beam Lithography

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ABSTRACT

In the present work we report about a parallel-processing ion beam fabrication technique whereby high-density sub-superficial graphitic microstructures can be created in diamond. Ion beam implantation is an effective tool for the structural modification of diamond: in particular ion-damaged diamond can be converted into graphite, therefore obtaining an electrically conductive phase embedded in an optically transparent and highly insulating matrix.

The proposed fabrication process consists in the combination of Deep Ion Beam Lithography (DIBL) and Focused Ion Beam (FIB) milling. FIB micromachining is employed to define micro-apertures in the contact masks consisting of thin (<10 μm) deposited metal layers through which ions are implanted in the sample. A prototypical single-cell biosensor was realized with the above described technique. The biosensor has 16 independent electrodes converging inside a circular area of 20 μm diameter (typical neuroendocrine cells size) for the simultaneous recording of amperometric signals.

1. Introduction

In the last decade diamond has attracted interest for the development of electronic devices with promising performances [1] owing to its extreme electrical properties. Significant effort has been made to optimize the interfacing of diamond with conventional electronics, resulting in the development of techniques for the fabrication of electrical contacts and electrodes in this material. Different approaches have been adopted, ranging from surface processing such as metallization [2] or hydrogen termination [3,4], to bulk doping achieved by ion implantation [5]. Moreover, high power pulsed laser was employed to promote the diamond graphitization both on the surface and in the bulk [6].

Besides the above-mentioned techniques, ion-beam-induced graphitization of diamond has been extensively investigated with Deep Ion Beam Lithography (DIBL) [7,8]. This approach takes advantage of the metastable nature of diamond, which can be converted into the stable allotropic form of carbon in ambient temperature and pressure conditions (i.e., graphite) by inducing high defect concentration in the lattice and by subsequently processing the material via thermal annealing [9]. The damaging with energetic ions in matter occurs mainly at the end of ion range, where the cross section for nuclear collisions is strongly enhanced [10], while the effects of electronic energy loss can be neglected in this material. In order to connect the buried implanted structures to the sample surface, a three-dimensional masking technique was developed to modulate the penetration depth of the ions from their range in the unmasked material up to the sample surface with increasing thickness of stopping material [7,8]. The permanent conversion of ion-implanted diamond to a graphite-like phase upon thermal annealing at high temperature (>900 °C) occurs when a critical damage density (usually referred to as "graphitization threshold") is overcome. Such threshold value has been estimated as 9×10^{22} vacancies cm^{-3} [11]. MeV ion beams focused to micrometric spot sizes have been employed in DIBL and opened the way to the fabrication of micro-structures in diamond.

Ion beam lithography in diamond was extensively applied for the fabrication of a broad range of devices: waveguides [12–14], photonic structures [15–17], micromechanical resonators [18–20]. The possibility of creating graphitic conductive regions allowed the fabrication of infrared radiation emitters [21], field emitters

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[22] and ionizing radiation detectors [23–24]. Moreover, it is worth mentioning that diamond offers an intrinsic biocompatibility, a property that is functional for the realization of cellular biosensors [25].

All of the above-mentioned techniques are versatile tools for diamond modification but offer a spatial resolution limited to few micrometers due to the high currents necessary to implant the samples at the desired fluences. In the present paper we report on a parallel three-dimensional lithographic technique based on the combination of broad-beam DIBL with contact masking by means of metallic layers microfabricated by Focused Ion Beam machining.

2. Experimental

The sample consists of a commercial synthetic single-crystal diamond grown by chemical vapor deposition (CVD) by Element-Six. The diamond is 3 × 3 × 0.5 mm^3 in size and it is classified as type IIa ("optical grade") with substitutional nitrogen and boron concentrations lower than 1 ppm and 0.05 ppm, respectively. The sample is cut along the 100 crystal direction and it is optically polished on the two opposite large faces.

Our DIBL technique in diamond is based on the implantation with MeV ion beams through metal masks suitably microfabricated by Focused Ion Beam (FIB).

