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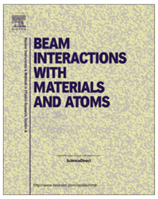

BEAM INTERACTIONS WITH MATERIALS AND ATOMS

Fabrication of monolithic microfluidic channels in diamond with ion beam lithography

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ABSTRACT

In the present work, we report on the monolithic fabrication by means of ion beam lithography of hollow micro-channels within a diamond substrate, to be employed for microfluidic applications.

The fabrication strategy takes advantage of ion beam induced damage to convert diamond into graphite, which is characterized by a higher reactivity to oxidative etching with respect to the chemically inert pristine structure. This phase transition occurs in sub-superficial layers thanks to the peculiar damage profile of MeV ions, which mostly damage the target material at their end of range.

The structures were obtained by irradiating commercial CVD diamond samples with a micrometric collimated C^+ ion beam at three different energies (4 MeV, 3.5 MeV and 3 MeV) at a total fluence of $2 \times 10^{16}\ \mathrm{cm}^{-2}$. The chosen multiple-energy implantation strategy allows to obtain a thick box-like highly damaged region ranging from 1.6 μm to 2.1 μm below the sample surface. High-temperature annealing was performed to both promote the graphitization of the ion-induced amorphous layer and to recover the pristine crystalline structure in the cap layer. Finally, the graphite was removed by ozone etching, obtaining monolithic microfluidic structures.

These prototypal microfluidic devices were tested injecting aqueous solutions and the evidence of the passage of fluids through the channels was confirmed by confocal fluorescent microscopy.

1. Introduction

'Lab-on-a-chip' technology is an emerging field exploited for a wide range of applications. The benefits of these platforms for the study of biological systems or chemical reactions have been widely reviewed in previous works [1–6]. Microfluidic lab-on-a-chips consist of small devices equipped with channel having size in the range of micrometres, which facilitates handling of volumes below the microliter range.

Microfluidic systems can be integrated with analytical detection techniques [7,8], such as electrochemical and optical methods including absorption, chemoluminescence and fluorescence. Moreover, they can be employed in biosensing devices both as active component for cell activity monitoring [9–11] or as perfusion system for drug/solution transport [12].

The vast majority of microfluidic devices consist, however, of simple planar microchips fabricated by photolithography on standard substrates such as glass, silicon or polymers.

The employment of diamond as a substrate material would represent a significant improvement for hard environment applications (i.e. high temperature, strong acid or basic solutions) since it guarantees to the final device high chemical inertness, mechanical stability, wide transparency window from IR to near UV and long term biocompatibility [13,14]. To this scope, the fabrication of microfluidic structures in diamond has already been explored with standard lithographic techniques, for which the definition of three-dimensional structures typically requires multiple processing steps in non-monolithic polycrystalline substrates [15–17].

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Deep Ion Beam Lithography (DIBL) represents a versatile fabrication tool for the structural modification of diamond [18,19], with significant applications in the realization of different types of integrated devices, as already demonstrated in previous works [20–22]. In particular, the use of DIBL for the fabrication of thin microfluidic structures was preliminary explored by Strack et al. with a single-energy ion implantation strategy [23].

In the present paper, we report on the employment of a three-dimensional lithographic technique based on multiple-energy collimated MeV ion beams for the realization of thick monolithic microfluidic structures in diamond.

2. Experimental

The sample under analysis consists of an artificial single-crystal diamond grown by chemical vapor deposition (CVD) by ElementSix. The diamond is $3 \times 3 \times 0.5\ \text{mm}^3$ in size and it is classified as type IIa ("optical grade") with a concentration of substitutional nitrogen lower than 1 ppm. The sample is cut along the (100) crystal direction and it is optically polished on the two large opposite faces.

The diamond was implanted with a collimated MeV $C^{\pm}$ ion beam at room temperature at the Laboratory for Ion Beam Interaction of the Ruđer Bošković Institute (LIBI-RBI).

The high damage density produced by ion implantation, which occurs mainly at ion end of range in correspondence of the Bragg-peak, promotes the conversion of the diamond lattice into an amorphous phase once a critical damage density threshold is overcome [24–26]. Thermal treatment (950 °C for 2 h, in this study) induces the transition of this modified region to polycrystalline graphite, which represents the thermodynamically stabile allotropic form of carbon [27,28]. The thickness of this graphitic layer is determined by the ion damage profile and usually ranges from 100 nm to 250 nm, depending on the implantation parameters.

With the purpose of creating channels with appropriate thicknesses (i.e. significantly larger than the above-mentioned values) for applications in micro-fluidics, a multiple-energy implantation approach was adopted instead of a single-energy implantation [23]. Carbon ions with three different decreasing energies (i.e.: 4 MeV, 3.5 MeV and 3 MeV) were employed, by suitably tuning the respective fluences (i.e.: $2 \times 10^{16}\ \text{ion cm}^{-2}$ for the former energy and $1.2 \times 10^{16}\ \text{ion cm}^{-2}$ for the latter ones) in order to have a uniform "box-like" above-threshold damage density profile. Particularly, the energies chosen guarantee the creation of a buried graphitic layer at 1.6 μm below the surface having an overall thickness of ≃500 nm, as confirmed by the SRIM Monte Carlo [29] simulation reported in Fig. 1.

The implantation was performed using a broad ($25\ \text{mm}^2$) ion beam [30] which was collimated by employing a metal mask which guarantees the control of the structures geometry with micrometric spatial resolution (see the schematics in Fig. 1). The thickness of the collimator element is defined to fully stop the incoming ions with the highest energy. We employed a 15 μm thick aluminium foil fabricated using a high power laser system [31] which allows to define apertures with micrometric resolution. This approach allows to perform multiple-energy irradiation of the desired region while avoiding beam refocusing and beam sample drift or misalignments during the required implantation times.

The obtained graphitic structures were fully embedded into the diamond matrix. Thus, Focused Ion Beam milling was necessary to create access holes to expose their endpoints to the external environment. This post-processing step was performed with a Quanta 3D™ dual-beam system by FEI, available at the "NanoFacility Piemonte" laboratories of the Italian National Institute of Metrological Research (INRiM), employing 30 keV $Ga^{\pm}$ focused-ion-

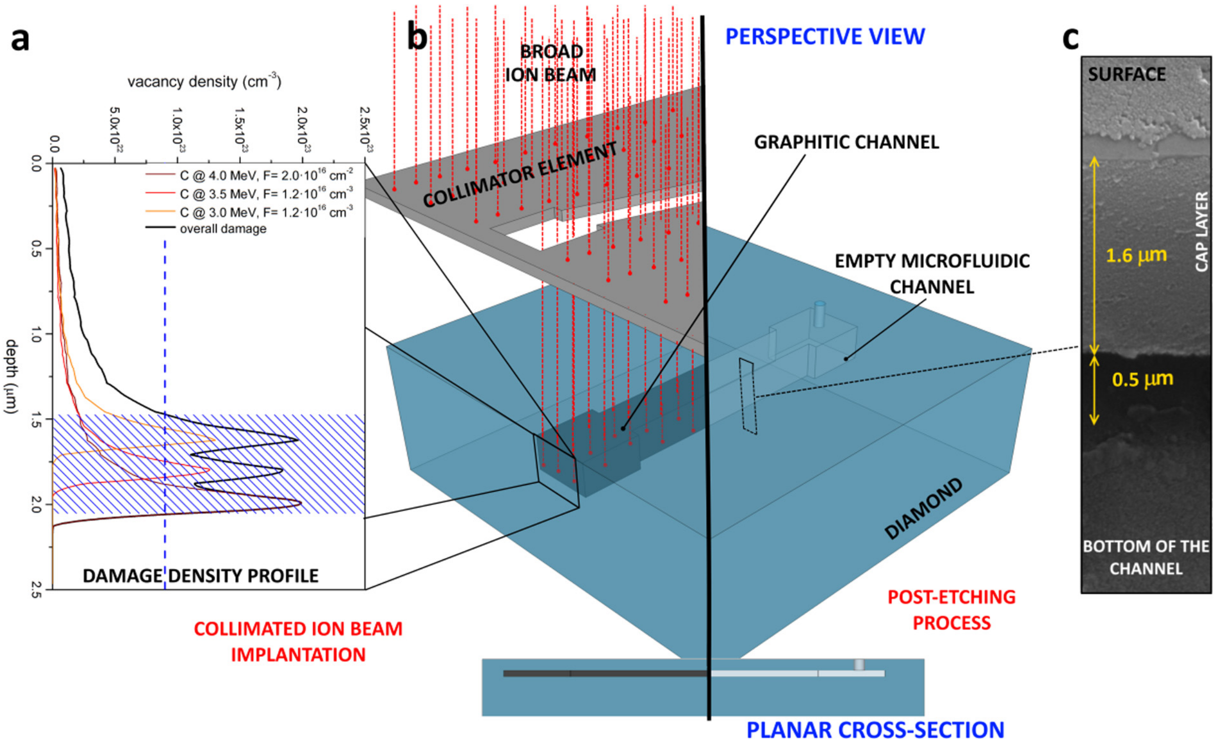

Fig. 1. (a) SRIM simulation of box-like implantation of $C^{\pm}$ ions at 4 MeV, 3.5 MeV and 3 MeV (brown, red and orange lines, respectively): the total damage (black line) and the graphitization threshold (blue dashed line) are also reported; the region where the threshold is overcome is highlighted (blue lined area). (b) Schematics of collimated beam implantation process and microfluidic channel obtained after etching treatment. (c) Cross-section SEM micrograph of a microfluidic structure. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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