The sample micro-machining consisted of the following fabrication steps

Firstly, a uniform 4-μm-thick copper film was deposited directly onto the diamond surface by thermal evaporation

Subsequently, a high-resolution mask, which defines the geometry of the implanted graphitic structures, was realized in the above-mentioned layer. The mask apertures were performed by means of FIB milling. The instrument employed is a dual beam FEI Quanta 3D™ equipped with Nanometer Pattern Generation System from J.C. Nabity. It is essential to obtain buried channels with surface-exposed end-points which will act as multiple bio-sensing electrodes for cellular *in vitro* recordings, and at the sample periphery, which will provide contacts for chip-bonding. Therefore, the milling process must produce variable thickness holes which act as graded implantation masks [7,8]. Such configuration is achievable by opportunely tuning the milling dose during the FIB machining: the employed instrument is equipped with NPGS software, a dedicated environment for the delineation of complex structures [26]. As shown in Fig. 1 highly resolved 3D metal masks for ion beam lithography were fabricated. Typical dimensions of the milled holes were width of ~2.2 μm and length of ~200–235 μm. A protective thin layer (~200 nm) was leaved on the bottom of the milled aperture in order to avoid the superficial damage induced by Ga

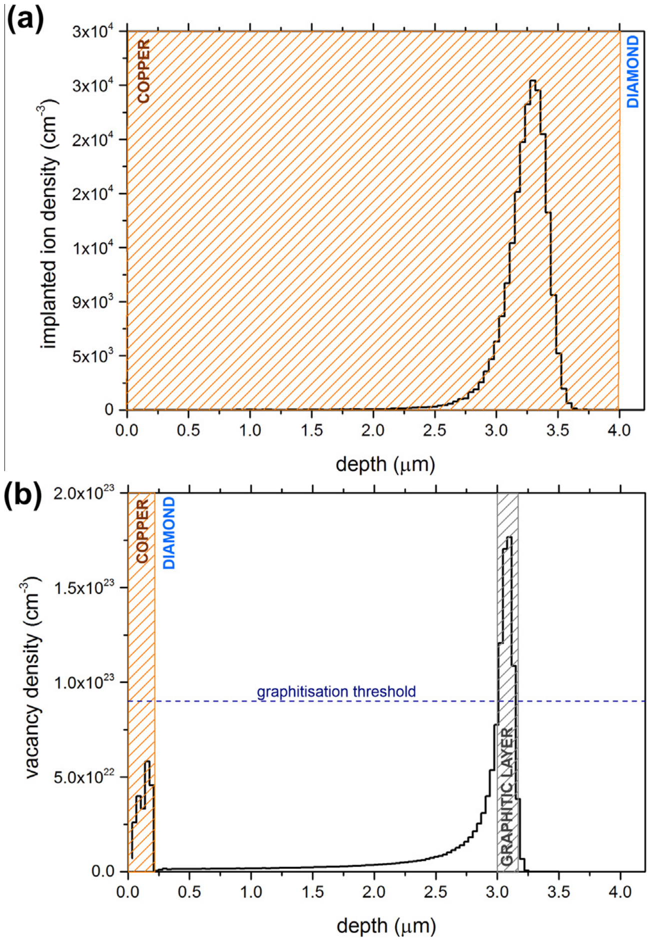

Fig. 2. SRIM Monte Carlo simulations of: (a) ion distribution of 1.8 MeV He^+ implanted in a 4 μm thick copper layer: the Bragg pick is entirely located within the metal mask; (b) vacancy density profile induced in diamond substrate covered with 200 nm Cu by 1.8 MeV He^+. The graphitization threshold is reported in dashed line. The graphitic region is highlighted in correspondence of the intersection of the Bragg peak with the graphitization threshold.

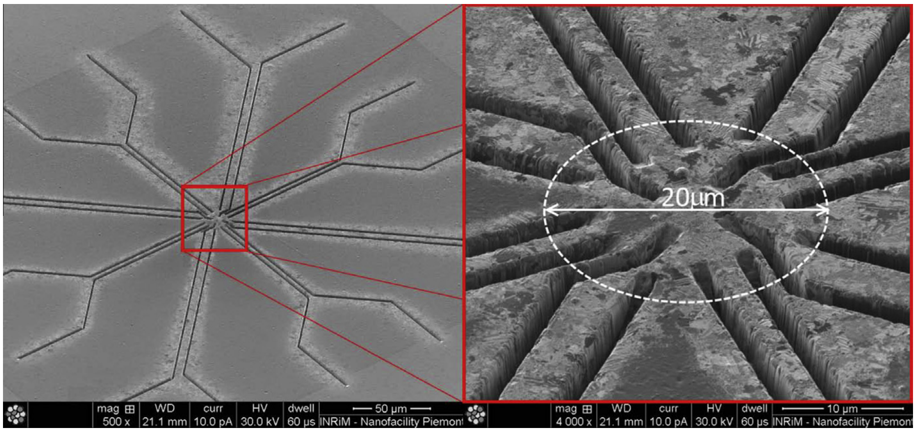

Fig. 1. SEM micrographs of the FIB micro-machined copper mask: (a) overview of the whole milled region; (b) zoom of the 300 $μm^2$ circular area where the 16 holes are converging; the slowly-thinning Cu ramps are visible.